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EXPERIMENTAL INVESTIGATION ON THE PERFORMANCE CHARACTERISTICS OF DI-CI ENGINE FUELLED WITH METHYL ESTERS AND PINE OIL BLENDS

A THESIS Submitted by PANNEERSELVAM N

in partial fulfillment of the requirements for the degree of

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FACULTY OF MECHANICAL ENGINEERING

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CERTIFICATE

The research work embodied in the present Thesis entitled “

EXPERIMENTAL INVESTIGATION ON THE PERFORMANCE CHARACTERISTICS OF DI-CI ENGINE FUELLED WITH METHYL ESTERS AND PINE OIL BLENDS”

has been carried out in the

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Department of Mechanical Engineering, K.S. Rangasamy College of Technology, Tiruchengode.

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ABSTRACT

In the recent years most commonly fossil fuels are more than 27% of the world's major consumption of energy. However, due to fluctuating price, environmental pollution and limited reserves. The development of alternative energy sources has become inevitable. In India domestic demand of petroleum requirements is completed by importing of nearly 75% curde oil. In order to fulfill the requirements, in the recent years researchers have made various efforts for different energy sources for alternative fuels in diesel engine without any engine modification.

In the present work biodiesel is prepared through the transesterification process by using ceibapentandra seed oil and water melon seed oil. From the transesterification results observed that a maximum yield esters CPME product reached 92% and 88% for catalyst quantity of 14 grams of KOH and NaOH respectively are methanol as a solvent.

The amount of KOH used is less than that of NaOH for same mass of feed stock oil therefore KOH is the best catalyst when compare to NaOH.

For transesterification of watermelon seed oil using KOH as a catalyst and methanol as a solvent. The maximum yield of ester WME 88% for catalyst quantity of 15grams.

As per ASTM standard the physical and chemical properties of raw oil and methyl ester were compared with diesel such as viscosity, relative density, cloud, flash point, pour point and calorific value are measured and presented in the report. The Gas chromatography and Mass spectrum analysis (GC-MS) proved that more esters content with high purity of CPME and WME are best suitable fuel for compression ignition engine. This clearly shows that prepared methyl esters are very good fuel property.

The

pine oil

has low viscosity and higher calorific value as compared

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with diesel. So the low viscosity of pine oil is blended with

B25 CPME and diesel. The results proved that the blended fuel properties closer to diesel. So,

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it can be directly used in diesel engine without any modification.

A single cylinder, naturally aspirated, direct injection, water cooled, kirloskar make AV1model diesel engine capable of developing a power output of 3.7kW (5HP), at rated speed of 1500 rpm engine is equipped with an combustion analyzer and it is loaded by an eddy current dynamometer. A piezoelectric pressure transducer is associated with PC based data acquisition system. The cylinder pressure for each crank angle is measured by PC based data acquisition system. The data of cylinder pressure were accustomed to calculate the combustion parameters. The combustion parameters like peak pressure and heat release were calculated based on the pressure data. The AVL 444 di gas analyzer is used to measure the carbon monoxide (CO), carbon dioxide (CO2), oxides of nitrogen (NOx) and unburned hydrocarbon (HC) emissions. The AVL 437 continuous flow smoke meter is used to measure the smoke emission. The inlet and exhaust gas temperatures and water temperatures are measured by using thermocouples.

The performance and emission test is conducted by two phases such as Phase I and Phase II. In phase1 three blend ratios likes B25, B50, B75 and B100 are prepared from CPME and WME to experimentally evaluate the performance and emission characteristics. The results proves that the brake thermal efficiency of B25 CPME and B25WME were 30.69% and 30.35% respectively. It is nearly close to diesel (31.88%). The emissions of CO, CO2

HC, and Smoke are reduced. The NOx emissions are slightly higher compared with B100 CPME and B100 WME. From the experimental result B25 CPME proved that best blend ratio compared with other blends.

In phase II the best fuel of B25 CPME is blended with pine oil (PO) and diesel (DI). The blends of B 25

CPME +

PO 50+DI 25 ratio, brake thermal efficiency (30.71%) is close to diesel (31.88%) and pine oil (31.97). The smoke emission is nearly close to diesel and slightly reduceNOx emission compared with diesel.

The overall combustion characteristics of B 25 CPME +

PO 50+DI 25 blend is proved that the cylinder pressure and heat release rate is (63.85bar and 59.58 kJ/m3 deg.) higher than B100CPME (47.44 bar and 43.58kJ/m3 deg.) and closer to diesel (65.16 bar and 62.57 kJ/m3 deg.). From the experimental result proved that 25% of ceibapentandra methyl ester blended with 25% of diesel and 50% of pine oil blends ratio is a best alternative fuel for naturally aspirated direct injection diesel engine producing a 3.7kW at a rated speed of 1500 rpm without any modification of the engine.

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LIST OF SYMBOLS AND ABBREVIATIONS

Symbols

μ - Dynamic viscosity

μm - Micro meter

$ - US Dollar

Abbreviation

ASTM - American standard for testing methods

B100 WME - Biodiesel 100 WME B100 CPME - Biodiesel 100% CPME

B25 CPME - Biodiesel 25% CPME and diesel 75%

B25CPME+

PO75 - Biodiesel 25% CPME and pine oil 75% B25CPME+PO25+DI50 - Biodiesel 25% CPME, pine oil 25% and diesel

50%

B25CPME+PO50+DI25 - Biodiesel 25% CPME, Pine oil 50% and diesel

25%

B25 WME - Biodiesel 25% WME and diesel 75% B50 CPME - Biodiesel 50% CPME and diesel 50% B50 WME - Biodiesel 50% WME and diesel 50% B75 CPME - Biodiesel 75% CPME and diesel 25% B75 WME - Biodiesel 75% WME and diesel 25% BSEC - Brake specific energy consumption BSFC - Brake specific fuel consumption

BTE - Brake thermal efficiency

CO2 - Carbon dioxides

CPME - Ceibapentandra oil methyl ester cSt - Centi Stoke

CN - Cetane number

CP - Cloud point

CI Engine - Compression ignition engine

CA - Crank angle

DI - Diesel

EU - European Union

EGT - Exhaust gas temperature

FP - Flash point

FFA - Free fatty acid

GC - Gas chromatography

GC-MS - Gas chromatography mass spectrum

HSU - Hartridge smoke unit HHV - Higher heating value HP - Horse power

IOCL - Indian Oil Corporationlimited IEA - International energy agency IN - Iodine number

IV - Iodine value kW - kilo Watt

Kg/ha - kilogram per hectare kJ/Kg - Kilojoule per kilogram LPG - Liquefied petroleum gas LNG - Liquid natural gas

LHV - Lower heating value

M & HCV - Medium and heavy duty commercial vehicles

MMT - Million metric tons

NMOG - Non-methane organic gases

NOx - Oxide of nitrogen SOx - Oxide of sulphur PM - Particulate matter ppm - Parts per million

PO - Pine oil

KOH - Potassium hydroxide

PP - Pour point

RPM - Revolutions per minute NaOH - Sodium hydroxide NaoCH3 - Sodium methoxide

SVO - Straight vegetable oil

HC - Unburned hydrocarbon WME - Watermelon oil methyl ester Wt - Weight

CHAPTER 1

INTRODUCTION

1.1 GENERAL

In day to day life, the energy demands becomes increasing and fossil fuels are getting depleting, so we are in need to search renewable alternate fuels. At that time, the petroleum fuels acts an important role in the development of agricultural sector, transportation, industrial growth and to satisfy the primary necessitates of humans. When the

consumption is increasing rapidly these fuels are limited and depleting day by day.

The utilization of fossil fuels is disturbing the environment and it makes so much of environmental problems in the society (i.e.)

when the fossil fuel are burned it releases various smog –causing

pollutant and greenhouse gases that lends to global warming. The conventional fossil fuels (diesel) used in a diesel engine, it releasing higher amounts of carbon dioxides (CO2), oxide of sulphur (SOX) particulate matter (PM), oxides of nitrogen (NOX) and unburned hydrocarbon (HC). Moreover, CO2 is the greenhouse gas but NOX and SOX causes “acid rain” (Altun & Lapuerta 2014). The particulate matter (PM) which emits from petroleum diesel engine causes several health hazards such as respiratory diseases, cardiovascular diseases and lung cancer.

In addition to that, the fast depleting the petroleum fuels cause the threat of climatic change and fluctuating fuel cost. The expecting future populations about top ten countries are pointed out in Table 1.1 and

Table 1.2.

From the periods of

1950 to 2050 and 2100 to 2300 respectively. In a clear evident manner it shows that India will be the first position in terms of population with in the

year 2050. (U.N. http://www.un.org/esa/populations/longrange 2/ world pop

2300 final. Pdf: 2004). While the population increases the usage of automobiles also increasing exponentially as well as the need of fossil fuel is also increasing rapidly.

1.1.1 Current Status of Automobile Industry in India

Agarwal (2016) reported the confederation of India industry survey about automobile sector status as follows,

¾ Largest three wheelers market

¾ Second largest two wheelers market

¾ Tenth largest passengers car market

¾ Fourth largest tractor market

¾ Fifth largest commercial vehicle market

¾ Fifth largest bus and truck segment

The number of vehicles producing an industry is increasing year by year. As such in the year of April-March 2015, the industry produced a total number of 2,33,66,246 vehicles including two wheelers, passenger vehicles and three wheelers in direct contrast to 2,15,00,165 in the period April-March 2014. This record shows the development of 8.68% over the same period compared with last year presented in Table 1.3.

Renewable energy sources like nuclear, solar, wind, hydro power, nuclear, biofuel and biodiesel are evolving as feasible alternative and research in using them is going on in a developing stage. The photo voltaic cells convert the solar energy in to thermal energy and indirectly using as electric energy. The origin of renewable energy depends on the nature and it is

difficult to store except bio-energy. So the researchers are looking for the alternative fuel, however bio-diesel from vegetable oil compromises all those solutions. In the 19th century (1858-1913), ran the engine using vegetable oils as a substitute for diesel fuel for Rudolf Christian Karl diesel (Talebian- Kiakalaieh et al. 2013).

The term bio-diesel is hosted in the United States during 1992 by the National soy diesel Development Board (presently national bio-diesel board) which has founded the commercialization of bio-diesel in the United States. At any ratio the bio-diesel can be used with petroleum diesel but it has lower exhaust emissions and also it has very similar characteristics. When compare with petroleum diesel properties the bio-diesel holds well fuel properties such as nontoxic, renewability, biodegradability, and free of sulfur and aromatics (Martini & Schell 2012).

1.2 FUEL DEMAND AND ITS INCREASE IN PRICE

Diesel fuel plays a major role in different sectors like agriculture industries, agriculture and transportation. Diesel fuel is also one of the unavailable and developing energy liquid fuel in India , due to the reason the diesel demand increasing, the fuel price also increasing. Since, we are importing approximately 75% of petroleum from other countries reported by Shukla et al. (2015). It becomes a great setback for the economy development of India against US dollars. Ale & Bade Shrestha (2008) reported that the year by year increased demand ratio of petrol, diesel and kerosene and it’s evidently indicated that as compare with petrol and kerosene the usage and increasing the demand in diesel is higher as shown in Figure 1.1. So, the alternate source of energy research is a need of the day.

Table 1.1 Top ten largest countries and their populations, selected years

1950 to 2050 in millions

Rank

Country Population in millions up to 1950 year

Rank

Country Population in millions up to 2000 year

Rank

Country Population in millions up to 2050 year 10 Italy 47.1 10 Nigeria 114.7 10 Congo, DR 151.6 9 United 49.8 9 Japan 127.0 9 Ethiopia 171.0 8 Brazil 54.0 8 Bangladesh 138.8 8 Brazil 233.1 7 Germany 68.4 7 Pakistan 142.7 7 Bangladesh 254.6 6 Indonesia 79.5 6 Russian 145.6 6 Nigeria 258.5 5 Japan 83.6 5 Brazil 171.8 5 Indonesia 293.8 4 Russian 102.7 4 Indonesia 211.6 4 Pakistan 348.7 3 U.S.A 157.8 3 U.S.A 285.0 3 U.S.A 408.7 2 India 357.6 2 India 1016.9 2 China 1395.2 1 China 554.8 1 China 1275.2 1 India 1531.4

Table 1.2 Top ten largest countries and their populations, selected years

2100 to 2300 in millions.

Rank

Country Population in millions up to 2100 year

Rank

Country Population in millions up to 2200 year

Rank

Country Population in millions up to 2300 year 10 Congo, DR 203.3 10 Congo, DR 173.0 10 Congo, DR 182.7 9 Brazil 212.4 9 Ethiopia 196.9 9 Ethiopia 206.5 8 Ethiopia 222.2 8 Brazil 208.8 8 Brazil 222.6 7 Bangladesh 259.9 7 Bangladesh 232.0 7 Bangladesh 242.7 6 Indonesia 272.8 6 Indonesia 263.0 6 Indonesia 276.2 5 Nigeria 302.5 5 Nigeria 268.4 5 Nigeria 282.8 4 Pakistan 408.5 4 Pakistan 342.5 4 Pakistan 359.1 3 U.S.A 437.2 3 U.S.A 470.0 3 U.S.A 493.0 2 China 1181.5 2 China 1200.7 2 China 1285.7 1 India 1458.4 1 India 1304.5 1 India 1371.7

Table 1.3 Indian automobiles industry production and sales trends

11

Figure 1.1 Petrol, Diesel and Kerosene demand 2006 to 2020

Table 1.4 India's petrol, diesel, kerosene and LPG prices in Rupees

(August 01 2015 - July 01 2016)

Month & Year Petrol Rs. Diesel Rs. Kerosene Rs. LPG Rs. Aug 01, 2015 64.47 46.12 14.96 417.82 Nov 01, 2015 61.38 47.10 -- -- Feb 01, 2016 59.95 44.68 -- --

May 01, 2016 62.19 50.95 -- 419 sub/ 527.5 nonsub July 01, 2016 64.76 54.7 -- --

Source:http://in.reuters.com/article/2015/08/31/india-fuel-prices-idinl4n1163j420150831

The petrol, diesel, kerosene and LPG price has monthly greater than before and also fluctuations. As on August 2016, the price of petrol, diesel, kerosene and LPG were 64.47, 46.12, 14.96 and 417.82 rupees respectively. The differences in the oil price modification in India (01.08.2016 to 01.07.2016) are shown in Table 1.4. At present the cost of petrol, diesel, kerosene and LPG were 64.76, 54.7, 14.96 and 419 rupee per liter and 14.5kg per cylinder respectively. This needs the developments and commercialization of alternative fuel from bio-sources as reported by Reuters. Source:http://in.reuters.com/article/2015/08/31/india-fuel-prices-idinl4n1163 j420150831

1.3 PRODUCTION, IMPORT AND EXPORT OF CRUDE OIL AND COST OF PETROLEUM PRODUCT

1.3.1 Production and Consumption of Petroleum Products

The production of petroleum products is 221.136 MMT in the year

2014-15 as against 220.756 MMT achieved in 2013-14, shows a marginally increase of about 0.17%. However, production was affected due to planned outages in IOCL-Panipat, Koyali and Bongaigaon refineries during April- July, 2014. During the year 2014-15, the consumption of petroleum products in India was 164.987 MMT with a growth of 4.15% as compared to consumption of 158.407 MMT during 2013-14. Production and consumption of petroleum products during the years 2009-10 to 2014-15 have been shown in the Figure 1.2.

Figure 1.2 Production and consumption (indigenous sales) of petroleum products

1.3.2 Imports of Crude Oil

Import of crude oil during the year 2014-15 was 189.432 MMT valued at rupees 6,87,350 crore which marked a decrease of 0.10% in quantity terms and 20.53% decrease in value terms over the same period of last year. The decline in value terms released to reduced crude oil prices by around 50% from June 2014 to December 2014. During the year 2013-14 the import of crude oil was 189.238 MMT valued at rupees 8, 64,875 core.

The average international crude oil oil price (Indian Basket) was US$ 84.20/bbl. During the period of April-march in the year 2014-15 as compared to US$ 105.52/bbl in the same period of 2013 -14, ie lower by

20.20% as compared to the same period last year. During the last 6 months there is continuous declining trend in the price of crude oil in the international market. The price of Indian basket crude oil which was at $111.84/bbl on 19th June 2014, it has continuously decreased thereafter to US$ 53.64/bbl on 31st March, 2015. The trend in growth of crude oil imports and crude oil international (Indian Basket) prices is shown in Figure 1.3.

Figure 1.3 Quantity of crude oil importsand average international crude oil prices

1.3.3 Imports and Exports of Petroleum Products

During the period April-March, in the year 2014-15 imports of petroleum products were 20.423 MMT 72778 crore which shows an increase of 22.16% in quantity terms and 2.45% decrease in value terms against the same period of previous year. The quantity of petroleum products imported during the year 2013-14 was 16.718 MMT at rupees 74,605 crore. During the period April-March in the year 2014-2015 the exports of petroleum products were 63.928 MMT valued at rupees 2,88,563 crore which shows a decreased of 5.80% in quantity terms and 21.65% decreased in value terms against the experts of 67.864 MMT valued at rupees 3, 68, 279 crore for the same period of last year. The trends in quantity of petroleum products and LNG imports and export are depicted shown in the Figure1.4.

Figure 1.4 Trend in imports & exports of petroleum products

1.4 GLOBAL STATUS

The developed and developing countries alike have shown interest in developing and effective utilization of renewable energy. This interest will produce better prospects for the biofuels and the advantages of these fuels are, which reducing the vehicle emission and it reduces the global warming and enhancing rural development, which is an achievement towards energy security, renewable and reduce pollution to environment. The European biodiesel board (2011) reported that European Union produced over 24.7x10-9

Lyr-1of biodiesel in the year 2011. Due to new production capacity, projected

Canadian biodiesel production in the year 2013 (0.471x10-9) was more than double estimated for in 2012 (0.210x109 Lyr-1). A raised bio-diesel production of 0.65x109 L in 2014. It was partly attributed to the expected completion of a plant producing 2.65x108Lyr-1. The major biodiesel feed stack in Europe, United States, and Canada are soybean, rape-seed oil and canola oil respectively (Adewale et al. 2015). For decreasing the greenhouse effect and oil import Canada and European Union (EU) have already started this replacement with bio-diesel at a certain percentage. The European

parliament would increasing the biofuel market share of 10% by the year

2020.The Canadian government announced a new policy for a new biofuel advocated the blend of 2% of bio-diesel and 5% of heated bio-diesel in the replacement of diesel for transportation sector in between the years 2012 to

2015 respectively (Roy et al. 2013).A new AEDP 2012-2021 project recently announced by the Royal Thai Government has regulated that the blending ratio of bio-diesel in petrol-diesel must achieve 10% (ie) B10 bio fuel, in

2021, corresponding to a 5.97ML/day market in Thailand (Chen et al. 2014).

1.4.1 Effect of Engine Emissions in Environment and Human Health

The emissions produced by combustion of diesel must have a contrary effect on environment and human health. It is depicted by the unite nation inter-governmental panel that because of greenhouse gas emission including methane, carbon dioxides and nitrogen oxides increases the global warming. Due to green gas effect the global temperature is increased by more than 2 C and about hundreds of millions of people will lose their lives. hydrocarbon (HC), oxide of nitrogen (NOx), Carbon monoxide (CO), and organic gases other than methane (Non-methane organic gases, (ie) NMOG) which are emitting from internal combustion engine has been identified as harmful to the human health and environment degradation. Table 1.5 shows

the

impact of exhaust emissions on human health.

Table 1.5 Shows the engine emissions impact on human health

S. No Exhaust emissions Reason for formation Impact in health 1 NOx The temperature inside the combustion chamber becomes high due to higher calorific value (or) presence of a higher amount of oxygen, leading to increase in the formation of NOx. Irritate the lungs and cause edema bronchitis and pneumonia; and result in increased sensitivity to dust and pollen in asthmatics 2 CO Incomplete formed due to shortage of air (or) due to low gas temperature

CO is the formed.

It affects fetal growth inside pregnant women and tissue development of young children. It has a synergistic action with other pollutants to promote morbidity in people with respiratory (or) circulatory problems. 3 HC HC appears in exhaust gas due to local rich mixture pockets at much lower temperature than the combustion and due to flame quenching near to metallic walls. Eye irritation, coughing and sneezing, drowsiness and symptoms akin to drunkenness. Some hydrocarbons have a close affinity for diesel particulates and many contribute to lung disease

1.5 NATIONAL STATUS

1.5.1 Indian Biomass Energy Conversion Policy

In recent years, due to population and economic growth India's energy consumption has been increasing a relatively fast rate. By way of rapid

urbanization and improving standards of living for million Indian households, the demand is probable raise a lot. Consequently, Indian government is now making various planning and polices in energy sector. Since, the key target of the world is now sustainable development, so renewable energy resources are considering for power generation. Ministry of new and renewable energy of India (MNRES) has developed many projects and polices in this filed and promoting to adopt these methodologies by providing various subsidies and incentives.

Diesel engines are located in power sector and transportation, with a high thermal efficiency and extended durability. To accomplish the domestic demand, India has to import the 75% of petroleum requirements. The major part of these petroleum products is utilized by the diesel vehicles play a major role in transport sector. Hence, to satisfy the domestic energy demand in a suitable manner alternative fuel search plays crucial role in India. Developing countries like India have a big fleet of old vehicles still running on their roads which usually emitting comparatively higher emissions compare with latest vehicles. Emissions from older vehicles need to be cut down, in order to satisfy the contemporary emissions legislations, using one or early emission reduction techniques (Shukla et al. 2015).

1.5.2 Food-Fuel Conflict

Pro-biofuels experts declare that the impacts of biofuels can be covered by two major type of technological innovation. World countries can be focusing more on cellulosic biofuel technologies that use waste products or food spin-offs and other crops to create biofuels. Technologies are essential for this option be possible are just starting to become available but are comparatively expansive. Similarly, investments in existing technologies increasing the agricultural productivity could mitigate the biofuels impact specially well-organized with technologies for cellulosic biofuel production.

The selection is not necessarily between food and fuel. But, the exception of this struggle is mainly depends on each country's willingness in new skills.

1.6

NECESSITY OF ALTERNATIVE FUEL

Major attention is taken care to produce biodiesel for a promising alternative to diesel, due to its renewable, biodegradable, sustainable, carbon neutral, nontoxic gases release and environmental friendly characteristics.

With or without major modification, biodiesel can be directly used in a diesel engine in replacement of diesel fuel, which can reduce importing oil requirement of the country (Subramaniam et al. 2013).

Mofijur et al. (2013) reported that the use of biodiesel in an internal combustion engine resulted in a reduction of unburned hydrocarbon (HC), particulate matter (PM) and carbon monoxide (CO) emissions. At the same time, the nitrogen oxides had (NOx) slightly increased its dependence on the nature of oil and engine type. The chemical and thermo physical properties of biodiesel were entirely different from the fossil fuel, which resulted in the variation of the combustion characteristics. The cetane number of the biodiesel was higher than that of diesel; it gives shorter ignition delay time. The viscosity of biodiesel was 1.5 to 2.5 times higher than that of diesel, which resulted in the increase of the combustion duration.

1.7

VEGETABLE OIL SELECTION

Bio fuels produced from vegetable oil and animal fats such as rapeseed, soybean, sunflower, safflower, cotton seed oil, peanut, jatropha, coconut, palam kernel, neem oil, Honne oil, cardinal oil, rubber seed oil, orange oil, mahua oil, koroch oil, karanja oil, cotton seed oil, waste cooking, waste plastic oil, tallow and lard (Kwon et al. 2015 and Subramaniam et al.

2013). The present research work focuses on producing the biodiesel from

waste, useless seeds like watermelon, ceibia pentandra seeds crushed in the oil expeller by extracting in the

oil, and pine oil to meet the requirement of the alternative fuels because of

limited research and publications.

1.7.1 Watermelon Seed Oil

The seed were extracted from the fruits, washed and dried in an oven at 70º C for a period of 48 hours for easy removal of the husk (Rao et al.

2012) The oil is extracted in the laboratory using soxhlet extractor and hexane as solvent. Finally ground seeds dried on oven at a temperature of

100º C for two hour were subjected to extraction with hexane for a period of

24 hours. The removal of solvent by rotary evaporation and drying afforded oil and yield is calculated on dry weight basis (Adebanjo & Kehinde 2013). The watermelon fruits and seed as shown in the Figure 1.5 & Figure 1.6.

Figure 1.5 Watermelon fruits Figure 1.6 Watermelon seeds

1.7.2 Ceiba Pentandra Seed Oil

The ceiba pentandra pods were gather together from Palamedu village, Namakkal district Tamilnadu, India. In the agriculture land, it has been grown naturally. In each fruit, ceiba pentandra seed occupy nearly

35- 42% (W/W). To remove the moisture the collected seeds were dried in

the sun light for two (or) three days depending upon the sunlight. To extracting the oil with the help of

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steam treatment process shadowed by mechanical crushing process. The oil seed yield is producing on average

2850 kg/ha.

0: http://isi-dl.com/downloadpdf/22054 82%

Ceiba pentandra seed were low feeding value due to its higher fiber content. Moreover, the opening of kapok (ceiba pentandra) fiber as bio- ethanol feed stock.

It found that kapok fiber contains 34 to 64% of cellulose higher potential to produce cellulosic ethanol.

According to the Tradition

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1: http://isi-dl.com/downloadpdf/22054 89%

kapok fiber is utilized as stuffing material for pillows and bed (Tye et al.

2012,

Silitonga et al. 2013).

The ceiba pentandra pods in tree, and seed as

shown in the Figure 1.7 and Figure 1.8.

Figure 1.7 Ceiba pentandra pods

in tree

Figure 1.8 Ceiba pentandra seeds

1.7.3 Pine Oil

Pine oleoresin, the raw material for pine tree synthesis, is tapped from pine tree and is subjected to steam distillation, before being treated with acids to synthesis the required pine oil. In general, pine oil, by its appearance and nature, can be regarded as one of the less viscous fuels in the likes of eucalyptus oil, ethanol and methanol. Chemically speaking, pine oil, an

alicyclic hydrocarbon, consists of mainly cyclic terpene alcohols, known as terpineol ($C_{10}H_{18}O$) along with alpha-pinene ($C_{10}H_{16}$). Further from its molecular structure, it is evident that it possesses inherent oxygen, with lower molecular weight and shorter carbon chain length than diesel (or) biodiesel. Notably, pine oil, despite being a higher alcohol, does not produce any cooling effects like methanol and ethanol as the latent heat of vaporization of it is not higher like lower alcohols

1.8 TECHNOLOGY USED IN THE CONVERSION OF SVO INTO BIODIESEL

The following techniques are available for the conversion of SVO

in to bio-diesel.

x Micro-emulsion

x Thermal cracking

x Preheating

x Blending

x

Transesterification

1.8.1 Micro-emulsion

To solve the problem of high viscosity of the vegetable oils, micro- emulsions with solvents such as methanol, ethanol and 1-butanol have been investigated. A micro-emulsion is defined as a colloidal equilibrium dispersion of optically isotropic fluid microstructure with dimensions in the

1-150 Nano meter range, formed spontaneously from two normally immiscible liquids and one or more ionic immiscible. They can improve spray characteristics by explosive vaporization of the low boiling constituents

in the micelles. A brief study shows that the performance of micro emulsions of aqueous ethanol in vegetables oil was nearly as good as that of diesel, inspite of lower cetane number and energy content (Balakrishna 2012 & Subramaniam et al. 2013).

1.8.2 Thermal Cracking (Pyrolysis)

Pyrolysis can be defined as the conversion of one substance into another by means of heat in the absence of air (or oxygen) or by heat in the presence of a catalyst which results in the cleavage of bonds and formation of a variety of small molecules. The thermal cracking of vegetable oil to produce bio fuels has been studied and found to produce alkanes, alkenes, alkadienes, aromatics and carboxylic acids in various proportions (Balakrishna 2012 & Subramaniam et al. 2013).

1.8.3 Preheating

Preheating the vegetable oils prior to injection can reduce the viscosity of vegetable oils. By preheating the vegetable oils about 55°C, the viscosity becomes almost equal to that of diesel. This will improve the spray characteristics of the fuel. Preheated vegetable oils results in improved performance with a reduction of emissions (Balakrishna 2012 & Subramaniam et al. 2013).

1.8.4 Blending

A mixture of 10% vegetable oils is use to run engine without any modifications. At present, it is not practical to substitute 100% vegetable oil in diesel engines, but a blend of 20% vegetable oil and

80% of diesel fuel can be used. Some short-term experiments are conducted with 50% blend of Jatropha oil in diesel engine without any major operational difficulties, but

further study is required for the long term durable operation of the engine. Direct use of vegetable oil and the use of higher percentage of blends of oil have generally been considered not satisfactory for both direct and indirect diesel engines. High viscosity, acid composition, free fatty acid content, as well as gum formation due to oxidation, polymerization during storage and combustion, carbon deposits and lubricating oil thickening are some of the problems (Balakrishna 2012 & Subramaniam et al. 2013).

1.8.5

Transesterification

Transesterification is the process of exchanging the alkoxy group of an ester compound by alcohol and the reaction often catalyzed by

an acid or base. Transesterification is important

for producing bio-diesel from bio- lipids. The transesterification of vegetable oil

is needed for reaction of triglycerides (fat/oil) with bio-alcohol to from ester and glycerol (Balakrishna

2012 & Subramaniam et al. 2013).

1.9 COMPRESSION IGNITION ENGINES

Diesel engines are main workhorses for heavy-duty vehicles because of their fuel economy and durability. They have higher thermal efficiencies, resulting from high compression ratio and lean fuel combustion. Diesel engines are the backbone of the world industries with high torque. They dominate significant sectors like road, train transport, agricultural, military, mining, construction, propulsion, maritime and stationary electricity production (Akarte 2004).

1.10 PRESENT ISSUES IN FOUR STROKE DI-CI ENGINE

There have been continuous researches carry out for the most efficient prime movers to be invented right from the beginning, which will do

less harm to the environment. The research and development mainly focuses on the fuel economy and the exhaust emissions of the four stroke compression ignition engines.

The high levels of smoke and oxides of nitrogen (NOx) emissions make the diesel engines difficult to pass through the strict emission norms. The high level of smoke emissions is due to the diffusive combustion of diesel engine, whereas oxides of nitrogen emissions are mainly due to high combustion chamber temperature and dissociation. It is very difficult to control simultaneously both the smoke and oxides of nitrogen (NOx) emission in a diesel engine due to their trade off.

1.11 ORGANIZATION OF THESIS

The thesis is organized in to six chapters:

Chapter 1 introduces the essential and need of this research work. Chapter 2 brings out the review of literature related to bio diesel production, fuel properties, advantages and disadvantages of bio-diesel, engine performance, emission and combustion analysis of different types of vegetable esters. The Chapter 3 explains the objectives and research methodologies adopted throughout the study for achieving the set objectives. Chapter 4 brings out the description of bio diesel production unit, instruments used for measuring the fuel properties, experimental engine set up, gas analyzer, smoke meter, data acquisition system and procedure for conducting experiments. Chapter 5 demonstrates the results and discussion of various methods of bio diesel production techniques, properties of raw oil, bio diesel- diesel blend fuel properties, performance emission and combustion characteristics of the DI-CI engine fuelled with bio-diesel-diesel blends mixed with pine oil. Chapter 6 concludes the major share of the research work and guidelines for future work.

CHAPTER 2

REVIEW OF LITERATURE

2.1 INTRODUCTION

The literature survey of this research work has a discussion on

the present status of using bio diesel as the best suitable alternative fuel for diesel.

While for an instant touching the history in the wake of alternative fuels for compression ignition engines, it considers some of the current techniques for the production of bio diesel as well as some new technologies under the research.

This research work is primarily focused on the development of bio diesel production, fuel properties,

comparison of two types of biodiesel, and performance, emissions and combustion

characteristics of DI-CI engine fuelled with biodiesel-diesel blends.

2.2

HISTORICAL BACKGROUND

In day to day life,

bio diesel production is not something new, since the method of using vegetable oil as a fuel

was invented in the year 1895.

In the year of 1900 the

Rudolf Diesel developed the first engine which operates with vegetable oil. The Rudolf Diesel used groundnut oil as fuel for the first engine. In 1911, Rudolf Diesel told that the diesel engine can be fed with vegetable oil and would substantially serve the agriculture development of the country. In the year 1912, Rudolf Diesel stated that “The use of vegetable oils as engine fuel a relatively might be unimportant today. But in a certain time period such oils may consider as significant resource like as crude oil and the coal tar products of the present time” (Babu & Devaradjane 2003). In the

20th century, world energy use age has increased significantly due to the effect of industrialization, and

increased

in use age has resulted in inadequate petroleum reserves, such as in the 1970s oil crisis. All of those problems have motivated the scientific society to seek new, alternative energy sources that have decrease global warming and air pollution effects. The past experiences by Stuart staniford

was reported in the

year 2008.

The

energy production from

1970s to 2050 strongly supports the increasing trend of renewable energy consumption. The sustainability of biofuels is progressively promoting its wide acceptance and market demand will go up in the near future. Approximately 27% of transport fuel will be totally replaced by bio fuels by

2050 accordant with the international Energy Agency IEA by (Sanjid et al.

2014). Many researchers and scientists had tried out different types of biodiesel production process, from these fatty acid alkyl esters derived from transesterification of triglycerides, which are commonly known as biodiesel, are well accepted renewable alternatives of petroleum diesel. In last two decades, several studies from across the globe have reported successful operation of compression ignition (C.I) engines with biodiesel derived from a host of feed stocks and their blends with mineral diesel.

2.3 FATTY ACIDS IN THE VEGETABLE OIL

Generally, vegetable oils such as canola oil, soybean oil, sunflower oil and corn oil are used as raw materials for biodiesel production. Because these oils contain relatively many unsaturated fatty acids, they can exist as liquid at room temperatures and show good fluidity at low temperatures. However, vegetable oils have little output and are expansive. In contrast animal fats such as beef tallow, lard and chicken oil animal fats are more stable than vegetable oils in oxidation and have higher heating values and cetane numbers, they have the drawback of being solids at room temperature. Thus depending on the fatty acids contend in the oil / fat used in biodiesel

production, the produced biodiesels can have different fuel properties. Oils/fats mainly consists of saturated fatty acids such as myristic acid (C14:0), palmatic acid (C16:0), stearic acid (C18:0), and saturated fatty acids such as palmitoleic acid (C16:1), linoleic acid (C18:2), linolenic acid (C18:3) oleic acid (C18:1) (Hong et al. 2015).

Table 2.1 Common fatty acids Chemical structures

S. No. Chemical Formula Structure (xx:y)

Fatty acid name Fatty acids Chemical name 1

C18H30O2 18:3

Alpha-linolenic acid Octadecatrienoic 2

C16H32O2 16:1 Palmitoleic Hexadec-9-enoic

acid 2

C20H44O2 20:0 Behenic Docosanoic 3 C10H20O2 10:0 Capric acid Decanoic acid 4 C18H32O2 18:2 Linoleic Cis-9,cis-12- Octadecadienoic 5 C8H16O2 8:0 Caprylic acid n-octanric acid 6 C32H42O2 22:1 Eurucle Cas-13-docosenoic 7 C18H30O2 18:3 Linolenic Cis-9,cis-12,Cis-15-Octadecatrienoic 8 C6H12O2 6:0 Caproicacid Hexanoic acid 9 C24H48O2 24:0 Lignoceric Tetracosanoic 10 C18H36O2 18:0 Stearic Octadecanoic 11 C14H28O2 14:0 Myristic Tetradecanoic 12 C18H34O2 18:1

Oleic Cis-9- Octadecanoic 13 C12H24O2 12.0 Lauric Dodecanoic 14 C16H32O2 16:0 Plamitic Hexadecanoic 15 C20H40O2 20:0

Arachidic Eicosanoic

Where, xx indicate carbonsnumbers and y the quantity of double bonds in the fatty acid chain

The diesel engine can also use

the unprocessed vegetable oil

with needed modification in the engine driving habits. In general vegetable oil

contains saturated hydrocarbon and Triglycerides which possess fatty acids. Vegetable oils

also have

different chemical structures and it is occupied with three fatty acids connected by glycerol molecules on ester linkage. The fatty acids vary depending on

the carbon chain length and its double bond amount. Table 2.1 represents the about the fatty acids with their chemical name, structural information and formula.

Vegetable oil contains a very high structure of fatty acids, so it

gives higher cetane number and it has less prone oxidation, due to its high percent of saturated fatty acid and free acids.

2.4

VEGETABLE OIL PROPERTIES

Barnwal & Sharma (2005), Gashaw & Lakachew (2014) described that the vegetable oil fuel properties in Table 2.2. It suggests that the vegetable oil kinematic viscosity differs at 380C with the range of 30-40 cSt

.The

causes for

the high viscosity of these oils is primarily due to their large molecular mass in the range of 600-900. This is about 10-20 times higher than that of diesel fuel (Rakopouls 2013). The vegetable oil has very high flash point (above 2000C) and the heating value are in the range of 39-40 MJ/kg as compare to diesel fuel (about 44 MJ/kg). The presence of chemically bound oxygen in vegetable oil lowers their calorific values by about 10%. The range of cetane numbers in the vegetable oil is 32-40.

The blends of diesel fuel with vegetable oils would solve the problems developed in the operation of C.I engine with pure vegetable oils. Diesel fuel dissolves

rather than the vegetable oils (Gashaw & Lakachew (2014), (Wang et al. 2006)).

Table 2.2 Properties of Vegetable oil (Barnwal & Sharma (2005); Gashaw &

Lakachew (2014))

Vegetable oil

Density (kg/l) Kinematic viscosity at 38oC (mm2/s) Heating value (MJ/kg) Flash point

o (C) Cloud point o (C)

Pour point (oC)

Cetane no. Diesel 0.855 3.06 43.8 56 - -16 50 Palm 0.9180 39.6 - 267 31 - 42 Soya bean 0.9138 32.6 39.6 254 -3.9 -12.3 37.9 Sunflower 0.9161 33.9 39.6 274 7.2 -15.0 37.1 Corn 0.9095 34.9 39.5 277 1.1 -40.0 37.6 Sesame 0.9133 35.5 39.3 260 -3.9 -9.4 40.2 Cotton seed 0.9095 33.5 39.5 234 1.7 -15.0 41.8 Safflower 0.9144 31.3 39.5 260 18.3 -6.7 41.3 Crambe 0.9048 53.6 40.5 274 10.0 -12.2 44.6 Rapeseed 0.9115 37.0 39.7 246 -3.9 -31.7 37.6 Linseed 0.9236 27.2 39.3 241 1.7 -15.0 34.6 Peanut 0.9026 39.6 39.8 271 12.8 -6.7 41.8

2.5 APPROPRIATE

Table 2.2 clearly proved that using vegetable oil

converted in to the bio-diesel is to replace the partial requirement of

diesel because of the following appropriate

1.

Energy density has similar (or) nearby to fossil diesel fuel.

2. Calorific values of different vegetable oils are approximately

90% that of petroleum diesel fuel.

3.

Cetane number is similar or close to

that of diesel fuel.

4.

Compared with

diesel, the normal chain length for plant oils is from 12 to 22 carbon atoms, with 0 to 3 double bonds that are responsible for the vegetable oil Physico-chemical properties.

Long chain saturated, unbranched hydrocarbons are especially suitable for conventional diesel fuel. The long, unbranched hydrocarbon chains in the fatty acids meet this requirement. (

Barnwal & Sharma (2005); Gashaw & Lakachew (2014))

2.6

EFFECT OF ENGINE CHARACTERISTICS USING STRAIGHT VEGETABLE OIL IN COMPRESSION IGNITION ENGINE.

It has been found that natural vegetable oil can be used as a alternative for diesel fuel in C.I engine but it has the complying problems,

as

in the main first

it have phospholipids, sterols , water,odorants and other impurities.

x The injection process, leads to poor atomization which may be the reason of high viscosity.

x The high flash point attributes to lower volatility.

x Vegetable

oil’s has

improper air fuel mixing that produces

incomplete combustion contributed heavy smoke emission.

x Injection nozzle failure.

x

High carbon deposits

x Lubricating oil dilution

x

vegetable oil‘s

could and pour points are significantly higher than those of petorelum diesel. These high values may do problems in cold weather.

x Dragged engine liner.

x

Ring is beyond a surface.

x

In C.I engine, the SVO characteristics of injection,atomization

and combustion are significantly different from the petroleum fuel due to

poor physical properties.

x

Due to above stated problems the SVO are in need of modification.

To solve these problems, the oil is in need of small

chemical modification. These problems can be cleared if the vegetable oil are chemically changed in to bio-diesel, which

has similar character to diesel

reported by Lokanatham & Ravindranath (2013); Balakrishna (2012); Balat & Balat (2008).

2.7 TECHNOLOGY USED IN THE CONVERSION OF SVO IN TO THE BIODIESEL

In common, micro-emulsion, thermal cracking, preheating, blending and transesterification are the main methods available for the conversion of SVO in to biodiesel. Amongst, the

common systematic way to treat transesterification is SVO.

2.7.1 Transesterification

Transesterification is the method of replacing the alkoxy group of an ester compound by alcohol and the chemical reaction was frequently catalyzed by a base. Transesterification plays a vital role for producing bio- diesel from bio-lipids. The transesterification of vegetable oil implicates reaction of triglycerides (fat/oil) by alcohol to from ester and glycerol (Subramaniam et al. 2013).

2.7.1.1 Transesterification reaction

Transterification, also

named as alcoholics. It is the method of replacing

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alcohol from an ester by some other alcohol, which resembles to

hydrolysis anticipate before that an alcohol is used as a substitute of water. This has been

usually applied to cut down the viscosity of the triglycerides. The transesterification is denoted as

RCOOR’ + R’’OH R’COOR’’ + ROH

ESTER ALCOHOL ESTER Alcohol (1)

Equation (1)

demonstrates the

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transesterification process. The methanol is used in this process, so it is called methanolysis. Methanolysis of triglycerides is represented in Equation (2).

CH2-COOR1 CH2OH R1COOCH3

CH-COOR2 + 3C H3OH CH OH + R2COOCH3

CH2-COOR3 CH2OH R3COOCH3 Vegetable Oil Alcohol Glycerol Methyl-ester (2)

Equation (2) Methanolysis of Tri-glycerides

Transesterification

process is probably considered as

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one of the reversible reactions and proceeds basically by mixing the reactants. But, the presence of a catalyst (a strong acid or base) speed up the conversion (

Adewale et al. 2015).

2.7.1.2 Chemistry of transesterification process

The Equation (2) denotes the complete transesterification reaction. Though;

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three successive and reversible reactions are conceived to occur. These reactions are indicated in Equation (3).

ROH + Triglycerides R1COOR + Diglycerides

ROH + Diglycerides

ROH + Monoglycerides R2COOR + Monoglycerides

R3COOR + Glycerol

(3)

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Equation (3) Chemistry of transesterification

The first step transition presents the triglycerides to diglycerides, adopted by the conversion of diglycerides to monoglycerides controlling one methyl ester molecule from each glycerides at each step

reported by Meher et al. 2004.

2.7.2

Variables Affecting Transesterification Process

The process of transesterification brings about drastic change in viscosity of the vegetable oil. The high viscosity component, glycerol is removed and hence the product has low viscosity like the fossil fuel. The biodisel produced is totally miscible with mineral diesel in any proportion. Flash point of the biodiesel is lowered after transesterification and the cetane number is improved. The yield of bio-diesel in the process of transesterification is assected by several process parameters which include; presence of moisture and free fatty acid (FFA), reaction time, reaction temprature, catalyst, molar ratio of alcohol and oil & mixing intensity

2.7.2.1 Reaction temperature

The rate of reaction is strongly affected by the reaction temperature.

When the reaction temprature is high, the viscosities of oil is decreased and the outcomes with an increased rate of reaction needs larger amount of enegry

for the reaction to happen. Hence, the percentage of biodiesel production is raised. However, the temperature of reaction must be less than

the alcohol boiling point (boiling point of methanol is at 60°C-70°C at

atmospheric pressure) to ensure the alcohol will not be lost through vaporization. Also, the yield of bio-diesel decreases if the reaction temperature goes beyond its optimum level because a higher reaction temperature will accelerate the saponification reaction which results in a lower yield. Depending upon the type of oil, the maximum yield is obtained at temperature varies from 60°C

to 80°

C . (Amit Agarwal et al. 2015).

2.7.2.2 Catalyst concentration

Catalyst concentration can affect the yield of the bio-diesel production. As mentioned earlier, basic catalysts are usually preferred to acid catalysts because of the higher reactivity and the lower process temperature required. The sodium hydroxide is less effective than sodium methoxide because of the decreased amount of water produced upon mixing methanol with sodium hydroxide. As the concentration

of catalyst increased, the conversion of triglycerides and the yield of methyl esters

also increased. A concentration of NaOH in the range of 1.0-1.4%(W/W) has been found to produce 90-98% conversion from jatropha oil to methyl ester. As for the concentration of KOH ranging from 0.55 to 2.0% (W/W) 95-99% of jatropha yield of bio-diesel has been obtained. However, the yield of

bio-diesel is

reduced if the alkali catalyst were added above their optimum concentration as this causes more soap formation (Amit Agarwal

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et al. 2015).

2.7.2.3 Methanol to oil molar ratio

Alcohol to triglyceride is one of the most important factor

for affecting the bio-diesel yield.

Stoichiometrically 3 moles of alcohol and 1 mole of triglyceride are needed for transesterification to yield 3 moles of fatty acid methyl ester and 1 mole of glycerol is used. It varied from 5.6

to 7.8:1 for both the catalyst systems. Bio-diesel

yield could be elevated by introducing an excess amount of methanol to shift the equilibrium to the right hand side. Ethanol, propanol, methanol, butanol, and any alcohol can be used in the transesterification reaction, amongst these alcohols methanol

has physical and chemical advantageous (shortest chain and polar alcohol) and the cost is also low. At 1:6 oil/methanol results exhibit highest methyl ester yield is nearly 99.5% (Gashaw & Teshita 2014; Leung et al. 2010).

2.7.2.4 Reaction time

The conversion time increases with the rate of fatty acid esters also increase. In the starting stage

the reaction is slow due to the mixing and dispresion of alcohol in to oil. After some time the reaction

continues in a very fast manner. In general, within 90 mins the yields reached

a maximum reaction time. In addition that the excess reaction time will result

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to a reduction in the product yield due to the backward reaction of transesterification, resulting in loss of ester as well as causing more fatty acids to form

soaps. (Gashaw & Teshita 2014; Leung et al. 2010).

2.7.2.5 Free fatty acid and water content

The FFA and moisture contents have significant effects on the transesterification of glycerides with alcohol using catalyst. The high FFA content (<1% W/W) will happen soap formation and the separation of products will be exceedingly difficult, and as a result, it has low yield of

bio- diesel

product. In addition, formation of gels and foams hinders the separation of glycerol from bio-diesel. For instance, water content in waste cooking oil will stimulate the hydrolysis reaction and simultaneously reduce the amount of ester formation. To overcome this problem, supercritical method

is

proposed. It may be noted that water has less influence. In supercritical methanol method. Therefore water substance should not always exceed 0.5% to obtain 90% yield of biodiesel and it is more crucial for an acid-catalyzed reaction than base catalyst reaction. The moisture levels of the collected waste

chicken fats vary widely, being as high 18%. Therefore, it is not possible to convert these oils

in

to biodiesel by using single process. One drawback of bio-diesel is that there is an inverse relationship between

bio-diesels

oxidative stability and its cold flow properties. Saturated compounds are less prone to oxidation than unsaturated compounds but they rise the cloud point of the fuel. The reaction of FFAs with alcohol produces ester, but also water that inhabits of the transesterification glycerides. This is due to the effect of the water produced when the FFAs react with alcohol to from esters. The coincidence of the lines indicates that water formation is the primary mechanisms limiting the completion of the acid catalyzed esterification reaction with FFAs (Gashaw & Teshita 2014).

2.7.2.6 Mixing intensity

Oil and alcohols are not totally miscible, thus reaction can occur in the interfacial region between the liquids and transesterification reaction is a moderately slow process. So mixing is very important in the transesterification process, adequate mixing between these two of feed stock is necessary to enhance the transesterification reactions to occur. Mechanical mixing is commonly used in the transesterificarion process. The intensity of the mixing could be varied depending on its necessity in the transesterification process. In general, the mixing intensity must be increased to ensure good and uniform mixing of the feed stock. When vegetable oil with high kinematic viscosity are used as the feed stock, intensive mechanical mixing is required to overcome the negative effect of viscosity to the mass transfer between oil, alcohol and catalyst. Agitation speed plays an important role in the formation of end product (mono alkyl ester (or)

bio-diesel),

because agitation of oil and catalyst mixture enhances the reaction. For example the mixing intensities chosen were 200rpm, 400rpm, 500rpm,

600rpm and 800rpm for 60 minutes while other parameters were kept

constant, because lower stirring speed shows lower product formation. On the other hand, higher stirring speed favors formation of soap (

Gashaw &

Teshita

2014).

2.7.3 Advantages and Disadvantages of Transesterification Process

The transesterification procedure has its

advantages and disadvantages can be completed by using three different methods like chemical catalysts, enzymatic catalyst, and supercritical fluid techniques are presented in Table 2.3 (Huang et al. 2010).

Table 2.3 Advantages and disadvantages of transesterification process

S. No Type of transesterification Advantages Disadvantages 1 Enzymatic catalysis (i) The small amount of methanol required in reaction. (ii) Moderate reaction condition. (

iii) In natural environment it have no pollution. (i) The presence of chemical

in the production process are poisonous to enzyme. (ii) In the conversion of fatty acid short chain enzyme is one of the limitations. 2

Chemical catalysis (i) Large scale production. (ii)

Reaction condition can be well controlled. (iii) The methanol produced in the process can be recycled. (

iv) The cost of production process is low. (v) The production has

higher conversion. (i) Reaction temperature is relatively high and process is complex. (

ii) The process

requires more energy. (

iii) The methanol recycles process needs an separate installation. (iv) The later disposal process is complex. (

v)

The waste water pollutes the environment. 3 Supercritical fluid techniques (i) Safe and fast. (

ii)

Easy to be controlled. (

iii)

Friendly to environmental. (i) High cost of production and waste energy due

to high temperature and high pressure in the reaction.

2.8 STUDIES ON CONVERSION OF VEGETABLE OIL IN TO BIO-DIESEL

Kafugu et al. (2010) stated that in moringaoleifera oil the maximum yield

of biodiesel

was 82%, when the 1wt% of potassium hydroxide and

30wt% of methanol reacted at thetemperature, time, andstirrer speed of 60°C,

60min and 400rpm. It was found that for the above consideration if more than

1% catalyst

would be

added then there is

no clear separation, solubility of methanol increased and more soap was developed during washing. The cause for that is hydrolysis of ester had more fatty acid.

Atapour &

Kariminia (2011) proposed that

Iranian bitter almond oil was carried out using methanol to oil molar ratio varies from 3:11 to 11:1. When the reaction temperature fluctuated from 30°C to 70°C the KOH concentration range varied from 0.15% to 1.8%wt , and the optimal value of methanol, KOH, temperature and yield were 7:1, 0.9, 50°C and 97.2% to

90.8%. When the amount of methanol used was high, it would acquire glycerol as a by-product. The not used up

part of the catalyst becomes soap increase saponification reaction and more than 50°C temperature large number of bubbles produced by vaporization of methanol.

Kaya et al. (2009) declared that the Crude peanut seed oil 89% of bio-diesel

was

obtained at the reaction temperature and time of 65°C, 120 min with 0.5 wt. % of NaOH by using methanol.

Lin et al. (2009) had created bio-diesel

in a two-step process by using crude rice bran oil. The first step procedure

is pretreatment. If processes Free Fatty Acid (FFA) when the values were reduced by using an acid catalyst of $H2SO4$ with less than 1mg and 1%w/w. The alkaline transesterification process

was

the second step. In this process 98.7% bio- diesel yield was got at a temperature and reaction time of 60°C and 60min.

Whereas the methanol/oil ratio is 6:1, and the KOH concentration was

0.9%w/w.

Anwar et al. (2010) stated that in okra seed oil the bio-diesel yield was 96.8%. In this methyl ester four distinct type of catalyst were used like sodium meth oxide(NaoCH3), sodium hydroxide(NaOH), potassium meth oxide(KOCH3), potassium hydroxide(KOH),. The maximum yield was got from

the

meth oxides (NaOCH3 ,KOCH3). Whereas methanol/oil molar ratio was 7:1 and the NaOCH3 Concentration 1%w/w by the reaction temperature and time of 65°C, 120min. From the above condition, okra methyl ester yield was 96.8%.

Suppalakpanya et al. (2010) specified that crude palm oil ester content was received with 1.5% wt. KOH, and the molar ratio of oil to ethanol was 1:8.5. From the same content the bio-diesel production was increased up to 98% wt. by the compression of conventional heating and microwave heating at 70°C. Higher reaction rate is obtained from lowest enzyme concentration 0.5, and the methanol to oil molar ratio was increased (1:1 to

1:25) as well as yield of bio-diesel was also increased (up to 1:15m).

Dhar et al. (2012) has developed bio-diesel by two-step process

method from neem oil. The first process method is esterification and the other process method was

transesterification process. In esterification process method, sulphuric acid was used as a catalyst with neem oil. As a result 20% FFA was reduced.

The

Second step was transesterification process method, in that process, the oil to alcohol molar ratio was 1:6 and the wt of NaOH was

4.5%.with the catalyst reaction time and temperature of 90 min and 45°C, using gravity separation method product

was kept in to a separating funnel up to 5

to 6hrs. Optimum reaction conditions were 1% NaOH catalyst methanol to oil molar ratio was 6:1 and reaction temperature 60°C.

Qiu et al. (2011) stated that biodiesel could be produced from mixed soybean oil and rapeseed oil.

In that process the homogeneous processes were in solid state like

NaOH and KOH. It will react 4000 times quicker than acid catalyst reaction. The acid catalyst was obtained by using a co-solvent n hexane, which

was

given a pre-treatment of soyabeen oil and rape seed oil. The benefit of this method was lower reaction temperature at

55°C. The optimum reaction temperature, molar ratio of oil to methanol, catalyst reaction time and yield of bio-diesel was 55°C, 5:1,08wt%, 120min, and 94% of yield.

Karavalakis et al. (2011) also suggested that use of the homogeneous catalyst like KOH, NaOH soap was formed and the hard of removing the catalyst species after reaction required a big amount of water used to separate and clean the glycerol and the esters. New catalyst and environmentally began processes of tetramethylghanidine optimum catalyst amount of 3wt% molar ratio of methanol to oil of 12:1, reaction time was

90min, yield is 93.8%. Sodium (or) potassium based catalyst required intermediate two stage reaction, increase glycerol and removing time from waste frying oils.

2.9

FUEL PROPERTIES OF ESTERS (BIODIESEL)

Giakoumis (2013) reported that the following

physical and chemical properties of biodiesel from various feed stocks were analyzed; a lot of data'

s

was carefully collected from

various papers published in international Journals, Conferences and

the

reports issued by well-known research centers. Mostly, the whole electronic databases of IEEE, Elsevier, American Chemical Society, Taylor and Francis, Wiley International, Springer and SAGE Publishing were scanned. For exciting papers, amount of data sets contributed from papers presented in ASABE, SAE International and various other indexed conferences around the world.

As a final point, 26 feed

stocks

0: https://www.researchgate.net/profile/Evangelos_Giakoumis/publication/261009368_An_insight_into_biodiesel_physico-chemical_properties_and_exhaust_emissions_based_on_statistical_elaboration_of_experimental_data/links/54e5e5f80cf2cd2e028b399c.pdf?inViewer=0&pdfJsDownload=0&origin=publication_detail 76%

were designated for the statistical investigation, namely four animal fats (beef tallow, chicken, lard and fish), twenty-one oils derived from various plants (canola, castor, coconut, corn (maize), cottonseed, croton, hazelnut, karanja (honge), jatropha, linseed (flax), mahua, neem, olive, palm, peanut, rapeseed, rice bran, rubber seed, safflower, soybean and sunflower), as well as waste cooking oil. These 26

feed stocks finally

provided 945 different data series of biodiesel properties and 700 data series of fatty acid compositions. On the

other hand, the dataset contains a good junk of American, European and Asian animal or vegetable feedstock-both edible and non-edible

as tabulated in Table 2.4. It is also attractive that soybean methyl ester or methyl soyate (SME) had been investigated.

in general

worldwide (although the majority of the research had been conducted in America) and with a continuous interest over the last three decades

in oils included recently.

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Rapeseed and the closely related canola (10.4+3.1=13.5%) were the next best familiar feedstocks, with a significant interest also found in palm (7.8%), the

non-edible

jatropha (6.9%;

mainly during the last years in India), sunflower (5.6%) andwaste cooking (9.5%). On the other hand, all animal fats (including fish) are of the order of 8.1%, with the majority

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of the research (50%) focused on beef tallow. The non-edible

cotton seed with 3.1% (more popular in the Mediterranean countries),

karanja with 3.8% (primarily during the last few years), as fine as rice bran (2.5%), castor (3.3%), coconut (2.4%),

corn (used for ethanol production too í 2.0%),safflower (1.8%) linseed

(1.9%), and neem and mahua (each 1.5%) comprise smaller but still important shares of the world wide bio-diesel research various other feedstocks such as grape seed, tobacco seed, jojoba (simmondsia chinensis), babassu (orbignya oleifera), polanga/punnai (calophyllum inophyllum), sesame (sesamum indicum)

chinese pistache (pistachio chinensis), hemp (cannabis sativa), and mustard were not in the

part of

statistical analysis.

Table 2.4 Physical and chemical properties of all biodiesels

Table 2.4 (Continued)

Density (kg/m3) Viscosity (mm2/s HHV (kJ/kg) LHV (kJ/kg) CFP point (°C) Pour point (°C) Cloud point (°C) Flash Point (°C) Cetane number Oxidation Stability (h) Iodine number Distillation temp.50% vol. (°C) Distillation Temp.90% Vol.(°C)

Sulfur content (ppm) Acid number (mg KOH/g)

Molecular weight (kg/kmol) O (% w/w) H (% w/w) C (% w/w)

Sunflower 882.9 (4.70) 4.53 (0.51) 40,000 (647) 37,800 (895) -3.5 (2.0) -3.8 (2.3) 0.9 (2.4) 172.0 (7.9) 51.9 (4.73) 1.3 (0.42) 128.6 (2.1) 355.7 (7.2) - 356.5 (2.1) 0.32 (0.14) 294.05 (0.50) 10.98 (0.15) 11.84 (0.29) 76.90 (0.37)

Palm 874.7 (4.01) 4.61 (0.61) 39,985 (688) 37,080 (855) 11.4 (4.7) 11.8 (2.4) 13.3 (2.3) 161.9 (17.3) 61.2 (4.90) 11.4 (2.38) 52.7 (5.6) 327.7 (4.0) 339.3 (6.7) 3.1 (2.7) 0.27 (0.11) 284.12 (0.14) 11.27 (0.43) 12.44 (0.50) 76.09 (0.37)

Neem 874.5 (7.54) 5.06 (0.54) 40,180 (879) 36,880 (53) _ 4.0 (2.2) 4.0 (1.0) 150.6 (24.1) 56.9 (6.07) 2.1 70.8 (13.1) _ _ _ 0.41 (0.09) _ 10.72 12.10 76.7

Soybean 882.8 (5.12) 4.29 (0.37) 40,020 (671) 37,750 (944) -3.6 (3.4) -3.0 (2.7) 0.1 (2.0) 158.8 (19.5) 51.8 (3.93) 5.0 (2.59) 126.0 (6.9) 336.3 (6.1) 345.0 (6.6) 2.7 (3.8) 0.32 (0.28) 290.83 (2.17) 10.95 (0.52) 11.90 (0.31) 77.03 (0.50)

Mahua 874.5 (7.54) 5.06 (0.54) 40,180 (879) 36,880 (53) _ 4.0 (2.2) 4.0 (1.0) 150.6 (24.1) 56.9 (6.07) 2.1 70.8 (13.1) _ _ _ 0.41 (0.09) 294.4 _ _ _

Rice bran 880.9 (5.71) 4.70 (0.44) 40,475 (1201) 38,045 (1335) 0.3 (2.4) -0.9 (2.7) 5.0 (4.8) 157.8 (23.3) 56.3 (5.49) _ 91.8 (13.0) 336.0 346.0 (1.7) 5.3 (4.7) 0.28 (0.18) _ 11.26 12.38 76.22

Rapeseed 882.2 (4.06) 4.63 (0.37) 40,335 (743) 37,625 (663) -11.3 (5.9) -9.7 (3.4) -3.3 (2.4) 164.4 (15.5) 54.1 (3.32) 7.4 (1.81) 111.7 (7.4) 340.7 (10.1) 344.3 (4.6) 4.6 (3.6) 0.24 (0.11) 294.80 10.93 (0.48) 11.84 (0.46) 77.07 (0.49)

Peanut 882.9 (2.62) 4.77 (0.26) 39,930 (205) 38,050 16.3 (0.6) -2.7 (8.4) 4.3 (3.8) 174.5 (5.9) 54.9 (2.74) _ 80.5 (15.1) _ _ 5.9 (7.8) 0.19 (0.13) _ _ _ _ Waste cooking 880.6 (6.04) 4.75 (0.46) 39,805 (607) 37,880 (1088) -2.8 (3.3) -0.3 (5.5) 5.3 (5.1) 161.7 (19.0) 56.2 (3.91) 5.0 (3.28) 85.1 (17.1) 340.4 (6.7) 350.3 (8.6) 8.6 (14.6) 0.41 (0.14) 289.31 (3.86) 10.77 (0.47) 12.02 (0.44) 76.90 (0.55)

Safflower 883.8 (5.21) 4.10 (0.12) 40,155 (644) 38,145 (862) -8.3 (2.1) -8.1 (2.9) -4.9 (1.7) 169.9 (17.6) 51.8 (2.53) _ 136.7 (5.7) _ _ _ 0.32 (0.05) _ _ _ _

Olive 881.2 (1.56) 5.05 (0.39) 40,280 (1048) 37,290 -5.3 (3.8) -5.0 (1.7) -2.0 (0) 171 (6.5) 58.9 (2.00) 1.7 (2.33) 80.3 (3.2) _ _ 7.8 (3.1) 0.37 (0.33) _ _ _ _

Linseed 891.5 (3.61) 4.06 (0.20) 40,410 (676) 37,830 (1713) -7.7 (1.7) -8.0 (4.2) -1.7 (2.0) 170.3 (5.6) 51.3 (4.24) 0.40 (0.25) 184.5 (17.9) 338 361 8.2 (8.5) 0.29 (0.15) 290.07 (3.49) 11.29 (0.31) 77.75 (0.37)

Rubber seed 882.3 (4.37) 4.79 (0.63) 40,350 (1018) 37,820 (807) -1.0 -7.0 (1.3) 3.6 (1.5) 158.3 (24.8) 50.4 (1.14) 7.2 (0.93) 139.7 (6.2) _ _ _ 0.22 (0.10) _ _ _ _ Source: Evangelos 2013, Physical and chemical properties of various biodiesel.

2.9.1

0: https://www.researchgate.net/profile/Evangelos_Giakoumis/publication/261009368_An_insight_into_biodiesel_physico-chemical_properties_and_exhaust_emissions_based_on_statistical_elaboration_of_experimental_data/links/54e5e5f80cf2cd2e028b399c.pdf?inViewer=0&pdfJsDownload=0&origin=publication_detail 67%

Viscosity

Viscosity is a measure of the fluid resistance which is being distorted by either tensile stress

or shear. In the case of liquid fuels, the less viscosity of fluid makes high easier movement (fluidity). The diesel engine has higher viscosity which contributes to minimum of exact operation for the fuel injectors, and the lesser atomization of the fuel spray has high viscosity which stands for

that cannot be used carefully as fuels in a C.I.

engine. However, without earlier heating (decreases viscosity exponentially with temperature increasing), and only for moderately small mixing ratios.

A European stipulation says that an adequate viscosity of biodiesel range is 3.5 to 5 mm2/s and that is excluded from B100 form. In the US, the specification

of the range is (1.9-6mm2/s), much high and the concept depicts all 25

analyzed feedstock’s acceptable by Giakoumis (2013).

2.9.2 Density

As comparedto petroleum diesel,

bio-

diesel fuels have high viscosity, which intends that with respect to volume the

fuel pumps will inject high mass of methyl ester than petroleum diesel fuel. These have an effect

upon the fuel

í

air ratio thus the exhaust gas temperatures and NOx

emissions.

In actual fact, there is a link among the NOx emissions and density,

lower density preferring lower NOx, Eventhough, no other investigators have not supported such an unambiguous trend. Investigation of

25 types of bio-diesels

and again excluding of castor, the ranges are starting

from 870.8 to 891.5 kg/m3, with the total average value being 880.2 kg/m3 (i.e. almost 5% higher than the corresponding

conventional diesel value). The average density of

caster

bio-diesel is 917.6kg/m3 it is totally contradictory from other 25 bio-diesel

feedstocks. In US, there is no particular specification for density of

bio-diesel

but in the EU suitable range is little bit varied from 860-900kg/ m3. The average density of

caster biodiesel is

917.6kg/m3 it is completely different from other 25 feedstocks of bio-diesel.

When the amount of double bonds increased the density

is

also increased,

which means that, the more unsaturated the originating oil, the higher the density of the derived bio-diesel, and the larger the fuel mass that will be injected

in a diesel-tuned engine is run on bio-diesel.

The correlation, however, among the cetane number and density

from all studied feedstock is represented with higher densities imparting lower cetane numbers by Giakoumis (2013).

2.9.3 Heating Value

The higher heating value (HHV) and lower heating value (LHV) of fuel is measured based on the fuel’s combustion, with the difference among them constituting the water’s heat vaporization.

The average value of bio-

diesel has 10í12% w/w oxygen, which ensues to a proportionally lower

heating value and energy density. As a result it indicates that in order to reach the same engine power output more fuel is needed for injection compared with the diesel. In the most recent research work technology, using

bio-diesel

resulted in

the decrease in PM/smoke emissions or from other

bio fuel

such as alcohols derived from the higher oxygen content of the

bio fuel-

diesel fuel blend that enhances the in-cylinder soot oxidation rate.

From another point of view,

the fact that larger mass of biodiesel requires to be injected in order

that

to achieve the same engine power as with conventional diesel fuel.

The resulting oil contained

the HHV, accordingly, it is 38,980 (coconut), 40,550 kJ/kg (fish) and the overall average value is 40,100 kJ/kg. The effect of unsaturation on the heating values shows that increasing the unsaturation or the number of double bonds results in a moderate increase in the FAME heating value.

Both heating values correlate rather well with the oxygen content, whereas LHV seems to have a significant inter-dependence with viscosity and cloud point

by Giakoumis (2013).

2.9.4 Flash Point

The flash point

temperature of fuel can be measured by heating process.in this process the vapor and air mixtures make the fuel ignited. The value of flash point and fuel’s volatility are related reciprocally. The above reason clearly states that the storage of pure biodiesel is much safer than diesel. In detail, the present works represents the average values of flash point for all 26 analyzed feed stocks, the ranges are from 127.7 °C to 174.5°C and the mean value from all feedstocks constituting 163.3°C.

In us

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specifications require biodiesel fuels to have at least 93°C flash point, whereas in the European the minimum essential level is 101°C; both are

intended to determine a lower purity limit in the final FAME, and are easily

coped with by all examined feedstocks of

biodiesel. It should be observed that some researchers have described about very low

flash point values.In general, the originating oil is not straightly linked to the

composition of fatty acid. the Relationship between flash point and unsaturation degree (excluded methyl ester of coconut oil), while with a little increasing trend is observed that the double bonds number is raised

by

Giakoumis (2013).

2.9.5 Cetane Number

Cetane number plays a crucial role in diesel fuel and it is expressible by a pure number (

dimensionless cetane number (CN)), which denotes the fuel ignitability, primarily critical on cold starting conditions. Low cetane numbers makes long ignition delay that means

a long time among the start of combustion and fuel injection. In accordance with, the Cetane Number islower for sudden premixed combustion phase and the contribution of combustion noise radiation is high. As an alternative the higher CNs encourage lower NOx emissions and faster fuel auto-ignition. In general the

bio-diesel

CNs is higher

than that of the petroleum diesel.

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Excluding castor, the (average) cetane numbers of the 25 feed stocks range from 50.4 for rubber seed methyl ester to 61.2 for PME, with the mean value from all feedstocks

being 54.8;

that is 8

í10%

higher than that of petro-diesel. biodiesel Cetane

Number

may be caused by i) differences in the fractional conversion of triglycerides to ester, and ii) by presence of residual methanol and glycerol in the final fuel; the second argument, however, has not been confirmed by the present statistical analysis, which included methanol and (free and total) glycerol effects. It has also been argued that the effect of blending biodiesel on the CN is approximately linear for mixtures of esters with diesel fuel

reported by Giakoumis (2013).

2.9.6 Iodine Number

The

iodine value (IV) or iodine number (IN) is a parameter used to resolve the unsaturation degree in a vegetable oil or animal fat. This number specifies the iodine mass (I2) in grams that is necessary for complete saturation by means of a stoichiometric reaction, the molecules of 100 g of given oil. The average values of

iodine for the examined feedstock's ranges are

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from 7.8 (for the most saturated, coconut) to 184.5 (for the most unsaturated one, linseed), with an overall average value

of 98.4

reported by Giakoumis (2013).

2.9.7

0: https://www.researchgate.net/profile/Evangelos_Giakoumis/publication/261009368_An_insight_into_biodiesel_physico-chemical_properties_and_exhaust_emissions_based_on_statistical_elaboration_of_experimental_data/links/54e5e5f80cf2cd2e028b399c.pdf?inViewer=0&pdfJsDownload=0&origin=publication_detail 100%

Oxidative Stability

One of the major issues that limit the use of

bio-diesel as a fuel in compression ignition engines is its poor oxidative stability. A brief assessment of

this work

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reveals that only a few of the investigated bio-diesels

fulfill the European specification of 6 hours oxidation stability

using the Rancimat method (EN 14112). The US specifications are also in this case

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more lenient, hence biodiesel produced from feedstocks such as corn, coconut, fish, jatropha and karanja could be approved.

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Many researchers used additives or did not apply the same esterification clean-up or storage conditions, all of which influence considerably the oxidation values

reported by Giakoumis (2013).

2.9.8 Carbon Content and Stoichiometric Air-fuel Ratio

This result provides that

the correlation between FAME carbon content and number of carbon atoms with the respective degree of unsaturation.

The number of carbon atoms for

the corresponding sample is narrower compared to the previous properties. It seems that the higher the unsaturation of the originating feedstock, the higher the carbon content although the absolute differences are small. However, no such correlation could be established for the hydrogen content. Apart from the degree of unsaturation, the FAME carbon content correlates rather well with the cetane number, density, viscosity and all cold flow temperatures. The FAME, AFst (Air- Fuel Stoichiometric) correlates well with the cetane number, density, viscosity, higher heating value

and interestingly, oxidation stability. In general, the stoichiometric air–fuel ratio is of the order of 12.5:1 for the neat

bio-diesel,

whereas it is approximately 15:1 for the neat diesel fuel. This means that the air–biodiesel mixtures can reach the stoichiometric conditions nearly 15% faster than their air–diesel counterparts. It is expected that this difference in the air–fuel ratio, caused by the presence of oxygen in the bio-diesel, must influence combustion. More specifically, if the combustion process is mixing controlled, the use of biodiesel injection has a beneficial effect, while if the combustion process is evaporation controlled, the effect is adverse

reported by Giakoumis (2013).

2.9.9

Distillation Temperature

Distillation is a method of separating mixtures based on differences in volatilities of components in a boiling liquid mixture.

The diesel contains hundreds of different compounds. It was demonstrated that most of the bio-diesels

generally contain a few number of major fatty acid compounds; these boil at roughly the same temperature (approximately 330°C

to 357°

C at atmospheric pressure). The distillation temperature specification has been

incorporated to ensure that fuels have not been contaminated with high boiling materials such as used motor oil. Hence, the 20 distillation of biodiesel fuel is a method that actually provides a demonstration of the FAME’s quality.

however, indicate that increasing the number of double bonds increases also the distillation temperature, with highly unsaturated feedstocks, such as linseed and fish (Rich in tri- and higher unsaturated acids) exhibiting the highest values

reported by Giakoumis (2013).

2.9.10

Low-Temperature Flow Properties

The input flow properties for winter fuel specification

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are cloud and pour point. Cloud point (CP) is the temperature at which wax form a cloudy appearance.

It is measured as the temperature of first formation of wax as the fuel is cooled. Pour point (PP)

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is the lowest temperature at which the fuel becomes semi solid and loses its flow characteristics being no longer pumpable;

hence it is a measure of the fuel gelling point.

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The pour point is always lower than the cloud point.

The cloud point is not generally affected by additives called flow improvers. However, flow-improver additives can decrease the size, or extent of the formation, from the wax crystallites formed on cooling the fuel, and so it lowers the temperature at which wax plugging becomes a problem. Poor cold flow properties may result in fuel line and pump blockage, ultimately leading to fuel starvation. These problems are particularly making better during cold starting particularly at low ambient temperatures.

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There are no European or US specifications for low temperature properties (Each country is free to determine its own limits according to local weather conditions), but it is well known that biodiesel fuels suffer from cold flow properties

way more (i.e. they are higher)

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than mineral diesel fuel. The (average) values of CP for the 26 examined feedstocks in the present work confirm this fact; they range from

í15.1

oC (castor ME; however with 12.5oC

standard deviation, which indicates contradicting results) to 13.8°C (Tallow

methyl ester (

ME)), with the mean value from all feedstocks being 2.1oC (almost 20oC higher than for conventional diesel fuel). For PP, it is

í23.4

oC (again castor ME; standard deviation 3.9oC) up to 11.8oC (palm ME), with an overall mean value of

í2

oC (More than 30oC higher than conventional diesel fuel).

In order to enhance the poor cold flow properties of biodiesel, various

measures have been suggested, such as the use of cold-flow improvers, ethyl or iso-propyl esterification (Higher cost than the use of methanol), removal of those components that have high melting points (Stearic and palmitic), or even modification of the fatty acid composition of the originating oil in order to produce an ester with optimum characteristics (concerns arise in this case over genetic modification issues

reported by Giakoumis (2013).

2.10 OPTIMIZATION PROCESS

In general, there are three categories of catalysts used for

bio-diesel

production: alkalis, acids and enzymes. Enzyme catalysts have become more attractive recently since it can avoid soap formation and the purification process is simple to accomplish. However, they are less often used commercially because of the longer reaction times and higher cost. To reduce the cost, some researchers developed new

bio catalysts

in recent years. Compare with enzyme catalysts, the alkali and acid catalysts are more commonly used in biodiesel production. The alkali and acid catalysts include homogeneous and heterogeneous catalysts. Due to the low cost of the materials, sodium hydroxide and potassium hydroxide are usually used as alkali homogeneous catalysts and alkali-catalyzed transesterification is most commonly used commercially. These materials are the most economic because the alkali-catalyzed transesterification process is carried out under a low temperature and pressure environment, and the conversion rate is high with no intermediate steps. However, the alkali homogeneous catalysts are highly hygroscopic and absorb water from air during storage. They also from water when dissolved in the alcohol reactant and effect the yield. Therefore, they should be properly handled. On the other hand, some heterogeneous catalysts are solid and it could be rapidly separated from the product by filtration, which reduces the requirement. In addition, solid heterogeneous catalysts can stimulatingly catalyze the transesterification and eaterification reaction that can avoid the pre-esterification step, thus the catalysts are particularly useful for those feed stocks with high free fatty acid content. However, using a solid catalysts, the reaction proceeds at a slower rate because the reaction mixture constitutes a three-phase system, which due to diffusion reasons, inhibits the reaction.

The three categories of catalysts with their advantages and disadvantages shown in Table 2.5 (Leung et al. 2010).

Table 2.5 Advantages and disadvantages at different types of catalysts in the biodiesel production

Type Advantages Disadvantages Example Alkai Homogeneous Favorable kinetics, High catalytic activity, modest operation conditions,low cost. Emulsion formation, low FFA requirments,more wastewater from purification,

anhydrous conditions, disposable. NaOH,KOH. Heterogeneous Enviormentally benign, non coressive, easily sepration, fewer disposal problems, longer catalyst life times, recyclable, higher selectivity. More waste water from purification, Low FFA requirment, high molar ratio of alcohol to oil requirment, anhydrous conditions,diffusion limitations, high pressure, reaction temperature and high cost. CaO, CaTiO3,CaZrO3, Cao-CeO2,

Ca2Fe2O5, CaMnO3, KOH/Al2O3, Al2O3/KI, KOH/NaY, alumina/silica supported, K2CO3, .

ETS-10 Zeolite.

Table 2.5 (Continued)

Type Advantages disadvantages Example

Acid Homogeneous Catalyze esterification and transesterification simultaneously, avoid soap formation. Equipment corrosion,

long reaction time,waste water from neutrlization, higher reaction temperature, weak catalytic activity,difficult to recycle. Concentracted sulphuric acid Heterogeneous Catalyze esterification and transesterification simultaneously recyclable, eco- friendly. Low microporosity, low acid site concentrations,high cost, diffusion limitations. TiO2/SO24,

ZrO2/SO2 , 4 ZnO/I2, , carbon-based solid acid catalysts, carbohydrate-derived catalysts, Vanadyl phosphate, Nafion-NR50, Sulphated zirconia, Vanadyl phosphate, Amberlyst-15.

Enzymes Avoid soap formation, non polluting, easier purification. Expansive and denaturation. Rhizomucor mieher lipase, candida antarctical fraction B lipase.

Atapour and Kariminia (2011)

prepared biodiesel from bitter almond oil (BAO). In their investigation 0.9% w/w of KOH concentration produced maximum yield of 90.8% w/w of bio-diesel.

Karnwal et al. (2010) prepared bio-diesel from Thumba oil by transesterification in the presence of potassium hydroxide as catalyst. In their study for 0.75% of KOH concentration maximum ester conversion of 97.8% was obtained.

2.11 QUALITATIVE ANALYSIS (

GC-MS)

An effective and proven combination for fatty acid and bio diesel analysis is Gas chromatography (GC) and mass spectrometry (MS), to identify and quantify the various components of the sample mixture simultaneously. Due to the high sensitivity and high specific detection of this technique, it allows a much finer degree of substance detection than either unit used one by one.

The capacity of GC-MS to confirm identity and separate by mass spectral structure elucidation has facilitated researchers to learn the influence of fatty acids on the quality of

bio-diesel

fuel. For example, methyloleate was proposed as a appropriate major

bio-diesel

component. Knothe also noted that, due to low-temperature properties methyl palmitoleic acid can be used an alternative to methyloleate. The identification of components by GC-MS has continued to gain importance in establishing the oil composition and quality reported by Iko et al. (2015).

Koria & Nithya (2012) reviewed that, fatty acid composition of the bio-diesel samples synthesized from beef tallow oil and soybean has influence on density, viscosity, and cloud point. Physical and fuel properties of

bio- diesel

for several oils have been tested and compared with conventional diesel fuel. Daturastramonium (

Tamil name is umathan phu) bio-diesel

is analyzed for its fatty acid composition using GC-MS and important properties such as density, kinematic viscosity, cloudpoint, heat of combustion and cetane index (ignition quality) are studied.

2.12

USE OF BIO DIESEL IN COMPRESSION IGITION ENGINE

Performance parameters like Brake Thermal Efficiency and Brake

Specific Fuel Consumption (BTE & BSFC) and emission parameters of

Carbon monoxide (CO), Hydrocarbon (HC), Nitrogen Oxide (

NOx)

and smoke density of various bio diesel blends have been reviewed briefly for different bio diesel and blends with diesel in the subsequent sections.

2.12.1 Soyabean Oil

Qi et al. (2010) reported the performance and combustion characteristics of a direct injection C.I engine fueled with

bio-diesel

produced from soya bean oil and its various blends (B0, B30, B50, B80, B100). The test result showed a small increase in Break Specific Fuel Consumption (BSFC) for

bio-diesel

and its blends are slightly lower than that of diesel keeps the same trend for low engine loads and higher engine load. The carbon monoxide (CO) and smoke emissions are significant improvement in decrease of biodiesel and its blends and it have little variance from diesel fuel. Bio- diesel and its blends emit slightly higher nitrogen oxides (Nox). This may be due to higher oxygen content in

bio-diesel.

The authors concluded that the excess oxygen contents present in the biodiesel plays a vital role in engine performance and

bio-diesel

is proved to be a potential fuel for partially (or) complete alternative of diesel fuel.

2.12.2 Sunflower Oil

Ulusoy et al. (2009) reported that the emission characterisics of adirect injection, four cylinders, four stroke turbo diesel engine with using sunflower oil methyl ester at different speeds and full load condition. The result showed that, for all engine speeds the smoke, CO and HC emissions of sunflower oil methyl ester are generally lower than diesel fuel. The oxygen content of

bio-diesel

fuel and the test engines turbo charger mechanisms have caused lower emission values by increasing the combustion quality. The author’s concluded that exhaust emissions of

bio-diesel

are lower that of base

diesel fuel, which shows that sun flower oil methyl esters has more favorable effects on air quality.

2.12.3 Mahuan (Madhuca indica)

Godiganur et al. (2009) reported that the performance and emission characteristics of a cummins 6BTA 5.9 G2-1, 158 HP rated power, water cooled, direct injection,

turbo charged,

constant speed (1500 rpm) diesel engine using diesel fuel, mahua bio-diesel and its blends at different load conditions. The test results showed that increased biodiesel blends of proportion significantly reduces the HC and CO emissions due to the

bio- diesel

complete combustion. Though, slightly increased Nox emissions (11.6%) as compared with that diesel. This could be attributed to the exhaust gas temperature increasing and the fact of some oxygen content present in the

bio-diesel

which facilitated NOx formation. It was observed that as compare with diesel the BSEC for B20 was lower but in case of B40, B60 and B100, the BSEC was higher. This reverse trend was observed due to lower heating value with increased

bio-diesel

percentage blends. At B20 (32.5%) the maximum brake thermal efficiency was higher than that of pure diesel. This may be due to calorific value reduction and increased BSFC as compared with B20 . There is a slight increase in BTE (brake thermal efficiency) and small reduction in BSEC (brake specific energy consumption) of B20 percentage blend in engine as compared to diesel fuel. When the concentration of

bio-diesel

blends increased the exhaust gas temperature (EGTs) also increased. Among those the mean EGTs of 7%, 9%, 10% and

12% are with respectable to B20 B40 B60 and B100 and it was higher than the mean EGT of diesel. This may be due to heat loss increased for higher bio-diesel blends and also it is evident from the lower BTE as compared to diesel. As of these findings, it is concluded that without significantly moving the engine performance (BSFC, BSEC, EGT) and emissions (CO, HC, and

NOx) the up to 20% of mahua biodiesel could be safely blended with diesel and it could be suitable substitutable fuel for heavy duty engines.

2.12.4 Mahuan Oil

Puhan et al., (2005) reported that the performance and emission of a constant speed, direct injection ,single cylinder, four stroke compression ignition diesel engine (kirloskar) usingmahua biodiesel. The result exposed that diesel engineperformance with biodiesel neither does not vary significantly. The brake thermal efficiency is 13% lower than that of diesel and brake specific fuel consumption is 20% higher than that of diesel. Exhaust emission pollutant are reduced when compared to diesel. Carbon monoxide, Hydro carbon, smoke and NOx are compared with diesel and it is decreased by 30%, 35%, 11% and 4% respectively. This experiment study observed by several researchers that NOx emission was increased with

bio- diesel.

2.12.5

Karanja Oil

Baiju et al. (2009) reported that the performance and exhaust emission characteristics using several blends of diesel and

bio-diesel

from karanja oil and by using base line fuel of petro diesel in a diesel engine. The result depicted that performance of engine does not vary greatly than diesel fuel. A little power loss, higher BSFC and lower BTE was observed due to lower calorific value of esters slightly produced higher power and emissions was lower than ethyl esters. They also found that at part loads when compare with conventional diesel fuel the emissions of NOx is increased by 10-25% when fuelled with diesel-biodiesel fuel blends. At maximum load, diesel emitted more NOx than esters and most of the major exhaust pollutants such as CO, HC and smoke were reduced with the use of neat

bio-diesel

and the blends. The study concluded that both methyl and ethyl ester of karanja oil

can be used as a fuel in compression ignition engine without any engine modification.

2.12.6 Cotton Seed Oil

Nabi et al. (2009) studied the effect of cotton seed oil

bio-diesel

on engine performance and emission. It was found that thermal efficiency of

bio- diesel

fuel was lower than that of diesel fuel due to the poor spray characteristics, higher density, viscosity and lower heating values of

bio- diesel

fuel. The emissions of CO, PM and smoke from biodiesel was lesser than that of neat diesel fuel. But, NOx emission with

bio-diesel

mixtures was higher compared to neat diesel fuel. For instance, with 10%

bio-diesel

mixtures (B10) PM and smoke emission reduced by 24% and 14% respectively. While

bio-diesel

mixture of 30% (B30) reduced CO emissions by 24% and NOx emissions increased by 10%. The authors attributed the reduction in PM, smoke and CO emissions and increased in NOx emission with biodiesel mixtures to the presence of oxygen in their molecular structure as well as low aromatics in the

bio-diesel

blend.

2.12.7

Jatropha Oil

Chauhan et al. (2012) evaluated the performance and exhaust emissions using 5%, 10%, 20% and 30% jatropha

bio-diesel blended

with diesel fuel on an unmodified diesel engine. The experimental results showed the engine performance with

bio-diesel

of jatropha and its blends were comparable to the performance of diesel fuel. In case of all fuel blends, brake thermal efficiency, HC, CO, CO2 and smoke density were lower while BSFC and NOx were higher than that of diesel. The authors concluded that biodiesel derived from jatropha and its blends could be used in a conventional diesel engine without any modification. However, there were various parameters which could

be evaluated in future such as the prediction of best blend with

respect to the various engine parameters by varying spray time of fuel using common rail fuel injection.

2.12.8 Tobacco Seed Oil

Usta et al. (2005) evaluated the performance and exhaust emissions of a turbo charged indirect injection diesel engine using 10%, 17.5% and 25% tobacco seed oil methyl ester blend (TSOME). The authors found that the addition of up to 25% in volume TSOME did not cause any significant variation in the engine torque and power. Although the heating value of the TSOME is 10% less than that of the diesel fuel. Moreover, the blending of tobacco seed oil methyl ester to the diesel fuel reduced CO due to the fact that TSOME contained about 11.4% oxygen by weight and SO2 emissions due to low sulphur content while causing slightly higher NOx emissions due to higher combustion temperature.

2.12.9 Polanga Oil

Sahoo et al. (2007) evaluated the performance and emission of polanga

oil methyl

ester (POME) blends in a single cylinder diesel engine at different loads. Tha main findings of the study showed that the performance of

bio-diesel

fuelled engine was marginally better than the diesel fuelled engine in terms of thermal efficiency, brake specific energy consumption, smoke opacity, and exhaust emissions including NOx emission for entire range of operations. The 100%

bio-diesel

was found to be the best, which improved the thermal efficiency of the engine by 0.1%. Similar trend was shown by the brake specific energy consumption and the exhaust emissions were reduced. Smoke emissions also reduced by 35% for B60 as compared to neat petro diesel. Decrease in the exhaust temperature of a bio-diesel fuelled engine led to approximately 4% decrease in NOx emissions for B100

bio- diesel

at full load. It was conclusively proved that excess oxygen content of

bio-diesel played a key role in engine performance. However, long term endurance test and other tribological studies need to be carried out before suggesting long term application of polanga oil based

bio-diesel.

2.12.10 Jojoba Oil

In an experimental investigation conducted by Shehata

and

Razek (2011) on the performance and emission characteristics using jojoba blend indicated BTE for pure diesel higher than B20 due to the higher heating value of diesel and higher BSFC due to the same reason. An increase in CO and CO2 concentration due to higher carbon / hydrogen ratio and reduction in NOx due to lower gas temperature were also observed.

2.13.11 Koroch Oil

Gogoi & Baruah (2011) used koroch seed oil methyl ester (KSOME) in a single cylinder diesel engine and reported that the engine BTE at full load for Numaligarh refinery limited (NRL) diesel, B10, B20, B30 and B40 were found to be 25.63%, 24.86%, 24.34%, 24.09% and 22.32% respectively . The BTE of various fuel blends were found to be lesser than NRL diesel due to its lower calorific value. Due to higher viscosity and density of KSOME blends, the rate of fuel consumption was found to be more for the methyl esters. Their study revealed the use of KSOME blends up to B30 as fuel for diesel engine without any significant drop in the performance characteristics.

2.12.12 Rubber Seed Oil

Ramadhas et al. (2005

a)

evaluated the emissions and performance of a diesel engine fuelled with

bio-diesel

of rubber seed oil. Their results showed that the maximum brake thermal efficiency obtained is about 28% for

B10, which was quite higher than that of diesel (25%). Moreover, it was observed that using lower percentage of

bio-diesel in bio-diesel-

diesel blends, the brake specific fuel consumption of the engine is lower than that of diesel for all loads. Under all loading conditions, it was observed that the engine emits more CO using diesel as compared to that of

bio-diesel blends.

However, using higher concentration of biodiesel blends increase CO2 and NOx emission. Smoke density for biodiesel blend is noticed to be generally lower than that of diesel. For instance, B20 blends gave smoke density of

20% as compared to 45% in the case of diesel. The experimental results proved that the use of biodiesel produced from unrefined rubber seed oil in compression ignition engines is a sustainable alternative to diesel.

2.12.13 Rice Bran Oil

Saravanan et al. (2009) studied the feasibility of crude rice bran oil methyl ester (CRBME) in diesel engine. The results showed that, with CRBME blend the brake thermal efficiency decreases only marginally due to the lower heating value of the methyl ester. Moreover, a significant reduction in CO, HC and particulate emission were observed with a marginal increase in NOx emission than that of diesel due to higher oxygen contents of methyl ester.

2.12.14

Mahua

Oil

Saravanan

et al. (2010) investigated the performance and emission of a diesel engine fuelled with

Madhuca indicabiodiesel. Experiments were conducted on a single cylinder, air cooled, four stroke, direct injection, compression ignition engine using mahua oil methyl ester and diesel as a fuel. The result showed that at full load, the power loss was around 13% combined with 20% increase in fuel consumption with mahua oil methyl ester due to the lower heating value and higher viscosity of biodiesel fuel.

Emissions such as carbonmonoxide, hydrocarbons were lesser for mahua ester compared to diesel by 26% and 20% reapectively, due to the higher oxygen contents which promoted combustion. Oxides of nitrogen were lesser by 4% for the ester compared to diesel due to the lower in cylinder temperature. A smoke intensity reduction of 15% for MOME (Mahua oil methyl ester) was also observed at full load. It was also observed that the exhaust gas temperature for MOME operation is lower compared to that of diesel.

2.12.15

Turpentine Oil

Anand et al. (2010)

reported that the performance emission of diesel engine operated using turpentine oil. The result showed that the higher brake power and heat release rate produced for 30% of TPOF with 70% of diesel fuel. Nox, CO, HC and smoke emission are reduced. Further increased

40% and 50% of TPOF blends, produced lower heat releasr rate and brake power.

2.12.16 Pine

Oil

Vallinayagam et al. (2013) evaluated the combustion, performance and emission of a diesel engine fuelled with

pine oil.

From the resin of pine tree the pine oil was extracted. It could be directly used in diesel engine without any modification.

The result showed that the pure pine oil (100%) at full load condition, the brake thermal efficiency and heat release rate increased by 5% and 27% respectively, and reduced hydrocarbon, carbon monoxide and smoke emissions by 30%, 65% and 70% respectively. However, slightly increased NOx emissions.

2.12.17

Camphor Oil and Cashew Nut Shell Oil

Kasiraman et al. (2012) studied the effect of cashew nut shell oil blended with camphor oil. The experimental results at full load condition shows the camphor oil 30% and 70% cashew nut shell oil blends (CMPRO30) produced good performance and heat release rate compared with diesel.CMPRO30 blend brake thermal efficiency was 29.1% compared to diesel of 30.14% and slightly reduced NOx emission compared to diesel.

2.12.18

Ceiba Pentandra Oil

Kathirvelu et al. (2014) reported that bio-diesel was

produced from curde ceiba pentandra seed oil through transesterification processes, using H2SO4 as a catalyst (1%v/v) methanol as a solvent, 45min reaction time and

60°C reaction temperature. The maximum methyl ester yield obtained was

93%. The property of methyl ester viscosity was nearly close to diesel. BTE of B100 fuel at maximum load 6.26% reduced compared with diesel. The emissions of CO, CO2, HC and NOx were reduced. In compared

to

diesel similar cylinder pressure and heat release rate are observed.

2.12.19

Watermelon Oil

Santhosh Kumar et al. (2016) reported that bio-diesel

was produced from watermelon seed oil through transesterification process. B20 biodiesel blend at maximum load 9kg, standard injection timing (23°BTDC) with compression ratio of 18 has optimum engine performance of brake thermal efficiency was 33.55%. CO, HC, CO2 and NOx were 0.02% vol.,

7ppm, 1.1%vol. and 123ppm-vol. respectively.

2.13

SUMMARY

The key points from the above discussion are,

• The four-stroke

DI-CI engine

are indispensable in major areas of transport, agriculture, etc. and it will be very difficult to replace it with any other type of power plant.

• The present situation for four-stroke DI-CI engines is a difficult one and the future of the engine depends on meeting the emission norms that are being imposed.

• Modification of the fuel is one of the best ways to reduce emissions without any modification on the existing engines and exhaust gas treatment.

- Raw oils are not suitable for diesel

engines.

- Preheating, direct use blending, micro emulsion, pyrolysis (Thermal cracking) of straight vegetable oils (SVO) are used only for short term experimental purpose only.

- For long term application of non-edible oils chemically modified into bio diesel is known as transesterification process.

- Aalkaline catalyzed transesterification is the best way and most accepted method which have been used to prepare bio diesel from high free fatty acid content vegetable oils like Jojoba oil, Rice bran oil pungamia, soyabean oil etc.,

-

Very few research publications on production of

bio-diesel from ceiba pentandra seed oil and watermelon seed oil.

No publication in CG-MS analysis of watermelon methyl ester.

-

Increased bio-diesel blends ratio with decrease the brake thermal efficiency of bio-diesel operated engine. This may be due to the more specific fuel consumption of bio-diesel due to heavier oxygen molecule structure present in the fuel.

- The increased bio-diesel percentage of blends significantly reduced CO, HC and smoke due to the presence of oxygen in the fuel. However, slightly increased NOx emission due to the higher combustion chamber temperature and Exhaust Gas Temperature (EGT)

- It is obvious in few of the researches that the neat orange oil, Pine oil, karanja oil, and turpentine oil brake thermal efficiency were slightly higher than diesel fuel due to less viscous and better atomization of those oils.

From the above literature review, it is pointed out that more work is needed for optimize the parameters of bio diesel production towards maximum yield, to analyze the blend properties and to find the best blend in an engine. The research problem for the current work is selected based on the above literature review. The following chapter briefly explains the problem definition, its objectives and scope of present work.

CHAPTER 3

OBJECTIVE AND METHODOLOGY

3.1

PROBLEM DEFINITION

The chapter one and two discussed the serious description of the review of the literature. The study and investigation is based on the problems, production of methyl esters from non-edible oils such as alcohols, catalyst, reaction temperature and reaction time of different vegetable oils. The physic-chemical properties of the diesel, vegetable oil, methyl esters and its blends are to be analyzed. The performance, combustion and emission characteristics of different methyl esters, pine oil and diesel blends are to be analyzed.

3.2 OBJECTIVES OF THE STUDY

By under viewing the review of literature, the following objectives were outlined for the present work:

x

To investigate the potential suitability of ceiba pentandra oil, watermelon seed oil and pine oil as a substitute fuel for DI-CI engine.

x To produce the bio diesel from ceiba pentandra seed oil and watermelon seed oil using transesterification process.

x To optimize the stirrer speed, amount of catalyst and solvent required for maximum yield of biodiesel production.

x To study the quantitative analysis for methyl esters of ceiba

pentandra and watermelon .

x To study

and compare the physical and chemical

fuel properties of pine oil, CPME and WME with its diesel blends as per ASTM standards.

x To study the DI-CI engine performance and emission characteristics fuelled with pine oil-CPME-WME and its diesel blends.

x To study the DI-CI engine combustion characteristics of fuelled with pine oil-CPME- WME and its diesel blends

The

current work focus on experimental investigations to achieve the above objectives. The experimental investigation of methyl ester production is made and the properties, performance, combustion, and emission characteristics were analyzed.

3.3 SCOPE OF THE PRESENT WORK

The scope of the present investigation includes:

x Increase the conversion efficiency of

bio diesel production ceiba pentandra seed oil and watermelon seed oil by using

homogeneous catalyst.

x Qualitative analysis of GC-MS for methyl esters of bio-diesel from ceiba pentandra oil and watermelon oil.

x Comparing CPME, WME and PO blends with diesel tested in

DI-CI engine.

x Performance and emission analysis of PO-biodiesel-diesel.

x

Combustion analysis of PO - bio diesel-diesel blends in DI-C.I

engine.

The current work focus involves experimental investigations to achieve the above objectives.

3.4

METHODOLOGY

The methodology used in the present work is outlined below in a step-by- step manner as shown in Figure 3.1.

Optimization of the important parameters like speed of the stirrer, catalyst concentration like KOH, NaOH and reaction time for producing higher yields of methyl esters are discussed. Ceiba pentandra seed oil, watermelon seed oil have been converted into biodiesel of the concerned oil. The important fuel properties like specific gravity, viscosity, flash point, cloud point, pour point, and calorific value of SVO and methyl esters of bio diesel – diesel blends were studied using ASTM specified equipment. The qualitative analysis of diesel and

two

esters were done using gas chromatography and mass spectrum analysis. A single cylinder,

direct injection

diesel engine experimental setup is fabricated with the necessary instrumentation to study the performance, emission and combustion characteristics of the diesel engine fueled by CPME, WME & pine oil and its blends with diesel.

Figure 3.1 Research methodologies for bio diesel production, study of fuel properties, engine performance, emission and combustion analysis

3.5

SALIENT FEATURES OF THE PRESENT WORK

Following are some of the significant features of the present work:

x Bio-diesel is produced

from ceiba pentandra seed oil and watermelon seed oil using

electronically controlled batch type production unit.

x The present investigation has been carried out on a production model

DI-CI engine.

x

Pines, bio-diesel from ceiba pentandra seed oil, watermelon seed oil and diesel oil have been used as fuel.

x The physical and chemical properties of bio-diesel – diesel blends like flash point, fire point,relative density, viscosity, calorific value, cloud and pour point were studied.

x Exhaust emissions such as carbon monoxide, carbon dioxide, unburned hydrocarbon (UBHC), Nitrogen oxides (

NOx),

smoke, cylinder pressure and combustion characteristics have been measured at each operating point.

To achieve the above stated objectives and methodology, a detailed experimental work has been carried out. The details of the experiments and equipment's used are explained in Chapter 4.

CHAPTER 4

EXPERIMENTAL SETUP AND PROCEDURE

4.1

INTRODUCTION

This chapter discusses

the experimental setup for bio diesel production unit, measurement of fuel physical chemical properties instruments and finally experimental engine setup along with necessary instrumentation to evaluate the engine performance, emission and combustion analysis of the engine with two types of bio diesel blends

with pine oil operating conditions.

4.2 BIO DIESEL PRODUCTION

The vegetable oil usually contains free fatty acids, phospholipids, sterols, water, odorants and other impurities. Because of their presence, the oil cannot be used as fuel directly. To overcome these problems, the oil requires chemical modifications like transesterification, emulsification and pyrolysis. Among these, the transesterification is an significant process to produce clean and environment friendly fuel from vegetable oil and it seems to be more suitable because the by product (glycerol) has got commercial value.

4.2.1 Process of Bio Diesel Production

The process flow chart for bio diesel production from ceiba pentanndra seed oil and water melon seed oil is shown in Figure 4.1.

Figure 4.1 Process flow chart for bio diesel production

4.2.2 Experimental Setup for Bio Diesel Production Unit

A small batch type unit is designed and fabricated keeping in mind the objective of the present work for producing bio diesel from non-edible ceiba pentandra seed oil and watermelon seed oil. It is very compact type and can maximum produce five liter bio diesel from non-edible oils in a period of 11/2 hours. The production of bio diesel is approximately 70 to 80 liters per day. It has the following advantages x Cost is low

x

Easy to operate

x Electronic temperature control monitor system

x Digital Speed monitoring system.

x

According to the requirements the variable and constant speed can be maintained.

The container and blades are made up of stainless steel. It can able to produce the bio diesel about 80 to 96% of yield based on the nature of the oils and reactants.

4.2.2.1 Description of bio diesel production unit

The transesterification unit used for bio diesel production is planned and fabricated in the laboratory. It consists of stainless steel container, with a capacity of six liters and a variable speed D.C motor is fixed on the top of the container. The motor is coupled with a mechanical stirrer. The operating speed of the mechanical stirrer range from 200 rpm to 900 rpm. The stirrer speed can be easily varied with the help of an electronic speed regulator and it is monitored using digital speed sensor along with a digital speed indicator. The stirrer consists of eight stainless steel blades at an angle

of 45

q

oriented to the base.

At bottom of the container a capacity of 1.5 kW is fixed to heat the oil. A temperature control unit is used to vary the temperature of oil from

30qC to 250qC. The oil temperature is measured by using Chromel Alumel

(k-type) thermocouple along with digital temperature indicator. Control the motor speed and temperature an electronic microprocessor is used and the cooling fan which is inside the electronic kit is used to absorb the heat from the transformer and integrated circuits. The bottom of the container has one drain valve and the top of the container has one inlet valve. The line diagram and photographic view of the bio diesel production unit are shown in Figures

4.2 and 4.3.

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Figure 4.2 Line diagram of the bio diesel production unit

1. Container

2. DC motor

3. Heater

4. Thermocouple

5. Blades

6. Speed sensor

7. Inlet valve

8. Outlet valve

9. Microprocessor kit

10. Electrical connection for motor, eater and thermocouple

11. Cooling fan formicroprocessor kit

12. Temperature indicator

13. Speed (rpm) indicator

14. ON/OFF switch

15. Speed regulator for DCMotor

16. Transformer

17. Sensor controller

18. Shaft

Figure 4.3 Photographic view of bio diesel production unit

4.2.2.2

Features of the bio diesel production unit

a) The automatic temperature controller is provided in the unit to vary the temperature of oil from 30° C to 250°C. This feature is highly useful for pre-treatment, transesterification and evaporating of water molecules present in the

bio

diesel after washing.

b) It consists of eight numbers of stainless steel blades with an angle of 45° and rotating in clockwise direction to produce the centrifugal effects to separate the fatty acids from the oil more effectively.

c) The volume of the tank is six liters and it is capable to produce maximum of five liters or minimum of two liters of oil depending upon the requirements.

4.2.3

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Procedure for Transesterification Process

x

Mixing of reactants is found to be an important factor which effects the transesterification and yield of

bio diesel.

x

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Operation is carried out at a speed of 600 rpm in the first stage of reaction (10 min) and a lower stirring speed of 550 rpm for

the

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remaining period.

x 2.5

litre of ceiba pentandra oil is filtered using cloth to remove the impurities in the oil and poured into a bio diesel production unit

and 500 ml of methanol is used as a solvent for 2.5 litre oil based on literature review in first phase of work.

x Under agitation, the raw oil is heated up to 60ºC (nearer to boiling point of methanol) for production of methyl ester by using heating coil.

x The amount required for catalyst pellets (based on literature)

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KOH is quickly weighed, protecting it as much as possible from atmospheric moisture and carbon di-oxide.

x The pellets are quickly moved to the dry mixture grinder to change into powder form.

x The

powdered form of

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catalyst and solvents (methanol) solution is then vigorously shaken in a conical flask for homogeneous mixing. At this point dissolved catalyst (KOH) is presumed to have been converted into potassium/ sodium/methoxide.

x A fixed amount of solvent and catalyst solution is poured into the oil

to mark the beginning of the reaction.

x When the reaction reached the preset reaction time, the

process of heating and stirring are stopped. The oil has taken

60 minutes for obtaining reaction products.

x

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Once the reaction period is over, then the product is allowed to settle

for 24 hours. In the output two distinct liquid phases are identified. At the top phase is Crude ester and the bottom phase is glycerol. That is bio diesel

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process turns the oil into esters, separating out the glycerol. The glycerol sinks at the bottom and the bio diesel floats on top and can be syphoned off.

x The crude ester phase is separated and glycerol phase is then washed by warm de-ionized (double distilled) water for four times

to remove un-reacted oil, catalyst and methanol until the water becomes clear.

x The solution mixture was again allowed to settle under gravity and the impurities were drained out along with water at the bottom of the funnel. So there is no need for further wash.

x Heating the oil in an open container nearer to 110°C to evaporate the water content in the washed ester is done.

x

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The excess methanol and water in the ester phase is removed (or recycled) by evaporation under atmospheric condition after being measured for product yield calculation.

x The fuel properties analysis like flash point, pour point, cloud point, density, calorific value, viscosity, acid value and cetane number of ester are as per the ASTM standard.

The final product is analyzed by gas chromatography and mass spectrum to determine its constituents.

4.2.4 Production of Mthyl Esters of Bio Diesel from Ceiba pentandra

Oil and watermelon seed oil

Methanolysis of triglycerides is represented in Equation (4.1).

CH2 - COO - R

|

CH3

OH

Catalyst

R - COO - CH3

CH2 - OH

|

CH - COO - R

|

CH3

OH

m o

R - COO- CH3

CH - OH

|

CH2 - COO - R

CH3OH

R - COO - CH3

CH2 - OH

triglyceride + 3 methanol 3 bio diesel + glycerol (4.1)

Tranesterification

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is

one of the reversible reactions and proceeds essentially by mixing the reactants. however, the presence of a catalyst (a strong acid or base) accelerates the conversion

of

esters (Adewale et al.

2015).

4.2.5 Optimization

The increase bio diesel production requires optimize production processes that are compatible with high production capacity, feature simplified operations, high yields, quantity of catalyst, solvent requirements and waste streams reaction are investigated systematically. The optimal value of each parameter in the process is determine while the rest of parameters are kept constant. After each optimal value is attained, this value is adopted for the optimization of the next parameter. It should be noted that, once the reaction takes place, it would continue even at room temperature, as long as the catalyst exist in the reactant mixture, which is favorable for bio diesel production. In the present experiment, when heating is finished, no acid is added into the product mixture to neutralize the catalyst and to stop the

reaction at once, but rather the catalyst is kept in the mixture and experiences a long settling time together with the mixture. Better conversion is obtained by extended reaction time. The reaction will stop completely when water washing is finished, because the catalyst in product mixture would have been removed during this process. The PH value of both the product and washed water was ‘5’ at that time. Therefore, in order to compare the product yield under different reaction conditions, the product mixture is allowed settle for a period of 15 hours and washed for three to four times in an interval of one hour each.

4.2.6 Washing of Biodiesel

When the reaction is completed the bio diesel and glycerol are separated. Consequently, with hot distilled water the bio diesel is washed, to remove entrained glycerol, catalyst, soap and excess methanol. The excess methanol in bio diesel corrodes the fuel injection system and hence it should be separated from the bio diesel (Naik et al. 2008).

4.2.7 Drying of Bio Diesel

By heating the washed fuel approximately to 110qC in an open container the drying is determined up to that there is no more steam from the fuel, which should be a clear, amber- colored liquid as shown in Figure 4.4. This heating process will also drive off any traces of remaining alcohol as well. Then it is allowed to cool to room temperatures, it can be pumped directly into vehicles, or into storage containers. If the fuel still appears somewhat cloudy after drying, the drying cycle should be repeated but the likely culprit is probably the presence of non-water soluble contaminant in the

fuel (such as mono and di-glycerides). The pure bio diesel is collected and stored in separating funnel as shown in Figure 4.5.

Figure 4.4 Photographic view of drying the bio diesel in open container

Figure 4.5 Photographic view of pure bio diesel

4.3 FUEL PROPERTIES

The Fuel properties of vegetable oil, methyl esters (Bio diesel) can be grouped conveniently into physical, chemical and thermal properties. The physical properties have flash point, pour point, cloud point, viscosity,

density, boiling range, freezing point and refractory index. The chemical properties contains chemical structure, saponification value, hydroxyl value, peroxide value, acid value, iodine, acetyl value, overall heating value, ash and sulphur contents, sulphur and copper corrosions and ignitability of products. Thermal properties are distillation temperature, thermal degradation point, carbon residue, specific heat value, thermal conductivity etc (Demirbas 2008; Verma et al. 2016).

The present work gives the details about the following properties.

- Relative density

- Cloud and Pour point

- Calorific value

- Viscosity

- Flash point

- Water and sediments

- GC-MS Analysis

4.3.1 Specific Gravity

Specific gravity is also one of the most important fuel properties, because injection system, pumps and injectors must deliver the correct amount of fuel precisely adjusted to provide proper combustion (Dzida & Prusakiewic (2008); Verma et al. (2016)).

Specific gravity or else relative density refer

the ratio of the density of a fuel to the density of

water at the same temperature. Specific gravity of methyl esters and diesel blends was measured by means of hydrometer (ASTM D1298 method), which is shown in Figure 4.6.

Figure 4.6 Photographic view of hydrometer

4.3.2 Viscosity

Viscosity is one of the major factors for establishing their suitability to the metering requirements of engine operation. Figure 4.7 shows the redwood viscometer (ASTM D 445 method) (Verma et al. 2016). The redwood viscometer is mainly for the measurement of dynamic viscosity of pine oil, bio diesel and diesel blends. The experiments were performed at

40ºC and the apparatus works on the principle of measuring the time of gravity flow in seconds of the sample through a specified hole. The following Equations (4.3) and

Equation (4.4)

represent dynamic viscosity calculation (4.2) and (4.3).

$\mu = 0.0026\ t - 1.79/t$ for t upto 100 second (4.2) μ = 0.00247 t-0.5/t for t < 100 second (4.3)

where μ = dynamic viscosity in centistokes t = flow time in second

Figure 4.7 Photographic view of Red wood viscometer

4.3.3 Flash Point

It plays a crucial role for determining the fire hazard of the fuel. Flash point is the lowest temperature at which the vapor given off by a fuel will flash with a test flame held above the surface without continuous burning. The fire point is the minimum temperature at which the inflammable vapours will continue to form and steadily burn once ignited. Depends upon the original crude oil nature the flash and fire points may vary. Figure 4.8 shown the flash apparatus of Pensky Martens (ASTM D93 method). It is used to measure the flash point of the pine oil, bio diesel and diesel blends (Verma et al. 2016).

Figure 4.8 Photographic view of pensky Martens Apparatus

4.3.4 Cloud and Pour Point

Cloud points are one of the main guides to evaluate the wax precipitation potential of a fluid. Petroleum oil when cooled may become plastic solids as a result of partial separation of wax congealing of the hydro carbons, with some oils. The separation of wax becomes visible at temperature slightly above the solidification point and this temperature is known as the cloud point (ASTM D-2500). The pour point (ASTM D-97) indicates the temperature at which oil stops flowing to the pump, bearings or cylinder walls. It is particularly important for immediate oil circulation with respect to starting of engines in very cold climates with gravity lubricating system.

The fluidity is a factor of pour point and viscosity of the cold oil. Pour point depressants may be added to wax containing oils to lower the pour points instead of de-waxing the oil. The cloud and pour point temperatures have much importance in knowing the behavior of fuels in cold weather. The apparatus used for measurement of cloud and pour point of bio diesel and diesel blends, which is shown in Figure 4.9.

Figure 4.9 Photographic views of cloud and pour point apparatus

4.3.5 Higher Calorific Value

The calorific value or heat of combustion can be defined as the standard measure of the energy content of a fuel and its heating value (H.V). The heating values are obtained by the complete combustion of a unit quantity of liquid fuel in an oxygen-bomb calorimeter under carefully defined conditions. The gross heat of combustion or heating value (GHC or HV) is obtained by oxygen-bomb calorimeter method as the latent heat of moisture in the combustion product is recovered. The heating value is one of the most important properties of a fuel. Figure 4.10has shown the “Advance (ARICO) bomb calorimeter”. It is used to measure the heating values of bio diesel- diesel blends. As per the ASTM D2015 standard method, an oxygen-bomb

was pressurized to 25 kg/cm2 with oxygen. The bomb is fired automatically

after the jacket and bucket temperatures equilibrated within acceptable accuracy of each other. The calibration of the bomb calorimeter has been carried out by using the standard benzoic acid samples which were obtained from Advance Research Instrument Company (ARICO) New Delhi.

Figure 4.10 Photographic view of Bomb Calorimeter

4.3.6 Qualitative Analysis

The chemical composition is very important to determine the quality of bio diesel like the amount of conversion in fatty acid alkyl esters, and presence glycerol, free fatty acid, some metal alkyl hydrocarbons, presence of unreacted alkyl alcohol and KOH/NaOH

reported by

Khan (2002). GC-MS is a method that combines the features of gas-liquid chromatography and mass spectrometry to identify different substances within a test sample and identification of unknown samples. Additionally, it can identify trace elements in materials that were previously thought to have disintegrated beyond identification. The schematic diagram of GC-MS is shown in Figure 4.11. The GC-MS is composed of two major building blocks namely

- Gas chromatograph and

- Mass spectrometer.

The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties (example 5% phenyl) polysiloxane. The difference in the chemical properties between different molecules in a mixture will separate the molecules as the sample travels through the length of the column. The molecules take different period of time (called the retention time) to come out of (elute from) the gas chromatograph and this allows the mass spectrometer downstream to capture, ionize, accelerate, deflect and detect the ionized molecules separately. The mass spectrometer does this by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio.

The qualitative analysis of GC-MS is performed for the observed compounds present in every step and also the nature of compounds change during the various processes during bio diesel formation. It also identifies the components in the compounds, molecular weight and purity of compounds by this analysis it

is known

that conversion or interchange in the compound.By the purification and transesterificationprocess http://en.wikipedia.org/wiki/ Gas_chromatography%E2%80%93mass_spectrometry-2014). Transesterification process is the conversion of triglycerides into fatty acid alkyl esters (bio diesel) that needs to be separated from several impurities and compounds. The conversion of triglyceride to FAME takes place in different phases and is determine by knowing the compound and its quality. Hence, it is required to perform the GC-MS to identify unknown compounds in the samples at each phase. The compounds were identified mainly by comparing their mass spectra with those of the standard compounds in the NIST and Wiley library, together with the comparison of their GC retention times with those of standard compounds and were confirmed by the standard addition technique. Tentatively the components are identified based only on

comparison of the mass spectra with the reference spectra of the NIST and

WILEY library that yielded more than 90% matches (Plonjarean et al. 2007).

The GC-MS analysis was performed on a FISON (GC) 8000 series/ MD 800 which is shown in Figure 4.12. Separation was achieved using helium as the carrier gas (1ml / min) with a fused silica capillary column 30m long, inner diameter 5 micron and 0.5 micron film thickness. The GC injector was in a split less mode. Injector and detector temperature were

250°C and 260°C respectively. The oven temperature was ranges from 100°C to 250°C. The effluent from the capillary column went directly into mass spectrometer. This instrument was operated is the full scan and electron impact ionization mode with an ionizitionation voltage 70eV and an acceleration voltage of 70V the iron source temperature was 200ºC and the GC-MS transfer line was set to 250 °C. The sample was diluted with petroleum ether the data is compared with NIST and WILEY library to know the peak compounds.

Figure 4.11 Schematic diagrams of GC-MS

Figure 4.12 Photographic view of gas chromatography mass spectrum analyzer. FISON (GC) 8000 SERIES / MD 800

4.4 ENGINE EXPERIMENTAL INVESTIGATIONS

An experimental set up is made to evaluate the performance, emission and combustion parameters of diesel engine with essential instrumentation at different operating conditions.

4.4.1 Description of Engine Experimental Set up for Comparative of Different Methyl Esters

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Naturally aspirated (NA- DI) engine is selected for evaluation of bio diesel, pine oil fuels based on two specific reasons. First, a NA-DI engine represents a larger population of engines sold in agricultural and construction equipment for the last 55 years. Secondly, a naturally aspirated engine is more sensitive due to longer ignition delays and lower pressure injection equipment.

The Kirloskar engine is

one of the

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widely used engines in agricultural, pump sets, farm machinery, transport-vehicles, small and

medium scale commercial purposes. The engine can withstand higher pressures encountered during tests because of its rugged construction. Further, the necessary modification on the cylinder head and piston crown can be easily carried out in this type of engine. Hence this engine is selected for the present research work.

A single cylinder, water cooled, four stroke direct injection compression ignition engine

with compression ratio of 16.5: 1, developing 3.7 kW at a rated speed 1500 rpm is used for the present study. The specifications of the engine setup are tabulated in the appendix 1. The layout of the experimental setup is shown in Figure 4.13. The photographic view of the experimental setup is shown in Figure 4.14. Variable load tests are conducted for no load, 0.74, 1.48, 2.22, 2.96 and 3.7 kW power output at a constant rated speed of 1500 rpm, with fuel injection pressure of 200 bar and cooling water exit temperature at around 60ºC. The injection timing recommended by the manufacturer is 23° BTDC. The governor was used to maintain constant speed under varying load conditions, which control the fuel flow as load changes over engine. The engine had an open combustion chamber with over head valves operated through push rods. A provision is made in the cylinder head surface to mount an Optrand make pressure transducer for measuring the cylinder pressure.

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AVL-444 DI Gas analyzer is used to measure the exhaust tail pipe emissions such as HC, CO, CO2, and NOx. The smoke opacity is measure with the aid of AVL 437 standard smoke meter.

Initially,

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before starting experimental tests, the engine is made to run under ideal condition as warm up phase and then the

tests were conducted. The engine is started and allowed to warm-up for about 10 minutes.

The engine is tested under five discrete part load conditions Viz; No load, 20%, 40%, 60%, 80% and 100%. The combustion characteristic varies in terms of pressure traces, maximum pressure and heat release. These performance and emission characteristics are compared with the results of base line diesel.

Figure 4.13 The layout of the experimental setup

Figure 4.14 shows the experimental setup photographic view

4.4.2 Fuel Supply Measurement

The fuel from the tank is connected to a solenoid valve, the outlet of the solenoid valve is connected to a glass burette and the same is connected to the engine through a manual ball valve. The fuel solenoid of the tank remain open, until the burette is filled to the high level sensor, during this time the fuel is flowing to the top level, the fuel solenoid closes fuel tank outlet line now the fuel in the burette gets discharged to the engine. When the fuel level reached the high level optical slot sensor, the sequence running in the computer records the time of this event. Likewise when the fuel level crossed the low level optical slot sensor, the sequence running in the computer records the time of this event and immediately the fuel solenoid opens filling up the burette and the cycle is repeated. The volume of the fuel between the high level and low level sensors is 20 CC. Figure 4.15 shows the photographic view of fuel supply measurements.

Figure 4.15 Fuel Supply measurement photographic view

4.4.3 Temperature Measurement

Figure 4.16 shows the photographic views of temperature measurements. The thermocouples are a widely used as a temperature sensor and can also be used as to convert thermal potential difference into electric potential difference. It can be measured a wide range of temperatures and system error of less than 1°C is difficult to achieve. K-Type thermocouples are located at the following points in the engine test bed.

(i) Engine exhaust

(ii) Engine cooling water inlet

(iii) Engine cooling water outlet

Figure 4.16 Temperature measurements photographic views

The output temperature transmitters are connected to the Data

Acquisition Card.

4.4.4 Air Flow Measurements

Figure 4.17 Air flow measurements photographic views

The air flow to the engine is routed through cubical air tank. The air tank fulfills the purpose of regulating the flow of air to the tank. The inlet of the air tank is provided with an orifice, the airflow rate is measured using the mass air flow sensor. Figure 4.17 shows the photographic views of air flow measurements. Mass air flow sensor is used to measure the airflow rate. A hot wire mass airflow sensor determined the mass of air flowing into the engine's air intake systems. This is achieved by heating a wire with an electric current that is suspended in the engine's air stream, not unlike a toaster wire. The wire's electrical resistance increase with the wire's temperature, which limits electrical current flowing through the circuit. When air flows through the wire, the wire cools decreasing its resistance, which in turn allows more current to flow through the circuit. As more current flows, the wire's temperature increases until the resistance reaches equilibrium again. The amount of current required to maintain the wire's electrical resistance is directly proportional to the mass of air flowing past the wire. The integrated electronic circuit converts the measurement of current into a voltage signal when is sent to the computer.

4.4.5 Speed and Load Measurements

Figure 4.18 shows the photographic views of speed and load measurements. The engine was set to run at a constant speed of 1500 rpm. The load of the engine is obtained from load cell reading. The speed of the engine is monitored using photo sensor along with digital speed indicator.

Figure 4.18 Speed and load measurements Photographic views

4.4.6 Eddy Current Dynamometer

The test engine is directly coupled by using a tyre coupling to an eddy current dynamometer with suitable switching and control facility for loading the engine. It is air cooled and the length of torque arm of the dynamometer is 0.195 m. The output shaft of the Eddy Current Dynamometer is fixed to a strain gauge type load cell for measuring applied load to the engine. Figure 4.19 shows the photographic view of Eddy Current Dynamometer and specifications are given in Appendix.2.

Figure 4.19 Eddy Current Dynamometer Photographic view

4.4.7 Combustion Analysis with Data Acquisition System

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The combustion analysis of diesel and bio diesel-diesel pine oil blends is carried out with the assistance of combustion pressure sensor (Optrand make combustion pressure sensor range 0-200 bar),

Combustion parameters were analyzed using a combustion analyzer. The cylinder pressure and maximum rate of pressure rise were analyzed. The combustion analyzer used for the present research work is shown in Figure 4.20

and specifications are given in an appendix 3.

Figure 4.20 Photographic views of Encoder and combustion pressure sensor

It is easy to operate, it contains menu driven parameter editor for setting up the system, utilized for TDC detection, the numerical monitor for on-line display of calculated results like Indicated Mean Effective Pressure (IMEP) or mass burn fractions as well as monitor program for oscilloscope like graph display. This versatile software designed by legion brothers written in “Visual C” was used for on-line data acquisition from the pressure transducer and crank angle Encoder (Make Kubler, 360 PPR coupled to crank shaft).

4.4.8 Exhaust Emission Measurements

The intensity of smoke is measured by light obscuration method, in which the intensity of a light beam is reduced by smoke, which is a measure of smoke intensity. The oxides of nitrogen (NOx) are measured by chemical reaction method. The carbon monoxide emission is measured by Non-Dispersive Infra-Red (NDIR) analyzer and unburned hydrocarbons (UBHC) by Flame Ionization Detector which is a well-established and accepted method for measuring unburned hydrocarbons. The AVL five-gas analyzer and smoke meter are shown in Figure 4.21 and specifications are given in an Appendix 4 &

Appendix 5.

Figure 4.21 AVL 437 standard smoke meter and AVL 444 digas analyzer photographic views

4.4.9 Experimental Procedure

Initially the engine is allowed to run with diesel at a constant speed of 1500 rpm for nearly 30 minutes to attain the steady state conditions at the lowest possible load. During the investigation, the temperature of lubricating oil and temperature of the engine cooling water were held constant to eliminate their influence on the results. The speed of the engine was stabilized with injected fuel to attain the temperature of lubricating oil as

65°C and temperature of cooling water as 60°C. Then the following observations were made twice for concordance: The exhaust gas analyzer and smoke meter is switched on quite early so that all its systems will get stabilized before the commencement of the experiment and the following observations were documented.

- Time for 20 cc of fuel consumption. (s)

- Exhaust gas temperature. (°C)

- Measurement of smoke using AVL 437 smoke meter

- Measurement of CO, CO2, HC, O2, and NOx using AVL 444 digas analyzer.

- Combustion parameters were analyzed using Optrand make combustion pressure sensor.

- Bio diesel is the pure or 100% bio diesel fuel. It is referred to as

B100 or neat fuel.

- Bio diesel-diesel blend means, pure bio diesel blended with diesel.

- Bio diesel blends are referred to as Bxx.

- The xx indicates the amount of bio diesel blend i.e. B25 blend is 25% bio diesel and 75% diesel.

After completing the experiments with neat diesel, the same experimental procedure was repeated with B25 blend of the WME. The experiments and similar observations were repeated with other four blends in order B50, B75, and B100 for CPME, WME and, B25CPME pine oil

and

diesel.

After completing the experiment with the bio diesel, the engine was allowed to run for about half an hour with diesel to eliminate the interference of the previous bio diesel fuel. The entire bio diesel, diesel pine oil blends were tested by the similar procedure. The diesel fuel is then retested to check repeatability. The results tended to confirm the result of the first run with diesel fuel.

4.4.10 Experimental Uncertainty

This work, several measurements are taken only one sample type i.e., the

one instruments made used for measurements.

Necessary care has been taken to maintain the original accuracy by frequent calibration wherever possible. The uncertainties for the basic measurements liketime, barometer pressure,temperature,weight, length, etc., are equal to the least count of individual instruments. The errors on measures likeviscosity, specific gravity, characteristic gas constant, density etc., are taken from hand books and tables.

The uncertainty on the derived quantity is calculated by using the method suggested by Holman (2012), which is based on the works of Kline and McClintock. The uncertainties in the measured values like engine power, fuel flow,

pressure andairflow

are calculated and the results are presented in Table 4.1.

Table 4.1 Uncertainties of experimental

S.No Variable Units Uncertainties 01 Engine power kW ± 0.9434 % 02 Airflow kg/hr ± 0.6481 % 03 Fuel flow kg/hr ± 0.7319 % 04

Cylinder pressure Bar ± 0.61644 05

Viscosity centiStokes ± 0.7 % 06 Smoke HSU ± 1 % 07 HC Ppm ± 0.2

It can be found from Table 4.1, except for measurement

of

emission, that the uncertainties involved in the various parameters are less than 1%. The emission calculation involves many parameters such as

engine

power, temperature and emission level. So the uncertainty rises in particular emission values. However, the uncertainty of 1.5% is still a minor value and may not affect the accuracy of results.

Chapter 5 presents the results and discussion of various methods of bio diesel production techniques, properties of raw oil, bio diesel-diesel blends with pine oil. Performance, combustion and emission studies were studied.

CHAPTER 5

RESULTS AND DISCUSSION

PHASE 1

5.1 BIO DIESEL PRODUCTION FROM RAW OIL

Methyl esters of watermelom seed oil and ceiba pentandra seed oil were produced. The parameters influencing bio diesel production such as speed of the stirrer, reaction time catalyst concentration and methanol to oil ratio were studied and presented in the following sections.

5.1.1 Production of Bio Diesel from Ceiba Pentandra Seed Oil

5.1.1.1 Effect on

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speed of the stirrer

Mixing is very important in the transesterification reaction, as an oil and fat are immiscible with potassium hydroxide-methanol solution.

Once the two phase are mixed the reaction is started.

Methanolysis is conducted with different stirrer rates such as 350, 400, 450, 500, 550,600, 650 and 700 revolutions per minutes (rpm).

The variation of methyl esters yield with different rate of stirrer is shown in Figure 5.1. It is observed that

the reaction is incomplete up to 500 rpm, and rate of mixing is insufficient for methanolysis. Further increasing the speed of 550 rpm

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the yield of 92% obtained and again further increase in the speed of stirrer to 600 rpm, the yield of methyl esters was reduced to 88%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel for this batch type production unit.

This occurrence was reported with experimental work

performed by (Meher et al. 2006, Rashidand Anwar 2008, Ong et al. 2011, and Venkanna & Venkataramana 2011).

Figure 5.1 Variation of different stirrer speed with methyl esters yield for transesterification of ceiba pentandra oil

5.1.1.2

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Effect of reaction time

The reaction time is required for the completion of alkaline-catalyst transesterification depends not only on the reaction temperature, but also on the degree of mixing in the process. The reaction rate of transesterification increases with increase

in

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temperature and degree of mixing. The reaction time of transesterifaction process is conducted at 60 C ie. Nearer to the boiling point of the methanol.The variation of methyl ester yield with different reaction time as shown in Figure 5.2.The figure

shows that

yield of bio-diesel

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is less at the beginning and reaches a maximum at the reaction time of 60 minutes (min) at 60 C and relatively decrease afterwards. The reaction time of the product yield after more than 65 minute to 75 minute has no

significant effect on the conversion triglycerides, but leads to a reduction in the product yield. This is because, longer reaction time enhances the hydrolysis of esters (reverse reaction of transesterification) which results in the

loss of esters as well as causing more fatty acids to form

soap. The gradual increase in reaction time shows the negative effect on the

productyield. It is clear that the reaction time is an important controlling factor for the product yields. The similar observation is reported by Eevera et al. (2009),

Karavalakis et al. (2011), Sbihi et al. (2014).

94

92

CPME

90

88

86

84 Reaction time : 60 min

Reaction temp : 60 °C

82 Speed : 550 rpm

No. of wash : 5

80 Amount of KOH : 14 g

78 Amount of Methanol : 500 ml

76

30 40 50 60 70 80

Reaction time (min.)

Figure 5.2 Variation of different reaction time with methyl esters yield for transesterification of ceiba pentandra oil

5.1.1.3

Quantity of catalyst

The effect of two catalyst used in the transesterification were compared through examining the esters content in the bio diesel product obtained. Two catalyst exhibited similar trends on the conversion of triglycerides to esters but different quantity of catalyst were required for

achieving the same conversion. The effect of NaOH and KOH quantity on the transesterification of the ceiba pentandra oil was investigated with its quantity varying from 8 g to 16g. The operating conditions during the reaction process were fixed as reaction temperature is 60ºC, reaction time is 60 minute and amount of methanol used for the study is 500ml.

Figure 5.3 present the result of variation of yield with different quantities of NaOH and KOH as catalysts.

Two catalyst exhibited similar trends on the conversion of triglycerides to esters but different amount yields were obtained for the same

amount of catalyst used.

Figure 5.3 Variation of different catalyst quantity with methyl esters yield for transesterification of ceiba pentandra oil

Maximum esters content of the bio diesel product reached at 92% and 88% yield for catalyst quantity for 14g of KOH and NaOH respectively. The amount of KOH used was less than that of NaOH for same mass of feedstock oil. Hence, KOH is the best catalyst when compared to NaOH.

The

similar observation is reported by Karmee and Chadha 2005, Meher et al. (2006); Rashid & Anwar (2008); Fahad Ahmed (2016).

It can be analyzed that from Figure 5.3, increase in NaOH/KOH concentration beyond the optimal value which results drop in yield of bio diesel production and soap formation. This

is

observed during the experimental study. This is because of addition of excess of alkaline catalyst (NaOH/KOH) caused more triglycerides participating in the saponification reaction with NaOH/KOH producing more soap there by reducing the yield of esters. The another possible reason for the rise is formation of emulsion may be the addition of excess amount of catalyst gave rise in the formation of an

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emulsion, which increased the viscosity and led to the formation of

gels. The similar results were observed by Karmee & Anju Chadha (2005); Ramadhas et al. (2005 b) & Meher et al. (2006);

Nur Syakirah Talha & Sarina Sulaiman (2016).

5.1.1.4 Production of bio diesel from watermelon oil

The above procedure is followed by production of biodiesel from watermelon seed oil. It is

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observed from Figure 5.4

the reaction is incomplete up to 500 rpm, and rate of mixing was insufficient for methanolysis. Further increasing the speed of 550 rpm

is yield 88% is obtained

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and again further increase in the speed of stirrer to 600 rpm. The yield of methyl esters is reduced to 86%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel

from watermelon seed oil for this batch type production unit.

Figure 5.4 Variation of

different catalyst quantity with methyl esters yield for transesterification of

watermelon oil.

PHASE II

5.2

FUEL PROPERTIES

Laboratory tests were carried out to determine the properties such as relative density, viscosity, flash point, cloud & pour point and calorific values for two different blend ratios ofesters in varying proportions viz., 25%,

50%, 75%, with diesel and 100% of esters (bio diesel). Gas chromatography

and mass spectrum

studies were also employed for qualitative analysis. The result of this study is presented below.

5.2.1 Specific Gravity

The variation of specific gravity of ceiba pentandra oil methyl ester (CPME) and watermelon oil methyl ester (WME) and diesel blends with different percentage in volume are shown in Figure 5.5.

It can be observed from

the figure

specific gravity increases linearly with increasing concentrations of CPME, WME in diesel blends. The higher specific gravity of CPME and WME

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compared to diesel

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may be attributed to the higher molecular weight of triglyceride molecules present in the oil.

Slightly higher specific gravity of the

ester blends compared to base diesel makes their energy content on volume basis nearly closer to diesel than the energy content on mass basis. The fuel injection equipment operates on a volume metering system hence a higher specific gravity for esters and its blends with diesel results in the delivery of a slightly higher mass of fuel to engine.

As compared with WME specific gravity of CPME is slightly low for the same blend ratio.

These results are in agreement with work done by Alamu et al. (2007), Raheman and Ghadge 2007, Demirbas 2008,Srivastava, PK and Verma,

M 2008, Ijaz et al.., 2016).

Figure 5.5 Variation of percentage of bio diesel in diesel blends with specific gravity

5.2.2 Viscosity

The variation of viscosity of the of ceiba pentandra oil methyl ester and watermelon oil methyl ester and diesel blends with different percentage in volume is shown

in

Figure 5.6. After the transesterification process,

the viscosity of the FAME is reduced considerably to levels comparable to (But still higher than) that of

diesel. All of the biodiesel blended with diesel have a slightly higher viscosity; particularly B100 of CPME and WME have higher viscosity than diesel and other blends. The viscosity rises with the increasing concentrations of methyl ester with diesel blends.

As compared to WME the values of viscosity is less for CPME for the same blend ratio at a temperature of 40° C.

This is due to the higher molecular mass in the structure of esters (Mittel 1996; Karmee & Chadha 2005; Joshi & Pegg 2007; Demirbas 2008; Rao 2011& Kaisan et al. 2013).

Figure 5.6 Variation of percentage of bio diesel in diesel blends with viscosity

5.2.3 Flash Point

Figure 5.7 shows the variation of flash point with percentage of

two types

of methyl ester and bio diesel in diesel blends.With reference to the figure, all blends have the flash point, which is higher than that of diesel fuel. Flash point increases linearly with increasing concentration of methyl ester and bio diesel-diesel in the blends. The flash point of diesel is 54ºC, the flash point of CPME and WME are 148ºC, and 154ºC respectively.It is also higher than that of diesel but lower than that of before transesterification of the oil. This is due to the larger molecular mass in the structure of oil and methyl ester companied with unsaturated and saturated fatty acids present in their structure. . This leads to rises

in

the flash point. This higher flash point of methyl esters and its blends with diesel makes storage safer. It also called as “safe fuel”. These results are in accordance with work done by Alamu et al. (2007); Srivastava & Verma (2008); Naik et al. (2008); Suresh Kumar et al. (2008) & Pradeesh et al. (2016).

Figure 5.7 Variation of percentage of bio diesel in diesel blends with flash point

5.2.4 Cloud and Pour Point

Figure 5.8 and Figure 5.9 shows the variation of cloud and pour point with percentage of bio diesel in diesel blends. From the

Figure 5.8 and Figure 5.9,

it was found that, cloud and pour points of diesel fuel are 2º C and

1º C respectively, whereas biodiesel at 100% volume (B100) becomes a gel at approximately 7ºC. In such a state it becomes problematic to use in CI engine, it may clog filters and causes difficulties in pumping from the fuel tank to the engine. This is due to presence of unsaturated and saturated fatty acids present in the structure. The cloud and pour points of oils and bio diesel can be reduced by blending it with diesel fuel. In general, the pour and cloud points of diesel were lower than that of bio diesel. Both pour point and cloud point of bio diesel and its blends with diesel fuel increase with increasing the concentrations of bio diesel. Three trials were conducted for each (biodiesel- diesel blends) sample to check the consistency of results. The difference among all the three measurements are never more than 1ºC. The presence of alcohol made the pour and cloud points of bio diesel slightly lower than that of raw oil. Upto B25 blend, these properties were found to be almost closer to that of diesel and as the blend ratio increases, the value of cloud point is also increases. This might be due to the presence of wax in oil, which begins to crystallize with the reduction in temperature. This difficult can be overcome by using lower blends up to 25% (Alamu et al. 2007;

Joshi & Pegg 2007; Srivastava & Verma 2008; Sajjad et al. 2014).

Figure 5.8 Variation of percentage of bio diesel in diesel blends with cloud point

Figure 5.9 Variation of percentage of bio diesel in diesel blends with pour point

5.2.5 Calorific Value

Figure 5.10 Variation of percentage of bio diesel in diesel blends with calorific value

Figure 5.10 shows the variation of calorific value with percentage of bio diesel in diesel blends. It is clearly observed from the

figure the calorific values of blends uniformly decrease with the increase in bio diesel percentage in blends. The calorific values of diesel and B100 of CPME and WME were 42,800 kJ/kg, 40016 kJ/kg and 39,850 kJ/kg respectively. Measured calorific values of diesel were found to be higher than that of bio diesel blends, due to the difference in their chemical composition from that of diesel or the difference in the percentage of carbon and hydrogen content of oil and esters (bio diesel) or due to the oxygen content as suggested (Naik et al. 2008). Tranesterification process improves the calorific value of raw ceibea pentandra seed oil compared to esters (bio diesel). This is due to unwanted fats presents in the raw oil were removed in

the tranesterification process. These results are in accordance with the work done by (Srivastava & Verma 2008; Suresh Kumar et al. 2008;

Giakoumis

2013)

5.2.6 Fuel Properties of Bio-Diesel-

Pine Oil-Diesel Blends

In general, when high viscous fuels are blended with low viscous fuels such as

bio-diesel (

or) diesel the overall viscosity of the resulting blend could be lower. In the same way, the blend of high viscous CPME, less viscous pine oil with diesel has brought a reduction in biodiesel and diesel viscosity, increasing in pine oil viscosity

as shown in Table 5.1

which in- turns has improved the its atomization and mixing with air.

Table 5.1 Fuel properties of bio diesel, pine oil and diesel

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blends

Properties DIESEL PI B25 CPME+PI75 B25 CPME +PI50+DI25 B25 CPME +PI25+DI50 Kinematic viscosity (cSt) @ 40°C 3.21 2.5 3.05 3.22 3.38 Specific gravity 0.838 0.841 0.852 0.851 0.85 (C.alorific value kJ/kg) 42800 42961 42227 42188 41147 Flash point

° C 52 49 73.5 74.5 75.5

PHASE III

5.3

QUALITATIVE ANALYSIS

5.3.1 Diesel

The identified peaks and compounds in the diesel are shown in Figure 5.11 and Table 5.2 respectively. It is clearly shown from the above results it contains large peaks and high purities of –ane- group (octadecane, nondecane, furan etc.,) present, which leads to good combustion properties.

Figure 5.11 GC-MS analysis for diesel

Table 5.2 Major compounds in the diesel

Peak No

Compound Chemical formula MW (Mol. Weight) Retention time RT (min) 1. 2-Hydrobicyclo[3,1,1] heptan-6-one

C7H10O2 126 6.262 2. 3-Methyl-2-(2-oxopropyl)furan C8H10O2 138 8.067 3. Octadecane(cas)$$ N- Octadecane C18H36 254 10.021 4. Octadecane(cas)$$ N- Octadecane C18H36 254 11.968 5. 2-Hydrobicyclo[3,1,1]heptan-6-one C7H10O2 126 12.525 6. Butyl 2,4- dimethyl-2-nitro-4- pentenoate C11H19O4N 229 13.535 7. Octadecane(cas)$$ N- Octadecane C18H36 254 13.876 8. Butyl 2,4- dimethyl-2-nitro-4- pentenoate C11H19O4N 229 15.692 9. Nondecane(cas)$$ N- Nondecane C19H40 268 17.450 10. Octadecane(cas)$$ N- Octadecane C18H36 254 19.130 11. Butyl 2,4- dimethyl-2-nitro-4- pentenoate C11H19O4N 229 20.743 12. Nondecane(cas)$$ N- Nondecane C19H40 268 22.291 13. Octadecane(cas)$$ N- Octadecane C18H36 254 23.880 14. Nondecane(cas)$$ N- Nondecane C19H40 268 25.600 15. Octadecane(cas)$$ N- Octadecane C18H36 254 27.341 16. Docosane(cas)$$ N- Docosan$$C22H46 standard C22H46 310 28.327 17. Nondecane(cas)$$ N- Nondecane C19H40 268 29.270 18. Doctricontanol(cas)$$doctricontanol$$ N-Dot C32H66O 466 31.674 19.

Docosane(cas)$$ N- Docosane$$C22H46 standard C22H46 310 34.731 20. 1-

Tetradecanol(cas)$$ alfol 14 $$ tetradecanol$$loxanol V $$ lanette K $$ lanette

C22H46

214

35.224

5.3.2 Methyl Esters of Ceiba Pentandra Oil

Methanol (CH3OH) is used as the solvent alcohol and KOH as a catalyst for production of methyl esters (bio diesel) from ceiba pentandraoil. Some compounds are found in esters having good fuel properties of after transesterification. The identified peaks and compounds in the biodiesel of ceiba pentandra oil are shown in Figure 5.12 and Table 5.3 respectively. The majority of bio diesel (methyl ester) is in highly purified form and in large quantities. The compounds found in high quantities are shown in peak Nos.

1,2,3,7 as the esters compound (bio diesel), but some compound in

peak Nos.

4,5,6 and 8

remain as the impurities in less amount. These compounds of

peak

Nos 6 and 8

are the fatty acid alcohols.

Figure 5.12 GC-MS analysis for methyl esters of ceiba pentandra oil

Table 5.3 Major compounds in the methyl esters of ceiba pentandra oil

Peak No

Compound Chemical formula MW (Molecular Weight) Retention time RT (min) 1. Tetradecanoic acid,10,13- dimethyl,methyl esters

C17H34O2 270 19.499 2. Docasnedioic acid, dimethyl esters (cas) $$ methyl Docosane-1, 22-dioate. C24H46O4 398 22.664 3. Heptacosanoic acid, 26- methyl,- methyl esters C29H58O2 438 22.752 4. 3, 4 Epoxy-6, 9- octadecadiene. C18H32O 264 23.049 5. Pentane, 3-ethyl-(cas) $$ ethyl pentane. C7H16 100 23.910 6. 1-Tetradecanol(cas)$$ 14 $$ tetradecanol $$ loxanol V $$ lanette K $$ C14H30O 214 26.022 7. Heptacosanoic acid, 26- methyl,- methyl esters C29H58O2 438 26.161 8. 1-Dotricontanol. C32H66O 466 30.523

5.3.3

Methyl Esters of Watermelon Oil

Methanol (CH3OH) is used as the solvent alcohol and KOH as a catalyst for production of methyl esters (bio diesel) from watermelon oil. The identified peaks and compounds in the methyl esters of watermelon oil are shown in Figure 5.13 and Table 5.4 respectively. According to the methyl esters results

as shown in

Table 5.4, it is clearly seen that a high percentage of esters compound are formed as in peak Nos. 3, 4, 5, and 8. However, some compounds in peak Nos. 1, 2, 6, 7, 9, and 10 are in less percentage as impurities or supporting compound and may be found in dissolved form in esters or alcohol.

Figure 5.13 GC-MS analysis for methyl esters of watermelon oil

Table 5.4 Major compounds in the watermelon methyl esters

Peak No

Compound Chemical formula MW (Mol. Weight) Retention time RT in min 1 Heptanone, 4 Methyl (CAS) $$ 4- methyl – 3 Heptanone

C8H16O 128 4.927 2 3-Heptanone 4-methyl (Cas) $$ 4 methyl – 3- Heptano C8H16O 128 5.048 3 Hexadecanoic acid, ethyl esters (cas) $$ ethyl palm C18H36O2 284 20.396 4 9-Octadecenic acid (z). Methyl esters (cas) $$ Met C19H36O2 296 22.406 5 8.11.14 eicosatrienoic acid, methyl esters (cas) $$M C21H36O2 320 22.522 6 2.5.8 Heptadecatrien – 1OL C17H30O 250 24.391 7 3.Methyl – 2 (2-Oxopropyl) furan C8H10O2 138 25.935 8 Hexadecanoic Acid, Ethyl Esters (cas) $$ Ethylpalm C18H36O2 284 33.019 9 4Isoxazolamine, 3 Phenyl C9H80

N2 160 34.068 10 3,4 Benzo – 6, 7-Dihydro – 6,7 Dimethyl (1H) Azepine – 2,5–Dio C12H13O2

N 203 34.466

PHASE IV

5.4

PERFORMANCE AND EMISSION CHARACTERISTICS OF COMPRESSION IGNITION ENGINE FUELLED WITH CEIBA PENTANDRA AND WATERMALON METHYL ESTERS

A series of experiments has been conducted on a single cylinder four stroke 3.7kW direct injection diesel engine fuelled with diesel, pine oil, bio-diesel

from CPME, WME and

diesel blends. The tests were carried out in the standard injection timing (23º BTDC).

The important results of the experimental work are presented in the following sections. The configurations of different fuel blends are given in Table 5.5

Table 5.5 Configuration of the different fuels

S.No Description Abbreviation 1 Diesel DI 2 Pine oil PO 3 Ceiba pentandra oil methyl ester CPME 4 Watermelon oil methyl ester WME

5.4.1 Brake Thermal Efficiency (Performance Parameters)

The following sections describe

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the disparity of brake thermal efficiency with respect to brake power

for biodiesel-diesel

blends of CPME, WME and diesel.

Figure 5.14 and Figure 5.15

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shows

the

variation of brake thermal efficiency with brake power for CPME, WME and its blends.

The calorific value of bio-diesel is lower and the BSFC of biodiesel was much higher

compared to base diesel operation condition.

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Since, the brake thermal efficiency is inversely proportional to its BSFC and heating value of fuel. For all the blends, the brake thermal efficiency

has the propensity to increase with increase in applied brake power. At full load conditions, among the B25, B50, B75 and B100

bio-diesel

blends up to B25 has a maximum brake thermal efficiency of 30.69%, 30.35% and 31.88% respectively, for CPME, WME and diesel.At full load conditions,the brake thermal efficiency up to B25 is almost closer to that of base diesel

which is depicted in figure 5.14 and 5.15. As

comparedto B100 bio-diesel

operation the brake thermal efficiency of B25 has increased by 3.76% and 3.28% for CPME and WME. It may be due to enhanced spray characteristics and dissolved oxygen molecule structure in bio-diesel of B25 blend of CPME and WME in the combustion chamber, which leads to effective utilization of air resulting in complete combustion of the fuel.

Figure 5.14 The variation of brake thermal efficiency for CPME

It is also evident from the plots that when the brake thermal efficiency is gradually decrease, the percentage of bio-diesel (CPME and WME) from B50 to B100 is gradually increase. The reason for that is the supply of biodiesel fuel to the engine is high as compare with base diesel, in order to maintain the equal energy input to the engine. The high viscous of the blended fuels inhibits the fuel vaporization, proper atomization and combustion. This trend is also due to the combined effect of lower heating value, higher viscosity and density of the blended fuel.

In addition to this, studies conducted by pervious researchers Kumar et al. (2001), Nazar et al.

2004, Suresh Kumar et al. (2008), Dinesha and Mohanan (2015) and Abedin et al. (2016).

From the experimental results proved that performance of ceiba pentandra methyl ester (CPME) B25 blend is the best fuel compared to WME and other blends.

Figure 5.15 The variation of brake thermal efficiency for WME

5.4.2 Exhaust Emissions

Diesel engine emits carbon monoxide, carbon dioxide, hydrocarbon, oxides of nitrogen and smoke are considered to be the major emissions from exhaust. From the above emission stated NOx and smoke are most important emissions from a C.I engine that has to be efficiently controlled due to diffusive combustion and high flame temperature in the combustion chamber. The lesser amount of CO and HC emission produces C.I engine than S.I engines. Moreover, in case of bio-diesel-

diesel blended fuelled engines, presence of airborne oxygen as well as its presence in the molecule structures of bio-diesel-diesel blended fuel aids nearly complete combustion of fuel. From the above study, CO, CO2, HC, NOx and smoke analysis presented in the following sections.

5.4.2.1 Carbon monoxide (CO)

The variation of carbon monoxide (CO) emissions with respect to brake power presented in

Figure 5.16 and Figure 5.17

are discussed for CPME and WME and its blends with diesel. The characteristics of

DI-CI

engine shows when the load increases the air fuel ratio decreased. Usually, lower CO emissions emits CI engine

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due to excess air. CO is mainly dependent on the air-fuel ratio relatively to the chemically correct proportion and increase the air-fuel ratio becomes more than stoichiometric air-fuel requirements stated by (Kalam et al. 2003).

It could be seen from Figure 5.16 and

Figure 5.17 that the CPME and WME fuelled engine emits less CO emission in compare with diesel fuelled. Because bio-diesel is an oxygenated fuel, which helps for complete combustion; reduction of CO emissions were observed in the experimental work. It is also clear from the plots the minimum CO emissions were observed with B100 for CPME and WME becomes maximum of 0.14 and 0.15 by %

in

vol. at full load condition. The combustion of biodiesel presence of extra oxygen molecule in the bio-diesel

chain might have converted some of the CO in to CO2 and reduced CO formation. Similar finding were noted with CO in the experimental work conducted by Kumar et al. (2001), Sureshkumar et al. (2008), Datta and Mandal(2016).

5.4.2.2 Carbon dioxide

Figure 5.18 and

Figure 5.19 show

the variation of carbon dioxide (CO2) emissions in percentage in volume with brake power for diesel, CPME, WME and its blends. This measurements reveals that the brake power increases with increases in CO2 emissions; it indicates the combustion chamber efficiency, When compared with diesel the B25 percentage of blends emit almost same amount of CO2 emission. This is because

bio-diesel

is generally a lower elemental carbon to hydrogen ratio and has a low carbon fuel than base diesel fuel. The ratio of the blends increases from B50 to B100, there is a slight increase in CO2 emission which is due to more oxygen content of methyl esters. In B100, the value of CO2 for CPME and WME were emits 6.7% and 6.9 % by vol. respectively and more CO2 emissions in compression with base diesel. The insignificant differences were observed between

bio-diesel blends and diesel from B25 to B100. In general, bio diesel themselves are considered carbon neutral because, all the CO2 released during combustion has been sequestered from the atmosphere during the process of photosynthesis for the growth of vegetable oil crops, which are later processed into fuel. Hence, bio diesel also helps to mitigate global warming, as carbon dioxide levels are kept in balance. These results are in accordance with experimental work done by Barsic and Humke (1981), Ramadhas et al. (2005 a), Alamu et al. (2007), Suresh Kumar et al. (2008) andKumar and Sharma (2016).

Figure 5.16 The Variation of COEmission for CPME

Figure 5.17 The Variation of COEmission for WME

Figure 5.18 The Variation of CO2 Emission for CPME

Figure 5.19 The Variation of CO2 Emission for WME

5.4.2.3 Hydrocarbon

Figure 5.20 and

Figure 5.21show

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the variation of hydrocarbon (HC) emissions in parts per million (ppm) with respect to brake power

for diesel, CPME, WME and its blends. Experimental study exposed that the HC emission is reduced for all blended fuels (B25 to B100) as compare with diesel at full load. For all the blends, up to partial load the HC emission are lower, but increase at maximum load. This is due to the availability of relatively a lesser amount of oxygen content

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for the reaction, when more fuel is injected into the cylinder at higher

load. The ratio of the bio-diesel blends increases from B25 to B100, the HC emission decreases. This is due to the fact that cetane number of diesel fuel is generally lower than that of bio- diesel.

The HC emission for diesel at full load is 63ppm where as for B100 of CPME and WME were 45ppm and 48ppm respectively. This is due to

poor physical properties; high surface tension and viscosity affect the penetration rate, maximum penetration and droplet size which in turn affect the mixing of air-fuel. Apart from this the diesel has higher volatility compared to B100 which may also responsible for more HC emission; the engine operations are being affected. These results are good association with the previous researchers reported by (Kumar et al. 2001; Nazar et al. 2004; Srivastava & Verma 2008; Banapurmath et al. 2008; Agarwal et al. 2015).

However, HC emission was lower for blended fuels (B25 to B75) as compared with B100 and diesel at maximum load.

The ignition delay period effects causes in lower HC emission. In addition, the intrinsic oxygen contained in the biodiesel is responsible for the reduction in HC emission value for 22ppm which is less than the diesel. This is due to the sufficient oxygen presence in the biodiesel than base diesel. This phenomena also observed by the previous researchers (Sureshkumar et al. 2008 & Abedin et al. 2016).

Figure 5.20 The Variation of HC Emission for CPME

Figure 5.21 The Variation of HC Emission for WME

5.4.2.4 Oxides of nitrogen

The oxide of nitrogen (NOx) in the exhaust emission consists of nitrogen dioxide (NO2) and nitric oxide (NO).

The

NOx formation is highly dependent on engine technology. In general concentration of the NOx emissions varies linearly with the load, and when the engine

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load increases the overall air-fuel ratio increase resultant in an increase in the average temperature

of the gas in the combustion chamber and more NOx formation.

The variation of NOx emissions (ppm) with respect to engine brake power

shown in Figure 5.22 and 5.23

for diesel, bio-diesel and its blends.The NOx emission of diesel at full load was 875ppm, whereas B100 of CPME and WME

is 768

ppm and 754ppm respectively. This is due to reduced premixed combustion rate and hence reduces in combustion temperatures. High viscosities of B100 bio-diesels resulted in poor atomization, decrease cone angle, reduce spray penetration and larger mean droplet size than base diesel, it resulting in lesser amount of air entrainment and to poor combustion characteristics leading to lower combustion temperatures. These results are in agreement with the experimental study done by (Patterson et al. 2006;

Agarwal & Agarwal 2007; Rashedul et al. 2015).

The

bio-diesel

blends ratio increases (B25 to B50) with increase in NOx emissions. This is due to complete combustion of fuel increasing exhaust gas temperature and also by the presence of oxygen molecules in the

bio-diesel. when the emissions of NOx increased up to B50, bio-diesel blended fuel is attributed to difference in the compressibility of thediesel andbio-diesel

blends fuel, which results in inadvertent advance in dynamic injection timing when

bio-diesel-

diesel blends are used in

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the engine. Apart from this, the bio-diesel-

diesel blends has longer combustion duration compared to diesel resulting in higher exhaust gas temperature.

These results were in good association with the experimental results of previous researchers (Patterson et al. 2006; Nazer et al. 2004;

Prabakaran & Dinoop Viswanathan,

2016).

Figure 5.22 The Variation of NOx Emission for CPME

5.4.2.5 Smoke

Smoke is nothing but solid soot particles suspended in the exhaust gas. The quantity of soot formed depends upon the type of fuel and fuel ratio. In general NOx and smoke are trade-off. The variation of smoke emissions with respect to brake power shown in Figures 5.24 and

Figure 5.25

discussed for diesel, CPME, WME and its blends. the smoke variations are given in Hartridge Smoke Unit (HSU). For all the bio-diesel blends, the brake power increase with increase in smoke emission. The more smoke emissions are forme is indicative of incomplete combustion of fuel. The smoke quantity present in the exhaust gas is measured to amount the particulate matter present in the

exhaust gas . The smoke emission of base diesel at full load is

55 HSU whereas B100 of CPME and WME is 67 HSU

and 71HSU respectively. This is due to lower volatility and higher viscosity can result in poor air-fuel mixture formation, resulting in higher smoke emissions. Another probable reason is B100 biodiesel has stronger molecule structures, which leads to poor atomization of fuel there by increasing smoke emission.

These results are in good agreement with the pervious researchers reported by (Hebbal 2006; Pradeep & Sharma 2007; Datta & Mandal 2016).

However at full load condition the smoke emission is reduced for lower blends (B25 and B50) as compared with the higher blends (B75 and B100) due to betterfuel combustion. The lower viscosity of blended fuels (B25 and B50) as well as oxygen present in the molecules lower the smoke emission

as reported by (Kalam et al. 2003; Banapurmath et al. 2008). When the bio-diesel blend ratio increase (B75 and B100)

the value of

smoke emission also increased. This is may be due to high viscosity

of B100,

which reduce the fuel jet Reynolds number, decrease in

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air fuel mixing rate and leading to larger mean fuel

drop sizes at higher blends (B75 and B100)

as suggested by (Barsic & Humke 1981; Devan & Mahalakshmi 2009).

Figure 5.24 The variation of Smoke Opacity for CPME

From Phase IV experimental results proved that B25 CPME is the best blend ratio for DI-CI engine as compared with other bio-diesel blends. Because brake thermal efficiency of the engine is not affected and emissions are reduced (Expect NOx). So, in this phase concludes B25 CPME is best blend ratio compared with WME and its blends.

In general, when high viscous fuels are blended with low viscous fuels such as

bio-diesel (

or) diesel the overall viscosity of the resulting blend could be lower. In the same way, the blend of high viscous CPME, less viscous pine oil with diesel has brought a reduction in

bio-diesel

and diesel viscosity, increasing in pine oil viscosity, which in-turns has improved the its atomization and mixing with air.

In Phase V pine oil mixed with B25 CPME blend. Because pine oil has a less viscosity

compared to

diesel. On the other hand, B25 CPME is higher viscosity compare with diesel (Devan & Mahalakshmi et al. 2009; Vallinayagam et al. 2014).

PHASE-V

5.5 PERFORMANCE AND EMISSIONS CHARACTERISTICS OF DI-CI ENGINE FUELLED WITH B25 CPME-PO- DIESEL

BLENDS

In this phase

same test engine were used without any modification, to study the,

performance, emissions and combustion characteristics of PO, B25 + PO75 (B25%CPME and Pine oil 75%), B25+ PO50+DI25 (B25%CPME, Pine oil 50% and Diesel 25%), B25+PO25+DI50 (B25% CPME, Pine oil 25% and Diesel 50%) and diesel.

5.5.1

Brake Thermal Efficiency

Figure 5.26

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shows

the disparity of brake thermal efficiency (BTE) with respect to brake power. For diesel, pine oil,

bio-diesel-

pine oil-diesel and its blends. B25+PO50+DI25 blends brake thermal efficiency (30.71%) is close to diesel (31.88%) and PO (31.98%) due to viscosity and heating value close to diesel. The oxygen content in

bio-diesel

and pine oil slightly larger fuel droplets which contribute better atomization, leading to proper mixing, vaporization, ignition and as a result complete combustion. The engine brake thermal efficiency depends upon number of factors but the most meaningful property is viscosity, density and heating value

suggested by (Kasiraman et al. 2012).

Figure 5.26 The variation of brake thermal efficiency for biodiesel, PO, and its blends

PO brake thermal efficiency is higher than diesel, and PO75+DI25 blends brake thermal efficiency also nearly close to diesel. This is may be due to higher heating value of PO than diesel. From this experimental investigation with

bio-diesel-

PO-diesel blends in diesel engine, it is observed that 50% of PO is found to be optimize blend because for blends beyond 50% of PO, the engine suffered some vibration, particularly at full load condition. However, lesser cetane number of PO affected the fuel injection suggested by Vallinayagam et al. (2014). From the experimental results,

it is

proved that performance of B25+PO50+DI25 blend is the best fuel compared to other

blends.

5.5.2

Exhaust Emission

The exhaust emissions such as CO, CO2, HC, NOx and smoke are measured and analyzed. For above mentioned emissions, NOx and smoke is the most significant emissions for diesel engine due to high flame temperature and diffusive combustion in the combustion chamber. Since NOx and smoke emissions from current diesel technologies are closer to the limits permitted by regulations and both limits will be even more stringent in the near future, these two emissions will be critical factors in the development of new diesel engine. Apart from this, diesel engine produces lesser amount of CO and HC emissions then spark ignition engine.From the above facts, emission from

B25 CPME-

pine oil blended with diesel focused on NOx and smoke.

5.5.2.1

Oxides of nitrogen

Figure 5.27 describes

the variations of NOx emissions with respect to brake power for diesel, pine oil, biodiesel-PO-diesel and its blends. The emissions of NOx for diesel at maximum load is 875ppm, where PO, B25+PO75, B25+PO50+DI25 and B25+PO25+DI50 are 913, 899, 869 and

865ppm respectively. POand B25+PO75 blend NOx emission was higher than diesel. This is due to the sudden burning of PO air mixture by the auto- ignition of diesel, lower cetane number (longer ignition delay) of fuel is higher heat release rate, could have resulted higher in-cylinder temperature and presence of inbuilt oxygen with in the PO has produced more NOx formation

suggested by (Lee et al. 2011 & Zhang et al. 2012).

B25+PO50+DI25 blend NOx emission is lower than diesel, PO, and B25+PO75 blends. The blend of less viscous pine oil and high viscous CPME (lower calorific value) with diesel has brought a reduce in

bio-diesel

and diesel viscosity, increasing in pine oil viscosity, which in-turns has improved the atomization and mixing with air

suggested (Devan & Mahalakshmi et al.

2009; Vallinayagam et al. 2014).

Figure 5.27 The variation of NOx for biodiesel, PO, and its blends.

5.5.3

Smoke Emission

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The variation of smoke emission with respect to brake power for diesel,

PO, bio-diesel-PO-diesel blends as shown in Figure 5.28.

Smoke is attributed to either mixture of

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fuel-air that are too lean to auto ignite (or) to support a

propagating flame (or) mixture of air fuel that are also rich to ignite

is suggested by (yashiyuki et al. 2000).

PO, biodiesel-PO- diesel blends smoke emission was (51 HSU) lower compared to diesel (55HSU). This may be due to higher calorific value,

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lower viscosity and boiling point of PO is effectual mixing with air forming locally lean regions limiting the primary smoke formation, responsible for decreasing smoke emission

reported by Anand et al. (2010); Vallinayagam et al. (2014).

Figure 5.28 The variation of

smoke for biodiesel, PO, and its blends

PHASE- VI

5.6 ENGINE COMBUSTION ANALYSIS

FUELLED WITH B25

CPME - PINE OIL – DEISEL

The combustion investigation of pine oil-

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bio-diesel blends with diesel is carried out with the help of combustion pressure sensor (optrand make pressure range 0-200bar), and software (Engine test express, legion brothers,written “visul C”), crank angle Encoder (Make kubler, 360 PPR coupled to crankshaft).

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In a C.I engine, cylinder pressure depends on the burned fuel fraction during the premixed burning phase, ie, initial stage of combustion. This stage of combustion depends upon the ability of the fuel to mix well with air and burn.

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Heat release diagram is a quantitative description of timely burning fuel in engine. It has a major effect on cycle efficiency and maximum cylinder pressure. The heat release program is based on the first law of thermodynamics.

5.6.1

Cylinder Pressure

Figures 5.29 depicts the variation of cylinder pressurewith respect to the crank angle

of diesel, B25+PO50+DI25 and B100 CPME at full load and standard injecton timing (23° BTDC).

It is proved that, at same operating condition as compared to diesel, the methylester percentage blends rises the peak cylinder pressure were reduced and also the peak pressures of diesel, B25 +PO50+DI25 and B100 CPME were recored as 65.16bar, 63.85 bar and

47.44 bar respectively. The major possible reason for decreased cylinder pressure when compared to diesel is greater molecular structure, lesser heating value and volatility slightly higher viscosity of the bio-diesel. Additional possible reason owing to slow burnning rates of the methyl ester blends. The supplementary imaginable reason is during the combustion of diesel, it released more amount of energy.

Another possible explanation the combination of slow burning rates at a lower minimum pressure rise of the bio-diesel fuels, which might have caused lower mechanical stress in the blended fuel operation.

Therefore combustion noise is lower when bio diesel- diesel blends operation.

Figure 5.29 The variation of cylinder pressure with crank angle for biodiesel, PO, and its blends.

5.6.2 Heat Release Rate

Figure 5.30

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shows the variation of maximum heat relase rate (kJ/m3

degree) with respect to the crank angle

fordiesel, B25+PO50+DI25 and B100

CPME.The combustion rate difference, start of combustion and the fraction of fuel burned in the premixed modes of fuel are identified by the heat release rate. At full load and standard injection timing (23 BTDC). The variation of

heat release rate with crank angle were recorded. The maximum and minimum heat release rate for diesel, B25+PO50+DI25 and B100 CPME were recorded as 62.57kg/m3deg., 59.58 kg/m3deg., 43.58 kg/m3deg. The bio- diesel blends had shorter ignition delay, When compared to diesel, Because of heat relase ratereduced, the shorter ignition delay is caused. Diesel is longer ignition delay to allowed for more air/fuel prepartion, which is ready to auto ignite and result a higher premixed peak (Ozener

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et al. 2014).

Figure 5.30 The variation of heat release rate with crank angle for

biodiesel, PO, and its blends

CHAPTER 6

CONCLUSIONS

work

The following conclusions were drawn in the present experimental

6.1

BIO-DIESEL PRODUCTION

x

Biodiesel is produced from ceiba pentandra seed oil and watermelon seed oil. The catalyst KOH is found to be superior than NaOH, So, KOH is the best suitable catalyst of methanolysis process.

x The maximum yields of 92% and 88% bio-diesel obtained from ceiba pentandra oil and watermelon oil.

6.2

PHYSICO-CHEMICAL PROPERTIES AND QUANTITIVE ANALYSIS

x The transesterification

process improved the fuel properties of the oil with respect to relative density,

viscosity, flash point, cloud point, pour point and calorific value.

The methyl esters properties of ceiba pentandra with pine oil blends physic-

chemical characteristics are near to ASTM standards.

x When the blends ratio of bio-diesel increased, increased kinematic viscosity, specific gravity, flash point and reduced

calorific value when compared to diesel.

The methyl esters properties of ceiba pentandra with pine oil blends physic- chemical characteristics are near to ASTM standards.

x Quantitive analysis proved that produced CPME and WME

have more ester content high purity and good fuel property.

6.3

PERFORMANCE AND EMISSION CHARACTERISTICS

x

The engine performance of B25 blend (30.69%) is nearly similar to that of diesel (31.88%) at the same time not affected the engine performance.

x Compared with diesel, bio-diesel-diesel blends emit lower CO, HC, smoke and slightly increased NOx emission.

From the experimental results proved that performance of ceiba pentandra methyl ester B25 best blended fuel compared to WME and other

blends.

x B 25

CPME +

PO50 + DI25 blends brake thermal efficiency (31.06) is close to diesel(31.88%) due to viscosity and heating value are close to diesel.

x

Up to B25 CPME + PO50 + DI25

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blends can be used in a diesel engine without any engine modification. It does not affect performance, emission and combustion characteristics.

The ceiba pentandra with pine oil-diesel blends improved the fuel

atomization and easily mixed with air result in increased in brake thermal efficiency compared with CPME-WME.

6.4

COMBUSTION ANALYSIS

The overall combustion characteristics of B 25

CPME +

PO 50 + DI 25 blend is proved that the cylinder pressure and heat release rate is (63.85bar and 59.58 kJ/m3 deg.) higher than B100CPME (47.44 bar and

43.58kJ/m3 deg.) and closer to diesel (65.16 bar and 62.57 kJ/m3 deg.).

6.5 OVERALL CONCLUSION

From the experimental result proved that 25% of ceiba pentandra methyl ester blended with 25% of diesel and 50% of pine oil blends ratio is a best alternative fuel for naturally aspirated direct injection diesel engine producing a 3.7 kW at a rated speed of 1500 rpm without any modification of the engine.

6.6

FUTURE WORK

x Addition of

adding some anti-freezing additives is needed to improve the cold flow properties of bio diesel to use under severe winter conditions.

x Engine performance characteristics on the

blends of bio diesel and pine oil can be investigated on the various injection timing

in DI-CI engine.

x

Measurement of aldehyde emissions can be carried out.

x

Study the characteristics of LHR engine fuelled with the

B25CPME+PO50+DI25 blend ratio.

x Study the NOx emission on DI-CI

engine can be operated with the different percentage of EGR.

APPENDIX 1

SPECIFICATIONS OF THE ENGINE

MAKE Kirloskar AV1

Type Single cylinder, Direct Injection,Naturally Aspirated, 4 Stroke,water cooled Engine BHP 3.7 kW = 5HP @ 1500 rpm BORE 80 mm STROKE 110 mm COMPRESSION RATIO 16.5:1 TYPE OF IGNITION Compression Ignition INJECTION TIMING 23º bTDC METHOD OF LOADING Eddy Current Dynamometer AVL 444-5 GAS ANALYZER Emission (HC, CO, CO2 ,O2, &

NOX)

AVL 437 SMOKE METER Smoke Density FUEL USED Diesel, Bio-diesel, pine oil Blends

APPENDIX 2

SPECIFICATIONS OF THE DYNAMOMETER

Make PowerMag Type Air cooled eddy current dynamometer

Loading Auto loading system, the engine loading is automatically done through the computer Coupling type Tyre Load measurement method Strain gauge Maximum Power 5hp @ 2000 rpm

APPENDIX 3

SPECIFICATION OF THE COMBUSTION ANALYSER

Make Optrand make combustion pressure sensor Type AutoPSI-S AutoPSI-TC and Auto PSI-HT Sensors

1. Specifications

Over Pressure : 2 x Pressure Range (typical)

Non-Linearity & Hysteresis : ±0.5% FS under non-combustion

Conditions, under constant temperature

±1% FS under combustion conditions,

i.e., varying temperature within one

Combustion cycle

Frequency Range : 0.1 Hz to 20 kHz (Standard) Diaphragm Resonant Frequency : 120 kHz min.

Cable Operating Temperature : -40°C to 200°C Sensor Housing Temperature Range: -40°C to 350° C

Fiber optic Cable Length : 1.5m (5’) Fiber optic Cable Min.

Sensor Type : Sealed Gauge

Interface Unit : Integrated with Sensor

Bending Radius : 5mm (3/16”) (Analog) 5V DC input : 0.5 - 4.5 V

Diagnostic Output Signal : 9-18V DC input: 0.5 – 2.5 V (Analog) 5V DC input : 0.5 – 2.5 V

Pressure Output Signal : 9-18V DC input: 0.5 – 4.5 V Power Supply Voltage : 9-18V DC or 5V DC

Interface Temperature Range : AutoPSI-S,A,TC:-20°C to 60°C Current Draw : 85 mA Max, 50 mA Typical AutoPSI-HT : -20°C to 125°C

Vibration : 100G

Pressure Media : Gaseous or Liquid

Guaranteed Lifetime : 1, 2 or 3 Years depending on sensor package or application

APPENDIX 4

SPECIFICATIONS OF THE AVL DIGAS 444 ANALYSER

Make AVL Type AVL Digas 444 Power supply 11…22 voltage | 25 W Connector gas in | 180 l/h, max. overpressure 450 hPa Warm up time | 7 min. Storage temperature 0…50 qC Operating temperature 5 …45 qC Response time t95

d 15s Inclination 0…90q‘ Relative humidity d 95%,

non-condensing Weight 4.5 kg net weight without accessories Dimension (w x d x h) 270 x 320 x 85 mm3 Interfaces RS 232 C, Pick up, Oil temperature probe

APPENDIX 5

SPECIFICATIONS OF THE AVL 437 SMOKE METER

Make AVL 437 Smoke meter Type IP 52 Measuring range 0 to 100 opacity in % 0 to 99.99 absorption m-1 Measurement chamber Effective length 0.430 m r 0.005m Accuracy and reproducibility

r 1 %

full scale reading Light source Halogen bulb 12 V/5W Heating time 220 V approximately 20 min. Dimensions 570mm u 500mm u 1250mm Power supply 190 – 240 V AC, 50…60 Hz, 2.5 A Maximum smoke temperature 250 qC Weight Approx. 50 kg net

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Category 2009-2010 2010-2011 2011-2012 2012-2013 2013-2014 2014-2015 Production Sales Production Sales Production Sales Production Sales Production Sales Production Sales Passenger Vehicles 23,57,411 19,51,333 29,82,772 25,01,542 31,46,069 26,29,839 32,31,058 26,65,015 30,87,973 25,03,509 32,20,172 26,01,1 Commercial vehicles 5,67,556 5,32,721 7,60,735 6,84,905 9,29,136 8,09,499 8,32,649 7,93,211 6.99,035 6,32,851 6,97,083 6,14,961 Three wheelers 6,19,194 4,40,392 7,99,533 5,26,024 8,79,289 5,13,281 8,39,748 5,38,290 8,30,108 4,80,085 9,49,021 5,31,927 Two wheelers 1,05,12,903 93,70,951 1,33,49,349 1,17,68,910 1,54,27,532 1,34,09,150 1,57,44,156 1,37,97,185 1,68,83,049 1,48,06,778 1,84,99,970 1,60,04,581 Total 1,40,57,064 1,22,95,397 1,78,92,409 1,54,81,381 2,03,82,026 1,73,61,769 2,06,47,611 1,77,93,701 2,15,00,165 1,84,23,223 2,33,66,246 1,97,52,580

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Density (kg/m3) Viscosity (mm2/s HHV (kJ/kg) LHV (

kJ/kg) CFP point (°C) Pour point (°C) Cloud point (°C) Flash Point (°C) Cetane number Oxidation Stability (h) Iodine number Distillation temp.50% vol. (°C) Distillation Temp.90% Vol.(°C)

Sulfur content (ppm) Acid number (mg KOH/g)

Molecular weight (kg/kmol) O (% w/w) H (% w/w) C (% w/w)

Castor 917.6 (6.98) 14.52 (1.78) 39,900 (1926) 37,630 (243) 5.9 (18.5) -23.4 (4.0) -15.1 (12.5) 160.9 (37.5) 42.1 (6.83) 12.9 (6.1) 85.2 (3.3) 327 (8.5) 381.6 (23.4) 0.5 (0.45) 0.39 (0.18) 270 _ _ _ Chicken fat 876.3 (6.37) 4.81 (0.50) 39,890 (405) 37,610 2.7 (0.6) 4.40 (1.8) 7.5 (1.3) 162.2 (9.8) 57 (5.66) 8.7 (3.21) 78.7 (1.30) _ _ _ 0.32 (0.15) _ _ _ _ Beef tallow 874.3 (3.82) 4.83 (0.50) 40,040 (216) 37,220 (274) 12.2 (2.3) 10.0 (2.0) 13.8 (1.8) 157.2 (8.6) 60.9 (5.55) 0.45 (0) 54.5 (10.3) 329.2 (3.5) 344.2 (6.6) 14.3 (5.5) 0.30 (0.18) _ 11.35 (0.39) 12.60 (0.81) 76.09 (0.69)

Canola 881.6 (3.51) 4.40 (0.25) 39,975 (150) 37,980 (860) -8.8 (3.3) -8.0 (2.6) -1.8 (1.4) 159.0 (14.0) 54.8 (3.37) 11.0 (0.95) 104.0 (4.6) 352.0 (1.7) 354.3 (4.9) 2.2 (1.2) 0.22 (0.14) _ 11.04 12.84 _ Cotton seed 879.0 (5.49) 4.70 (0.88) 40,480 (435) 38,175 (1135) 4.8 (2.5) -0.2 (4.1) 1.2 (4.4) 165.4 (14.0) 53.3 (2.75) 1.8 105.7 _ 349.5 (9.1) 5.3 (4.2) 0.29 (0.18) _ 11.21 (0.62) 11.89 (0.16) 76.86 (0.77)

Karanja 882.9 (5.50) 5.04 (0.60) 40,275 (671) 36,490 (426) -7.0 2.5 (3.4) 7.6 (4.3) 163.6 (14.7) 55.4 (2.49) 4.1 (2.44) 85.5 (2.2) 334 345.4 (11.1) 13.5 (9.2) 0.44 (0.05) _ 11.51 (0.96) 11.95 (0.70) 76.35 (1.51)

Coconut 870.8 (4.71) 2.78 (0.26) 38,985 (1369) 35,985 (921) -4.7 (0.6) -3.8 (1.2) -1.2 (3.9) 127.7 (29.1) 61 (6.46) 11.0 (5.64) 7.8 (2.5) _ _ 3.3 (1.3) 0.16 (0.11) _ _ 11.65 (0.50) 72.75 (0.07)

Croton 883.2 (4.95) 4.48 (0.28) 40,280 37,825 (1578) 11.0 -6.3 (4.6) -4.0 174.5 (20.5) 50.6 (5.66) 4.0 142 352 358 _ 0.31 (0.26) _ 10.20 11.97 77.83

Fish 887.3 (7.15) 4.30 (0.33) 40,550 (955) 38,800 (1131) -3.0 (3.7) -4.0 (0) -0.3 (2.41) 162.6 (8.8) 51 (1.52) 8.2 (3.46) 142.6 (13.81) 339 360.2 (18.9) 5.7 (4.2) 0.39 (0.35) _ _ 11.85 77.40

Lard 873.0 (2.25) 4.89 (0.11) 39,950 (212) 36,910 9.0 (4.2) 11.0 15 153.5 (9.2) _ _ 64.5 (11.4) 329 337 _ 0.36 (0.26) _ 11.71 12.11 76.01

corn 882.2 (4.71) 4.32 (0.37) 40,190 (826) 38,600 (69) -7.5 (3.2) -5.1 (2.5) -3.0 (1.6) 165.7 (5.9) 52.5 (2.64) 3.0 (1.34) 120 (16.9) - 345.8 (7.0) 3 (2.1) 0.19 (0.06) _ 10.98 (0.03) 11.52 (0.54) 76.71 (0.56)

Jatropha 878.7 (6.52) 4.72 (0.56) 40,380 (1259) 38,050 (956) -1.2 (3.9) -0.9 (4.7) 5.7 (4.3) 158.5 (19.5) 55.7 (2.84) 5.0 (1.88) 99.0 (5.4) 333.3 (0.6) 341.2 (12.1) 12.9 (15.4) 0.34 (0.12) _ 11.32 (0.66) 12.21 (0.28) 76.57 (0.37)

Hazelnut 877.9 (15.2) 4.55 (0.91) 39,800 (469) 37,230 -10.2 (5.4) -14.0 (1.4) -12 (3.8) 163.5 (30.8) 53.8 (1.70) 53.8 (1.70) 7.6 92.5 (2.1) _ _ 0.22 _ _ 11.94 76.57

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Yield (%)

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Figure 5.23

The Variation of NOx Emission for WME

Figure 5.25 The variation of Smoke Opacity for

WME

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The variation of cylinder pressure with crank angle for biodiesel, PO, and its blends 133 5.30 The variation of heat release rate with crank angle for	the variations of maximum cylinder pressure (bar) with respect to the crank angle for different pine oil and diesel, CPME, Fig. 8. The variation of heat release rate with crank angle for
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Ceiba pentandra seed were low feeding value due to its higher fiber content. Moreover, the opening of kapok (ceiba pentandra) fiber as bio- ethanol feed stock.	Ceiba pentandra seed were low feeding value owing to its higher fiber content. Furthermore, the possibility of kapok (ceiba pentandra) fiber as bio- ethanol feed stock.
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kapok fiber is utilized as stuffing material for pillows and bed (Tye et al. 2012,	kapok fiber is utilized as stuffing material for pillows and bed (Panneerselvam et al., 2016). 2.
54: http://isi-dl.com/downloadpdf/22054 41%	54: http://isi-dl.com/downloadpdf/22054 41%
Naturally aspirated (NA- DI) engine is selected for evaluation of bio diesel, pine oil fuels based on two specific reasons. First, a NA-DI engine represents a larger population of engines sold in agricultural and construction equipment for the last 55 years. Secondly, a	Naturally aspirated (NA) engine was selected for evaluation of bio-diesel fuel based on two particular reasons. First, a NA engine represents a more population of engines sold in construction and agricultural equipments of the previous few dec- ades. Second, a

naturally aspirated engine is more sensitive due to longer ignition delays and lower pressure injection equipment.

The Kirloskar engine is

naturally aspirated engine is more sensitive due to lengthier ignition delay and lesser pressure injection equipment. The kirloskar engine is

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widely used engines in agricultural, pump sets, farm machinery, transport-vehicles, small and

medium scale commercial purposes. The engine can withstand higher pressures encountered during tests because of its rugged construction. Further, the necessary modification on the cylinder head and piston crown can be easily carried out in this type of engine. Hence this engine is selected for the present research work.

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widely used in agricultural, farm machinery, pumps sets, transport vehicles, small and medium commercial purpose. The engine can withstand higher pressure encountered during tests because of its rugged con- struction. Moreover, the necessary modification on the cylinder head and piston crown can be easily carried out in this type of engine. So, this engine is preferred for the current research work.

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AVL-444 DI Gas analyzer is used to measure the exhaust tail pipe emissions such as HC, CO, CO2, and NOx. The smoke opacity is measure with the aid of AVL 437 standard smoke meter.

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AVL-444 DI gas analyzer is used to record the exhaust tail pipeline emissions such as CO, CO 2 , HC and NOx. The smoke emission was measured with the aid of AVL 437 standard smoke meter.

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before starting experimental tests, the engine is made to run under ideal condition as warm up phase and then the

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Before starting the test procedure the engine was made to run without load condition for 20 min as a warm up phase and then the

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The combustion analysis of diesel and bio diesel-diesel pine oil blends is carried out with the assistance of combustion pressure sensor (Optrand make combustion pressure sensor range 0-200 bar),

The combustion analysis of

bio-diesel blends and diesel was carried out with the assistance of combustion pressure sen- sor (optrand make pressure range 0–200 bar),

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may be attributed to the higher molecular weight of triglyceride molecules present in the oil.

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may be due to the higher molecular weight of triglycerides mole- cules present in the oil.

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blends

Properties DIESEL PI B25 CPME+PI75 B25 CPME +PI50+DI25 B25 CPME +PI25+DI50 Kinematic viscosity (cSt) @ 40°C 3.21 2.5 3.05 3.22 3.38 Specific gravity 0.838 0.841 0.852 0.851 0.85 (C.alorific value kJ/kg) 42800 42961 42227 42188 41147 Flash point

° C 52 49 73.5 74.5 75.5

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blends. Properties Diesel Pine oil B25 B50 B75 B100 Kinematic viscosity (cSt) at 40 *C 3.21 1.10 2.56 3.24 3.92 4.58 Specific gravity 0.838 0.841 0.852 0.863 0.875 0.887 Calorific value (kJ/kg) 42,800 42,961 42,218 41,485 40,746 40,016 Flash point *C 52 48 57 73 114 148

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the disparity of brake thermal efficiency with respect to brake power

for biodiesel-diesel

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the variation of brake thermal efficiency (

BTE) with respect to brake power for diesel,

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shows

the

variation of brake thermal efficiency with brake power for CPME, WME and its blends.

shows

the variation of brake thermal efficiency (

BTE) with respect to brake power for diesel,

pine oil,

CPME and CPMEP blends.

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Since, the brake thermal efficiency is inversely proportional to its BSFC and heating value of fuel. For all the blends, the brake thermal efficiency

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Since, the brake thermal efficiency is inversely proportional to its BSFC and heating value of fuel. Fig. 3

shows

the variation of brake thermal efficiency (

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due to excess air. CO is mainly dependent on the air-fuel ratio relatively to the chemically correct proportion and increase the air-fuel ratio becomes more than stoichiometric air-fuel requirements stated by (Kalam et al. 2003).

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due to excess air. Carbon monoxide is mainly dependent on the air-fuel ratio relative to the chemically correct proportion and increases when the air-fuel ratio becomes greater than stoichiometric air-fuel requirements reported by Kalam et al. (2003).

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the variation of hydrocarbon (HC) emissions in parts per million (ppm) with respect to brake power

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the variation of unburned hydrocarbon (HC) emissions in parts per million (ppm) with respect to brake power.

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for the reaction, when more fuel is injected into the cylinder at higher

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for the reaction, when more fuel is injected into the engine cylinder at higher

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load increases the overall air-fuel ratio increase resultant in an increase in the average temperature

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load increases the overall fuel-air ratio increased, resulting in an increase in the average gas temperature

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the engine. Apart from this, the bio-diesel-

diesel blends has longer combustion duration compared to diesel resulting in higher exhaust gas temperature.

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the efficiency. Apart from this the pine oil has longer combustion duration compared to diesel and biodiesel resulting in higher exhaust gas temperature (

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air fuel mixing rate and leading to larger mean fuel

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air fuel mixing rate and leading to larger mean fuel

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shows

the disparity of brake thermal efficiency (BTE) with respect to brake power. For diesel, pine oil,

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shows

the variation of brake thermal efficiency (

BTE) with respect to brake power for diesel, pine oil,

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The variation of smoke emission with respect to brake power for diesel,

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The variation of smoke emission with respect to brake power for diesel,

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fuel-air that are too lean to auto ignite (or) to support a

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fuel-air mixtures that are too lean to auto ignite or to support a

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lower viscosity and boiling point of PO is effectual mixing with air forming locally lean regions limiting the primary smoke formation, responsible for decreasing smoke emission

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lower viscosity and higher calorific value of pine oil is efficient mixing with air forming locally lean regions limiting the primary smoke formation, responsible for decreasing smoke emission.

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bio-diesel blends with diesel is carried out with the help of combustion pressure sensor (optrand make pressure range 0-200bar), and software (Engine test express, legion brothers,written “visul C”), crank angle Encoder (Make kubler, 360 PPR coupled to crankshaft).

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bio-diesel blends and diesel was carried out with the assistance of combustion pressure sen- sor (optrand make pressure range 0–200 bar),

and software(Engine test express, make legion brothers, written ‘ ‘visul C”), crank angle Encoder (Make kubler, 360 PPR coupled to crankshaft). 4.1.

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In a C.I engine, cylinder pressure depends on the burned fuel fraction during the premixed burning phase, ie, initial stage of combustion. This stage of combustion depends upon the ability of the fuel to mix well with air and burn.

In a C.I engine, cylinder pressure depends on the burned fuel fraction during the premixed burning phase, that is, first stage of combustion. This stage of combustion depends upon the ability of the fuel to mix well with air and burn.

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Heat release diagram is a quantitative description of timely burning fuel in engine. It has a major effect on cycle efficiency and maximum cylinder pressure. The heat release program is based on the first law of thermodynamics.

5.6.1

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Heat release diagram is a quantitative description of timely burning fuel in engine. It has a major effect on cycle efficiency and maximum cylinder pressure. The heat release program is based on the first law of thermodynamics.

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shows the variation of maximum heat relase rate (kJ/m3

degree) with respect to the crank angle

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shows the variations of maximum heat release rate (kJ/m 3 degree) with respect to the crank angle

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et al. 2014).

Figure 5.30 The variation of heat release rate with crank angle for

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et al. (2004). Fig. 10. The variation of cumulative heat release rate with crank angle for

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blends can be used in a diesel engine without any engine modification. It does not affect performance, emission and combustion characteristics.

blends can be used in a diesel engine without any engine modification. It does not affect performance, emission and combustion characteristics.

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steam treatment process shadowed by mechanical crushing process. The oil seed yield is producing on average

2850 kg/ha.

Ceiba pentandra seed were low feeding value due to its higher fiber content. Moreover, the opening of kapok (ceiba pentandra) fiber as bio- ethanol feed stock.

It found that kapok fiber contains 34 to 64% of cellulose higher potential to produce cellulosic ethanol.

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kapok fiber is utilized as stuffing material for pillows and bed (Tye et al.

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Silitonga et al. 2013).

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kapok fiber is utilized as stuffing material for pillows and bed. (Tye et al., 2012., and Ong et al., 2013) 2.2

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alcohol from an ester by some other alcohol, which resembles to

hydrolysis anticipate before that an alcohol is used as a substitute of water. This has been

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transesterification process. The methanol is used in this process, so it is called methanolysis. Methanolysis of triglycerides is represented in Equation (2).

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one of the reversible reactions and proceeds basically by mixing the reactants. But, the presence of a catalyst (a strong acid or base) speed up the conversion (

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three successive and reversible reactions are conceived to occur. These reactions are indicated in Equation (3).

alcohol from an ester by another alcohol in the process similar to hydrolysis except then that an alcohol is used instead of water. This has been

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transesterification process. If the methanol is used in this process then it is called methanolysis. Methanolysis of triglycerides is represented in equation -2. (2)

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one of the reversible reactions and proceeds essentially by mixing the reactants. However, the presence of a catalyst (a strong acid or base) accelerates the conversion. 2.3

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three consecutive and reversible reactions are believed to occur. These reactions are represented in equation-3. (3)

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Equation (3) Chemistry of transesterification

The first step transition presents the triglycerides to diglycerides, adopted by the conversion of diglycerides to monoglycerides controlling one methyl ester molecule from each glycerides at each step

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Equation -3.Chemistry of transesterification The first step in the conversion is triglycerides to diglycerides, followed by the conversion of diglycerides to monoglycerides yielding one methyl ester molecule from each glycerides at each step. (

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et al. 2015).

2.7.2.3 Methanol to oil molar ratio

Alcohol to triglyceride is one of the most important factor

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et al., 2014) . 3.4 Effect of methanol to oil ratio The methanol to oil ratio is one of the most important factor

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to a reduction in the product yield due to the backward reaction of transesterification, resulting in loss of ester as well as causing more fatty acids to form

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to a reduction in the product yield. This is because, longer reaction time enhances the hydrolysis of esters (reverse reaction of transesterification) which results in the loss of esters as well as causing more fatty acids to form

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Figure 4.2 Line diagram of the bio diesel production unit

1. Container
2. DC motor
3. Heater
4. Thermocouple
5. Blades
6. Speed sensor
7. Inlet valve
8. Outlet valve
9. Microprocessor kit
10. Electrical connection for motor, eater and thermocouple
11. Cooling fan formicroprocessor kit
12. Temperature indicator
13. Speed (rpm) indicator
14. ON/OFF switch
15. Speed regulator for DCMotor

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Figure1 Schematic diagram of the bio-diesel production unit 1. Container 2. DC motor 3. Heater 4. Thermocouple 5. Blades 6. Speed sensor 7. Inlet valve 8. Outlet valve 9. Microprocessor kit 10. Electrical connection for motor, heater and thermocouple Cooling fan for microprocessor kit 11. Temperature indicator 12. Speed (rpm) indicator 13. ON/OFF switch 14. Speed regulator for DC Motor 15. Transformer 16. Sensor controller 17. Shaft 3

16. Transformer

17. Sensor controller

18. Shaft

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Procedure for Transesterification Process

x

Mixing of reactants is found to be an important factor which effects the transesterification and yield of

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procedure for the transesterification process Mixing of reactants is found to be important factor which affects the transesterification and yield of

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Operation is carried out at a speed of 600 rpm in the first stage of reaction (10 min) and a lower stirring speed of 550 rpm for

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Operation was carried out up to a speed of 600 rpm in the first stage of reaction (10min) and a lower stirring speed of 550 rpm for

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remaining period.

x 2.5

litre of ceiba pentandra oil is filtered using cloth to remove the impurities in the oil and poured into a bio diesel production unit

remaining period. A 2.5 liters of Ceiba pentandra raw oil is filtered using musline cloth to remove the impurities in the oil and poured in to a biodiesel production unit.

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KOH is quickly weighed, protecting it as much as possible from atmospheric moisture and carbon di-oxide.

x The pellets are quickly moved to the dry mixture grinder to change into powder form.

x The

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KOH) is quickly weighed, protecting it as much as possible from the atmospheric moisture and carbondi-oxide. The pellets are quickly transferred to the dry mixture grinder to convert it in to powder form. The

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catalyst and solvents (methanol) solution is then vigorously shaken in a conical flask for homogeneous mixing. At this point dissolved catalyst (KOH) is presumed to have been converted into potassium/sodium/methoxide.

x A fixed amount of solvent and catalyst solution is poured into the oil

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catalyst and solvent of methanol solution is then vigorously shaken in a conical flask for homogeneous mixing. At this point dissolved catalyst of potassium hydroxide (KOH) is presumed to have been converted into potassium methoxide. A fixed amount of solvent and catalyst solution is poured into the raw oil

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Once the reaction period is over, then the product is allowed to settle

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Once the reaction period is over the product is allowed to settle

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process turns the oil into esters, separating out the glycerol. The glycerol sinks at the bottom and the bio diesel floats on top and can be syphoned off.

x The crude ester phase is separated and glycerol phase is then washed by warm de-ionized (double distilled) water for four times

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process turns the oil into esters, separating out the glycerol. The glycerol sinks at the bottom and the biodiesel floats on top and can be syphoned off. The crude esters phase is separated and glycerol phase is then washed by warm de-ionized (double distilled) water for several times

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The excess methanol and water in the ester phase is removed (or recycled) by evaporation under atmospheric condition after being measured for product yield calculation.

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Chemistry of

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speed of the stirrer

Mixing is very important in the transesterification reaction, as an oil and fat are immiscible with potassium hydroxide-methanol solution.

Once the two phase are mixed the reaction is started.

Methanolysis is conducted with different stirrer rates such as 350, 400, 450, 500, 550,600, 650 and 700 revolutions per minutes (rpm).

The variation of methyl esters yield with different rate of stirrer is shown in Figure 5.1. It is observed that

the reaction is incomplete up to 500 rpm, and rate of mixing is insufficient for methanolysis. Further increasing the speed of 550 rpm

speed of the stirrer Mixing is very important in the transesterification reaction, as an oil and fat are immiscible with potassium hydroxide-methanol solution. Once the two phase of mixed the reaction is started. Methanolysis was conducted with different stirrer rates such as 350, 400, 450, 500, 550 and 600, 650 and 700 revolutions per minutes (rpm). The variation of methyl esters yield with different rate of stirrer is shown in the figure 2. It was

observed from the figure 2 the reaction was incomplete up to 500 rpm, and rate of mixing was insufficient for methanolysis. Further increasing

the speed of 550 rpm

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the yield of 92% obtained and again further increase in the speed of stirrer to 600 rpm, the yield of methyl esters was reduced to 88%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel for this batch type production unit.

the speed of 550 rpm

was 92%

and again further increase in the speed of stirrer to 600 rpm. The yield of methyl esters was reduced to 88%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel for this batch type production unit.

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Effect of reaction time

The reaction time is required for the completion of alkaline-catalyst transesterification depends not only on the reaction temperature, but also on the degree of mixing in the process. The reaction rate of transesterification increases with increase

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Effect of reaction time The reaction time is required for the completion of alkaline-catalyst transesterification depends not only on the reaction temperature, but also on the degree of mixing in the process. The reaction rate of transesterification increase with increase

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temperature and degree of mixing. The reaction time of transesterifaction process is conducted at 60 C ie. Nearer to the boiling point of the methanol.The variation of methyl ester yield with different reaction time as shown in Figure 5.2.The figure

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temperature and degree of mixing. The reaction time of transesterifaction process is conducted at 60˚C ie. Nearer to the boiling point of the methanol. The variation of methyl ester yield with different reaction time as shown in figure 3. From the figure

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is less at the beginning and reaches a maximum at the reaction time of 60 minutes (min) at 60 C and relatively decrease afterwards. The reaction time of the product yield after more than 65 minute to 75 minute has no

significant effect on the conversion triglycerides, but leads to a reduction in the product yield. This is because, longer reaction time enhances the hydrolysis of esters (reverse reaction of transesterification) which results in the

loss of esters as well as causing more fatty acids to form

soap. The gradual increase in reaction time shows the negative effect on the

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is less at the beginning and reaches a maximum at the reaction time of 60 minutes (min) at 60˚C and relatively decrease afterwards. The reaction time of the product yield after more than 65 minute to 75 minute has no significant effect on the conversion triglycerides, but leads

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emulsion, which increased the viscosity and led to the formation of

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emulsion which increased the viscosity and led to the gels. The formation of

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observed from Figure 5.4

the reaction is incomplete up to 500 rpm, and rate of mixing was insufficient for methanolysis. Further increasing the speed of 550 rpm

observed from the figure 2 the reaction was incomplete up to 500 rpm, and rate of mixing was insufficient for methanolysis. Further increasing

the speed of 550 rpm

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and again further increase in the speed of stirrer to 600 rpm. The yield of methyl esters is reduced to 86%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel

from watermelon seed oil for this batch type production unit.

Figure 5.4 Variation of

and again further increase in the speed of stirrer to 600 rpm. The yield of methyl esters was reduced to 88%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel for this batch type production unit.

Figure 2 Influence of

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compared to diesel

may be attributed to the higher molecular weight of triglyceride molecules present in the oil.

compared to diesel. It may be due to attribution of the higher molecular weight of triglyceride molecules present in the oil.

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were designated for the statistical investigation, namely four animal fats (beef tallow, chicken, lard and fish), twenty-one oils derived from various plants (canola, castor, coconut, corn (maize), cottonseed, croton, hazelnut, karanja (honge), jatropha, linseed (flax), mahua, neem, olive, palm, peanut, rapeseed, rice bran, rubber seed, safflower, soybean and sunflower), as well as waste cooking oil. These 26

feed stocks finally

provided 945 different data series of biodiesel properties and 700 data series of fatty acid compositions. On the

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Rapeseed and the closely related canola (10.4+3.1=13.5%) were the next best familiar feedstocks, with a significant interest also found in palm (7.8%), the

non-edible

jatropha (6.9%;

Rapeseed and the closely related canola (10.4+3.1=13.5%) are the next most common feedstocks, with a considerable interest also found in waste cooking (9.5%), palm (7.8%), the non-edible jatropha (6.9%),

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of the research (50%) focused on beef tallow. The non-edible

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Viscosity

Viscosity is a measure of the fluid resistance which is being distorted by either tensile stress

viscosity Viscosity is a measure of the resistance of a fluid which is being deformed by either shear or tensile stress.

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specifications require biodiesel fuels to have at least 93°C flash point, whereas in the European the minimum essential level is 101° C; both are

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specifications require biodiesel fuels to have at least 101˚C flash point; in the US the minimum required level is 93˚C. Both specifications are

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Excluding castor, the (average) cetane numbers of the 25 feed stocks range from 50.4 for rubber seed methyl ester to 61.2 for PME, with the mean value from all feedstocks

being 54.8;

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excluding castor, the (average) viscosity values of the 25 examined feedstocks, range from 2.78 mm 2 /s (for the most saturated coconut ME) to 5.06 mm 2 /s (for mahua ME), with the mean value from all feedstocks being 4.55

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from 7.8 (for the most saturated, coconut) to 184.5 (for the most unsaturated one, linseed), with an overall average value

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Oxidative Stability

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fulfill the European specification of 6 hours oxidation stability

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more lenient, hence biodiesel produced from feedstocks such as corn, coconut, fish, jatropha and karanja could be approved.

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Many researchers used additives or did not apply the same esterification clean-up or storage conditions, all of which influence considerably the oxidation values

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many researchers used additives during the preparation process or did not apply the same esterification clean-up or storage conditions, all of which influence considerably the oxidation values. 2.3.8

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are cloud and pour point. Cloud point (CP) is the temperature at which wax form a cloudy appearance.

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are the cloud (CP) and the pour point (PP). The cloud point is the temperature at which wax forms a cloudy appearance.

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is the lowest temperature at which the fuel becomes semi solid and loses its flow characteristics being no longer pumpable;

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The pour point is always lower than the cloud point.

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There are no European or US specifications for low temperature properties (Each country is free to determine its own limits according to local weather conditions), but it is well known that biodiesel fuels suffer from cold flow properties

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than mineral diesel fuel. The (average) values of CP for the 26 examined feedstocks in the present work

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Judicious Recycling of Biobased Adsorbents for Biodiesel Purification: A Critical Review Alagumalai Avinash a and Arthanarisamy Murugesan b a Department of Mechanical Engineering, KPR Institute of Engineering and Technology, Arasur, Coimbatore Tamil Nadu, India; avinashandromeda@gmail.com (for correspondence) b Department of Mechanical Engineering, K.S. Rangasamy College of Technology, Tiruchengode Tamil Nadu, India Published online 00 Month 2018 in Wiley Online Library (wileyonlinelibrary.com). DOI 10.1002/ep.13077 Reusing biomass waste is one objective of environmental sustainability and has turned into a choice to increase the value of producers. Biosorbent, the most fitting illustrative of biomass waste, is as of now acquiring adequate attraction, because of its convenience and potentiality in protection of nature. Affirming the statement, this article presents a cutting-edge review of different biosorbents in dry cleaning of biodie- sel impurities. Moreover, the progress on the preparation of these biodegradable adsorbents, their formidable challenges in concert with the future prospect, has been featured and dis- cussed. Finally, the examination of biomass-derived adsor- bents in the area of adsorption science and technology represents a possible alternative, prompting the widespread change of pollution control. ? 2018 American Institute of Chemical Engineers Environ Prog, 2018 Keywords: biomass, biological residues, biosorbents, dry washing, adsorption INTRODUCTION In recent days, crude oil prices are shooting through the roof [1,2]. This triggered primary interest in the development of biomass-derived fuels as surrogate to petroleum-based fuels [3,4]. This trend has also made the biofuels such as biodiesel a viable option [5–7]. Biodiesel is a renewable and sustainable biofuel which is an outstanding substitute to diesel and it possesses significant advantages such as nontoxicity [8], inherent lubricity [9], biodegradability [10], high cetane number, and nonexistence of sul- fur [11]. The most widely used method to produce biodiesel is transesterification process, where the oil or fat is converted to biodiesel and glycerol in the presence of alcohol and catalyst [12,13]. In addition to glycerol, several impurities are present in biodiesel depending on the production technology (Table 1) [14]. After glycerol removal in biodiesel production process, the soap and trace amounts of glycerin are usually removed by wet washing method [15]. Research works have portrayed that

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the use of water to purify biodiesel causes several problems such as producing a large quantity of wastewater that must be treated

prior to reuse and there is a great deal of time and energy for drying [16]. Also, the water used for purification must be deionized in order to avoid the transfer of metal ions (especially calcium and magnesium ions) to the fuel [17]. Both of these trace metals possess toxic potential to the environ- ment [18]. Alternative to water washing, several purification methods have been reported in the literature. Researchers have used membrane filtration [19,20], ion exchange resins [21], and adsorbents [22] to purify biodiesel. Nevertheless, each of these techniques has their own advantages and drawbacks. In mem- brane filtration technique, high retention of free glycerol, total glyceride, as well as soap is achievable. Recent studies portray that biodiesel purification by polymeric composite nanofiltra- tion membranes are highly resistant to harsh conditions and are said to have an exceptional stability for biodiesel perme- ation along with high flux recovery ratio [19]. On the other hand, membrane fouling during cleaning is a major problem in membrane filtration technique [23]. Although ion exchange resins provide exceptional performance in the removal of soap, water, glycerol, and catalyst, this technique lacks in the effective remove of methanol [24]. Also, ion exchange is not cost-effective. At last, to make the adsorption process more efficient and effective, a filter unit must be provided and the process generates spent adsorbent that needs to be regener- ated [25]. Based on the above considerations, successful biodiesel purification by cost-effective process which imparts less harm to the environment is the most challenging topic to deal with, and it is more likely to be motivated by further research and development in upcoming days. In this perspective, biosor- bents are biomass-derived materials that have appeared to be invaluable asset primarily in the third-world economies. Recently, various research studies have dealt with the conver- sion of biomass into valuable products. With the aforemen- tioned, this article aspires to depict the recent advancements and prospective use of various biosorbents. This review is par- ticularly intended to present brief and up-to-date information of the different biosorbents in purification of biodiesel. AFFINITY-BASED SEPARATION PROCESSES Adsorption and ion exchange are the most common affinity-based separation methods, also known as dry washing methods for biodiesel purification. In these methods, a suitable adsorbent is used to specifically adsorb certain impurities from the liquid phase onto its surface [26]. In addition, dry washing technique offers several advantages over wet washing, includ- ing easiness of integration into an existing plant, shorter purifi- cation time, no water consumption and wastewater production, and smaller unit sizes [27]. The nonexistence of water in purification of biodiesel through dry washing results ? 2018 American Institute of Chemical Engineers Environmental Progress & Sustainable Energy DOI 10.1002/ep 1

in biodiesel with acceptable water content (less than 500 ppm) based on ASTM D6751 [28]. Adsorption In the last few decades, there has been a vast progression in adsorbent design and cyclic adsorption process develop- ments, thus making adsorption an imperative separation tech- nique [29]. Adsorptive separation is the process by which atoms, ions, or molecules, known as adsorbates, from a sub- stance (generally liquid or gas) adhere to a solid surface, called the adsorbent [30,31]. Adsorption is not the same as absorp- tion; though, the term “sorption” refers to both the methods. In the absorption process, the constituent dissolves in the bulk of absorbent while surface adhesion occurs during adsorption [30]. On the other hand, adsorbents are either natural or syn- thetic materials of amorphous or microcrystalline structures possessing basic and acidic adsorption sites [32], where polar substances such as glycerol and methanol can be adsorbed and filtered out of biodiesel [33–35]. Adsorption selectivity, cost, capacity, kinetics, compatibility, and regenerability are the most important factors that need to be considered for adsorbent selection. Thus, a proper selection of an adsorbent can certainly impact the process economy. The adsorption process for biodiesel purification can be also classified based on the type of adsorbent. Silica-based adsorbents, activated compounds, and biobased adsorbents are among the most common adsorbents for this process [32]. BIOBASED ADSORBENTS OR BIOSORBENTS A wide range of biowastes can be used as adsorbents such as starch-based and lignocellulosic substrates [36,37]. These biosorbents have many potential advantages such as low cost, lower regeneration temperature and less pollution through dis- posal [38]. In addition some of their disadvantages are supply fluctuation due to seasonal changes, bulky nature, and logistics constraints [39,40]. In order to expose the function of biobased adsorbents in the purification of biodiesel, this study aspires to present the possible and viable biosorbents for biodiesel puri- fication, their processing and production techniques, chal- lenges, and future prospects are discussed in the subsequent sections. Also, the process of biodiesel purification by biosorbent is illustrated (Figure 1). Activated Carbons In recent days, removal of heavy metals ions from aqueous effluents by adsorption method has gained interest and is typi- cally achieved by using activated carbon or activated alumina [41–44]. Activated carbon is a porous material with an extremely large surface area and intrinsic adsorption to several chemicals [45–48]. Several researchers have shown increased interest in the use of activated carbons in the dry washing method of biodiesel purification. Farid et al. [49] worked to improve the biodiesel quality produced from waste cooking oil using palm-derived biosorbent. They produced biosorbent by a two-step process, beginning with carbonization in a fur- nace and then by activation (700 * C for 2 h) and neutralization (0.1 M HCl). Hunsom and Autthanit [50] discussed the adsorp- tive purification of waste used-oil biodiesel using a set of 15 different activated carbons prepared from sewage sludge. The experimental results clearly showed that adsorption time and shaking rate are the two most influencing factors in the adsorptive purification. Studies by Fadhil and coworkers [51,52] showed that in general, using activated carbons of spent tea waste for waste frying oil biodiesel purification resulted in higher yields compared to the conventional silica gel and water washing method of biodiesel purification. In another study, Fadhil and Ahmed [53] reported that activated carbon produced from de-oiled fish waste can be used to purify the crude ethyl esters of biodiesel from fish oil. Thus, the exploit of different activated carbons improved the fuel properties of purified biodiesel. Furthermore, the properties of purified biodiesel met the standard limits [54,55]. Overall, activated carbon as an adsorbent facilitates better removal of con- taminants from biodiesel because of its higher surface area and existence of oxygen group in its surface. All these features aid in better interactions (either physical or chemical) between the adsorbent surface and the adsorbate. Banana Peel For years, Banana peel is thrown away as no longer useful or desirable material. However, recent studies have proved that banana peel show signs of adsorption potential to remove heavy metals [56,57] and dyes from waste water [58]. Also, the surface morphology studies with banana peel powder substan- tiate its microporous nature with crater-sized pores and the surface demonstrating microrough surface and asymmetrical particles, which promote its adsorption capacities [59]. A novel approach to purify Jatropha curcas-based biodiesel by using banana peel waste was discussed by Bang et al. [60]. They Table 1. Contaminants present in biodiesel depending on transesterification technology [14]. Contaminants Alkali catalyzed Acid catalyzed Supercritical Soaps By neutralization of free fatty acid (FFA) with catalyst After neutralization of the catalyst If feedstock treatment was ineffective Metals If feedstock treatment was ineffective If feedstock treatment was ineffective If feedstock treatment was ineffective FFAs No Due to incomplete esterification Due to hydrolysis of the feedstock Monoglycerides Product of transesterification Product of transesterification Product of transesterification Diglycerides Product of transesterification Product of transesterification Product of transesterification Triglycerides Due to incomplete conversion Due to incomplete conversion Due to incomplete conversion Glycerol Product of transesterification Product of transesterification Product of transesterification Figure 1.

Process of drying washing of biodiesel using biosorbents.[Color figurecanbeviewedatwileyonlinelibrary.com] 2 Environmental Progress & Sustainable Energy DOI 10.1002/ep

prepared the adsorbent by air drying banana peel for 72 h fol- lowed by drying in oven at 45 * C to constant weight. In this work, 3% (w/w) of prepared natural adsorbent was used in the purification of biodiesel at condition 65 * C and 25 min stir- ring. The study revealed that banana peel powder is efficient in the removal of impurities (352 mg/kg of soap, 1.132 mg/g of acid value, 1250 mg/kg of water, 2.6 mg/kg of potassium, and 0.17% of methanol) from unpurified biodiesel and the results are also similar with other commercial adsorbents (Purolite PD 206, Purolite PD 206, and Tulsion T-45BD mix- ture). These results are achievable because of the microporous nature of the banana peel powder and specific adsorptive functional groups present in their structure. Bentonite Clay Bentonite is a natural low silica adsorbent which is mainly composed of montmorillonite [61]. The adsorptive nature

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of bentonite is a function of the montmorillonite content and the number of interlayer cations. In order to remove impurities and various exchangeable cations, bentonite

can be modified by acid treatments [62,63]. Leeruang and Pengprecha [64] stud- ied the adsorptive properties of the acid activated bentonites in refined palm oil biodiesel purification process. This natural low silica was used in this study to remove glycerin from crude biodiesel by activating bentonite with 0.1 M H 2 SO 4 at 100 * C for 1 h. The experimental results disclosed that 47% of glycerin removal can be achieved with 5% of activated bentonite of 120–200 mesh size. Chamotte Clay Among several biobased adsorbents, chamotte clay is one of the low-cost alternative biosorbents which is a waste gener- ated from red ceramic industries [65]. Although chamotte clay's chemical composition varies with its origin, it is chiefly com- posed of silicon dioxide, aluminum, and iron oxide [66]. Santos et al. [67] assessed the potential of chamotte clay as an adsorbent to purify palm kernel oil biodiesel. The study showed that chamotte clay level of 2.5 w/v % at 45 * C maxi- mized glycerol removal (1282 mg/ g of glycerol) with minimum loss of adsorbate. The mineralogical characterization studies demonstrated that the adsorptive capacity of chamotte clay is because of the high content of silica and alumina in its compo- sition. The chamotte clay-purified biodiesel sample demon- strated comparable performance to water washing method. The study also reported unsatisfactory regenerability of the chamotte clay (30%) and the need to increase recycle ability by increasing regeneration temperature above 100 * C. Cow Dung Ash Cow dung possesses an important characteristic which has been in use since ancient times. It is used as a substance that deters mosquitoes from approaching or settling, a promising component part in the manufacture of mud brick housing, and as a low-priced thermal insulator [68]. It is used as agricultural manure [69] and in biogas production [70,71]. Furthermore, it is used in wastewater treatment process [72,73]. Avinash and Murugesan [74] exploited cow dung ash as an adsorbent to purify waste cooking oil biodiesel. In their work, cow dung ash was prepared by flaming cow dung cakes at 500 * C in the muffle furnace and then the produced ash was subjected to characterization analysis. From these analyses, the authors report that cow dung ash is an adsorbent with high SiO 2 con- tent along with trace amount of other metal oxides. The pre- pared cow dung ash at different concentrations such as 1% wt/wt, 2% wt/wt, 3% wt/wt, and 4% wt/wt were employed in purification of biodiesel, and eventually the purified biodiesel was passed through filter to remove the spent adsorbent. Through this research, the researchers were able to also find the ideal amount of adsorbent to be used for purification of biodiesel by noting the similarity and dissimilarity between the spectra. The experimental results revealed that cow dung ash concentration of 1% wt/wt showed satisfactory impurity removal (0.22 * 0.01 mg KOH/g of acid number, 205 mg KOH/g of saponification value, >0.01% of methanol, 1879.5 mg/kg of water, 0.00393 * 0.00002% of free glycerin, and 0.50 * 0.01% of total glycerin) from crude biodiesel com- pared to water purification method (0.21 * 0.01 mg KOH/g of acid number, 204 mg KOH/g of saponification value, 0.015 * 0.01% of methanol, 5365.2 mg/kg of water, 0.00035 * 0.00001% of free glycerin, and 0.49 * 0.01% of total glycerin). Fiber-Based Biosorbents The utilization of fiber-based biosorbents is an attractive and sustainable route to purify crude biodiesel as reported by Yang et al. [75]. The test results demonstrated that BD-Zorb, sawdust, and wood shavings can effectively remove soap (51.1, 24.4, and 9.4 mL/g, respectively) from crude camelina oil biodiesel. However, compared to commercial adsorbents, a lower purification capacity is achieved by fiber-based biosor- bents. This indicates continual replacements of adsorbents packed in purification towers, which might lead to an increase in labor costs. Also, the study indicated that subsidiary treat- ments are necessary to further reduce the acid index and water content of biodiesel purified by fiber-based biosorbents. Starch and Cellulose The use of starch and cellulose from different sources such as potato starch, corn starch, rice starch, and cassava starch for biodiesel purification exhibited that the application of these biosorbents is effective in removal of impurities such as free fatty acids, free glycerine, sodium, and water content from bio- diesel. However, the best results were reported for potato starch (5% and 10%) and cassava starch (1% and 2%) in removal of turbidity of biodiesel [76]. In addition to potato starch and cassava starch, studies by Squissato et al. [77] showed that cellulosic adsorbent from eucalyptus pulp is a promising material for metal removal from sunflower oil and used cooking oil biodiesel. The test results exhibited that high amounts of metal removal (Fe-13-81%, Mn-35-84%, Cu- 40-74%) are evident from the produced biodiesels. Although there is a positive advancement in purification of biodiesel using pretreated starch, its chemical treatment is not sufficiently studied. This concern the properties associated to the effectiveness of its practical application. For this reason, Ismaila et al. [78] investigated the adsorption of residual free fatty acids after the biodiesel production from palm fatty acid distillate, using KOH activated potato and cassava starch. The highest adsorption efficiency of these adsorbents was noted at 0.25:1 KOH/starch molar ratio. The authors of this study also noted that the performance of adsorption increased with increase in the amount of adsorbent quantity. On the other hand, the adsorption capacity of free fatty acid increases with the decrease in adsorbent quantity. Sugarcane Bagasse Several research studies have reported that sugarcane bagasse is gaining interest to be used as a low-cost adsorbent [79]. This lignocellulosic biomass is widely produced in Brazil as a sugar waste and waste from ethanol industries [80]. In addition to raw bagasse, chemically modified bagasses and activated carbons from bagasse have also been investigated [81]. In recent days, steam explosion is considered as one of the most commonly used physicochemical pretreatment methods to treat the composition of the lignocellulosic bio- mass and for degradation of lignin and hemicellulose. In adsorption process, the use of steam explosion to pretreat bagasse has the following advantages. This method proves that Environmental Progress & Sustainable Energy DOI 10.1002/ep 3

it has a higher surface area than raw bagasse, and by this method it is much easier to separate from the liquid medium than bagasse ash [82]. Alves et al. [82] compared the perfor- mance of three different forms of sugarcane bagasse (raw, exploded, and ash) as low-cost biosorbents to treat soybean biodiesel. The examination results revealed that wet washing method of biodiesel purification showed greater affinity to adsorb free glycerin (0.0002 * 0.00000 wt %) followed by commercial magnasol adsorption (0.0011 * 0.00010 wt %), and then by raw (0.0053 * 0.00000 wt %) and exploded sugar- cane bagasse (0.0067 * 0.00055 wt %). However, bagasse ash hardly removed the free glycerin content (0.0141 * 0.00364 wt %) due to low concentration of cellulosic substance. Also, the study clearly pointed out that all of the suggested purification methods did not considerably affect the properties of biodiesel such as acid value, viscosity, and density. However, they have substantially increased the water content. This increase is most likely related to the suitable addition of water during the wet washing method and to that of atmospheric water adsorption during the dry washing process. Mushroom Powder Apart from several agro-wastes, many microorganisms, fungi, algae, and green plants have confirmed huge potential as metal biosorbents [83]. In line, mushrooms are low-cost bio- sorbents that have been used to evaluate the environmental pollution level [84–86] and to remove heavy metals from con- taminated soil [87]. Also, mushrooms exhibit active sites for biosorption process [88]. Bang et al. [60] discovered the signifi- cance of mushroom powder as an adsorbent in J. curcas- based biodiesel by dry purification process. The morphology characteristics of mushroom powder before the utilization as adsorbent evidently give an idea about the existence of micro- porous structure at 2 μm magnification. However, the mor- phology result after mushroom powder has been used as an adsorbent obviously demonstrated the variation in the mor- phology. The chemical analysis of adsorbent after purification showed the presence of sodium demonstrating that the used adsorbent is more efficient in the soap removal and other con- taminants from biodiesel. Overall, the study disclosed that mushroom powder was highly effective in lowering the acid value (1.27 mg/g) of unpurified biodiesel in addition to removal of soap (320 mg/kg), water (2000 mg/kg), and resid- ual methanol content (0.27%). Rice Husk Ash Rice husk is a product of rice processing industries mainly characterized by its abrasive intrinsic resistance behavior [89] and silica-cellulose structural arrangement [90]. The main con- stituents of these agro-residues (rice husks) are cellulose, hemicellulose, lignin, hydrated silica, and ash content [91]. Over years, rice husks have been disposed in landfills, contrib- uting to esthetic pollution [92,93]. Increasing global environ- mental and public health concern paved way for generating energy from rice husks [94]. In this regard, rice husk ash is col- lected from rice husk fired boilers [95]. The rice husk ash is the most imperative renewable source of silica, which contains about 95% of silica and the trace amounts of other inorganic components such as K 2 O, CaO, and P 2 O 5 [96]. According to Foo and Hameed [97], rice husk ash is a potential adsorbent to control environmental pollution. They also offer a numbers of advantages and prevail over part of the issues related to agri- cultural wastes. Pioneering work by Manique et al. [98,99] demonstrated that rice hush ash can be successfully used as a biosorbent for purifying waste frying oil and soybean biodiesel similar to conventional method of biodiesel purification. Rice husk ash is very effective

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in removing glycerol, giving biodie- sel containing glycerol lower than 0.01% that meet the expected standard to be utilized as a fuel [100]. The

advantage of using rich husk ash as an adsorbent is that it is having high silica content which accounts for high adsorptivity and easy diffusion of adsorbed species. Research by Svitlana et al. [101] also stated that rice husk ash is much richer in silica and has a micro-/macroporous structure with a high specific area which aid in adsorption of impurities from nonedible oils (Balanites aegyptiaca, Azadirachta indica, and J. curcas) biodiesel. MAJOR CHALLENGES AND FUTURE PROSPECTS In recent days, a wide interest in the use of biobased adsor- bents is increasing as a result of extremely large waste produc- tion and bioresource conservation has directed a great deal of consideration toward the recuperation of input supply, contrib- uting new set of circumstances that makes it possible to vary the range of biobased adsorbents [102–104]. On the other hand, the major challenge in adsorbent purification process is the opti- mization of process parameters such as contact time, tempera- ture, and reusability. As reported in literature, the effectiveness of adsorbent purification process is enhanced when contact time is increased up to 20 min. Moreover, increasing the proces- sing time further may result in contamination by silicon due to processing by glass reactor [101]. Regarding temperature, increasing the temperature up to 65 * C actively supports reten- tion of large molecules (diacylglycerides and monoacylglycer- ides) as well as potassium. However, the other contaminants like sulfur, calcium, free glycerin, and mainly water are effi- ciently removed at ambient temperature (20 * C) [101]. Concern- ing the last challenge of adsorbent purification (reusability), several research studies have reported unsatisfactory perfor- mance in contaminants removal with reused adsorbents in dry purification method [98–101]. On the other hand, these adsor- bents have shown positive results in water removal when reused. This nature is mainly because of the longer contact time of adsorbent prompting primarily water adsorption [101]. Over- all, there is less or no potential in using most of the adsorbents in a continuous dry purification process. However, brick formu- lation and partial cement replacement are the viable options to reuse clay- and ash-based adsorbents. As suggested by Martí- nez-Martínez et al. [105], addition of glycerol to spent clay-based adsorbents will stimulate modifications in the microstructure and thus increases their thermal insulation properties. However, performance and economic feasibility of these spent adsorbents in brick formulation and partial cement replacement are needed to be explored further. In addition to the above issues, environmental and public concerns of biobased adsorbents are becoming a great signifi- cance to an increasing extent, stimulating in the direction of clean and sustainable technology [106]. In turn in the direction of reaching the place of green and sustainable energy policy, a compassionate situation to integrate energy conservation and energy quality measures has to be developed. Increasingly, there is a need to make breakthrough in sustainable technologi- cal advancements and improvements in biobased adsorbents production with the expressing necessity for environmental pro- tection and sustainability [107,108]. Although numerous research studies have reported the use of biomass-derived adsorbents for biodiesel purification, there have been issues in successful implementation. Generally, their industrial-scale production is facing different kinds of problems such as the accessibility of cost-effective method, innovatory and management of waste resources, suitable market tactics, and improvement and implementation of suitable policy to fruit- fully utilize these waste sources. Most importantly, full collabo- ration and mutual undertaking among different countries with innovative green and sustainable technologies are hopeful indi- cations for better and not too distant future. CONCLUSION AND OUTLOOK Based on the review conducted, the following major con- clusions are drawn: 4 Environmental Progress & Sustainable Energy DOI 10.1002/ep

* It became apparent that the exploit of adsorbents for bio- diesel purification by dry washing process offers greater benefit of considerably reducing wastewater, making the process eco-friendly. * As suggested by researchers, addition of higher amounts of biosorbent aids in greater removal of glycerine. However, the higher penalty in terms of biodiesel lost may be from this higher addition. * Additionally, the quantity and type of adsorbents did not notably impact the resultant acidity value, density, and vis- cosity showing no specific selectivity of the kind of adsor- bent employed. However, the adsorption time, temperature, and the shaking rate are the three most impor- tant parameters influencing the adsorptive purification process. * Despite various pros and cons that have been identified in the use of biosorbents in biodiesel purification, an exten- sive progress in this field can be anticipated in the not too distant future. LITERATURE CITED 1. Avinash, A., Subramaniam, D., & Murugesan, A. (2014). Bio-diesel—A global scenario, Renewable and Sustain- able Energy Reviews, 29, 517–527. https://doi.org/10. 1016/j.rser.2013.09.007. 2. Bhattarai, K., Stalick, W.M., Mckay, S., Geme, G., & Bhattarai, N. (2011). Biofuel: An alternative to fossil fuel for alleviating world energy and economic crises, Journal of Environmental Science and Health - Part A Toxic/Hazardous Substances and Environmental Engineering, 46, 1424–1442. https:// doi.org/10.1080/10934529.2011.607042. 3. Schnoor, J.L. (2006). Biofuels and the environment, Envi- ronmental Science & Technology, 40, 4042. https://doi. org/10.1021/es0627141. 4. Demirbas, A. (2007). Importance of biodiesel as transpor- tation fuel, Energy Policy, 35, 4661–4670. 5. Balat, M. (2009). Prospects for worldwide biodiesel mar- ket development, Energy Sources, Part B: Economics, Planning, and Policy, 4, 48–58. https://doi.org/10. 1080/15567240701423951. 6. Knothe, G. (2001). Historical perspectives on vegetable oil-based fuels, International News on Fats, Oils and Related Materials, 12, 1103. 7. Demirbas, A. (2010). Biodiesel for future transportation energy needs, Energy Sources, Part A: Recovery Utiliza- tion and Environmental Effects, 32, 1490–1508. https:// doi.org/10.1080/15567030903078335. 8. Purushothaman, K., & Nagarajan, G. (2009). Performance, emission and combustion characteristics of a compres- sion ignition engine operating on neat orange oil, Renew- able Energy, 34, 242–245. https://doi.org/10.1016/j. renene.2008.03.012. 9. Chang, D.Y.Z., Van Gerpen, J.H., Lee, I., Johnson, L.A., Hammond, E.G., & Marley, S.J. (1996). Fuel properties and emissions of soybean oil esters as diesel fuel, Journal of the American Oil Chemists' Society, 73, 1549–1555. https://doi.org/10.1007/BF02523523. 10. Romig, C., & Spataru, A. (1996). Emissions and engine performance from blends of soya and canola methyl esters with ARB 2 diesel in a DDC 6V92TA MUI engine. Bioresource Technology. 56 25–34. https://doi.org/10. 1016/0960-8524(95)00175-1. 11. Wang, W.G., Lyons, D.W., Clark, N.N., Gautam, M., & Norton, P.M. (2000). Emissions from nine heavy trucks fueled by diesel and biodiesel blend without

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Chemometric analysis of cow dung ash as an adsorbent for purifying

biodiesel from waste cooking oil A. Avinash 1 & A.

Murugesan 2

Taraditionally, the water-soluble contaminants of biodiesel are treated by water wash method.

However, water wash method ends up in an aqueous effuent, which might then cause a harmful environmental impact.

As a consequence, waterless purification of biodiesel has triggered primary interest in biodiesel manufacturing process.

To address this issue, an endeavour has been made in

this work to investigate the waterless purification of biodiesel from waste cooking oil using cow dung ash

at different concentration of 1, 2, 3

and 4

wt/wt %.

The optimum concentration of cow dung ash for biodiesel purification was found through chemometric analysis by comparing the Fourier

transform infrared transmission (FTIR) spectral characteristics of cow dung ash with the water treated FTIR. It was observed from the experimental study that 1 wt/wt % of cow dung ash exhibited similar structural characteristics

as that of traditional water treated method of biodiesel purification. Therefore, bio- waste cow dung ash is an effective adsorbent in purifying biodiesel analogous to traditional water washing technology.

In the past few decades, the rapid depletion of easily accessible underground carbon reserves

and detrimental effects on environmental pollution impose the use of alternative renewable energy sources 1, 2 . In this perspective, biodiesel is one among the alternative renewable energy sources which have been widely used in many countries of the world 3 . Biodiesel is a renewable and

biodegradable fuel for use in existing automobile and stationary engines 4 . It consists of mono-alkyl (methyl or ethyl) esters of long-chain fatty acids typically produced by chemically reacting lipids (

e.g., vegetable oil or animal fat) with an alcohol (methanol or ethanol) 5, 6 . Biodiesel is most commonly produced by transesterification of vegetable oil or animal fat feedstock through the different methods such as common batch process 7 , supercritical process 8 , ultrasonic irradiation 9 and microwave methods 10, 11 . Among these methods, transesterification by the batch process was noted as the most economic and simple

method to over- come problems

due to direct use of oil in diesel engines 12 .

Transesterification is the process of displacement of glycerol with simpler alcohol in the presence of a catalyst to

chemically break the molecule of fat into esters with glycerol as a by-product 13 . After glycerol removal by the separation method, the

trace impurities such as unreacted alcohol, unreacted glycerides and unreacted catalyst present in biodiesel are traditionally removed by water washing. Several studies have

reported

that multiple water washes are required to achieve satisfactory impurity removal from unpurified biodiesel. Some cases may require

as many as six individual washes 14 . Consequently,

water washing method pro- duces a large quantity of biodiesel treated wastewater that requires treatment before reuse and causes the

opera- tional

problem (

emulsion) 14 . Recently, several alternative “waterless”

purification methods have been developed, such as ion exchange resins and

adsorbent treatment 15 .

In the adsorbent method of biodiesel purification, commercial magnesium silicate (Magnesol) and silica were widely used 16, 17 . These adsorbents primarily consist of basic and acidic adsorption sites which can easily attract polar substances such as glycerol and unreacted methanol 17 . Kucek et al. 18 achieved a significant reduction in monoglyceride and bond glycerin by adding 2 wt/wt % of Magnesol in unpurified biodiesel by stirring the mixture continuously for 20 min at 65 °C and then the Magnesol was filtered from biodiesel. Predojevic et al. 19 assessed the use of silica gel to purify biodiesel and compared the results of silica gel purification to that of water 1

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washing method. In their study, it was found that there was a significant change in the acid value of biodiesel after purification by silica gel. However,

there was no significant change in density, kinematic viscosity, iodine number, water content, saponification number due to purification by silica gel.

Recent investigations by researchers por- tray that the use of silica- based agricultural waste products

have shown success in the removal of impurities from biodiesel

as reported by Márcia et al. 20 . In their work, the researchers have used rice husk ash as an adsorbent to purify waste frying oil biodiesel. On the whole, the adsorption process is one of the efficient methods to remove the water-soluble contam- inants present in biodiesel. However, there is a need to carry out further studies on biodiesel purification by adsorption using low-cost and eco-friendly adsorbents. In this regard,

cow dung has several imperative properties

which have been in use since ancient times. It is used as

manure for agricultural purpose and in the production of biogas. It is used to repel mosquitoes and as a low-cost thermal insulator. Cow dung is also a possible constituent in the manufacture of adobe mud brick housing. In addition, cow dung ash (

CDA)

is used as an adsorbent for sequestering heavy metals present in wastewater 21 . Thus, the advantage of utilising cow dung ash as an adsorbent is not only revolving around its low economic value but also can stop the environmental issue of foul odour ensuing from it.

Based on these considerations, the main objective of present work is to purify biodiesel produced from waste cooking oil using CDA as an adsorbent at different concentration of 1, 2, 3 and 4 wt/wt % and compare their FTIR structural characteristics with water treatment method. In this work, CDA was prepared by burning cow dung cakes in the muffe furnace (500 o C for 2 h) and adsorption studies were performed by the batch technique using CDA as an adsorbent. Also in this work qualitative analysis (principle component analysis and hierarchical cluster analysis) and quantitative analysis (partial least square regression) were done through chemometric analysis of FTIR spectral data to find the optimum concentration of cow dung ash which demonstrates similar structural characteristics as that of traditional water purification method. Results and Discussion

X-ray powder diffraction (XRD). The XRD analysis of the CDA was obtained with Copper (Cu) target at 40 kV, 30 mA under continuous scan mode with a scan range of 10–90 degree, and a scan speed of 10 degree/min- ute. The XRD pattern of CDA is shown in Fig. 1. This 2θ versus Intensity (I) (CPS - Counts per second) plot shows the profiles and peaks of compounds identified in XRD. Also, experimental XRD peaks

of

CDA at 500 o C were indexed with Joint Committee on Powder Diffraction Standards (JCPDS) file. Table 1 shows the comparison of planar spacing (d-spacing) and strongest peak intensities of CDA with standard compounds. The planar spacing values were calculated from Bragg’s law of diffraction (nλ = 2

d sinθ). It can be seen from Table 1

that silica (SiO 2) is a major phase in CDA followed by Al 2 O 3 , MgO, CaO and

Fe 2

O 3 .

Energy-dispersive X-ray spectroscopy (EDX). The percentage composition of various elements present in CDA was characterised by EDX. The EDX spectrum of CDA (Fig. 2) shows various elemental peaks with the major Si peak (Kα = 1.74 keV) and O peak (Kα = 0.525 keV). The percentage composition of various elements present in CDA is also shown in Fig. 2. The percentage of Si in the ash is higher with some metallic impurities in minor amounts.

FTIR characterisation of biodiesel treated wastewater.

In this work, pure distilled water before and after biodiesel treatment is characterised by FTIR to identify the transport of organic and inorganic impurities to pure distilled water after biodiesel purification. Figure 3(a) shows the FTIR spectrum of pure distilled water and the spectrum of biodiesel treated wastewater. Also, the functional groups present in pure and biodiesel treated wastewater are marked in Fig. 3(a). Pure distilled water showed a strong IR absorbance band at 3319.48 cm −1 (O-H stretching) and around 1636.63 cm −1 (H-O-H bending). Also, pure water exhibited combination band centred at 2117.78 cm −1.

On the other hand,

the strong band for biodiesel treated wastewater shifted to a higher wavelength, around 3331.57 cm −1 (O-H stretching) and around 1638.44 (H-O-H bending).

Also,

the combination band of bio- diesel treated wastewater shifted to 2120.26 cm −1 . This peak shift denotes the dilution and electron transfer effect of methyl ester in biodiesel to pure distilled water. This analysis discloses that

there is a need to go for

waterless purification of biodiesel to overcome the aqueous effuents released by water wash technique.

FTIR characterisation of water and CDA treated

biodiesel samples. The mid-infrared spectral data (4000–400 cm −1) have been used to identify functional groups and the bands

corresponding to vibration in the unpurified, water purified and ash treated biodiesel samples. Figure 3(b) shows peaks identified from the spectra of 6 biodiesel samples. The typical peak at 3397 cm −1 with stretching mode of vibration seen in the unpurified spectrum is attributed to the presence of alcohol (O-H) group. This peak is absent for the water purified and ash purified samples. This clearly indicates that the ash treatment of biodiesel removes the unreacted/impure alco- hol present in the unpurified biodiesel similar to water treatment process. The removal of impurities present in unpurified biodiesel was achieved by ash treatment

because of

the presence of high percentage of silica in cow dung ash. In this work, it was observed that FTIR spectrum of water treated and ash treated samples are

structur- ally similar and it is very difficult to visually discriminate the FTIR spectral data of the samples (Fig. 3(b)). Thus, the

chemometric analysis was carried out with the spectral data to classify the samples and to find the optimum weight percentage of cow dung ash

to be

used for biodiesel purification which exhibits similar structural charac- teristics as that of water purified biodiesel.

Characterisation of biodiesel. In this work, the methyl ester content of biodiesel was noted as 95.05 ± 0.26%. Indeed, the methyl ester content is not a very important parameter for determining the stage of purification because the purification process is no way associated with transesterification reaction and it does not have an effect on the methyl esters already formed. Thus, the biodiesel samples were characterised by acid value, saponification value,

www.nature.com/scientificreports/ 3 SCIENTIfIC REPOrTs | 7: 9526 | DOI:10.1038/ s41598-017-09881-z Figure 1. XRD pattern of cow dung ash.

Compound JCPDS hkl d-spacing (nm) Position (2θ) Relative intensity (%) Standard Experimental Standard Experimental Standard Experimental SiO 2 (46–1045) 101 0.33434 0.331583 26.639 26.8662 100 100 Al 2 O 3 (10–0173) 122 0.15140 0.15147 61.164 61.10 5 6 MgO (45–0946) 311 0.12698 0.126521 74.689 75.01 5 5 CaO (37–1497) 400 0.12025 0.121854 79.662 78.4178 6 7 Fe 2 O 3 (33–0664) 128 0.11896 0.119219 80.709 80.5 5 4

Table 1. Comparison of d-spacing, position and strongest peak intensities of CDA with standard compounds.

www.nature.com/scientificreports/ 4 SCIENTIfIC REPOrTs | 7: 9526 | DOI:10.1038/ s41598-017-09881-z methanol

content, water content, free and total glycerin.

The biodiesel characterisation results of unpurified, water purified and ash treated biodiesel samples are presented in Table 2.

It can be observed from Table 2 that 1 wt/wt % CDA on biodiesel exhibited results analogous to that of traditional water washing technology.

Chemometric analysis of spectral data. The chemometrics was constructed using preprocessing method- Savitzky-Golay (SG) filtering with second polynomial order and 11 smoothing points.

Among different preproc- essing methods, SG filtering was chosen because spectra in the dataset of all samples appear to have the

similar scatter level as shown in Fig. 4 and all spectra were recorded on the same day and on the same lab so there were no overlapping bands in the raw spectra.

Qualitative analysis. Before establishing quantitative model using partial least square regression (PLSR), unsupervised methods like

principal component analysis (PCA) and hierarchical cluster analysis (HCA) were used as classification tools for analysing FTIR spectra of unpurified, water treated and ash treated biodiesel samples. PCA is a well-known method

of dimension

reduction and data exploration 22 . To visualise the data trend of unpurified and purified biodiesel samples, a two-dimensional graph of samples using the first two principal com- ponents (PCs) was obtained, which is shown in Fig. 5(a). The

PC1 and PC2 explained 86% and 14% of variables, respectively. This means that in PCA the first two principal components could possibly explain 100% of all sample information.

Also from the score plot shown in Fig. 5(a) it can be seen

that unpurified biodiesel is an outlier in the lower right quadrant followed by close clustering of water treated and ash treated (1wt/wt %) samples in the upper right quadrant.

On the other hand,

ash treated biodiesel samples (2, 3, 4 wt/wt %) formed another cluster in the lower left quadrant of the score plot. Thus, PCA discriminates structurally similar unpurified and purified biodiesel samples from each other using the score plot visually.

HCA was performed using the Euclidean distance as the distance measure and complete linkage strategy

to link

clusters within the dataset. Figure 5(b) shows the dendrogram of the spectral data. It can be seen from the dendrogram that

the water purified and ash treated (1 wt/wt%) biodiesel formed one cluster and the ash treated (2, 3 and 4 wt/wt%) biodiesel samples formed another cluster. Overall, it is

clearly seen

from the PCA and HCA that the water purified and 1wt/wt% ash purified biodiesel sample exhibit similar structural characteristics.

Quantitative analysis.   Even though

PCA and HCA explore the relationships between the spectral data, these methods cannot be used for the quantitative analysis. For this purpose, PLSR method is employed to develop a model. The PLSR model was developed for acid value alone because it was found from the present work and available literature that

there was no significant change in

water content, saponification number, methanol and glycerin content due to purification by silica-based adsorbents. However, there was a significant change in acid value 19, 20 . In this context, the models were generated for the acid value of biodiesel and compared using PLSR from raw and SG preprocessed data. These models could be used

for the prediction of

the acid value of biodiesel. Between the raw and SG preprocessed model, preprocessing had the best performance with a high correlation

of

prediction (RP = 0.937) and low root mean square error of prediction (RMSEP = 0.010) as shown in Fig. 6(a,b).

Figure 2.

EDX spectrum of cow dung ash.

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Conclusion Based on the present investigation on the use

of cow dung ash as an adsorbent for purifying waste cooking oil,

the following major conclusions are drawn: •

In this study cow dung ash was successfully used

as an adsorbent for purifying biodiesel from waste cooking oil.

The test results clearly indicated that cow dung ash in a concentration of 1 wt/wt % showed an excellent result for removal of impurities from biodiesel similar to

that of water treatment. This result was possibly achieved in this work because of the

high silica content of the CDA as confirmed by XRD and EDX analysis, which also accounts for its high adsorptive capacity. • FTIR spectroscopy of biodiesel coupled with chemometrics was a helpful method to visually classify the highly correlated spectra and PLSR model developed from preprocessed spectra is considered

as a useful tool for rapid detection of the acid value of biodiesel.

Figure 3. (

a) FTIR spectra of pure water and biodiesel treated wastewater (b) FTIR spectra

of

biodiesel samples.

Purification method

Acid number (mg KOH/g) Saponification value (mg KOH/g) Methanol (%) Water (mg/kg) Free glycerin (%) Total glycerin (%)

Unpurified 0.30 ± 0.01 212 0.74 ± 0.02 2247.4 0.00782 ± 0.00001 0.59 ± 0.02 Water purified 0.21 ± 0.01 204 0.015 ± 0.01 5365.2 0.00035 ± 0.00001 0.49 ± 0.01 CDA treated 1 wt/wt % 0.22 ± 0.01 205 >0.01 1879.5 0.00393 ± 0.00002 0.50 ± 0.01 CDA treated 2 wt/wt % 0.27 ± 0.01 206 >0.01 1881.2 0.00451 ± 0.00001 0.48 ± 0.01 CDA treated 3 wt/wt % 0.27 ± 0.01 207 >0.01 1880.8 0.00412 ± 0.00001 0.47 ± 0.01 CDA treated 4 wt/wt % 0.29 ± 0.01 207 >0.01 1881.6 0.00442 ± 0.00001 0.48 ± 0.01

Table 2. Analysis of biodiesel samples.

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At last, it is suggested from

this work that biodiesel purification by cow dung ash has been shown to be envi- ronmentally friendly and a viable alternative substitute to water washing technology.

For these reasons, it is important to explore and exploit

other bio-waste silica-based adsorbents

to purify crude biodiesel. Materials and Methods Cow dung ash preparation. Cow dung was collected from a grazing field near KPR farms, Coimbatore, Tamil Nadu state, India. Cow dung was air-dried properly and grounded to powder. The grounded cow dung powder (100 g) was taken

in a silica cup and heated in the muffe furnace at 500 °C for 2 h. The resultant ash (CDA) was then allowed to cool to room temperature and stored in a desiccator to prevent it from absorbing moisture. In this work, the temperature of 500 °C

is chosen because it was noted from the available literature that the temperature at 500 °C will improve the adsorption property of cow dung ash 23 . The CDA was characterised by XRD followed by EDX.

Figure 4. Scatter plot of biodiesel

samples. Figure 5. (a) Score plot of PCA (b) Dendrogram.

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Production of biodiesel. The transesterification process of waste cooking oil was done with a base catalyst (NaOH) and methanol. The reaction was carried out at the stirrer speed of 500 rpm, the reaction time of 60 min with 9:1 molar ratio and 0.75% NaOH (wt/wt of oil) as a catalyst at fixed reaction temperature of 65 °C 24 . The transesterified oil was transferred to a conical flask for the gravity separation of the biodiesel and glycerol. The unpurified biodiesel was then taken for the purification process. Water purification. A sample of 100 g of biodiesel was transferred to a separating funnel and washed thrice with pure distilled water.

The washed biodiesel was heated at 90 °C to remove the water and stored for spectral analysis.

Cow dung ash purification. The biodiesel purification using CDA was performed in a batch mode under heating at 65 °C and stirring for 20–30 min 17 with different concentration of 1, 2, 3 and 4 wt/wt% of CDA. The concentration (wt/wt) percentage of CDA was calculated based on Equation (1). The purified biodiesel was fil- tered through a funnel with Whatman filter paper to remove the adsorbent and the samples were stored

for the spectral analysis. ? ? ? ? ? ? ? ? ? = × Concentration

wt wt mass of solute CDA mass of solution

biodiesel 100 % ( ) ( ) (1) Characterisation of biodiesel. Initially, the ester content of biodiesel was determined according to the method EN 14103. The acid value of biodiesel samples (unpurified and purified) was experimentally determined by titration procedure in accordance with EN 14104. In addition to acid value, saponification value (ASTM D 5558-95) of biodiesel samples was determined. The methanol content in biodiesel samples was determined according to method EN 14110

and water content by the Coulometric Karl Fischer method (EN 12937). Also, free glycerin and total glycerin in biodiesel samples were estimated by method EN 14105. All the procedures were done in duplicate and average values are presented.

Chemometrics. The

mid-infrared spectral data of the 6 biodiesel samples were used to perform the

chemo- metrics

using principal components analysis, hierarchical cluster analysis and partial least square regression using CAMO software (The Unscrambler X). The data from each sample contain 1667 variables; a total of 10002

varia- bles

were analysed. The analysis was based on a 6 × 1667 data matrix assembled so that

each

row corresponded to a sample and each column represented the spectral data at a given wavelength.

Figure 6. (

a) PLSR model for raw data (b) PLSR model for preprocessed data.

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Economic analysis of biodiesel production from waste cooking oil A. Avinash a and A. Murugesan b a

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ABSTRACT Renewabledieselfuel or biodiesel isone ofthe prospective growth inducers that could help to resolve a country’s economic problem and will decrease the nation’s dependence on foreign energy imports. In this context, an economic evaluation of biodiesel production from waste cooking oil is reported in this study. The biodiesel was produced from waste cooking oil by transesterification process. The by-product glycerol produced during transesterification is purified and recovered for its commercial value. Also, unreacted methanol after transes- terification is recovered by distillation and recycled for reuse. The biodiesel production facility developed in this study is dedicated to the production of 39,208 metric tons of biodiesel per year. The total capital investment charged to this project is estimated to be Rs. 1,615,133,000. At a value of Rs. 15/kg for feedstock waste cooking oil, a biodiesel cost of Rs. 55/kg was estimated. Along with mainrevenuefrombiodiesel, additionalrevenueofaroundRs. 22/kgcan be generated fromthe saleof glycerol. On thewhole, the profitability analysis ofthe study discloses that the modeled biodiesel production plant has the ability to produce biodiesel with unit production cost of Rs. 51/kg and unit production revenue of Rs. 58/kg. KEYWORDS Biodiesel; cost estimate; economic analysis; glycerol; waste cooking oil 1. Introduction In a developing country like India the import of fuels to meet the current energy demand triggers the need for biofuels to influence the indigenous sources of inputs, potentially increasing profits and opportunities in rural sectors (Talebian-Kiakalaieh et al., 2013). Based on these considerations, biodiesel is a suitable surrogate to diesel fuel. There are more than 350 oil bearing crops identified as potential alternative sources for biodiesel production (Balat, 2008). However, there are major obstacles in large-scale production of biodiesel including price competitiveness and production limitations. In addition, current production technologies force biodiesel prices to go higher than those of diesel fuel. As a means to lower prices, it is suggested to use waste cooking oil as a cost-effective source to produce biodiesel (Talebian-Kiakalaieh et al., 2013). The availability of waste cooking oil is from restaurants/hotels. Most of the waste cooking oils are collected from fried food which can produce as much as 15 L of waste cooking oil per day as reported by Abdullah et al. (2013). The high potential waste cooking oils can be collected from the hostel mess as reported by Burnwal et al. (2014). Biodiesel can be produced through different kinds of methods such as preheating, blending, micro-emulsification, pyrolysis, and transesterification (Hazar and Aydin, 2010.; Nainwal et al., 2015; Bora et al., 2016; Ito et al., 2012, and Bilgin et al., 2015).

Among these methods, transesterification process was noted as the most suitable and simple method to overcome problems due to direct use of oil in diesel engines (

Long and Fang, 2012).

Transesterification is the process of displacement of CONTACT A. Avinash avinashandromeda@gmail.com

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glycerol with simpler alcohol (methanol/ethanol)

in the presence of a catalyst to chemically break the molecule of

the fat/oil into esters with glycerol as a by-product (Chavan et al., 2013). Based on the above considerations, the main objective of this study is to perform an economic analysis of biodiesel from waste cooking oil by transesterification process with base catalyst (NaOH) and methanol. The economic analysis was done in this work with the SuperPro designer (Intelligen Inc., Scotch Plains, NJ, USA) The included SuperPro model is a slightly modified version of a process model developed by Haas et al. (2006). The optimum parameters (Molar ratio-9:1 and catalyst concentration 0.75 wt/wt of NaOH) for waste cooking oil biodiesel production were chosen based on the work carried out by Carlos et al. (2011). 2. Features of biodiesel production plant The model of the biodiesel plant is shown in Figure 1. Initially, waste cooking oil stored in tank is fed into transesterification reactor 1. Before entering the reactor, the oil is preheated to 60˚C to facilitate the reaction. The alcohol (methanol) and catalyst (NaOH mixed with methanol) stored in separate tanks are mixed in the mixer and enter the two-way flow splitter. The splitter splits 90% of mixture and is fed into transesterification reactor 1 and rest 10% is fed into transesterification reactor 2. In this plant, transesterificationis carried out in two stagesto increase the biodiesel conversion efficiency (~97%). In first stage, waste cooking oil is transesterified using 90% mixture of catalyst and alcohol. In the second stage, transesterified biodiesel with unreacted components from the first stage is again transesterified with 10% of catalyst–alcohol mixture. After transesterification process, the by-product glycerol and unreacted methanolare separated and recovered bydistillationprocess.Therecoveredmethanol is recycled back to the mixing vessel. Crude biodiesel is acid-washed (HCl) and purified by vacuum dryer system. 3. Bulk raw material required for entire process The annual operating days per year of the modeled plant = 330 and thus the annual operating time = 7,920 h. This plant has the capacity to produce approximately 39,208 metric tons of biodiesel per year and 3,128 metric tons of glycerol per year. The quantities of each raw material required to produce 39,208 metric tons of biodiesel per year are listed in Table 1, which shows the material requirements in both kg/year and kg/h. 4. Economic analysis of biodiesel production The economic evaluation report of the biodiesel production plant dedicated to production of 39,208 metric tons of biodiesel per year is shown in Table 2. Capital investment charged for such a plant is around Rs. 1,615,133,000. One-third (Rs. 538,378,000) of this capital cost was for actual equipment purchase, and two-thirds (Rs. 1,076,755,000) were based on the assumption of a construction/installation cost roughly double the equipment costs. The projected annual operating cost of modeled biodiesel production facility is shown in Table 3 and the percentage breakdown of the annual operating cost is depicted in Figure 2. FromTable 3 it is seen that annual operating cost of the biodiesel plant is Rs. 2,075,333,000 and this figure translates to a unit biodiesel production cost of around Rs. 51/kg. From Figure 2 it is seen that

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raw materials costs (80%) constitute the greatest component of overall production costs

followed by facility-dependent (11%), utilities (7%), and at last labor cost (2%). This analysis assumes that a new facility will be built for this process, and the project lifetime is 15 years. The main revenue is based on the selling price of biodiesel at Rs. 55.8/kg (Rs. 2,261,222,601/year). Additional revenue of Rs. 22.5/kg (Rs.104, 373,027/year) is generated from the sale of glycerol. Thus, the total revenue of this plant is Rs. 2,365,586,252/year. Overall, the gross profit from the facility is Rs. 290,253,000 /year. ENERGY SOURCES, PART B: ECONOMICS, PLANNING, AND POLICY 891 Downloaded by [Mr alagumalai avinash] at 21:39 11 December 2017

Figure 1. Model of biodiesel production plant. 892 A. AVINASH AND A. MURUGESAN Downloaded by [Mr alagumalai avinash] at 21:39 11 December 2017

5. Conclusion The main objective of this study is to model a biodiesel plant to produce methyl esters from waste cooking oil and to recover the co-products (glycerol and unreacted methanol). The economic analysis disclosed that it is possible to produce biodiesel from the modeled plant more profitably with unit production cost of Rs. 51/kg and unit production revenue of Rs. 58/kg. Based on this study, the following recommendations are drawn. Table 1. Bulk materials (entire process). Material kg/year kg/h HCl 83,762 10,576 Methanol 10,952,330 1,382,870 Sodium hydroxide 266,508 33,650 Waste cooking oil 33,634,775 4,246,815 Water 1,471,253 185,764 Table 2. Executive summary. Total capital investment Rs. 1,615,133,000 Capital investment charged to this project Rs. 1,615,133,000 Operating cost Rs. 2,075,333,000/year Main revenue Rs. 2,261,222,000/year Other revenues Rs. 104,373,027/year Total revenues Rs. 2,365,586,000/year Cost basis annual rate 40,499,171 kg MP/year Unit production cost Rs.

51.24/kg MP Net unit production cost Rs. 51.24/kg MP Unit production revenue Rs. 58.41/kg MP MP: main product – biodiesel. Table 3. Annual operating cost. Cost item Rs. Raw materials 1,665,650,000 Labor-dependent 33,405,000 Facility-dependent 219,820,000 Utilities 153,054,000 Miscellaneous 3,404,000 TOTAL 2,075,333,000 Figure 2. Percentage breakdown of annual operating cost. ENERGY SOURCES, PART B: ECONOMICS, PLANNING, AND POLICY 893 Downloaded by [Mr alagumalai avinash] at 21:39 11 December 2017

The installation of large-scale biodiesel production facility could have positive impacts and will generate new jobs for the poorest communities. Although biodiesel may be a good energy alternative opportunity in the future, there are lots of barriers that keep the biodiesel industry from taking off the ground in the present. The first and foremost of all is the level of subsidies provided by the government. Finally, the country's national policy should call for the creation of a biodiesel naviga- tion committee, which will lead this project with significant political clout to bring energy indepen- dence to the nation. References Talebian-Kiakalaieh, A., Amin,

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Keywords: Waste cooking oil Transesterification Biodiesel Response surface methodology Artificial neural network

ABSTRACT

The present work describes the comparison of biodiesel yield prediction by Response Surface Methodology (RSM) and Artificial Neural Network (ANN). The prediction models were developed based on three-level design of experiments conducted with waste cooking oil transesterified by varying four process parameters such as catalyst concentration, molar ratio, reaction time, and stirrer speed. The optimum reaction conditions were found to be 0.75% wt/wt catalyst concentration, 9:1 M ratio, 60 min reaction time and 500 rpm stirrer speed. For these optimum conditions, experimental fatty acid methyl ester (FAME) content of 95.05 ± 0.26% was ob- tained, which was in good agreement with the predicted yield. The RSM model was developed using Box- Behnken design and the ANN predictive model was developed using a feed-forward backpropagation neural network algorithm with 14 neurons in the hidden layer. The mathematical models of RSM and developed ANN were compared for biodiesel yield. The higher value of correlation coefficient (R 2 = 0.99) and lower value of root mean square error (RMSE = 1.97) for ANN compared to RSM (R 2 = 0.95 and RMSE = 2.71) evidently proved that ANN model is far better in predicting FAME content compared to the RSM model.

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avinashandromeda@gmail.com (A. Avinash). Fuel 216 (2018) 322–329 0016-2361/ © 2017 Elsevier Ltd. All rights reserved. T

1. Introduction Undoubtedly, depletion of easily accessible fossil fuels is currently one of the most challenging topics, which is gaining strict considera- tions during the past few decades. For this reason, biomass derived biofuels has emerged to be a priceless source primarily in the third- world economies [1]. Over the past several years, a number of research studies have been drastically addressed for the conversion of biomass into the high value-added products. In this regard, biodiesel is an in- valuable product which is renewable and biodegradable. [2]. Generally, biodiesel is described as monoalkyl esters of long chain fatty acids that are produced by chemically reacting lipids [3]. Al- though there are several noteworthy advantages of biodiesel, the manufacturers are still facing various challenges in producing high- quality biodiesel with consistent characteristics, regardless of feedstock being used [4]. Over the years, a number of studies and researches have reported that base-catalyzed transesterification can produce high quality biodiesel in a short span of time. Moreover, base-catalyzed transesterification is the method by which most of biodiesel produced today is made. As reported by Wang et al. [5], base-catalyzed transes- terification requires low temperature and pressure to produce high conversion yield with no intermittent compounds [5]. Previous re- search studies also validate that base-catalyzed transesterification can account for high conversion within a short span, usually ranging be- tween 30 and 60 min [6–7]. Among different feedstocks available for biodiesel production, bio- diesel production from highly polluting waste is of recent interest [8]. Waste cooking oil is one such waste that prohibits human improvement, and thus the social welfare [9]. Almost every nation in the world consumes million tons of edible cooking oil every year, out of which 20% is considered as waste, which could be possibly considered as a raw material for biodiesel production to cut down the petroleum im- ports [10]. Several research studies have reported that waste cooking oil is a promising feedstock for biodiesel production despite its dis- advantages such as high free fatty acid and water contents. Moreover, a comprehensive review of the pre-treatment and the usage of waste cooking oil for the production of biodiesel using several techniques, different types of reactors, and various types and amounts of alcohol and catalysts have been reported in literature [11]. In the past decades, several mathematical tools have been devel- oped for modeling and optimization. RSM and ANN are such tools suitable for modeling and simulation of real-world problems [12]. As reported by Jeong and Park [13], RSM can be used to optimize the process parameters of biodiesel production. This analysis by which the researchers were able to generate a multi level and multi factor RSM design and results disclosed that the generated model can predict the experimental range adequately. In another study carried out by Ferella et al. [14], RSM method established a good agreement between ex- perimental and predicted yield of biodiesel which suggests that RSM is a useful tool to optimize the biodiesel yield. ANN is a machine learning system to reproduce the neurological processing capability of the human brain [15]. Yuste and his coauthors [15] used ANN model to accurately simulate the production of biodiesel from Olive oil. The results depicted that ANN is a powerful tool to predict the experimental data. The similar observation was made by Ying et al. [16] in predicting biodiesel yield from Rapeseed soapstock. Based on the above considerations, RSM and ANN have been widely used for modeling and optimization of biodiesel, but the predictive capabilities of both RSM and ANN have been

rarely compared. Therefore, in the present work, RSM and ANN predictive models were developed and compared for the methanolysis reaction of waste cooking oil catalyzed by sodium hydroxide. 2. Materials and methods 2.1. Materials and chemicals Waste cooking oil was collected from the college hostel mess and filtered using a muslin cloth to remove suspended solid impurities. After filtration, the oil is water degummed to remove trace impurities like protein, phosphatides, colorants, etc [17]. The procedure of de- gumming employed in this work is same as the method described by Araújo et al. [18]. After degumming, there was a notable reduction in acid value from 1.34 mg KOH/g to 1.21 mg KOH/g. All other necessary materials like anhydrous methanol, anhydrous sodium sulfate, and so- dium hydroxide pellets were purchased from Merck Limited, India. 2.2. Biodiesel production and process conditions The transesterification reaction was carried out in a batch type re- actor with a base catalyst (NaOH) and methanol. The reactor was equipped with a stirrer and thermometer. The glass reactor vessel was maintained at fixed reaction temperature of 65 ˚C. The molar ratio of alcohol to oil was varied within the range of 3:1–9:1 with an interval of 3:1. The catalyst concentration was varied in the range of 0.75–1.25% (wt/ wt) by an interval of 0.25%. The reaction time was varied within the range of 30 min to 90 min with an interval of 30 min and the stirrer speed was varied in the range of 300 rpm to 700 rpm with an interval of 200 rpm. The chosen range of process parameters is based on the suggestion made by Carlos et al. [19]. The transesterification reaction was carried out in an open system under barometric pressure. The required concentration of catalyst (NaOH) was initially dispersed in the oil under desired stirring speed and heated to reach the reaction temperature of 65 ˚C. Then, the re- quired amount of pre-heated methanol was added to the mixture and the reaction was started. The reaction is carried out until the desired time and then the transesterified oil was transferred to a gravity se- paration funnel to separate crude or unpurified biodiesel and glycerol. The crude biodiesel was subjected to water wash method and finally, the refined biodiesel layer is collected for characterization and analysis of fuel properties. 2.3. Fuel properties and characterization The critical fuel properties like density, viscosity, calorific value, cetane number, flash and fire point were determined experimentally by standard procedures. The acid value and saponification value of bio- diesel was experimentally determined by titration procedure in ac- cordance with EN 14104 and ASTM D 5558-95. The methanol content in biodiesel was determined in accordance with EN 14110 and water content by the Coulometric Karl Fischer method (EN 12937). Furthermore, free glycerin and total glycerin content in the test sample were estimated by method EN 14105. All the tests were done in du- plicate and average values are presented. The chemical composition of produced biodiesel was investigated by Fourier transform infrared transmission (FTIR) spectrum. The in- frared spectrum of biodiesel was recorded on FT-IR-4100 type 'A' Spectrometer in the region of 4000–400 cm -1 . In this work, FTIR spectrum is used as a qualitative measurement to analyze the chemical structure of biodiesel. 2.4. Design of experiments 2.4.1. Response surface methodology Since this work describes three-levels and four factors, Box-Behnken Design (BBD) is used for regression and graphical analysis of experi- mental data. In fact, BBD is a three-level experimental design for RSM in which each selection parameter is studied at three-levels with equally spaced interval [20]. The Minitab software (version 16.1.1) was used to A. Avinash, A. Murugesan Fuel 216 (2018) 322–329 323 design the experiments and to perform the regression analysis. The coded and actual values of variables used in BBD design is given in Table 1. The experimental data from BBD was analyzed using general quadratic model expressed by Eq. (1). Σ Σ Σ Σ = + + + + = = = = + Y β β x β x β x x ε i k i i i k ii i i j i ij i j 0 1 1 2 1 1 (1) where, Y is the response (FAME %), β 0 , β i , β ii and β ij are regression coefficients for constant, linear, quadratic and interaction effects, respectively. whereas x i and x j are independent factors variables and ε is the error. The experimental data presented in this work is done in duplicate and average values are presented for the sake of better accuracy. 2.4.2. Artificial neural network model In this work, the input and target data used for training in neural network model was normalized in the limits −1 to +1. The data have been normalized in order to match the tangent sigmoid function (tansig) employed for ANN modeling which also ranges from −1 and +1. The data is normalized using the Eq. (2). = ⎡ ⎣ ⎢ − − ⎤ ⎦ ⎥ X X X X Normalized data (2 2) ( ) Ac min max min (2) where X Ac , X min , and X max are the actual, minimum, maximum data, respectively. A feed-forward backpropagation neural network algorithm with two layers was used. A neural network of two transfer function, where the first transfer function was tansig and the second transfer function was linear transfer function (purelin) was used. For this study, neural net- work tool box of MATLAB software version 7.10.0. 499 (R 2010a) was Table 1 Coded and actual values of BBD design. Coded variable levels Variables 1 0 -1 1.25 1 0.75 Catalyst% (wt/wt) 9:1 6:1 3:1 Molar ratio 90 60 30 Reaction time (min) 700 500 300 Stirrer speed (rpm) Table 2 BBD matrix of four independent variables along with experimental and predicted response. Run A B C D Yield (%) Residuals Catalyst% (wt/wt) Molar ratio Reaction time (min) Stirrer speed (rpm) Experiment RSM ANN RSM ANN 1 1 3:1 60 300 72.18 71.29 72.50 0.89 −0.32 2 1 6:1 90 700 84.32 82.30 84.09 2.02 0.23 3 1 6:1 60 500 90.98 89.81 88.79 1.17 2.19 4 1 3:1 30 300 59.42 62.86 59.44 −3.44 −0.02 5 1 9:1 30 500 91.72 90.63 91.72 1.09 0.00 6 1 6:1 90 300 83.70 87.71 83.74 −4.01 −0.04 7 1.25 3:1 60 500 55.02 52.61 56.49 2.41 −1.47 8 0.75 6:1 60 300 93.46 90.99 93.50 2.47 −0.04 9 1.25 6:1 60 300 86.99 83.27 87.00 3.72 −0.01 10 0.75 6:1 60 700 92.14 92.17 94.40 −0.03 −2.26 11 0.75 6:1 90 500 76.62 76.54 76.70 0.08 −0.08 12 0.75 6:1 30 500 87.39 82.47 94.35 4.92 −6.96 13 0.75 3:1 60 500 62.25 64.77 62.31 −2.52 −0.06 14 1 3:1 60 300 74.25 71.29 72.50 2.96 1.75 15 1.25 6:1 90 500 58.23 63.05 60.62 −4.82 −2.39 16 1 3:1 90 500 65.72 64.62 65.79 1.10 −0.07 17 1 6:1 60 500 88.80 89.81 88.79 −1.01 0.01 18 1 6:1 60 500 89.20 89.81 88.79 −0.61 0.41 19 1 9:1 90 500 92.60 86.97 92.58 5.63 0.02 20 1 6:1 30 300 90.94 92.82 92.11 −1.88 −1.17 21 1.25 6:1 60 500 65.26 67.80 65.27 −2.54 −0.01 22 1 3:1 60 700 75.12 76.51 79.76 −1.39 −4.64 23 1.25 6:1 60 700 60.32 59.11 63.88 1.21 −3.56 24 1 6:1 30 300 92.12 92.82 92.11 −0.70 0.01 25 1.25 6:1 30 500 55.20 55.18 55.20 0.02 0.00 26 1 9:1 60 700 84.98 86.78 84.94 −1.80 0.04 27 0.75 9:1 60 500 95.05 99.97 95.02 −4.92 0.03 Table 3 Analysis of variance for biodiesel yield. Source Degrees of freedom (DF) Sum of square (SS) Mean square F-value Model 11 4701.15 335.80 20.01 Linear 4 3023.66 808.44 48.19 Square 4 1134.33 312.40 18.62 Interaction 3 543.16 90.53 5.40 Residual error 15 201.33 16.78 Lack of fit 11 195.80 24.47 17.70 Pure error 4 5.53 1.38 Total 26 4902.48 Fig. 1. Normal probability plot of response yield. A. Avinash, A. Murugesan Fuel 216 (2018) 322–329 324

used to predict the biodiesel yield. 3. Results and discussion 3.1. RSM based predictive model The experiments were conducted employing RSM and the results are given in Table 2. A second order polynomial regression equation was developed using coded units. The final developed model based on coded units for biodiesel yield is represented by Eq. (3). = − + + −

− − − + − + − − − + Yield A B C D A B C D AB AC AD BC BD CD (%) 90.27 10.27 13.55 0.42 5.96 11.85 6.22 8.69 3.43 4 3.45 6.33 2.33 8.56 2.99 2 2 2 2 (3) where, A – Catalyst% (wt/wt), B – Molar ratio, C – Reaction time (min), D – Stirrer speed (rpm) The significance of each coefficient in Eq. (3) is determined by analysis of variance (ANOVA). The regression model was significant at 95% confidence level. The value of correlation coefficient (R 2 = 0.9589) was found to be reasonable which clearly indicated that 95.89% of the effect on the biodiesel yield was explained by the var- iation in the process variables. ANOVA results are shown in Table 3. The analysis indicated that the developed model is significant, which is confirmed by its P-value. The standard deviation value of the developed model was found to be 4.07. Overall, the analysis revealed that there was a good agreement between the experimental data and predicted data of RSM as shown in normal probability plot (Fig. 1). From Fig. 1 it can be clearly seen that all points are located close to the straight line representing that RSM predicts well the experimental data for the considered valid region. 3.2. Effect of process parameters The three-dimensional response surface plots of process parameters versus yield are shown in Fig. 2a–c. Fig. 2a shows the influence of catalyst and reaction time on biodiesel yield for the fixed molar ratio of Fig. 2. Response surface plots showing the relative effect of process variables on Yield (%) (a) effect of catalyst and reaction time; (b) effect of catalyst and molar ratio; (c) effect of catalyst and reaction speed. A. Avinash, A. Murugesan Fuel 216 (2018) 322–329 325

6:1 and stirring speed of 500 rpm. Fig. 2a clearly shows that there is a decrease in biodiesel yield when the concentration of catalyst is in- creased beyond the optimum value. This trend clearly indicates that with increase in catalyst concentration beyond the optimum quantity more triglycerides take part in the saponification reaction and thereby reducing the FAME content. As reported by Ramadhas et al. [21] and Meher et al. [22] addition of excess amount of catalyst will lead to the formation of gels. The similar observation was noted in the present work. Fig. 2a also shows the variation of biodiesel yield with respect to reaction time. Reaction time is an important process parameter in biodiesel production which decides the economy of transesterification process [23]. It is observed from Fig. 2a that the yield is less at the beginning and reaches maximum at the reaction time of 60 min. The reaction time of the biodiesel yield after 60 min decreased. This result indicates that if the reaction time is not correctly set, there are some possibilities for the backward reaction, which will reduce the product yield. Moreover, when the reaction is carried out for a longer time, specific gravity of biodiesel decreases in an exponential manner with respect to time. The similar trend was observed by Leung and Guo [24]. Fig. 2b shows the influence of catalyst and molar ratio on biodiesel yield for the fixed reaction time of 60 min and stirring speed of 500 rpm. Although methanol to oil molar ratio of 3:1 is required to produce biodiesel stoichiometrically, an excess amount of alcohol will always be required to drive the reaction faster based on Le Chatelier's principle [25]. Fig. 2b shows that the product yield increases with in- crease in molar ratio. The maximum yield was noted at 9:1 M ratio. This shows that for molar ratios less than 9:1, the reaction was incomplete. The similar trend was observed in the work conducted by Carlos et al. [19]. Fig.2c shows the influence of catalyst and stirrer speed on biodiesel Fig. 3. Comparison between the experimental values and yield predicted by ANN. Fig. 4. Box plot of experimental and predictive models. A. Avinash, A. Murugesan Fuel 216 (2018) 322–329 326 yield. In transesterification reaction, stirring is necessary to mix the oil, sodium hydroxide and methanol. Once these materials are mixed, the reaction starts. In this work, transesterification reaction was conducted at different stirrer rates such as 350, 500 and 700 rpm. The variation of yield with different stirrer rates is shown in the Fig. 2c. It was observed from the experiment that the reaction was incomplete at 350 rpm and rate of mixing was insignificant for methanolysis. On the other hand, beyond certain speed of stirrer, there was insignificant rise in the pro- duct yield. Hence, the stirrer speed of 500 rpm is sufficient for pro- duction of maximum biodiesel yield. 3.3. ANN based predictive model ANN is one of the most effective predictive models for optimization when compared to linear regression models [26]. In this work to de- velop ANN model, the experimental data was randomly partitioned into three subsets, namely training, validation and testing. Firstly, 70% of experimental data was randomly chosen for training stage, 15% of data for validation and remaining 15% for testing. Moreover, the weighted parameters of the connections were explained until minimum value of mean square error (MSE) was achieved between experimental and predicted yield. The training stopped with MSE of 0.0002 at 13 epoch, which is close to the acceptable limit for MSE to 0.001. Levenberg-Marquardt backpropagation algorithm can accomplish higher R 2 value with lower standard deviation and MSE compared to any other algorithm [27]. Therefore, in this work Levenberg-Marquardt feed-forward backpropagation algorithm with 14 neurons in hidden layer is employed for modeling of biodiesel yield. The optimum net- work topology of ANN is 4:14:1, which implies that input layer with four input variables, hidden layer with fourteen hidden neurons, and output layer with single output variable. Two transfer functions, where the first transfer function is tansig and the second transfer function is purelin is used to model the neural network. In ANN model, the interpretation of R 2 value of experimental and predicted response is one indicator to test the validity of the model. Fig. 3 shows the experimental versus predicted yield of developed ANN model. The R 2 values for training, validation, testing, and all obtained using ANN model were 0.9999, 0.9635, 0.9756, and 0.9851, respec- tively. The R 2 values of ANN model imply that there is good agreement between experimental response and ANN predicted response. Thus, the developed ANN model can precisely predict biodiesel yield owing to its greater flexibility and potential to develop non-linear relationships. 3.4. Comparison between RSM and ANN predictive models The developed predictive models (ANN and RSM) were compared to test their capability of predicting biodiesel yield. The models were compared based on two important parameters namely correlation coefficient and root mean square error.

The higher value of correlation coefficient (R 2 = 0.99) and lower value of root mean square error (RMSE = 1.97) for ANN compared to RSM (R 2 = 0.95 and RMSE = 2.71) clearly depicted

that ANN model is more accurate in predicting

yield as compared to the RSM model.

In addition, the Table 4 Complete prediction of response (yield) for input variables and levels. Catalyst% (wt/wt) Molar ratio Reaction time (min) Stirrer speed (rpm) Predicted Yield (%) 1.25 3:1 90 500 55.53 0.75 6:1 60 500 94.56 1.25 6:1 30 500 55.20 0.75 9:1 60 700 94.94 1.25 9:1 90 700 82.32 1 9:1 90 700 92.31 1 6:1 60 500 88.79 0.75 3:1 90 300 61.74 0.75 6:1 30 500 94.35 1

6:1 60 300 79.98 1 9:1 30 700 85.22 1.25 6:1 90 300 68.70 0.75 3:1 60 300 76.08 0.75 6:1 30 700 84.49 1 6:1 60 700 91.33 1.25 3:1 90 300 68.33 0.75 3:1 30 500 89.67 1.25 6:1 90 700 74.04 1 3:1 60 500 73.17 0.75 3:1 60 700 77.20 1.25 3:1 60 500 56.49 1 6:1 30 700 56.18 1 9:1 30 500 91.72 1 9:1 90 500 92.58 0.75 6:1 90 500 76.70 1 6:1 90 700 84.09 1.25 9:1 60 700 78.14 0.75 9:1 90 500 88.49 1 9:1 60 500 89.23 1 6:1 30 300 92.11 0.75 9:1 90 700 83.63 0.75 9:1 60 300 95.03 0.75 9:1 30 700 92.01 1.25 6:1 90 500 60.62 0.75 3:1 30 300 94.37 0.75 6:1 60 700 94.40 1 3:1 90 700 62.34 1 3:1 30 700 55.05 1.25 3:1 60 700 55.53 1.25 3:1 90 700 57.26 1.25 6:1 60 500 65.27 0.75 6:1 60 300 93.50 0.75 3:1 60 500 62.31 0.75 9:1 30 300 95.00 1 3:1 30 300 59.44 1.25 9:1 60 300 88.00 1.25 9:1 30 700 55.64 0.75 3:1 90 700 75.20 1 6:1 90 300 83.74 0.75 6:1 90 300 81.83 1 3:1 60 700 79.76 1 6:1 90 500 87.19 0.75 3:1 30 700 57.48 1.25 9:1 30 300 93.93 0.75 6:1 30 300 94.80 1 3:1 90 300 75.04 1.25 6:1 60 300 87.00 1.25 3:1 30 500 55.08 1.25 3:1 30 700 55.07 1.25 9:1 90 500 88.22 1.25 6:1 30 700 55.23 1.25 9:1 60 500 87.29 1 3:1 30 500 55.11 0.75 9:1 30 500 94.81 1.25 9:1 90 300 84.40 1.25 3:1 30 300 68.60 1.25 3:1 60 300 80.32 1.25 6:1 30 300 88.03 1 3:1 60 300 72.50 0.75 6:1 90 700 71.76 0.75 9:1 90 300 94.18 0.75 9:1 60 500 95.02 1 9:1 30 300 94.65 Table 4 (continued) Catalyst% (wt/wt) Molar ratio Reaction time (min) Stirrer speed (rpm) Predicted Yield (%) 1 9:1 60 700 84.94 0.75 3:1 90 500 67.11 1 6:1 30 500 60.71 1.25 6:1 60 700 63.88 1 9:1 60 300 87.74 1 9:1 90 300 93.42 1.25 9:1 30 500 63.98 1 3:1 90 500 65.79 A. Avinash, A. Murugesan Fuel 216 (2018) 322–329 327

experimental response data is compared with the predicted response data with a box plot shown in Fig. 4. From the Fig. 4, it is apparent that the trained neural network model has competent approximated ex- perimental values. Since ANN performed superior to RSM, full level prediction of response (3 4 = 81 runs) is done using ANN and the si- mulated result is provided in Table 4. 3.4.1 Multivariate analysis 3.4.1.1. Hierarchical cluster analysis (HCA). In this work, the response dataset (yield in %) of the experiment, RSM and ANN models were used to perform the HCA with the aid of Minitab software. HCA is an unsupervised method used to cluster the data [28]. HCA was performed using the Euclidean distance measure with single linkage method and standardized variables to link clusters within the dataset. Fig. 5 shows the dendrogram of the response data. It can be clearly seen from the dendrogram that experimental response data and ANN predicted data formed close clustering with similarity level of 47.88 or distance level of 4.54. On the other hand, RSM predicated data was found to have similarity level of 7.34 or distance level of 8.08. Overall, it is clearly seen from HCA that the experimental and ANN predicted response data exhibit a similar trend. 3.5. Fuel properties and characterization The produced biodiesel was tested and characterized to be used as an alternative fuel in compression ignition engine. After biodiesel purification, some of its critical fuel properties were measured and listed in Table 5. The density and viscosity of produced biodiesel was slightly higher than diesel owing to the larger molar mass and presence of oxygen in the structure of biodiesel. The higher flash point and fire point values of biodiesel offer safety during transportation, handling and storage. The high cetane number of biodiesel indicates better ig- nition quality of the fuel.

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The calorific value of biodiesel was around 4% lower than diesel due to the presence of oxygen in biodiesel.

As re- ported by Garis and Ohadi [29], the presence of electronegative ele- ment oxygen lowers the calorific value of biodiesel and makes biodiesel more polar. From Table 5 it can also be seen that acid number, sapo- nification value, free glycerin, total glycerin, methanol and water contents illustrate high degree of completion of transesterification re- action and thus the high quality of produced biodiesel. Furthermore, the mid-infrared spectral data (4000–400 cm −1) have been used to identify the functional groups and the bands Fig. 5. Dendrogram of experimental and predictive models. Table 5 Comparison of fuel properties. Properties Test method Diesel Biodiesel Viscosity at 40 ˚C (Cst) ASTMD445 3.2 4.9 Density at 15 ˚C (g/cm 3) ASTM D1298 0.84 0.88 Flash point (˚C) ASTM D93 58 175 Fire point (˚C) 71 189 Calorific value (MJ/kg) ASTM D2015 42.5 40.96 Cetane number ASTMD613 47 50 Acid number (mg KOH/g) EN 14104 – 0.21 ± 0.01 Saponification value (mg KOH/g) ASTM D 5558–95 – 204 Methanol (%) EN 14110 – 0.015 ± 0.01 Water (mg/kg) EN 12937 – 5365.2 Free glycerin (%) EN 14105 – 0.00035 ± 0.00001 Total glycerin (%) – 0.49 ± 0.01 Fig. 6. FTIR spectrum of biodiesel. A. Avinash, A. Murugesan Fuel 216 (2018) 322–329 328

corresponding to vibrations in produced biodiesel. The FTIR spectrum of biodiesel is shown in Fig. 6 and the identified functional groups are listed in Table 6. The low energy frequency region from 722 cm −1 to 851 cm −1 in- dicates the presence of]CeH functional groups with bending type of vibration. The band region of 1017 cm −1 –1244 cm −1 indicate the present of CeO alkoxy esters, ethers and CeOeC groups with the stretching mode of vibration. The characteristic peaks present in the region 1361 cm −1 –1462 cm −1 are attributed to the bending mode of vibration of CeH methyl groups present in the fuel. The ester functional group of biodiesel is observed at the infrared absorption band of 1741 cm −1 and the peaks at 2853 cm −1 and 2922 cm −1 and 3005 cm −1 indicate symmetric and asymmetric stretching vibrations of]CeH (alkenes), CeH (alkanes) and CeH (methylene), respectively. The similar trend was observed in the study conducted by Gunstone [30], Shuit et al. [31] and Saifuddin and Refai. [32]. 4. Conclusion The production of biodiesel by base-catalyzed transesterification of waste cooking oil is reported in this work. The following observations were made from the experimental results: • RSM was successfully applied for modeling and optimizing the ex- periments. The developed regression model based on coded units described that ~96% of the effect on the biodiesel yield can be well explained by the variation in the process variables. On the other hand, the developed ANN predictive model (4:14:1) demonstrated greater flexibility and potential to develop non-linear relationships. • The comparative analysis of prediction capabilities

of RSM and ANN disclosed that the correlation coefficient (R 2 = 0.99) and root mean square error (RMSE = 1.97) for ANN

is better in precise predicting of experimental yield as compared to the RSM (R 2 = 0.95 and RMSE = 2.71). This trend was also visually demonstrated by box plot and HCA. • The analysis on fuel properties revealed that the produced biodiesel is comparable to that of diesel. Also, biodiesel characterization by FTIR exposed the production of high-quality fuel. Overall, further investigation of this work is necessary to analyze the economics and storage stability of biodiesel and also testing of produced fuel in the engine. References [1] Agarwal AK. Bio fuels (Alcohols and bio diesel) applications as fuels for internal combustion engines. Prog Energy

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Understanding the interaction among the barriers of biodiesel production from waste cooking oil in India- an interpretive structural modeling approach

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abstract

Regardless of fragile biodiesel market of the present day, it is foreseen that biodiesel will draw a lot of public interest throughout the world in the not too distant future. Among different feed stocks available for biodiesel production, waste cooking oil is under a major prospective for large-scale biodiesel pro- duction as it can cut down the fuel costs than other alternative feed stocks. However, there are several barriers that hinder large-scale biodiesel production from waste cooking oil. Also, there might be contextual relationships among those barriers. In this perspective, this paper aspires to identify the most influential barrier and to describe the interactions among different barriers influencing biodiesel pro- duction from waste cooking oil. For this reason, an interpretive structural modeling approach is employed to determine relationships among barriers. MICMAC analysis has additionally been carried out to classify the barriers based on dependence and driving power. The results indicate that vehicle access problem, lack of processing technology, inconsistent supply quantity and inadequate production facilities are the top-level barriers. Also, the results show that lack of political will, lack of incentives and lack of policy implementation play a very important role in the effective implementation of biodiesel production from waste cooking oil. ? 2018

Elsevier Ltd. All rights reserved. 1. Introduction Biodiesel - a realistic surrogate to diesel fuel has gained signif- icant attention in recent years due to the brink of depletion of easily accessible fossil fuels and environmental catastrophes [1e5]. Because of widespread concern over the water-land-food nexus of first generation biofuels, a waste-to-energy system utilizing waste cooking oil has become very attractive in recent days [6,7]. Waste cooking oil can be recycled into useful industrial products such as biodiesel, soap, etc., [8]. In India, a large quantity of waste cooking oil can be collected from hotels, restaurants, hostel mess and even households. Indeed, a single branch of fast-food centre alone can generate as much as 15 L of waste cooking oil per day [9]. As a result, there is a necessity to connect with hotel chains and use the cooking oil which just gets wasted every day. The biodiesel derived from waste cooking oil offer a great potential in attenuating emissions by releasing only biogenic car- bon in the air and greater energy return on investment [10]. In contrast, to increase stakeholders and public involvement in waste cooking oil recycling, it is indispensable to identify what motivates to reprocess waste cooking oil. This includes a multifarious string of legal and market barriers such as government policies, subsidy, political support and consumer confidence [11]. Because of these barriers, there is a reluctance to adopt sus- tainable technology to convert waste cooking oil to renewable diesel fuel. On the other hand, analyzing these barriers will help a pilot biodiesel manufacturing industry to put into the operation of large-scale biodiesel production facility. It is indispensable to recognize the value of these barriers and their contextual re- lationships so that those barriers which hold up other barriers and those which are most dominated by few other barriers can be well recognized. Based on the above considerations, an endeavor has been made in this work to critically examine the relationships among the barriers for large-scale production of biodiesel from waste cooking oil by Interpretive Structural Modeling (ISM) approach. Indeed, ISM * Corresponding author. E-mail address: sasi_me75@yahoo.com (P. Sasikumar). Contents lists available at ScienceDirect Renewable Energy journal homepage: www.elsevier.com/locate/renene https://doi.org/10.1016/j.renene.2018.04.079 0960-1481/? 2018 Elsevier Ltd. All rights reserved. Renewable Energy 127 (2018) 678e684

is an interactive learning tool for recognizing relationships (direct & indirect) and inter-relationships among specific problems or issues.

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ISM is mostly intended as a group learning approach, but it can also be used individually [12

e15]. In this study, 11 barriers have been identified through the previously publishing works, interviews and questionnaire-based survey with experts from industry and academia. The survey was carried out in small scale biodiesel plants located in India. In agreement with the ISM methodology, the views of experts were sought to build up the structural self-interaction matrix, which was later on used in the formulation of ISM frame- work and application of Impact Matrix Cross e Reference Multi- plication Applied to a Classification (MICMAC) analysis for clustering the barriers. 2. Literature review In the present day scenario, biodiesel production cannot completely rely on virgin vegetable oils [16]. As the eagerness to promote the same to meet growing energy demands could cause more hunger and there will be nothing left to eat [17]. Therefore, biodiesel should be produced in a way that does not cut into food supplies. One of the promising way outs to resolve this problem is to use waste cooking oil collected from restaurants and food- processing industry to produce biodiesel. Moreover, these oils would be less expensive to use as feed stock for biodiesel produc- tion [18]. As reported by Carlos et al. [19], the exploit of waste cooking oil for biodiesel production can scale back up to 45% of direct production costs compared to untainted vegetable oils, even with the added costs of the pretreatment. Studies carried out by Javier et al. [20] and Tsai et al. [21] clearly imply that biodiesel production from waste cooking oil has greater potential to mitigate green house gas discharges into the atmosphere. On the other hand, there are several important bottlenecks that restrict the development of biodiesel production from waste cooking oil. Jiang and Zhang [22] identified that waste cooking oil has scattered collection centres and there is a need to ensure optimal distribution supply chain network for biodiesel produced from waste cooking oil. In addition to supply chain optimization, there is a requirement to assess the best practices of processing waste cooking oil into biodiesel as stated by Knothe [23], Leung and Guo [24]. These best practices include assessing the quality char- acteristics of oil and biodiesel processing methodologies. Further- more, legal limitations, financial aid, media and political support are other barriers that hinder the successful implantation and large-scale development of this project [25,26]. To analyze the barriers of this kind, a well-established model called ISM has been used by researchers over the past decade. ISM methodology has been widely used in green supply chain management strategies to develop the variables and to study the influential pressures in greening the supply chain [27e34]. Researchers have also used ISM approach in other areas like sustainable supply chain management and risk assessment [35,36], reverse logistics [37e40], mobile banking [41], total productive maintenance [42] design of business process orientation [43] and in environmentally conscious manufacturing [44]. Recently, Ansari et al. [45] developed a struc- tural model using ISM to figure out and eliminate the barriers in implementing solar powered stations in India. As suggested by Chun et al. [46], ISM can also be used as a risk control decision model for natural disasters. On the whole, ISM approach helps in better understanding of the barriers in the project implementation which would help an industry to determine their relative importance and manage the resources in a well-organized manner for the utmost number of such plans to be effectively implemented. Upon considering this, ISM framework has been used in this work to analyze

the interaction among the barriers in biodiesel production from waste cooking oil. 3.

Problem description “Despite actuality that there are several shortcomings in the biodiesel production industry, the demand for such fuel may in the due course of time become as important as underground carbon resources of the present time” [17]. Therefore, there will be setting up of new biodiesel production plants and increased production capacity within the existing plants. Alternatively, the society is becoming increasingly aware of the truth that the recycling of used cooking oil is an actual necessity [18,19]. For this purpose, we have identified major stumbling blocks or barriers in implementing large-scale biodiesel production facility in India on the basis of literature reviews and expert interviews. These barriers in imple- menting large-scale biodiesel manufacturing industry using waste cooking oil are discussed in Table 1. 4. Solution methodology In this study, ISM is suggested as a solution methodology to analyze the relationships amongst the barriers of biodiesel pro- duction from waste cooking oil. The central objective of ISM is to make use of experts' practical

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experience and skills to transform a complex system into a number of sub-systems (elements) and build a multilevel structural model [60,61]. ISM

methodology is additionally used

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to implement order and direction on the complexness of interactions among different components of a system [62,63]. The various steps involved in the ISM methodology are as fol- lows [64]: Step 1 Barriers affecting the system under consideration are listed. Step 2 From the barriers identified

in step 1, a contextual rela- tionship is established among the barriers

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with regard to which pairs of barriers would be examined.

Step 3

A Structural Self-Interaction Matrix (SSIM)

is developed for the identified barriers, which clearly indicates pair wise relationships

among barriers

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of the system into consideration. Step 4

Reachability matrix is framed

from the SSIM and the matrix is verified for transitivity. The transitivity

of

the contextual relation is a

basic assumption considered in ISM. It states that if a

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variable

A is related to B and B is related to

C, then A is necessarily associated to C.

Step 5 The

reachability matrix

attained in Step 4 is partitioned

into different levels. Step 6

Based on the relationships obtained in the reachability matrix, a directed graph is drawn and the transitive links are removed. Step 7 The resulting

digraph is transformed into

an

ISM, by replacing barrier nodes with statements. Step 8 The ISM

framework developed in Step 7 is examined to ensure for conceptual inconsistency and necessary modifi- cations are made.

These steps of ISM are exemplified in Fig. 1. 4.1. Structural self-interaction matrix (SSIM) ISM recommends making use of views of experts in developing the association amongst the barriers. Therefore, in this work for recognizing the appropriate associations

among the barriers of biodiesel production from waste cooking oil,

four experts, three A. Avinash et al. / Renewable Energy 127 (2018) 678e684 679

from the biodiesel production industry and one from the academia were consulted for the same. For analyzing the barriers, an appropriate relationship of “leads to” kind is selected. This implies that one variable guides to another variable. Based on this, in- teractions between the variables are framed. Considering in mind the contextual relationship for every variable, the extant of relation between any two variables (i and j) and therefore the linked di- rection

of the relation

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is questioned. Four symbols are used to indicate

the direction of relationship between the criteria (i and j): V: criteria i will help attenuate criteria j; A: criteria j will

be attenuated by criteria i;

X: criteria i and j will help accomplish each other;

and O: criteria i and j are

not related. The SSIM

for the barriers of biodiesel production from waste cooking oil is given in Table 2. Table 2 provides the inter relationship between 11 barriers of waste cooking oil biodiesel production. From Table 2 it is seen that Lack of collecting centres barrier will assist to attenuate the Inconsistent Supply Quantity barrier, thus the association of ‘V’ is represented for barriers 1 and 2 in the SSIM. Inadequate Production Facilities barrier can be attenuated by Lack of Incentives barrier. Therefore, the connection between these barriers is represented by ‘A’ in the SSIM. Inconsistent Supply Quantity barrier and Inade- quate Production Facilities barrier help out to attain each other. Consequently, the correlation between these barriers is Table 1 Description of barriers in biodiesel production from waste cooking oil. Barriers Description Source Lack of Collecting Centres Biodiesel production from waste cooking oil is highly influenced by the number of collection centres. In order to increase the biodiesel production quantity, it is necessary to identify adequate number of collection centres. Tsoutsos et al. [47] Kumaran et al. [48] GREENEA [49] Sheinbaum [50] Prussi [51] Kolyaei [52] Inconsistent Supply Quantity There is no regular supply of waste cooking oil for large scale production facility and also it is necessary to have huge supply quantity. Tsoutsos et al. [47] Kumaran et al. [48] Inadequate Production Facilities The number of large scale production facilities for biodiesel production to meet primary energy demands is still in its infancy in several countries. Haas et al. [26] Vehicle Access Problems This problem is mainly because of the distance between the collection points and the biodiesel production plant. Kumar et al. [53] Tsoutsos and Stavroula [54] Lack of Storage Space In a large scale biodiesel production, space constraints often make production prohibitive. Tsoutsos and Stavroula [54] Poor Quality Characteristics The difficulty in using used cooking oil as a biodiesel resource is the presence of impurities such as high free fatty acid and water. This causes adverse effects on the biodiesel production process. Otadi et al. [55] Wang [56] Karonis et al. [57] Lack of Processing Technology Biodiesel is generally produced through the transesterfication reaction because of economical causes; However, choosing an efficient method for biodiesel production has become imperative in recent years. € Ozbay et al. [58] Lack of Knowledge In some countries, biodiesel have been often considered as a negative reputation owing to one-sided media coverage on food Vs fuel conflict and not understanding that biodiesel is energy for sustainable development. Birger Kerckow [59] Lack of Incentives The profitability of a biodiesel production strongly depends on the level of subsidies provided by the Governments. Haas et al. [26] Lack of Policy Implementation Although each country has multi-pronged policy prescriptions for development and promotion of biodiesel. In some countries, the achievement of proposed target blending of biodiesel seems remote a possibility. Debbie Yaver [25] Lack of Political Will There is a lack of political will across each nation to provide support for large-scale biodiesel production. Thus, there is a need for significant political clout to bring energy independence to the nation Haas et al. [26] Fig. 1. Flow diagram for preparing ISM. Table 2 Structural self-interaction matrix (SSIM). Barriers 11 10 9 8 7 6 5 4 3 2 1 1. Lack of Collecting Centres

A A

A A

O V A V A V 2. Inconsistent Supply Quantity A A A A V V A A X 3. Inadequate Production Facilities A A A V A A

A V 4.

Vehicle Access Problems A A A O O V

A 5.

Lack of Storage Space A A A A O V 6. Poor Quality Characteristics A A A A V 7. Lack of Processing Technology A A A O 8. Lack of Knowledge A A A 9. Lack of Incentives A A 10. Lack of Policy Implementation A 11. Lack of Political will A. Avinash et al. / Renewable Energy 127 (2018) 678e684 680

represented by 'X' in the SSIM. No association exists between Lack of Collecting Centres barrier and the Lack of Processing Technology barrier and therefore the correlation between these barriers is represented by 'O' in the matrix. 4.2.

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Reachability

matrix The SSIM is converted

into a binary matrix, called the

initial reachability

matrix by substituting V, A, X, O by 1

and 0

as per

the

case. The rules for

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the substitution of 1's and 0's are

the following: *

If the (i, j) entry in the

SSIM is V, then the (i, j) entry in the reachability matrix becomes 1 and the (j, i)

entry becomes 0. * If the (i, j) entry in the SSIM is A, then the (i, j) entry in the reachability matrix

becomes 0 and the (

j, i) entry becomes 1. * If the (i, j) entry in the SSIM is X, then entry

for both (i, j) and (

j, i) in the reachability matrix becomes 1. * If the (i, j) entry in the

SSIM is O, then entry

for both (i, j) and (j, i) in the reachability matrix

becomes 0. Based on the above rules, initial reachability matrix for the barriers is provided in Table 3. Table 3

depicts

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the completed initial reachability matrix by completely changing the barriers relationship in binary numbers of 0'

sand 1's. The final reachability matrix for the different barriers, exhibited in Table 4, is acquired by including the transitivities as enumerated in Step 4 of the ISM framework. In Table 4, the driving power and dependence of every barrier are disclosed. The driving power of a specific barrier is that the total range of barriers (counting itself). The dependence is that the total number of bar- riers which can facilitate accomplishing it. These driving power and dependencies are utilized within the MICMAC analysis, where the barriers are classified into four groups such as dependent, inde- pendent (driver), linkage, and autonomous barriers. 4.3. Level partitions

The

reachability matrix and antecedent set for every barrier is attained

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from the

final reachability matrix.

The reachability set for a selected variable comprises of the variable itself and the other

variables that may impact it,

whereas the antecedent set comprises

of the variable

itself and the other variables, which may

help

in achieving

it [60]. Consequently,

the intersection of those sets is acquired for all variables. The variable for which the intersection sets

and reachability matrix are the identical is given the top-level variable in

the ISM,

which might not facilitate to realize any other variable on top of their own level. Once the discovery of the top- ranking variable, it is rejected from the other remaining variables. From Table 5, it is noted

that Vehicle Access Problems, Lack of Processing Technology, Inconsistent Supply Quantity and Inade- quate Production Facilities

barriers are seen at Level I. As a result, it might

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be put at the top of the ISM framework (Fig. 2). This iteration is repeated until the levels of every barrier are obtained. The recognized levels aid in constructing

the digraph and therefore the final model of ISM.

The barriers,

along with their reachability set, antecedent set, intersection set and

the levels, are shown in

Tables 5

e9. 4.4. Construction of ISM

The structural model is constructed from the

final

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reachability matrix. The association

between the barriers j and i is indicated by an arrow

pointing from i to j. This

resultant graph is known as

diagraph. Expelling the transitivities as depicted in the ISM pro- cedure, the digraph is at last transformed into the ISM approach as shown in Fig. 2. The figure demonstrates that lack of political will is an extremely huge hindrance for the biodiesel production from waste cooking oil as it occupies the bottom of the ISM framework.

Vehicle access problems, lack of processing technology, inconsis- tent supply quantity and inadequate production facilities are the

barriers on which the effectiveness of the biodiesel production from waste cooking oil depends. These barriers have showed up at the top of the ISM hierarchy. The particulars of full ISM approach for the barriers are depicted in Fig. 2. 5. Micmac analysis The MICMAC analysis is based on augmentation properties of matrices. The purpose of the

MICMAC analysis is to examine the driver and the dependence power of the variables [65]. The var- iables

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are classified into four

clusters (Fig. 3).

The

first cluster comprises of the ‘autonomous barriers’ that

have weak driver power and weak dependence. These

barriers are generally sepa- rated from the framework, with which they have

just couple of Table 3 Initial reachability matrix. Barriers 1 2 3 4 5 6 7 8 9 10 11 1. Lack of Collecting Centres 1 1 0 1 0 1 0 0 0 0 0 2. Inconsistent Supply Quantity 0 1 1 0 0 1 1 0 0 0 0 3. Inadequate Production Facilities 1 1 1 1 0 0 0 1 0 0 0 4. Vehicle Access Problems 0 1 0 1 0 1 0 0 0 0 0 5. Lack of Storage Space 1 1 1 1 1 1 0 0 0 0 0 6. Poor Quality Characteristics 0 0 1 0 0 1 1 0 0 0 0 7. Lack of Processing Technology 0 0 1 0 0 0 1 0 0 0 0 8. Lack of Knowledge 1 1 0 0 1 1 0 1 0 0 0 9. Lack of Incentives 1 1 1 1 1 1 1 1 1 0 0 10. Lack of Policy Implementation 1 1 1 1 1 1 1 1 1 1 0 11. Lack of Political will 1 1 1 1 1 1 1 1 1 1 1 Table 4 Final reachability matrix. Barriers 1 2 3 4 5 6 7 8 9 10 11 Driver Power 1. Lack of Collecting Centres 1 1 1 1 0 1 1 0 0 0 0 6 2. Inconsistent Supply Quantity 1 1 1 1 0 1 1 1 0 0 0 7 3. Inadequate Production Facilities 1 1 1 1 1 1 1 1 0 0 0 8 4. Vehicle Access Problems 0 1 1 1 0 1 1 0 0 0 0 5 5. Lack of Storage Space 1 1 1 1 1 1 1 1 0 0 0 8 6. Poor Quality Characteristics 1 1 1 1 0 1 1 1 0 0 0 7 7. Lack of Processing Technology 1 1 1 1 0 0 1 1 0 0 0 6 8. Lack of Knowledge 1 1 1 1 1 1 1 1 0 0 0 8 9. Lack of Incentives 1 1 1 1 1 1 1 1 1 0 0 9 10. Lack of Policy Implementation 1 1 1 1 1 1 1 1 1 1 0 10 11. Lack of Political will 1 1 1 1 1 1 1 1 1 1 1 11 Dependence Power 10 11 11 11 6 10 11 9 3 2 1 A. Avinash et al. / Renewable Energy 127 (2018) 678e684 681

links, which might be powerfully built. Second cluster comprises of the dependent barriers that

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have

weak driver power but strong dependence. Third cluster has the linkage barriers with strong driving power and

also

strong dependence. These barriers are

unbalanced in the actuality that any activity on these barriers will affect others and furthermore a feedback on themselves.

Fourth cluster has the

independent barriers with

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strong driving power but weak dependence. It is noticed that a variable with an extremely strong driving power called the primary variables, falls into the group of independent or linkage criteria. The

driving power

and the dependence of each of these barriers are shown in Table 4. Consequently, the driver poweredependence diagram for the barriers is constructed (Fig. 3). As delineation,

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it is seen

from Table 4 that vehicle access problems barrier is having a driver power of 5 and a dependence of 11. Accordingly, in Fig. 3, it is situated at a place relating to a driver power of 5 and a

de- pendency power of 11.

Table 5 Level partitions for barriers e Iteration 1. Barriers Reachability set Antecedent set Intersection set Level 1 1,2,3,4,6,7 1,2,3,5,6,7,8,9,10,11 1,2,3,6,7 2 1,2,3,4,6,7,8 1,2,3,4,5,6,7,8,9,10,11 1,2,3,4,6,7,8

I 3 1,2,3,4,5,6,7,8 1,2,3,4,5,6,7,8,9,10,11 1,2,3,4,5,6,7,8 I 4 2,3,4,6,7 1,2,3,4,5,6,7,8,9,10,11 2,3,4,6,7 I 5 1,2,3,4,5,6,7,8 3,5,8,9,10,11 3,5,8 6 1,2,3,4,6,7,8 1,2,3,4,5,6,8,9,10,11 1,2,3,4,6,8 7 1,2,3,4,7,8 1,2,3,4,5,6,7,8,9,10,11 1,2,3,4,7,8 I 8 1,2,3,4,5,6,7,8 2,3,5,6,7,8,9,10,11 2,3,5,6,7,8 9

1,2,3,4,5,6,7,8,9 9,10,11 9 10 1,2,3,4,5,6,7,8,9,10 10,11 10 11 1,2,3,4,5,6,7,8,9,10,11 11 11 Fig. 2. ISM e based model for the Biodiesel Production barriers. Table 6 Level partitions for

barriers e

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Iteration 2. Barriers

Reachability set Antecedent set Intersection set Level 1 1,6 1,5,6,8,9,10,11 1,6 II 5 1,5,6,8 3,5,8,9,10,11 5,8 6 1,6,8 1,5,6,8,9,10,11 1,6,8 II 8 1,5,6,8 5,6,8,9,10,11 5,6,8 9 1,5,6,8,9 9,10,11 9 10 1,5,6,8,9,10 10,11 10 11 1,5,6,8,9,10,11 11 11

Table 7

Level partitions for barriers e

Iteration 3. Barriers Reachability set Antecedent set Intersection set Level 5 5,8 3,5,8,9,10,11 5,8 III 8 5,8 5,8,9,10,11 5,8 III 9 5,8,9 9,10,11 9 10 5,8,9,10 10,11 10 11 5,8,9,10,11 11 11 Table 8 Level partitions for barriers e Iteration 4. Barriers Reachability set Antecedent set Intersection set Level 9 9 9,10,11 9 IV 10 9,10 10,11 10 11 9,10,11 11 11 Table 9 Level partitions for barriers e Iteration 5. Barriers Reachability set Antecedent set Intersection set Level 10 10 10,11 10

V 11 10,11 11 11

VI Fig. 3. Driving power and dependence power diagram. A. Avinash et al. / Renewable Energy 127 (2018) 678e684 682

6. Results and discussion Biodiesel production from waste cooking is a dynamic business activity with essential economic and environmental benefits. Subsequently, there is a need to promote sustainable biodiesel production from waste cooking oil and increase its local market intake by enhancing its supply chain network and transformation. However, recycling waste cooking oil into biodiesel emphasis on the most significant technical and practical barriers including collection practices, processing techniques, potential implications, political will and so on. These barriers hindering the biodiesel production fromwaste cooking oil make significant challenges both for manufacturers and policymakers in the biodiesel production industry. In this connection, the crucial goal of this work is to analyze the interactions among these barriers. Some of the signif- icant barriers have been featured here and put into ISM approach and these barriers need to be overcome for the success of biodiesel production from waste cooking oil. The driveredependence graph provides several important perceptions

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about the relative signifi- cance and the interdependencies among the barriers [66]. This can offer superior understanding to the manufacturers and policy makers so that they can proactively manage these barriers.

The imperative administrative ramifications rising up out of this ex- amination are as follows: * Fig. 3 exhibits that there is no autonomous barrier. Autonomous barriers

are weak drivers and weak dependents and

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do not have much impact on the system. The

nonexistence of autonomous barriers in the present examination demonstrates that all the considered barriers play

an important part. * Dependent barriers are lack of collecting centres (barrier 1), vehicle access problems (barrier 4) and lack of processing technology (barrier 7). These barriers are weak drivers but robustly rely on one another. Therefore, the manufacturers must take particular care to tackle these barriers. * Inconsistent supply quantity (barrier 2), inadequate production facilities (barrier 3), poor quality characteristics (barrier 6) and lack of knowledge (barrier 8) are found under the linkage element category having a strong driving power along with strong dependence. These barriers are unstable in nature due to the fact that any change occurring to them will affect other barriers and may themselves be affected through a feedback mechanism [67]. * It is additionally seen from Fig. 3 that lack of political will (barrier 11), lack of policy implementation (barrier 10), lack of incentives (barrier 9) and lack of storage space (barrier 5)

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are independent barriers, i.e. they encompass strong driving power and weak dependency on other barriers. They might be treated as the ‘key barriers’. The manufacturer and

policy maker in the government must put a high concern in handling these barriers. 7. Conclusions and recommendations The study reports on the use of ISM framework to discuss the relationship between various barriers that hinder the production of biodiesel from waste cooking oil. Based on the work, the levels of various barriers are imperative in superior understanding of their implications in effective accomplishment of Biodiesel production from waste cooking oil. An understanding of the ISM approach reveals

that vehicle access problems, lack of processing technology, inconsistent supply quantity and inadequate production facilities are the top-level barriers.

These are the barriers which are being influenced by lower level barriers. The second level barriers - lack of collecting centres and poor quality characteristics; and third level barriers e

Lack of Storage Space and lack of Knowledge are the operational level barriers that are most required for the effective operation of Biodiesel production from waste cooking oil.

Lack of political will, lack of incentives and lack of policy implementation

have highest drive power and lowest dependence; hence, they appear at the bottom level of the hierarchy. This sig- nifies

that lack of political will, lack of incentives and lack of policy implementation

play an important role

and work as the main driver in the

effective accomplishment of large-scale Biodiesel production from waste cooking oil. This fact is also true from the commer- cialization point of view, because if biodiesel manufacturers and Indian Government do not have a fiscal policy and willingness for the implementation of biodiesel production, others will have no vital impact. Thus, ISM methodology supports the practical views of waste cooking oil Biodiesel production industry and portrays an apparent depiction about the importance of production barriers. Like this, various barriers can be well recognized and dealt with greatest care for the effective accomplishment of Biodiesel production from waste cooking oil. The proposed ISM approach can be statistically validated using Structural Equation Modeling (SEM) technique. However, SEM cannot prepare a new model, while the ISM has the competence to develop a primary model. In the future, it is planned to validate the present ISM approach using SEM method. References [1] G. Knothe, L.F. Razon, Biodiesel fuels, Prog. Energy Combust. Sci. 58 (2017) 36e59, https:// doi.org/10.1016/j.pecs.2016.08.001. [2] S.H. Mohr, J. Wang, G. Ellem, J. Ward, D. Giurco, Projection of world fossil fuels by country, Fuel 141 (2015) 120e135, https://doi.org/10.1016/ j.fuel.2014.10.030. [3] B.P. Anand, C.G. Saravanan, C.A. Srinivasan, Performance and exhaust emission of turpentine oil powered direct injection diesel engine, Renew. Energy 35 (2010) 1179e1184, https://doi.org/10.1016/j.renene.2009.09.010. [4] K. Purushothaman, G. Nagarajan, Performance, emission and combustion characteristics of a compression ignition engine operating on neat orange oil, Renew. Energy 34 (2009) 242e245, https:// doi.org/10.1016/ j.renene.2008.03.012. [5]

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the completed initial reachability matrix by completely changing the barriers relationship in binary numbers of 0'		The source document can not be shown. The most likely reason is that the submitter has opted to exempt the document as a source in Urkund's Archive.	

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to implement order and direction on the complexness of interactions among different components of a system [62,63]. The various steps involved in	The source document can not be shown. The most likely reason is that the submitter has opted to exempt the document as a source in Urkund's Archive.

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be put at the top of the ISM framework (Fig. 2). This iteration is repeated until the levels of every barrier are obtained. The recognized levels aid in constructing the digraph and therefore the final model of ISM. The barriers, along with their reachability set, antecedent set, intersection set and the levels, are shown in Tables 5	The source document can not be shown. The most likely reason is that the submitter has opted to exempt the document as a source in Urkund's Archive.

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about the relative signifi- cance and the interdependencies among the barriers [66]. This can offer superior understanding to the manufacturers and policy makers so that they can proactively manage these barriers.

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do not have much impact on the system. The

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are independent barriers, i.e. they encompass strong driving power and weak dependency on other barriers. They might be treated as the ‘key barriers’. The manufacturer and

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experience and skills to transform a complex system into a number of sub-systems (elements) and build a multilevel structural model [60,61]. ISM	

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5	77%
of the system into consideration. Step 4 Reachability matrix is framed from the SSIM and the matrix is verified for transitivity. The transitivity	

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variable A is related to B and B is related to C, then A is necessarily associated to C. Step 5 The reachability matrix	

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attained in Step 4 is partitioned

into different levels. Step 6

Based on the relationships obtained in the reachability matrix, a directed graph

is drawn and the transitive links are removed. Step 7 The resulting

digraph is transformed into

an

8	90%

Reachability

matrix The SSIM is converted

into a binary matrix, called the

initial reachability

matrix by substituting V, A, X, O by 1

and 0

as per

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11 41%

from the

final reachability matrix.

The reachability set for a selected variable comprises of the variable itself and the other

variables that may impact it,

whereas the antecedent set comprises

of the variable

itself and the other variables, which may

help

in achieving

it [60]. Consequently,

the intersection of those sets is acquired for all variables. The variable for which the intersection sets

and reachability matrix are the identical is given the top-level variable in

the ISM,

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reachability matrix. The association between the barriers j and i is indicated by an arrow pointing from i to j. This	

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4 64%

with regard to which pairs of barriers would be examined.

Step 3

A Structural Self-Interaction Matrix (SSIM)

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9 62%

the substitution of 1's and 0's are

the following: *

If the (i, j) entry in the

SSIM is V, then the (i, j) entry in the reachability matrix becomes 1 and the (j, i)

entry becomes 0. * If the (i, j) entry in the SSIM is A, then the (i, j) entry in the reachability matrix

becomes 0 and the (

j, i) entry becomes 1. * If the (i, j) entry in the SSIM is X, then entry

for both (i, j) and (

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j, i) in the reachability matrix becomes 1. * If the (i, j) entry in the

SSIM is O, then entry

for both (i, j) and (j, i) in the reachability matrix

becomes 0. Based on the above rules, initial reachability matrix for the barriers is provided in Table 3. Table 3

14 57%

are classified into four

clusters (Fig. 3).

The

first cluster comprises of the ‘autonomous barriers’ that

have weak driver power and weak dependence. These

barriers are generally sepa- rated from the framework, with which they have

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18 47%

Iteration 2. Barriers

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Reachability set Antecedent set Intersection set Level 1 1,6 1,5,6,8,9,10,11 1,6 II 5 1,5,6,8 3,5,8,9,10,11 5,8 6 1,6,8 1,5,6,8,9,10,11 1,6,8 II 8 1,5,6,8 5,6,8,9,10,11 5,6,8 9 1,5,6,8,9 9,10,11 9 10 1,5,6,8,9,10 10,11 10 11 1,5,6,8,9,10,11 11 11

Table 7

Level partitions for barriers e

Iteration 3. Barriers Reachability set Antecedent set Intersection set Level 5 5,8 3,5,8,9,10,11 5,8 III 8 5,8 5,8,9,10,11 5,8 III 9 5,8,9 9,10,11 9 10 5,8,9,10 10,11 10 11 5,8,9,10,11 11 11 Table 8 Level partitions for barriers e Iteration 4. Barriers Reachability set Antecedent set Intersection set Level 9 9 9,10,11 9 IV 10 9,10 10,11 10 11 9,10,11 11 11 Table 9 Level partitions for barriers e Iteration 5. Barriers Reachability set Antecedent set Intersection set Level 10 10 10,11 10

V 11 10,11 11 11

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15 64%	15: http://doras.dcu.ie/20877/ 64%
have weak driver power but strong dependence. Third cluster has the linkage barriers with strong driving power and also strong dependence. These barriers are	have weak driver power but strong dependence. The third cluster (top right quadrant) has the linkage criteria as they have a strong driving power and also a strong dependence. These criteria are

16 60%	16: http://doras.dcu.ie/20877/ 60%
strong driving power but weak dependence. It is noticed that a variable with an extremely strong driving power called the primary variables, falls into the group of independent or linkage criteria. The driving power	strong driving power and weak dependence power and is the independent variables. It is observed that a variable with a very strong driving power called the key variables, falls into the category of independent or linkage criteria. The driver power–

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INVESTIGATION ON SUSTAINABLE BIODIESEL PRODUCTION AND ITS UTILIZATION IN A REACTIVITY CONTROLLED

COMPRESSION IGNITION ENGINE

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A SYNOPSIS Submitted by AVINASH A

in partial fulfillment of the requirements for the degree of DOCTOR OF

PHILOSOPHY FACULTY OF MECHANICAL ENGINEERING

ANNA UNIVERSITY CHENNAI 600 025

FEBRAURAY 2018

1 1 INTRODUCTION Fossil fuels from underground carbon resources continue to play a crucial role in global energy systems.

However, these non-renewable energy sources (fossil fuels) even have

a negative impact as they negatively affect human health, produce emissions

and other ozone-depleting substances. The earth should thus reconcile the role of energy in social and economic development with the need to decarbonize, reduce our dependence on fossil fuels and move towards lower-carbon energy

sources (Hannah & Max 2018).

For this reason, biofuels from biomass have proved to be an invaluable source, especially in the third-world economies. In recent days, many studies have been carried out on the conversion of biomass into high value-added products. In this regard, biodiesel is an invaluable product that is renewable and biodegradable (

Agarwal 2007).

Although biodiesel is produced worldwide with a wide variety of feedstocks, it’s production and marketing on large-scale faces a considerable challenge (Fargione et al. 2008).

Also,

due to widespread concerns about the water-land-food nexus of first-generation biofuels, a waste-to-energy system using waste cooking oil has become very attractive in recent days (Rulli et al. 2016

and Pimentel et al. 2009).

In biodiesel production, the yield is influenced by various process parameters.

The identification of the influencing parameters and their contribution to

the

proportion of biodiesel yield is one of the decisive steps in biodiesel production.

In this regard, several optimization techniques are applied for conducting

a

minimum number of experiments to optimize the process parameters for maximum biodiesel yield (

Sivaraos et al. 2014). After biodiesel production, the residual impurities are separated by water wash method. Studies have shown that using water for biodiesel purification triggers issues, including

the production of large amount of wastewater that needs to be treated before reuse.

2

Diesel-powered compression ignition engines are the main workhorses in agriculture, transportation, military, mining, and energy sectors because of their durability and improved fuel economy.

In the past decades, intense efforts by numerous researchers to advance the performance of

the conventional Internal Combustion (IC) engines

led to the development of non-conventional IC engines. Among the various non-conventional IC engines, this work mainly emphasis the dual-fuel engine because the dual-fuel engine has substantial advantages when compared to single fuel engine and requires very little modification. Moreover, the engine offers

a more significant reduction in exhaust emissions (Heywood 1988). 2 LITERATURE SURVEY

The rapid extraction of readily accessible crude oil is now leading us to look for other potential sources of energy to power our vehicles. The emissions from these vehicles also require immediate attention and effective control measures. The oil spill in the Gulf of Mexico is such an example of the threat posed to the environment by fossil fuels (Keshav et al. 2011). In this direction, several researchers are looking for a possible substitute for petroleum. A study by researchers at the Federal Institute reported

that

most of the biofuels could reduce greenhouse gas emissions by 30-50 percent compared to petroleum fuel (

Zah et al. 2007). Biodiesel is a potential biofuel obtained from several vegetable oils (edible or inedible) or animal fats (Karnwal et al. 2010).

In addition,

biodiesel production from highly polluting waste is of recent interest (Rulli et al. 2016). Waste cooking oil is one such waste that prohibits human improvement and thus social welfare (Pimentel et al. 2009). Almost every nation in the world consumes million tons of edible cooking oil each year, 20 percent of which is considered waste

and

that may be used as a raw material for biodiesel production to cut down the petroleum imports (Ghobadian 2012). Several

3 research studies have reported that waste cooking oil, despite its disadvantages such as high content of free fatty acids and water, is a promising feedstock for biodiesel production. Although these disadvantages can be addressed by pre-treatment techniques (such as degumming) as suggested by Diana da Silva Araújo et al. (2014), there are other notable barriers to large-scale production of biodiesel including price competitiveness and production limitations.

As discussed before, the biodiesel production process

parameters can be optimized using theoretical optimization methods to find optimum reaction conditions for the best response. Among various optimization techniques, the Response Surface Methodology (RSM) is the most widely used technique to optimize biodiesel process parameters (Singhal

et

al. 2012). Singhal et al. (2012) used RSM to optimize biodiesel produced by base- catalyzed transesterification of Karanja oil. The authors reported that maximum biodiesel yield (?98%) with shorter reaction time (1 hour less) is achievable with optimization of process parameters of karanja oil biodiesel through RSM. Their study also indicated that biodiesel production by RSM aids in lowering operating cost of biodiesel by optimizing the operating time.

Although RSM is a convenient tool to optimize process parameters, its results are not accurate for highly nonlinear processes. This limitation has led to the development of a model based on Artificial Neural Network (ANN). Betiku et al. (2014) tested the performance of RSM and ANN in optimizing the biodiesel production from neem oil. They evaluated the accuracy of both the models by comparing the regression coefficient and

the

absolute average deviation. In their observations, ANN was better than RSM in the modeling as well as in the optimization of

biodiesel production. After biodiesel production, the

by-product glycerol is usually removed by separation technique. Trace impurities such as unreacted alcohol,

4 unreacted glycerides and unreacted catalyst present in biodiesel are traditionally removed by water washing. Numerous studies have shown that several water washes are necessary to achieve

acceptable removal of impurities from unpurified biodiesel. (

Canakci & Van Gerpen 2003). Recently some

purification techniques have been developed, such as ion exchange resins and adsorption treatment (

Mazzieri et al. 2008). Adsorption

is the most commonly used method for purification of biodiesel. This process

use suitable adsorbent

to adsorb certain impurities from the liquid phase on its surface (Huang & Ramaswamy 2013).

Activated carbon and ash-based adsorbents are among the most common adsorbents for this process (LeVan & Carta 2008).

The major problem in diesel/biodiesel-powered CI engines is emissions (especially oxides of nitrogen and soot emissions). In

this regard, Reactivity Controlled Compression Ignition (RCCI) is a dual fuel strategy which uses high reactivity fuel in the direct injection and low reactivity fuel in the port fuel injection to substantially reduce NO x and soot emissions (Park

et

al. 2014). Park et al. (2014) evaluated the dual-fuel combustion of bioethanol/gasoline as port fuel injection fuel and biodiesel as direct injection fuel. The authors noted that NO x reduction in bioethanol-biodiesel mode is significant; however, NO x reduction in gasoline-biodiesel mode is limited. In addition to

NO x ,

soot emissions were also reduced to ultra-low level. On the other hand, HC and CO emissions increased in both the dual-fuel combustion modes.

From the literature survey, it is figured out that the biodiesel production from waste cooking oil can scale back the direct production costs when compared to other feedstocks. Furthermore, this review makes it clear that RSM is the most efficient mathematical tool to optimize the biodiesel process parameters by base catalytic transesterification and ANN

is the

5 powerful tool that can be used to predict the biodiesel yield accurately.

Further exploration of literature clarifies the need to conduct economic analysis and barrier analysis for large-scale biodiesel production using waste cooking oil. This review also suggests the need to purify the biodiesel by waterless purification technique (dry purification using bio adsorbents). By the way, a novel silica-based bioadsorbent (cow dung ash) is identified. Finally, this review recommends the necessity to evaluate biodiesel properties and test the engine characteristics fuelled with the biodiesel under conventional and RCCI mode. 3

EXPERIMENTAL METHODOLOGY

The optimization of biodiesel

process parameters like alcohol to oil molar ratio, catalyst concentration, reaction time and

mixing intensity for producing optimum biodiesel yield was done by

RSM model using Box-Behnken design, and the ANN predictive model was developed using a feed-forward back propagation neural network algorithm.

The waterless purification of biodiesel from waste cooking oil using cow dung ash at different concentration

was performed experimentally, and

the optimum quantity of cow dung ash for biodiesel purification was found through chemometric analysis by comparing the Fourier Transform Infrared (FTIR) transmission spectral characteristics of cow dung ash with the water treated

spectral characteristics. The process model to estimate the biodiesel production cost was developed using SuperPro designer

and

the barriers in large-scale biodiesel production from waste cooking oil were done by

Interpretive Structural Modeling (ISM) approach. Also, classification of barriers was done by the

Impact Matrix Cross–Reference Multiplication Applied to a Classification Analysis (MICMAC).

6 A single cylinder CI engine was modified to work in the RCCI mode with

the

manifold injection of low reactivity fuel (ethanol) and direct injection of high reactivity fuel (biodiesel). A vaporizer unit was mounted on the intake manifold to deliver low reactivity fuel as a premixed homogenous charge. A low-pressure fuel injector controlled by an Electronic Control Unit (ECU) was mounted on

the

top of the fuel vaporizer unit to supply exact quantity of ethanol. 4

OPTIMIZATION OF BIODIESEL PRODUCTION PROCESS PARAMETERS (a) RSM Based Predictive Model The experiments were performed using RSM, and the results are shown in Figure 1. A second order polynomial regression equation has been developed using coded units. The final model developed based on coded units for biodiesel yield is represented by Equation (1). * % = 90.27 − 10.27*+ 13.55 + 0.42? − 5.96 2 + 3.43 − 6.33* − 8.56

7

Figure 1 Response surface plots showing the relative effect of process variables on Yield (%) (a) Effect of catalyst and reaction time; (b) Effect of catalyst and molar ratio; (c) Effect of catalyst and reaction speed. (

b) ANN based predictive model The experimental versus predicted yield of developed ANN model is shown in Figure 2.

The R 2 values for training, validation, testing, and all obtained using the ANN model were 0.9999, 0.9635, 0.9756, and 0.9851, respectively. The R 2 values of

the

ANN model indicate that there is good agreement between experimental response and ANN predicted response. Therefore, the ANN model can accurately predict biodiesel yield due to its greater flexibility and potential to non-linear model relationships.

8 Figure 2 Comparison between the experimental values and yield predicted by ANN 5

BIODIESEL PURIFICATION BY COW DUNG ASH (CDA)

The purification of waste cooking oil biodiesel by CDA was carried out in a batch mode under fixed heating at 65 o C and continuous stirring for 20-30 minutes with different ash

concentration of 1, 2, 3 and 4 wt/wt%.

The optimum concentration of ash for biodiesel purification was found

as 1 wt% through chemometric analysis. 6

ECONOMIC ANALYSIS OF BIODIESEL PRODUCTION A process model was developed to produce biodiesel from waste cooking oil by

the

transesterification process.

The biodiesel production plant

9 developed in this study is devoted to the production of 39,208 metric tons of biodiesel per year. The total capital investment

for this plan is expected to be Rs.1,615,133,000. At an economic

value of Rs.15/kg for feedstock waste cooking oil, biodiesel cost of Rs.55/kg was estimated.

In addition to the main revenue from biodiesel, an additional revenue of about Rs.22/kg from the sale of glycerol can be produced. Overall,

the profitability analysis of the study unveiled that the developed biodiesel production plant can produce biodiesel with a unit cost of Rs.51/kg and unit production revenue of Rs.58/kg. 7

ISM APPROACH TO ANALYZE BARRIERS OF BIODIESEL PRODUCTION In this work, ISM approach was used to identify contextual relationships between barriers of biodiesel production by waste cooking oil. The

MICMAC analysis, in addition,

has been carried out to categorize the barriers based on dependence and driving power. The results show that the problem of

vehicle access, lack of processing technology, inconsistent supply quantity, and inadequate production facilities have top priority. The results also show that

a

lack of political will, lack of incentives and lack of policy implementation play a critical role in the

successful development

of

large- scale biodiesel production from waste cooking oil. 8 PERFORMANCE, EMISSION AND COMBUSTION

CHARACTERITSTICS OF RCCI ENGINE In this work, the combustion characteristics of the reactivity controlled compression ignition engine were investigated by manifold induction of vaporized ethanol and direct injection of waste cooking oil biodiesel. More or less, this work attempted to produce homogeneous charge using a fuel vaporizer unit. The research experiments were carried out with biodiesel-vaporized ethanol (10% and 20% ethanol substitution), and the

10 results were compared with

biodiesel and diesel engine operation. In reactivity controlled compression ignition

mode, the maximum rate of pressure rise and the maximum rate of heat-release were

noted. It was also seen that the increase of ethanol concentration by 10% in biodiesel prolongs the low temperature reactions by 1˚ crank angle. Also with the increase in ethanol concentration, the start of high temperature reactions retards by 3˚ crank angle. The experimental results showed that maximum brake thermal efficiency (31%) could be produced by reactivity controlled compression ignition engine running on biodiesel-vaporized ethanol compared to neat diesel (30%) and biodiesel (29%).

It has also been extrapolated from the experimental results that biodiesel-vaporized ethanol operation can significantly reduce oxides of nitrogen (

up to 90%) and smoke levels (up to 19%). The exhaust gas temperature also decreased by 60% for biodiesel-20% vaporized ethanol induction. However, unburned hydrocarbons and carbon monoxide emissions exhibited a reverse trend. It was noted that unburned hydrocarbons and carbon monoxide emissions increased up to 2% at full load for biodiesel-20% vaporized ethanol induction when compared to neat diesel and biodiesel. 9 CONCLUSION ? Response surface methodology

was successfully used to model and optimize the experiments. The regression model developed based on coded units describes that the

variation of the process variables can explain ~ 96% of the effect on the biodiesel yield. Conversely, the artificial neural network

prediction model developed (4:14:1) showed greater flexibility and the potential to establish nonlinear interactions

compared to response surface methodology.

11 ?

Cow dung ash was effectively used as an adsorbent for the purification of biodiesel from waste cooking oil. The test results showed that the

cow dung ash in a concentration of 1 wt/wt % showed an excellent effect for

the removal of contaminants from biodiesel similar to water treatment. ?

The economic analysis showed that it is possible to produce biodiesel from the modeled plant more profitably with

the

unit production cost of Rs.51/kg and unit production revenue of Rs.58/kg.

Also, the barrier analysis showed that

the levels of the various barriers are essential for a better understanding of their impact in effective implementation of

large-scale biodiesel production from waste cooking oil. ? The experimental results showed that maximum brake thermal efficiency of 31% could be produced by reactivity controlled compression ignition engine running on biodiesel-vaporized ethanol

compared to neat diesel (30%) and biodiesel (29%). ? The effect of the premix showed negative results in emissions of unburned hydrocarbons and carbon monoxide. In this study, emissions of unburned hydrocarbons and carbon monoxide emissions slightly increased with increasing vaporized ethanol induction. ?

There has been a remarkable reduction in oxides of nitrogen emission with

reactivity controlled compression ignition

mode. Almost 90% of nitrogen oxides reduced by 20% vaporized ethanol induction compared to base diesel at full load. The smoke intensity of the

reactivity controlled compression ignition

mode decreased by up to 19% when

12 compared to base diesel and by up to 17% compared to biodiesel.

The

exhaust gas temperature also decreased by 60% for 20% vaporized ethanol induction. ?

In the present work, combustion begins earlier, and the occurrence of peak pressure shifts towards TDC for

reactivity controlled compression ignition

operation. In this study, the maximum cylinder pressure was measured with biodiesel-vaporized ethanol (10% induction) in RCCI mode (81.733 bar) occurring at 5 ˚CA after TDC, while for conventional diesel operation the peak pressure (70.613 bar) occurred at 7 ˚CA after TDC.

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ii ANNA UNIVERSITY CHENNAI 600 025 CERTIFICATE The research work embodied in the present Thesis entitled “

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ABSTRACT In the recent years most commonly fossil fuels are more than 27% of the world’s major consumption of energy. However, due to fluctuating price, environmental pollution and limited reserves. The development of alternative energy sources has become inevitable. In India domestic demand of petroleum requirements is completed by importing of nearly 75% curde oil. In order to fulfill the requirements, in the recent years researchers have made various efforts for different energy sources for alternative fuels in diesel engine without any engine modification. In the present work biodiesel is prepared through the transesterification process by using ceibapentandra seed oil and water melon seed oil. From the transesterification results observed that a maximum yield esters CPME product reached 92% and 88% for catalyst quantity of 14 grams of KOH and NaOH respectively are methanol as a solvent.

The amount of KOH used is less than that of NaOH for same mass of feed stock oil therefore KOH is the best catalyst when compare to NaOH.

For transesterification of watermelon seed oil using KOH as a catalyst and methanol as a solvent. The maximum yield of ester WME 88% for catalyst quantity of 15grams. As per ASTM standard the physical and chemical properties of raw oil and methyl ester were compared with diesel such as viscosity, relative density, cloud, flash point, pour point and calorific value are measured and presented in the report. The Gas chromatography and Mass spectrum analysis (GC-MS) proved that more esters content with high purity of CPME and WME are best suitable fuel for compression ignition engine. This clearly shows that prepared methyl esters are very good fuel property.

The

pine oil

iv has low viscosity and higher calorific value as compared with diesel. So the low viscosity of pine oil is blended with B25 CPME and diesel. The results proved that the blended fuel properties closer to diesel. So,

it can be directly used in diesel engine without any modification.

A single cylinder, naturally aspirated, direct injection, water cooled, kirloskar make AV1model diesel engine capable of developing a power output of 3.7kW (5HP), at rated speed of 1500 rpm engine is equipped with an combustion analyzer and it is loaded by an eddy current dynamometer. A piezoelectric pressure transducer is associated with PC based data acquisition system. The cylinder pressure for each crank angle is measured by PC based data acquisition system. The data of cylinder pressure were accustomed to calculate the combustion parameters. The combustion parameters like peak pressure and heat release were calculated based on the pressure data. The AVL 444 di gas analyzer is used to measure the carbon monoxide (CO), carbon dioxide (CO2), oxides of nitrogen (NOx) and unburned hydrocarbon (HC) emissions. The AVL 437 continuous flow smoke meter is used to measure the smoke emission. The inlet and exhaust gas temperatures and water temperatures are measured by using thermocouples. The performance and emission test is conducted by two phases such as Phase I and Phase II. In phase1 three blend ratios likes B25, B50, B75 and B100 are prepared from CPME and WME to experimentally evaluate the performance and emission characteristics. The results proves that the brake thermal efficiency of B25 CPME and B25WME were 30.69% and 30.35% respectively. It is nearly close to diesel (31.88%). The emissions of CO, CO2 HC, and Smoke are reduced. The NOx emissions are slightly higher compared with B100 CPME and B100 WME. From the experimental result B25 CPME proved that best blend ratio compared with other blends.

v

In phase II the best fuel of B25 CPME is blended with pine oil (PO) and diesel (DI). The blends of B 25

CPME +

PO 50+DI 25 ratio, brake thermal efficiency (30.71%) is close to diesel (31.88%) and pine oil (31.97). The smoke emission is nearly close to diesel and slightly reduceNOx emission compared with diesel. The overall combustion characteristics of B 25 CPME +

PO 50+DI 25 blend is proved that the cylinder pressure and heat release rate is (63.85bar and 59.58 kJ/m3 deg.) higher than B100CPME (47.44 bar and 43.58kJ/m3 deg.) and closer to diesel (65.16 bar and 62.57 kJ/m3 deg.). From the experimental result proved that 25% of ceibapentandra methyl ester blended with 25% of diesel and 50% of pine oil blends ratio is a best alternative fuel for naturally aspirated direct injection diesel engine producing a 3.7kW at a rated speed of 1500 rpm without any modification of the engine.

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for biodiesel, PO, and its blends 131 5.29

The variation of cylinder pressure with crank angle for biodiesel, PO, and its blends 133 5.30 The variation of heat release rate with crank angle for biodiesel, PO, and

its blends 134

xxi LIST OF SYMBOLS AND ABBREVIATIONS Symbols µ - Dynamic viscosity µm - Micro meter $ - US Dollar Abbreviation ASTM - American standard for testing methods B100 WME - Biodiesel 100 WME B100 CPME - Biodiesel 100% CPME B25 CPME - Biodiesel 25% CPME and diesel 75%

B25CPME+

PO75 - Biodiesel 25% CPME and pine oil 75% B25CPME+PO25+DI50 - Biodiesel 25% CPME, pine oil 25% and diesel 50% B25CPME+PO50+DI25 - Biodiesel 25% CPME, Pine oil 50% and diesel 25%

B25 WME - Biodiesel 25% WME and diesel 75% B50 CPME - Biodiesel 50% CPME and diesel 50% B50 WME - Biodiesel 50% WME and diesel 50% B75 CPME - Biodiesel 75% CPME and diesel 25% B75 WME - Biodiesel 75% WME and diesel 25% BSEC - Brake specific energy consumption BSFC - Brake specific fuel consumption BTE - Brake thermal efficiency CO 2 - Carbon dioxides CPME - Ceibapentandra oil methyl ester cSt - Centi Stoke CN - Cetane number

xxii CP - Cloud point CI Engine - Compression ignition engine CA - Crank angle DI - Diesel EU - European Union EGT - Exhaust gas temperature FP - Flash point FFA - Free fatty acid GC - Gas chromatography GC-MS - Gas chromatography mass spectrum HSU - Hartridge smoke unit HHV - Higher heating value HP - Horse power IOCL - Indian Oil Corporationlimited IEA - International energy agency IN - Iodine number IV - Iodine value kW - kilo Watt Kg/ha - kilogram per hectare kJ/ Kg - Kilojoule per kilogram LPG - Liquefied petroleum gas LNG - Liquid natural gas LHV - Lower heating value M & HCV - Medium and heavy duty commercial vehicles MMT - Million metric tons NMOG - Non-methane organic gases NOx - Oxide of nitrogen SOx - Oxide of sulphur PM - Particulate matter ppm - Parts per million

xxiii PO - Pine oil KOH - Potassium hydroxide PP - Pour point RPM - Revolutions per minute NaOH - Sodium hydroxide NaoCH 3 - Sodium methoxide SVO - Straight vegetable oil HC - Unburned hydrocarbon WME - Watermelon oil methyl ester Wt - Weight

1

CHAPTER 1

INTRODUCTION 1.1 GENERAL In day to day life, the energy demands becomes increasing and fossil fuels are getting depleting, so we are in need to search renewable alternate fuels. At that time, the petroleum fuels acts an important role in the development of agricultural sector, transportation, industrial growth and to satisfy the primary necessitates of humans. When the

consumption is increasing rapidly these fuels are limited and depleting day by day.

The utilization of fossil fuels is disturbing the environment and it makes so much of environmental problems in the society (i.e.)

when the fossil fuel are burned it releases various smog –causing

pollutant and greenhouse gases that lends to global warming. The conventional fossil fuels (diesel) used in a diesel engine, it releasing higher amounts of carbon dioxides (CO 2), oxide of sulphur (SO X) particulate matter (PM), oxides of nitrogen (NO X) and unburned hydrocarbon (HC).

Moreover, CO 2 is the greenhouse gas but NO X and SO X causes "acid rain" (Altun & Lapuerta 2014). The particulate matter (PM) which emits from petroleum diesel engine causes several health hazards such as respiratory diseases, cardiovascular diseases and lung cancer.

In addition to that, the fast

depleting the

petroleum fuels cause the threat of climatic change and fluctuating fuel cost. The expecting future populations about top ten countries are pointed out in Table 1.1 and

Table 1.2.

From the periods of 1950 to 2050 and 2100 to 2300 respectively.

In a clear evident manner it shows that India will be the first position in terms of population

with in the

2 year 2050. (

U.N. http://www.un.org/esa/populations/longrange 2/ world pop 2300 final. Pdf: 2004). While the population increases the usage of automobiles also increasing exponentially as well as the need of fossil fuel is also increasing rapidly. 1.1.1 Current Status of Automobile Industry in India Agarwal (2016) reported the

confederation of India industry survey about automobile sector status

as follows, Largest three wheelers market Second largest two wheelers market Tenth largest passengers car market Fourth largest tractor market Fifth largest commercial vehicle market Fifth largest bus and truck segment The number of vehicles producing an industry is increasing year by year. As such in the year of April-March 2015, the industry produced a total number of 2,33,66,246 vehicles including two wheelers, passenger vehicles and three wheelers in direct contrast to 2,15,00,165 in

the period April-March 2014. This record shows the development of 8.68%

over the same period compared with last year presented in Table 1.3. Renewable energy sources like nuclear, solar, wind, hydro power, nuclear, biofuel and biodiesel are evolving as feasible alternative and research in using them is going on in a developing stage. The photo voltaic cells convert the solar energy

in to thermal energy and indirectly using

as electric energy. The origin of renewable energy depends on the nature and it is

3 difficult to store except bio-energy. So the researchers are looking for the alternative fuel, however bio-diesel from vegetable oil compromises all those solutions. In the 19 th century (1858-1913), ran the engine using vegetable oils as a substitute for diesel fuel for Rudolf Christian Karl diesel (Talebian- Kiakalaieh et al. 2013). The term bio-diesel is hosted in the United States during 1992 by the National soy diesel Development Board (presently national bio-diesel board) which has founded the commercialization of bio-diesel in the United States. At any ratio the bio-diesel can be used with petroleum diesel but it has lower exhaust emissions and also it has very similar characteristics. When compare with petroleum diesel properties the bio-diesel holds well fuel properties such as nontoxic, renewability, biodegradability, and free of sulfur and aromatics (Martini & Schell 2012). 1.2

FUEL DEMAND AND ITS INCREASE IN PRICE Diesel fuel plays a major role in different sectors like agriculture industries, agriculture and transportation. Diesel fuel is also one of the unavailable

and developing energy liquid fuel

in India , due to the reason the diesel demand increasing, the fuel price also increasing. Since, we are importing approximately 75% of petroleum from other countries

reported by Shukla

et al. (2015). It becomes a great setback for the economy development of India against US dollars. Ale & Bade Shrestha (2008) reported that the year by year increased demand ratio of petrol, diesel and kerosene and it’s evidently indicated that as compare with petrol and kerosene the usage and

increasing the

demand in diesel is higher as shown in Figure 1.1. So, the alternate source of energy research

is a need of the day.

4

Table 1.1 Top ten largest countries and their populations, selected years 1950 to 2050 in millions Rank Country Population in millions up to 1950 year Rank Country Population in millions up to 2000 year Rank Country Population in millions up to 2050 year 10 Italy 47.1 10 Nigeria 114.7 10 Congo, DR 151.6 9 United 49.8 9 Japan 127.0 9 Ethiopia 171.0 8 Brazil 54.0 8 Bangladesh 138.8 8 Brazil 233.1 7 Germany 68.4 7 Pakistan 142.7 7 Bangladesh 254.6 6 Indonesia 79.5 6 Russian 145.6 6 Nigeria 258.5 5 Japan 83.6 5 Brazil 171.8 5 Indonesia 293.8 4 Russian 102.7 4 Indonesia 211.6 4 Pakistan 348.7 3 U.S.A 157.8 3 U.S.A 285.0 3 U.S.A 408.7 2 India 357.6 2 India 1016.9 2 China 1395.2 1 China 554.8 1 China 1275.2 1 India 1531.4

Table 1.2

Top ten largest countries and their populations, selected years 2100 to 2300 in millions.

Rank

Country Population in millions up to 2100 year Rank Country Population in millions up to 2200 year Rank Country Population in millions up to 2300 year 10 Congo, DR 203.3 10 Congo, DR 173.0 10 Congo, DR 182.7 9 Brazil 212.4 9 Ethiopia 196.9 9 Ethiopia 206.5 8 Ethiopia 222.2 8 Brazil 208.8 8 Brazil 222.6 7 Bangladesh 259.9 7 Bangladesh 232.0 7 Bangladesh 242.7 6 Indonesia 272.8 6 Indonesia 263.0 6 Indonesia 276.2 5 Nigeria 302.5 5 Nigeria 268.4 5 Nigeria 282.8 4 Pakistan 408.5 4 Pakistan 342.5 4 Pakistan 359.1 3 U.S.A 437.2 3 U.S.A 470.0 3 U.S.A 493.0 2 China 1181.5 2 China 1200.7 2 China 1285.7 1 India 1458.4 1 India 1304.5 1 India 1371.7

5 Table 1.3 Indian automobiles industry production and sales trends Category 2009-2010 2010-2011 2011-2012 2012-2013 2013-2014 2014-2015 Production Sales Production Sales Production Sales Production Sales Production Sales Production Sales Passenger Vehicles 23,57,411 19,51,333 29,82,772 25,01,542 31,46,069 26,29,839 32,31,058 26,65,015 30,87,973 25,03,509 32,20,172 26,01,111 Commercial vehicles 5,67,556 5,32,721 7,60,735 6,84,905 9,29,136 8,09,499 8,32,649 7,93,211 6.99,035 6,32,851 6,97,083 6,14,961 Three wheelers 6,19,194 4,40,392 7,99,533 5,26,024 8,79,289 5,13,281 8,39,748 5,38,290 8,30,108 4,80,085 9,49,021 5,31,927 Two wheelers 1,05,12,903 93,70,951 1,33,49,349 1,17,68,910 1,54,27,532 1,34,09,150 1,57,44,156 1,37,97,185 1,68,83,049 1,48,06,778 1,84,99,970 1,60,04,581 Total 1,40,57,064 1,22,95,397 1,78,92,409 1,54,81,381 2,03,82,026 1,73,61,769 2,06,47,611 1,77,93,701 2,15,00,165 1,84,23,223 2,33,66,246 1,97,52,580

6 Figure 1.1 Petrol, Diesel and Kerosene demand 2006 to 2020

Table 1.4 India's petrol, diesel, kerosene and LPG prices in Rupees (August 01 2015 - July 01 2016) Month & Year Petrol

Rs. Diesel Rs. Kerosene Rs. LPG Rs. Aug 01, 2015 64.47 46.12 14.96 417.82 Nov 01, 2015 61.38 47.10 -- -- Feb 01, 2016 59.95 44.68 -- --

May 01, 2016 62.19 50.95 -- 419 sub/ 527.5 nonsub July 01, 2016 64.76 54.7 -- -- Source:http://in.reuters.com/article/2015/08/31/india-fuel-prices-idinl4n1163j420150831

7 The petrol, diesel, kerosene and LPG price has monthly greater than before and also fluctuations. As on August 2016, the price of petrol, diesel, kerosene and LPG were 64.47, 46.12, 14.96 and 417.82 rupees respectively. The differences in the oil price modification in India (01.08.2016 to 01.07.2016) are shown in Table 1.4.

At present the cost of petrol, diesel, kerosene and LPG were 64.76, 54.7, 14.96 and 419 rupee per liter and 14.5kg per cylinder respectively. This needs the developments and commercialization of alternative fuel from bio-sources as reported by Reuters. Source:http://in.reuters.com/article/2015/08/31/india-fuel-prices-idinl4n1163 j420150831 1.3 PRODUCTION, IMPORT AND EXPORT OF CRUDE OIL AND COST OF PETROLEUM PRODUCT 1.3.1 Production and Consumption of Petroleum Products The production of petroleum products is 221.136 MMT in the year 2014-15 as against 220.756 MMT achieved in 2013-14, shows a marginally increase of about 0.17%. However, production was affected due to planned outages in IOCL-Panipat, Koyali and Bongaigaon refineries during April- July, 2014. During the year 2014-15, the consumption of petroleum products in India was 164.987 MMT with a growth of 4.15% as compared to consumption of 158.407 MMT during 2013-14. Production and consumption of petroleum products during

the years 2009-10

to 2014-15 have been shown in the Figure 1.2.

8 Figure 1.2 Production and consumption (indigenous sales) of petroleum products 1.3.2 Imports of Crude Oil Import of crude oil during

the year 2014-15

was 189.432 MMT valued at rupees 6,87,350 crore which marked a decrease of 0.10% in quantity terms and 20.53% decrease in value terms over the same period of last year. The decline in value terms released to reduced crude oil prices by around 50% from June 2014 to December 2014. During the year 2013-14 the import of crude oil was 189.238 MMT valued at rupees 8, 64,875 core. The average international crude oil oil price (Indian Basket) was US$ 84.20/bbl. During

the period of April-march in the year 2014-15

as compared to US$ 105.52/bbl in the same period of 2013 -14, ie lower by 20.20% as compared to the same period last year. During the last 6 months there is continuous declining trend in the price of crude oil in the international market. The price of Indian basket crude oil which was at $111.84/bbl on 19 th June 2014, it has continuously decreased thereafter to US$ 53.64/bbl on 31 st March, 2015. The trend in growth of crude oil imports and crude oil international (Indian Basket) prices is shown in Figure 1.3.

9 Figure 1.3 Quantity of crude oil importsand average international crude oil prices 1.3.3 Imports and Exports of Petroleum Products During

the period April-March, in the year 2014-15 imports of petroleum products were 20.423 MMT 72778 crore which shows an increase of 22.16% in quantity terms and 2.45% decrease in value terms against the same period of previous year. The quantity of petroleum products imported during

the year 2013-14 was 16.718 MMT at rupees 74,605 crore. During the period April-March in the year 2014-2015

the exports of petroleum products were 63.928 MMT valued at rupees 2,88,563 crore which shows a decreased of 5.80% in quantity terms and 21.65% decreased in value terms against the experts of 67.864 MMT valued at rupees 3, 68, 279 crore for the same period of last year. The trends in quantity of petroleum products

and LNG imports and export are depicted shown in the Figure1.4.

10

Figure 1.4 Trend in imports & exports of petroleum products 1.4 GLOBAL STATUS The developed and developing countries alike have shown interest in developing and effective utilization of

renewable energy. This interest will produce better prospects for the biofuels and the advantages of these fuels are, which reducing the vehicle emission and it reduces the global warming and enhancing rural development, which is an achievement towards energy security, renewable and reduce pollution to environment. The European biodiesel board (2011) reported that European Union produced over 24.7x10 -9 Lyr-1of biodiesel in

the year 2011. Due to new production capacity, projected Canadian biodiesel production in the year 2013 (0.471x10 -9) was more than double estimated for in 2012 (0.210x10 9 Lyr-1). A raised bio-diesel

production of 0.65x10 9 L in 2014. It was partly attributed to the expected completion of a plant producing 2.65x10 8 Lyr-1. The major biodiesel feed stack in Europe, United States, and Canada are soybean, rape-seed oil and canola oil respectively (Adewale et al. 2015). For decreasing the greenhouse effect and oil import Canada and European Union (EU) have already started this replacement with bio-diesel at a certain percentage. The European parliament would increasing the biofuel market share of 10% by the

year

11 2020.

The Canadian government announced a new policy for a new biofuel advocated the blend of 2% of bio-diesel and 5% of heated bio-diesel in the replacement of diesel for transportation sector

in between the years 2012 to 2015 respectively (

Roy et al. 2013).A new AEDP 2012-2021 project recently announced by the Royal Thai Government has regulated that the blending ratio of

bio-diesel

in petrol-diesel must achieve 10% (ie) B10 bio fuel, in 2021, corresponding to a 5.97ML/day market in Thailand (Chen et al. 2014). 1.4.1 Effect of Engine Emissions in Environment and Human Health The emissions produced by combustion of diesel must have a contrary effect on environment and human health. It is depicted by the unite nation inter-governmental panel that because of greenhouse gas emission including methane, carbon dioxides and nitrogen oxides increases the global warming. Due to green gas effect the global temperature is increased by more than 2 *

H

C and about hundreds of millions of people will lose their lives. hydrocarbon (HC), oxide of nitrogen (NOx), Carbon monoxide (CO), and organic gases other than methane (Non-methane organic gases, (ie) NMOG) which are emitting from internal combustion engine has been identified as harmful to the human health and environment degradation. Table 1.5 shows

the

impact of exhaust emissions on human health.

12 Table 1.5 Shows the engine emissions impact on human health S. No Exhaust emissions

Reason for formation Impact in health 1 NOx The temperature inside the combustion chamber becomes high due to higher calorific value (or) presence of a higher amount of oxygen, leading to increase in the formation of

NOx. Irritate the lungs and cause edema bronchitis and pneumonia; and result in increased sensitivity to dust and pollen in asthmatics 2 CO

Incomplete formed due to shortage of air (or) due to low gas temperature

CO is the formed.

It affects fetal growth inside pregnant women and tissue development of young children. It has a synergistic action with other pollutants to promote morbidity in people with respiratory (or) circulatory problems. 3 HC

HC appears in exhaust gas due to local rich mixture pockets at much lower temperature than the combustion and due to flame quenching near to metallic walls.

Eye irritation, coughing and sneezing, drowsiness and symptoms akin to drunkenness. Some hydrocarbons have a close affinity for diesel particulates and many contribute to lung disease 1.5

NATIONAL STATUS 1.5.1 Indian Biomass Energy Conversion Policy In recent years, due to population and economic growth India's energy consumption has been increasing a relatively fast rate. By way of rapid

13 urbanization and improving standards of living for million Indian households, the demand is probable raise a lot. Consequently, Indian government is now making various planning and polices in energy sector. Since, the key target of the world is now sustainable development, so renewable energy resources are considering for power generation. Ministry of new and renewable energy of India (

MNRES)

has developed many projects and polices in this filed and promoting to adopt these methodologies by providing various subsidies and incentives. Diesel engines are located in power sector and transportation, with a high thermal efficiency and extended durability. To accomplish the domestic demand, India has to import the 75% of petroleum requirements. The major part of these petroleum products is utilized by the diesel vehicles play a major role in transport sector. Hence, to satisfy the domestic energy demand in a suitable manner alternative fuel search plays crucial role in India. Developing countries like India have a big fleet of old vehicles still running on their roads which usually emitting comparatively higher emissions

compare with latest vehicles.

Emissions from older vehicles need to be cut down, in order to satisfy the contemporary emissions legislations, using one or early emission reduction techniques (Shukla

et

al. 2015). 1.5.2 Food-Fuel Conflict Pro-biofuels experts declare that the impacts of biofuels can be covered by two major type of technological innovation. World countries can be focusing more on cellulosic biofuel technologies that use waste products or food spin-offs and other crops to create biofuels. Technologies

are

essential for this option be possible are just starting to become available but are comparatively expansive. Similarly, investments in existing technologies increasing the agricultural productivity could mitigate the biofuels impact specially well-organized with technologies for cellulosic biofuel production.

14 The selection is not necessarily between food and fuel. But, the exception of this struggle is mainly depends on each country's willingness in new skills. 1.6

NECESSITY OF ALTERNATIVE FUEL Major attention is taken care to produce biodiesel for a promising alternative to diesel, due to its renewable, biodegradable, sustainable, carbon neutral, nontoxic gases release and environmental friendly characteristics.

With or without major modification, biodiesel can be directly used in a diesel engine in replacement of diesel fuel, which can reduce importing oil requirement of the country (Subramaniam et al. 2013). Mofijur et al. (2013) reported that the use of biodiesel in an internal combustion engine resulted in a reduction of unburned hydrocarbon (HC), particulate matter (PM) and carbon monoxide (CO) emissions. At the same time, the nitrogen oxides had (NOx) slightly increased its dependence on the nature of oil and engine type. The chemical and thermo physical properties of biodiesel were entirely different from the fossil fuel, which resulted in the variation of the combustion characteristics. The cetane number of the biodiesel was higher than that of diesel; it gives shorter ignition delay time. The viscosity of biodiesel was 1.5 to 2.5 times higher than that of diesel, which resulted in the increase of the combustion duration. 1.7

VEGETABLE OIL SELECTION Bio fuels produced from vegetable oil and animal fats such as rapeseed, soybean, sunflower, safflower, cotton seed oil, peanut, jatropha, coconut, palam kernel, neem oil, Honne oil, cardinal oil, rubber seed oil, orange oil, mahua oil, koroch oil, karanja oil, cotton seed oil, waste cooking, waste plastic oil, tallow and lard (

Kwon et al. 2015 and Subramaniam et al. 2013).

The present research work focuses on producing the biodiesel from

15

waste, useless seeds like watermelon, ceibia pentandra seeds crushed in the oil expeller by extracting in the

oil, and pine oil to meet the requirement of the alternative fuels because of

limited

research and publications. 1.7.1

Watermelon Seed Oil The seed were extracted from the fruits, washed and dried in an oven at 70º C for

a period of 48

hours for easy removal of the husk (Rao et al. 2012) The oil

is

extracted in the laboratory using soxhlet extractor and hexane as solvent. Finally ground seeds dried on oven at

a temperature of 100

º C for two hour were subjected to extraction with hexane for a period of 24 hours.

The

removal of solvent by rotary evaporation and drying afforded oil and yield is calculated on dry weight basis (Adebanjo & Kehinde 2013). The watermelon fruits and seed as shown in the Figure 1.5 & Figure 1.6. Figure 1.5 Watermelon fruits Figure 1.6 Watermelon seeds 1.7.2 Ceiba Pentandra Seed Oil The ceiba pentandra pods were gather together from Palamedu village, Namakkal district Tamilnadu, India. In the agriculture land, it has been grown naturally. In each fruit, ceiba pentandra seed occupy nearly 35- 42% (W/W). To remove the moisture the collected seeds were dried in

16 the sun light for two (or) three days depending upon the sunlight. To extracting the oil with the help of steam treatment process shadowed by mechanical crushing process. The oil seed yield is producing on average 2850 kg/ha. Ceiba pentandra seed were low feeding value due to its higher fiber content. Moreover, the opening of kapok (ceiba pentandra) fiber as bio- ethanol feed stock.

It

found that kapok fiber contains 34 to 64% of cellulose higher potential to produce cellulosic ethanol. According to the Tradition kapok fiber is utilized as stuffing material for pillows and bed (Tye et al. 2012, Silitonga et al. 2013). The

ceiba pentandra pods in tree, and seed as shown in the Figure 1.7 and Figure 1.8. Figure 1.7 Ceiba pentandra pods in tree

Figure 1.8 Ceiba pentandra seeds 1.7.3 Pine Oil Pine oleoresin, the raw material for pine tree synthesis, is tapped from pine tree and is subjected to steam distillation, before being treated with acids to synthesis the required pine oil. In general, pine oil, by its appearance and nature, can be regarded as one of the less viscous fuels in the likes of eucalyptus oil, ethanol and methanol. Chemically speaking, pine oil, an

17 alicyclic hydrocarbon, consists of mainly cyclic terpene alcohols, known as terpineol ($C_{10}H_{18}O$) along with alpha-pinene ($C_{10}H_{16}$). Further from its molecular structure, it is evident that it possesses inherent oxygen, with lower molecular weight and shorter carbon chain length than diesel (or) biodiesel. Notably, pine oil, despite being a higher alcohol, does not produce any cooling effects like methanol and ethanol as the latent heat of vaporization of it is not higher like lower alcohols 1.8 TECHNOLOGY USED IN THE CONVERSION OF SVO INTO BIODIESEL The following techniques are available for

the conversion of SVO in to bio-diesel. Micro-emulsion Thermal cracking Preheating Blending Transesterification 1.8.1

Micro-emulsion To solve the problem of high viscosity of the vegetable oils, micro- emulsions with solvents such as methanol, ethanol and 1-butanol have been investigated. A micro-emulsion is defined as a colloidal equilibrium dispersion of optically isotropic fluid microstructure with dimensions in the 1-150 Nano meter range, formed spontaneously from two normally immiscible liquids and one or more ionic immiscible. They can improve spray characteristics by explosive vaporization of the low boiling constituents

18 in the micelles. A brief study shows that the performance of micro emulsions of aqueous ethanol in vegetables oil was nearly as good as that of diesel, inspite of lower cetane number and energy content (Balakrishna 2012 & Subramaniam et al. 2013). 1.8.2 Thermal Cracking (Pyrolysis) Pyrolysis can be defined as the conversion of one substance into another by means of heat in the absence of air (or oxygen) or by heat in the presence of a catalyst which results in the cleavage of bonds and formation of a variety of small molecules. The thermal cracking of vegetable oil to produce bio fuels has been studied and found to produce alkanes, alkenes, alkadienes, aromatics and carboxylic acids in various proportions (Balakrishna 2012 & Subramaniam et al. 2013). 1.8.3 Preheating Preheating the vegetable oils prior to injection can reduce the viscosity of vegetable oils. By preheating the vegetable oils about 55˚C, the viscosity becomes almost equal to that of diesel. This will improve the spray characteristics of the fuel. Preheated vegetable oils results in improved performance with a reduction of emissions (Balakrishna 2012 & Subramaniam et al. 2013). 1.8.4 Blending A mixture of 10% vegetable oils is use to run engine without any modifications. At present, it is not practical to substitute 100% vegetable oil in diesel engines, but a blend of 20% vegetable oil and 80% of diesel fuel can be used. Some short-term experiments are conducted with 50% blend of Jatropha oil in diesel engine without any major operational difficulties, but

19 further study is required for the long term durable operation of the engine. Direct use of vegetable oil and the use of higher percentage of blends of oil have generally been considered not satisfactory for both direct and indirect diesel engines. High viscosity, acid composition, free fatty acid content, as well as gum formation due to oxidation, polymerization during storage and combustion, carbon deposits and lubricating oil thickening are some of the problems (Balakrishna 2012 & Subramaniam et al. 2013). 1.8.5

Transesterification Transesterification is the process of exchanging the alkoxy group of an ester compound by alcohol and the reaction often catalyzed by

an acid or base. Transesterification is important

for producing bio-diesel from bio- lipids. The transesterification of vegetable oil

is needed for reaction of triglycerides (fat/oil) with bio-alcohol to from ester and glycerol (Balakrishna 2012 & Subramaniam et al. 2013). 1.9 COMPRESSION IGNITION ENGINES Diesel engines are main workhorses for heavy-duty vehicles because of their fuel economy and durability. They have higher thermal efficiencies, resulting from high compression ratio and lean fuel combustion. Diesel engines are the backbone of the world industries with high torque. They dominate significant sectors like road, train transport, agricultural, military, mining, construction, propulsion, maritime and stationary electricity production (Akarte 2004). 1.10 PRESENT ISSUES IN FOUR STROKE DI-CI ENGINE There have been continuous researches carry out for the most efficient prime movers to be invented right from the beginning, which will do

20 less harm to the environment. The research and development mainly focuses on the fuel economy and the exhaust emissions of the four stroke compression ignition engines. The high levels of smoke and oxides of nitrogen (NO x) emissions make the diesel engines difficult to pass through the strict emission norms. The high level of smoke emissions is due to the diffusive combustion of diesel engine, whereas oxides of nitrogen emissions are mainly due to high combustion chamber temperature and dissociation. It is very difficult to control simultaneously both the smoke and oxides of nitrogen (

NO x)

emission in a diesel engine due to their trade off. 1.11 ORGANIZATION OF THESIS The thesis is organized in to six chapters: Chapter 1 introduces the essential

and need

of this research work. Chapter 2 brings out the review of literature related to bio diesel production, fuel properties, advantages and disadvantages

of

bio-diesel, engine performance, emission and combustion analysis of different types of vegetable esters. The Chapter 3 explains the objectives and research methodologies adopted throughout the study for achieving the set objectives. Chapter 4 brings out the description of bio diesel production unit, instruments used for measuring the fuel properties, experimental engine set up, gas analyzer, smoke meter, data acquisition system and procedure for conducting experiments.

Chapter 5 demonstrates the results and discussion of various methods of bio diesel production techniques, properties of raw oil, bio diesel- diesel blend fuel properties, performance

emission and

combustion characteristics of the DI-CI engine fuelled with bio-diesel-diesel blends mixed with pine oil.

Chapter 6 concludes the major share of the research work and guidelines for future work.

21

CHAPTER 2 REVIEW OF LITERATURE 2.1 INTRODUCTION The literature survey of this research work has a discussion on

the present status of using bio diesel as the best suitable alternative fuel for diesel.

While for an instant touching the history in the wake of alternative fuels for compression ignition engines, it considers some of the current techniques for the production of bio diesel as well as some new technologies under the research.

This research work is primarily focused on the development of bio diesel production, fuel properties,

comparison of two types of biodiesel, and performance, emissions and combustion

characteristics of DI-CI engine fuelled with biodiesel-diesel blends. 2.2

HISTORICAL BACKGROUND In day to day life,

bio diesel production is not something new, since the method of using vegetable oil as a fuel

was invented in the year 1895.

In the year of 1900 the

Rudolf Diesel developed the first engine which operates with vegetable oil. The Rudolf Diesel used groundnut oil as fuel for the first engine. In 1911, Rudolf Diesel told that the diesel engine can be fed with vegetable oil and would substantially serve the agriculture development of the country. In the year 1912, Rudolf Diesel stated that “The use of vegetable oils as engine fuel a relatively might be unimportant today. But in a certain time period such oils may consider as significant resource like as crude oil and the coal tar products of the present time” (Babu & Devaradjane 2003). In the

22 20 th century, world energy use age has increased significantly due to the effect of industrialization, and

increased

in use age has resulted in inadequate petroleum reserves, such as in the 1970s oil crisis. All of those problems have motivated the scientific society to seek new, alternative energy sources that have decrease global warming and air pollution effects. The past experiences by Stuart staniford

was reported in the

year 2008.

The

energy production from 1970s to 2050 strongly supports the increasing trend of renewable energy consumption. The sustainability of biofuels is progressively promoting its wide acceptance and market demand will go up in the near future. Approximately 27% of transport fuel will be totally replaced by bio fuels by 2050 accordant with the international Energy Agency IEA by (Sanjid et al. 2014). Many researchers and scientists had tried out different types of biodiesel production process, from these fatty acid alkyl esters derived from transesterification of triglycerides, which are commonly known as biodiesel, are well accepted renewable alternatives of petroleum diesel. In last two decades, several studies from across the globe have reported successful operation of compression ignition (C.I) engines with biodiesel derived from a host of feed stocks and their blends with mineral diesel. 2.3 FATTY ACIDS IN THE VEGETABLE OIL Generally, vegetable oils such as canola oil, soybean oil, sunflower oil and corn oil are used as raw materials for biodiesel production. Because these oils contain relatively many unsaturated fatty acids, they can exist as liquid at room temperatures and show good fluidity at low temperatures. However, vegetable oils have little output and are expansive. In contrast animal fats such as beef tallow, lard and chicken oil animal fats are more stable than vegetable oils in oxidation and have higher heating values and cetane numbers, they have the drawback of being solids at room temperature. Thus depending on the fatty acids contend in the oil / fat used in biodiesel

23 production, the produced biodiesels can have different fuel properties. Oils/fats mainly consists of saturated fatty acids such as myristic acid (C 14:0), palmatic acid (C 16:0), stearic acid (C 18:0), and saturated fatty acids such as palmitoleic acid (C 16:1), linoleic acid (C18:2), linolenic acid (C18:3) oleic acid (C 18:1) (Hong et al. 2015).

Table 2.1 Common fatty acids Chemical structures S. No. Chemical Formula Structure (xx:y)

Fatty acid name Fatty acids Chemical name 1

C 18 H 30 O 2 18:3

Alpha-linolenic acid Octadecatrienoic 2 C 16 H 32 O 2 16:1 Palmitoleic Hexadec-9-enoic acid 2

C 20 H 44 O 2 20:0 Behenic Docosanoic 3 C 10 H 20 O 2 10:0 Capric acid Decanoic acid 4 C 18 H 32 O 2 18:2 Linoleic Cis-9,cis-12- Octadecadienoic 5 C 8 H 16 O 2 8:0 Caprylic acid n-octanric acid 6

C 32 H 42 O 2 22:1 Eurucle Cas-13-docosenoic 7 C 18 H 30 O 2 18:3 Linolenic Cis-9,cis-12,Cis-15-Octadecatrienoic 8 C 6 H 12 O 2 6:0 Caproicacid Hexanoic acid 9 C 24 H 48 O 2 24:0 Lignoceric

Tetracosanoic 10 C 18 H36O 2 18:0 Stearic Octadecanoic 11 C 14 H 28 O 2 14:0 Myristic Tetradecanoic 12 C 18 H 34 O 2 18:1

Oleic Cis-9- Octadecanoic 13 C 12 H 24 O 2 12.0 Lauric Dodecanoic 14 C 16 H 32 O 2 16:0 Plamitic Hexadecanoic 15 C 20 H 40 O 2 20:0

Arachidic Eicosanoic

Where, xx indicate carbonsnumbers and y the quantity of double bonds in the fatty acid chain

The diesel engine can also use

the unprocessed vegetable oil

with needed modification in the engine driving habits. In general vegetable oil

24 contains saturated hydrocarbon and Triglycerides which possess fatty acids. Vegetable oils

also have

different chemical structures and it is occupied with three fatty acids connected by glycerol molecules on ester linkage. The fatty acids vary depending on

the carbon chain length and its double bond amount. Table 2.1 represents the about the fatty acids with their chemical name, structural information and formula.

Vegetable oil contains a very high structure of fatty acids, so it

gives higher cetane number and it has less prone oxidation, due to its high percent of saturated fatty acid and free acids. 2.4

VEGETABLE OIL PROPERTIES Barnwal & Sharma (2005), Gashaw & Lakachew (2014) described that the vegetable oil fuel properties in Table 2.2. It suggests that the vegetable oil kinematic viscosity differs at 38 0 C with the range of 30-40 cSt .The

causes for

the high viscosity of these oils is primarily due to their large molecular mass in the range of 600-900. This is about 10-20 times higher than that of diesel fuel (Rakopouls 2013). The vegetable oil has very high flash point (above 200 0 C) and the heating value are in the range of 39-40 MJ/kg as compare to diesel fuel (about 44 MJ/kg). The presence of chemically bound oxygen in vegetable oil lowers their calorific values by about 10%. The range of cetane numbers in the vegetable oil is 32-40. The blends of diesel fuel with vegetable oils would solve the problems developed in the operation of C.I engine with pure vegetable oils. Diesel fuel dissolves

rather than the vegetable oils (Gashaw & Lakachew (2014), (Wang et al. 2006)).

25 Table 2.2 Properties of Vegetable oil (Barnwal & Sharma (2005); Gashaw & Lakachew (2014)) Vegetable oil Density (kg/l) Kinematic viscosity at 38

o C (mm 2 /s) Heating value (MJ/kg) Flash point (o C) Cloud point (o C) Pour point (o C)

Cetane no. Diesel 0.855 3.06 43.8 56 - -16 50 Palm 0.9180 39.6 - 267 31 - 42 Soya bean 0.9138 32.6 39.6 254 -3.9 -12.3 37.9 Sunflower 0.9161 33.9 39.6 274 7.2 -15.0 37.1 Corn 0.9095 34.9 39.5 277 1.1 -40.0 37.6 Sesame 0.9133 35.5 39.3 260 -3.9 -9.4 40.2 Cotton seed 0.9095 33.5 39.5 234 1.7 -15.0 41.8 Safflower 0.9144 31.3 39.5 260 18.3 -6.7 41.3 Crambe 0.9048 53.6 40.5 274 10.0

-12.2 44.6 Rapeseed 0.9115 37.0 39.7 246 -3.9 -31.7 37.6 Linseed 0.9236 27.2 39.3 241 1.7 -15.0 34.6 Peanut 0.9026 39.6 39.8 271 12.8 -6.7 41.8 2.5 APPROPRIATE

Table 2.2 clearly proved that using vegetable oil

converted in to the bio-diesel is to replace the partial requirement of

diesel because of the following appropriate 1.

Energy density has similar (or) nearby to fossil diesel fuel. 2. Calorific values of different vegetable oils are approximately 90% that of petroleum diesel fuel. 3.

Cetane number is similar or close to

that of diesel fuel. 4.

Compared with

diesel, the normal chain length for plant oils is from 12 to 22 carbon atoms, with 0 to 3 double bonds that are responsible for the vegetable oil Physico-chemical properties.

26

Long chain saturated, unbranched hydrocarbons are especially suitable for conventional diesel fuel. The long, unbranched hydrocarbon chains in the fatty acids meet this requirement. (

Barnwal & Sharma (2005); Gashaw & Lakachew (2014)) 2.6

EFFECT OF ENGINE CHARACTERISTICS USING STRAIGHT VEGETABLE OIL IN COMPRESSION IGNITION ENGINE. It has been found that natural vegetable oil can be used as a alternative for diesel fuel in C.I engine but it has the complying problems,

as

in the main first

it have phospholipids, sterols , water,odorants and other impurities. The injection process, leads to poor atomization which may be the reason of high viscosity. The high flash point attributes to lower volatility.

Vegetable

oil’s has

improper air fuel mixing that produces

incomplete combustion contributed heavy smoke emission.

Injection nozzle failure. High carbon deposits Lubricating oil dilution

vegetable oil‘s

could and pour points are significantly higher than those of petorelum diesel. These high values may do problems in cold weather. Dragged engine liner. Ring

is beyond a surface.

27 In C.I engine, the SVO characteristics of injection,atomization and combustion are significantly different from the petroleum fuel due to

poor physical properties.

Due to above stated problems the SVO are in need of modification.

To solve these problems, the oil is in need of small

chemical modification. These problems can be cleared if the vegetable oil are chemically changed in to bio-diesel, which has similar character to diesel

reported by Lokanatham & Ravindranath (2013); Balakrishna (2012); Balat & Balat (2008). 2.7 TECHNOLOGY USED IN

THE CONVERSION OF SVO IN TO THE

BIODIESEL In common, micro-emulsion, thermal cracking, preheating, blending

and transesterification are the main methods available for the conversion of SVO in to biodiesel. Amongst, the

common systematic way to treat transesterification is SVO. 2.7.1

Transesterification Transesterification is the method of replacing the alkoxy group of an ester compound by alcohol and the chemical reaction was frequently catalyzed by a base. Transesterification

plays a vital role

for producing bio- diesel from bio-lipids. The transesterification of vegetable oil implicates reaction of triglycerides (fat/oil) by alcohol to from ester and glycerol (Subramaniam et al. 2013). 2.7.1.1

Transesterification reaction Transterification, also

named as alcoholics. It is the method of replacing

alcohol from an ester by some other alcohol, which resembles to

28 hydrolysis anticipate before that an alcohol is used as a substitute of water. This has been usually applied to cut down the viscosity of the triglycerides. The transesterification is denoted as RCOOR’ + R’’OH R’COOR’’ + ROH ESTER ALCOHOL ESTER Alcohol (1) Equation (1)

demonstrates the

transesterification process. The methanol is used in this process, so it is called methanolysis. Methanolysis of triglycerides is represented in Equation (2). CH 2 -COOR 1 CH 2 OH R 1 COOCH 3 CH-COOR 2 + 3C H 3 OH CH OH + R 2 COOCH 3 CH 2 -COOR 3 CH 2 OH R 3 COOCH 3 Vegetable Oil Alcohol Glycerol Methyl-ester (2) Equation (2) Methanolysis of Tri-glycerides Transesterification

process is probably considered as

one of the reversible reactions and proceeds basically by mixing the reactants. But, the presence of a catalyst (a strong acid or base) speed up the conversion (Adewale et al. 2015). 2.7.1.2 Chemistry of transesterification process The Equation (2) denotes the complete transesterification reaction. Though; three successive

and reversible reactions are conceived to occur. These reactions are indicated in Equation (3).

29 ROH + Triglycerides R1COOR + Diglycerides ROH + Diglycerides R2COOR + Monoglycerides ROH + Monoglycerides R3COOR +

Glycerol (3) Equation (3) Chemistry of transesterification The first step transition presents the triglycerides to diglycerides, adopted by the conversion of diglycerides to monoglycerides controlling one methyl ester molecule from each glycerides at each step

reported by Meher et al. 2004. 2.7.2

Variables Affecting Transesterification Process The process of transesterification brings about drastic change in viscosity of the vegetable oil. The high viscosity component, glycerol is removed and hence the product has low viscosity like the fossil fuel. The biodisel produced is totally miscible with mineral diesel in any proportion. Flash point of the biodiesel is lowered after transesterification and the cetane number is improved. The yield of bio-diesel in the process of transesterification is assected by several process parameters which include; presence of moisture and free fatty acid (FFA), reaction time, reaction temprature, catalyst, molar ratio of alcohol and oil & mixing intensity 2.7.2.1 Reaction temperature The rate of reaction is strongly affected by the reaction temperature.

When the reaction temprature is high, the viscosities of oil is decreased and the outcomes with an increased rate of reaction needs larger amount of enegry

for the reaction to happen. Hence, the percentage of biodiesel production is raised. However, the temperature of reaction must be less than the alcohol boiling point (boiling point of methanol is at 60 ˚ C-70 ˚ C at

30 atmospheric pressure) to ensure the alcohol will not be lost through vaporization. Also, the yield of bio-diesel decreases if the reaction temperature goes beyond its optimum level because a higher reaction temperature will accelerate the saponification reaction which results in a lower yield. Depending upon the type of oil, the maximum yield is obtained at temperature varies from 60 ˚ C

to 80 ˚

C . (Amit Agarwal et al. 2015). 2.7.2.2 Catalyst concentration Catalyst concentration can affect the yield of the bio-diesel production. As mentioned earlier, basic catalysts are usually preferred to acid catalysts because of the higher reactivity and the lower process temperature required. The sodium hydroxide is less effective than sodium methoxide because of the decreased amount of water produced upon mixing methanol with sodium hydroxide. As the concentration

of catalyst increased, the conversion of triglycerides and the yield of methyl esters

also increased. A concentration of NaOH in the range of 1.0-1.4%(W/W) has been found to produce 90-98% conversion from jatropha oil to methyl ester. As for the concentration of KOH ranging from 0.55 to 2.0% (W/W) 95-99% of jatropha yield of bio-diesel has been obtained. However, the yield of

bio-diesel is

reduced if the alkali catalyst were added above their optimum concentration as this causes more soap formation (Amit Agarwal et al. 2015). 2.7.2.3 Methanol to oil molar ratio

Alcohol to triglyceride is one of the most important factor for affecting the bio-diesel yield.

Stoichiometrically 3 moles of alcohol and 1 mole of triglyceride are needed for transesterification to yield 3 moles of fatty acid methyl ester and 1 mole of glycerol is used. It varied from 5.6

to 7.8:1 for both the catalyst systems. Bio-diesel

yield could be elevated by introducing an excess amount of methanol to shift the equilibrium to the right

31 hand side. Ethanol, propanol, methanol, butanol, and any alcohol can be used in the transesterification reaction, amongst these alcohols methanol

has physical and chemical advantageous (shortest chain and polar alcohol) and the cost is also low. At 1:6 oil/methanol results exhibit highest methyl ester yield is nearly 99.5% (Gashaw & Teshita 2014; Leung et al. 2010). 2.7.2.4 Reaction time The conversion time increases with the rate of fatty acid esters also increase. In the starting stage

the reaction is slow due to the mixing and dispresion of alcohol in to oil. After some time the reaction

continues in a very fast manner. In general, within 90 mins the yields reached

a maximum reaction time. In addition that the excess reaction time will result

to a reduction in the product yield due to the backward reaction of transesterification, resulting in loss of ester as well as causing more fatty acids to form

soaps. (Gashaw & Teshita 2014; Leung et al. 2010). 2.7.2.5 Free fatty acid and water content The FFA and moisture contents have significant effects on the transesterification of glycerides with alcohol using catalyst. The high FFA content (<1% W/W) will happen soap formation and the separation of products will be exceedingly difficult, and as a result, it has low yield of

bio- diesel

product. In addition, formation of gels and foams hinders the separation of glycerol from bio-diesel. For instance, water content in waste cooking oil will stimulate the hydrolysis reaction and simultaneously reduce the amount of ester formation. To overcome this problem, supercritical method

is

proposed. It may be noted that water has less influence. In supercritical methanol method. Therefore water substance should not always exceed 0.5% to obtain 90% yield of biodiesel and it is more crucial for an acid-catalyzed reaction than base catalyst reaction. The moisture levels of the collected waste

32 chicken fats vary widely, being as high 18%. Therefore, it is not possible to convert these oils

in

to biodiesel by using single process. One drawback of bio-diesel is that there is an inverse relationship between

bio-diesels

oxidative stability and its cold flow properties. Saturated compounds are less prone to oxidation than unsaturated compounds but they rise the cloud point of the fuel. The reaction of FFAs with alcohol produces ester, but also water that inhabits of the transesterification glycerides. This is due to the effect of the water produced when the FFAs react with alcohol to from esters. The coincidence of the lines indicates that water formation is the primary mechanisms limiting the completion of the acid catalyzed esterification reaction with FFAs (Gashaw & Teshita 2014). 2.7.2.6 Mixing intensity Oil and alcohols are not totally miscible, thus reaction can occur in the interfacial region between the liquids and transesterification reaction is a moderately slow process. So mixing is very important in the transesterification process, adequate mixing between these two of feed stock is necessary to enhance the transesterification reactions to occur. Mechanical mixing is commonly used in the transesterificarion process. The intensity of the mixing could be varied depending on its necessity in the transesterification process. In general, the mixing intensity must be increased to ensure good and uniform mixing of the feed stock. When vegetable oil with high kinematic viscosity are used as the feed stock, intensive mechanical mixing is required to overcome the negative effect of viscosity to the mass transfer between oil, alcohol and catalyst. Agitation speed plays an important role in the formation of end product (mono alkyl ester (or)

bio-diesel),

because agitation of oil and catalyst mixture enhances the reaction. For example the mixing intensities chosen were 200rpm, 400rpm, 500rpm, 600rpm and 800rpm for 60 minutes while other parameters were kept

33 constant, because lower stirring speed shows lower product formation. On the other hand, higher stirring speed favors formation of soap (

Gashaw &

Teshita 2014). 2.7.3 Advantages and Disadvantages of Transesterification Process The transesterification procedure has its

advantages and disadvantages can be completed by using three different methods like chemical catalysts, enzymatic catalyst, and supercritical fluid techniques are presented in Table 2.3 (Huang et al. 2010). Table 2.3 Advantages and disadvantages of transesterification process

S. No Type of transesterification Advantages Disadvantages 1 Enzymatic catalysis (i) The small amount of methanol required in reaction. (ii) Moderate reaction condition. (

iii) In natural environment it have no pollution. (i) The presence of chemical

in the production process are poisonous to enzyme. (ii) In the conversion of fatty acid short chain enzyme is one of the limitations. 2

Chemical catalysis (i) Large scale production. (ii)

Reaction condition can be well controlled. (iii) The methanol produced in the process can be recycled. (

iv) The cost of production process is low. (v) The production has

higher conversion. (i) Reaction temperature is relatively high and process is complex. (

ii) The process

requires more energy. (

iii) The methanol recycles process needs an separate installation. (iv) The later disposal process is complex. (

v)

The waste water pollutes the environment. 3 Supercritical fluid techniques (i) Safe and fast. (

ii)

Easy to be controlled. (

iii)

Friendly to environmental. (i) High cost of production and waste energy due

to high temperature and high pressure in the reaction.

34 2.8 STUDIES ON CONVERSION OF VEGETABLE OIL IN TO BIO-DIESEL

Kafugu et al. (2010) stated that in moringaoleifera oil the maximum yield

of biodiesel

was 82%, when the 1wt% of potassium hydroxide and 30wt% of methanol reacted at thetemperature, time, andstirrer speed of 60˚C, 60min and 400rpm. It was found that for the above consideration if more than 1% catalyst

would be

added then there is

no clear separation, solubility of methanol increased and more soap was developed during washing. The cause for that is hydrolysis of ester had more fatty acid. Atapour &

Kariminia (2011) proposed that

Iranian bitter almond oil was carried out using methanol to oil molar ratio varies from 3:11 to 11:1. When the reaction temperature fluctuated from 30˚C to 70˚C the KOH concentration range varied from 0.15% to 1.8%wt , and the optimal value of methanol, KOH, temperature and yield were 7:1, 0.9, 50˚C and 97.2% to 90.8%. When the amount of methanol used was high, it would acquire glycerol as a by-product. The

not used up

part of the catalyst becomes soap increase saponification reaction and more than 50˚C temperature large number of bubbles produced by vaporization of methanol. Kaya et al. (2009) declared that the Crude peanut seed oil 89% of bio-diesel

was

obtained at the reaction temperature and time of 65˚C, 120 min with 0.5 wt. % of NaOH by using methanol. Lin et al. (2009) had created bio-diesel

in a two-step process by using crude rice bran oil. The first step procedure

is pretreatment. If processes Free Fatty Acid (FFA) when the values were reduced by using an acid catalyst of H_2SO_4 with less than 1mg and 1%w/w. The alkaline transesterification process

was

the second step. In this process 98.7% bio- diesel yield was got at a temperature and reaction time of 60˚C and 60min.

35 Whereas the methanol/oil ratio is 6:1, and the KOH concentration was 0.9%w/w. Anwar et al. (2010) stated that in okra seed oil the bio-diesel yield was 96.8%. In this methyl ester four distinct type of catalyst were used like sodium meth oxide(NaoCH 3), sodium hydroxide(NaOH), potassium meth oxide(KOCH 3), potassium hydroxide(KOH),. The maximum yield was got from

the

meth oxides (NaOCH 3 ,KOCH 3). Whereas methanol/oil molar ratio was 7:1 and the NaOCH 3 Concentration 1%w/w by the reaction temperature and time of 65˚C, 120min. From the above condition, okra methyl ester yield was 96.8%. Suppalakpanya et al. (2010) specified that crude palm oil ester content was received with 1.5% wt. KOH, and the molar ratio of oil to ethanol was 1:8.5. From the same content the bio-diesel production was increased up to 98% wt. by the compression of conventional heating and microwave heating at 70˚C. Higher reaction rate is obtained from lowest enzyme concentration 0.5, and the methanol to oil molar ratio was increased (1:1 to 1:25) as well as yield of bio-diesel was also increased (up to 1:15m). Dhar et al. (2012) has developed bio-diesel by two-step process

method from neem oil. The first process method is esterification and the other process method was

transesterification process. In esterification process method, sulphuric acid was used as a catalyst with neem oil. As a result 20% FFA was reduced.

The

Second step was transesterification process method, in that process, the oil to alcohol molar ratio was 1:6 and the wt of NaOH was 4.5%.with the catalyst reaction time and temperature of 90 min and 45˚C, using gravity separation method product

was kept in to a separating funnel up to 5

to 6hrs. Optimum reaction conditions were 1% NaOH catalyst methanol to oil molar ratio was 6:1 and reaction temperature 60˚C.

36 Qiu et al. (2011) stated that biodiesel could be produced from mixed soybean oil and rapeseed oil.

In that process the homogeneous processes were in solid state like

NaOH and KOH. It will react 4000 times quicker than acid catalyst reaction. The acid catalyst was obtained by using a co-solvent n hexane, which

was

given a pre-treatment of soyabeen oil and rape seed oil. The benefit of this method was lower reaction temperature at 55˚C. The optimum reaction temperature, molar ratio of oil to methanol, catalyst reaction time and yield of bio-diesel was 55˚C, 5:1,08wt%, 120min, and 94% of yield. Karavalakis et al. (2011) also suggested that use of the homogeneous catalyst like KOH, NaOH soap was formed and the hard of removing the catalyst species after reaction required a big amount of water used to separate and clean the glycerol and the esters. New catalyst and environmentally began processes of tetramethylghanidine optimum catalyst amount of 3wt% molar ratio of methanol to oil of 12:1, reaction time was 90min, yield is 93.8%. Sodium (or) potassium based catalyst required intermediate two stage reaction, increase glycerol and removing time from waste frying oils. 2.9

FUEL PROPERTIES OF ESTERS (BIODIESEL) Giakoumis (2013) reported that the following

physical and chemical properties of biodiesel from various feed stocks were analyzed; a lot of data’

s

was carefully collected from

various papers published in international Journals, Conferences and

the

reports issued by well-known research centers. Mostly, the whole electronic databases of IEEE, Elsevier, American Chemical Society, Taylor and Francis, Wiley International, Springer and SAGE Publishing were scanned. For exciting papers, amount of data sets contributed from papers presented in ASABE, SAE International and various other indexed conferences around the world.

As a final point, 26 feed

37 stocks

were designated for the statistical investigation, namely four animal fats (beef tallow, chicken, lard and fish), twenty-one oils derived from various plants (canola, castor, coconut, corn (maize), cottonseed, croton, hazelnut, karanja (honge), jatropha, linseed (flax), mahua, neem, olive, palm, peanut, rapeseed, rice bran, rubber seed, safflower, soybean and sunflower), as well as waste cooking oil. These 26

feed stocks finally

provided 945 different data series of biodiesel properties and 700 data series of fatty acid compositions. On the other hand, the dataset contains a good junk of American, European and Asian animal or vegetable

feedstock-both edible and non-edible

as tabulated in Table 2.4. It is also attractive that soybean methyl ester or methyl soyate (SME) had been investigated.

in general

worldwide (although the majority of the research had been conducted in America) and with a continuous interest over the last three decades

in oils included recently.

Rapeseed and the closely related canola (10.4+3.1=13.5%) were the next best familiar feedstocks, with a significant interest also found in palm (7.8%), the non-edible jatropha (6.9%; mainly during the last years in India), sunflower (5.6%) andwaste cooking (9.5%). On the other hand, all animal fats (including fish) are of the order of 8.1%, with the majority of the research (50%) focused on beef tallow. The non-edible cotton seed with 3.1% (more popular in the Mediterranean countries),

karanja with 3.8% (primarily during the last few years), as fine as rice bran (2.5%), castor (3.3%), coconut (2.4%), corn (used for ethanol production too 2.0%),safflower (1.8%) linseed (1.9%), and neem and mahua (each 1.5%) comprise smaller but still important shares of the world wide bio-diesel research various other feedstocks such as grape seed, tobacco seed, jojoba (simmondsia chinensis), babassu (orbignya oleifera), polanga/punnai (calophyllum inophyllum), sesame (sesamum indicum)

chinese pistache (pistachio chinensis), hemp (cannabis sativa), and mustard were not in the

part of

statistical analysis.

38 Table 2.4 Physical and chemical properties of all biodiesels

Density (kg/m3) Viscosity (mm2/s HHV (kJ/kg) LHV (kJ/kg) CFP point (˚C) Pour point (˚C) Cloud point (˚C) Flash Point (˚C) Cetane number Oxidation Stability (h) Iodine number Distillation temp.50% vol. (˚C) Distillation Temp.90% Vol.(˚C)

Sulfur content (ppm) Acid number (mg KOH/g)

Molecular weight (kg/kmol) O (%

w/w) H (% w/w) C (% w/w) Castor 917.6 (6.98) 14.52 (1.78) 39,900 (1926) 37,630 (243) 5.9 (18.5) -23.4 (4.0) -15.1 (12.5) 160.9 (37.5) 42.1 (6.83) 12.9 (6.1) 85.2 (3.3) 327 (8.5) 381.6 (23.4) 0.5 (0.45) 0.39 (0.18) 270 _ _ _ Chicken fat 876.3 (6.37) 4.81 (0.50) 39,890 (405) 37,610 2.7 (0.6) 4.40 (1.8) 7.5 (1.3) 162.2 (9.8) 57 (5.66) 8.7 (3.21) 78.7 (1.30) _ _ _ 0.32 (0.15) _ _ _ _ Beef tallow 874.3 (3.82) 4.83 (0.50) 40,040 (216) 37,220 (274) 12.2 (2.3) 10.0 (2.0) 13.8 (1.8) 157.2 (8.6) 60.9 (5.55) 0.45 (0) 54.5 (10.3) 329.2 (3.5) 344.2 (6.6) 14.3 (5.5) 0.30 (0.18) _ 11.35 (0.39) 12.60 (0.81) 76.09 (0.69) Canola 881.6 (3.51) 4.40 (0.25) 39,975 (150) 37,980 (860) -8.8 (3.3) -8.0 (2.6) -1.8 (1.4) 159.0 (14.0) 54.8 (3.37) 11.0 (0.95) 104.0 (4.6) 352.0 (1.7) 354.3 (4.9) 2.2 (1.2) 0.22 (0.14) _ 11.04 12.84 _ Cotton seed 879.0 (5.49) 4.70 (0.88) 40,480 (435) 38,175 (1135) 4.8 (2.5) -0.2 (4.1) 1.2 (4.4) 165.4 (14.0) 53.3 (2.75) 1.8 105.7 _ 349.5 (9.1) 5.3 (4.2) 0.29 (0.18) _ 11.21 (0.62) 11.89 (0.16) 76.86 (0.77) Karanja 882.9 (5.50) 5.04 (0.60) 40,275 (671) 36,490 (426) -7.0 2.5 (3.4) 7.6 (4.3) 163.6 (14.7) 55.4 (2.49) 4.1 (2.44) 85.5 (2.2) 334 345.4 (11.1) 13.5 (9.2) 0.44 (0.05) _ 11.51 (0.96) 11.95 (0.70) 76.35 (1.51) Coconut 870.8 (4.71) 2.78 (0.26) 38,985 (1369) 35,985 (921) -4.7 (0.6) -3.8 (1.2) -1.2 (3.9) 127.7 (29.1) 61 (6.46) 11.0 (5.64) 7.8 (2.5) _ _ 3.3 (1.3) 0.16 (0.11) _ _ 11.65 (0.50) 72.75 (0.07) Croton 883.2 (4.95) 4.48 (0.28) 40,280 37,825 (1578) 11.0 -6.3 (4.6) -4.0 174.5 (20.5) 50.6 (5.66) 4.0 142 352 358 _ 0.31 (0.26) _ 10.20 11.97 77.83 Fish 887.3 (7.15) 4.30 (0.33) 40,550 (955) 38,800 (1131) -3.0 (3.7) -4.0 (0) -0.3 (2.41) 162.6 (8.8) 51 (1.52) 8.2 (3.46) 142.6 (13.81) 339 360.2 (18.9) 5.7 (4.2) 0.39 (0.35) _ _ 11.85 77.40 Lard 873.0 (2.25) 4.89 (0.11) 39,950 (212) 36,910 9.0 (4.2) 11.0 15 153.5 (9.2) _ _ 64.5 (11.4) 329 337 _ 0.36 (0.26) _ 11.71 12.11 76.01 corn 882.2 (4.71) 4.32 (0.37) 40,190 (826) 38,600 (69) -7.5 (3.2) -5.1 (2.5) -3.0 (1.6) 165.7 (5.9) 52.5 (2.64) 3.0 (1.34) 120 (16.9) - 345.8 (7.0) 3 (2.1) 0.19 (0.06) _ 10.98 (0.03) 11.52 (0.54) 76.71 (0.56) Jatropha 878.7 (6.52) 4.72 (0.56) 40,380 (1259) 38,050 (956) -1.2 (3.9) -0.9 (4.7) 5.7 (4.3) 158.5 (19.5) 55.7 (2.84) 5.0 (1.88) 99.0 (5.4) 333.3 (0.6) 341.2 (12.1) 12.9 (15.4) 0.34 (0.12) _ 11.32 (0.66) 12.21 (0.28) 76.57 (0.37) Hazelnut 877.9 (15.2) 4.55 (0.91) 39,800 (469) 37,230 -10.2 (5.4) -14.0 (1.4) -12 (3.8) 163.5 (30.8) 53.8 (1.70) 53.8 (1.70) 7.6 92.5 (2.1) _ _ 0.22 _ _ 11.94 76.57

39 Table 2.4 (Continued)

Density (kg/m3) Viscosity (mm2/s HHV (kJ/kg) LHV (kJ/kg) CFP point (˚C) Pour point (˚C) Cloud point (˚C) Flash Point (˚C) Cetane number Oxidation Stability (h) Iodine number Distillation temp.50% vol. (˚C) Distillation Temp.90% Vol.(˚C)

Sulfur content (ppm) Acid number (mg KOH/g)

Molecular weight (kg/kmol) O (% w/w) H (% w/w) C (% w/w) Sunflower 882.9 (4.70) 4.53 (0.51) 40,000 (647) 37,800 (895) -3.5 (2.0) -3.8 (2.3) 0.9 (2.4) 172.0 (7.9) 51.9 (4.73) 1.3 (0.42) 128.6 (2.1) 355.7 (7.2) - 356.5 (2.1) 0.32 (0.14) 294.05 (0.50) 10.98 (0.15) 11.84 (0.29) 76.90 (0.37) Palm 874.7

(4.01) 4.61 (0.61) 39,985 (688) 37,080 (855) 11.4 (4.7) 11.8 (2.4) 13.3 (2.3) 161.9 (17.3) 61.2 (4.90) 11.4 (2.38) 52.7 (5.6) 327.7 (4.0) 339.3 (6.7) 3.1 (2.7) 0.27 (0.11) 284.12 (0.14) 11.27 (0.43) 12.44 (0.50) 76.09 (0.37) Neem 874.5 (7.54) 5.06 (0.54) 40,180 (879) 36,880 (53) _ 4.0 (2.2) 4.0 (1.0) 150.6 (24.1) 56.9 (6.07) 2.1 70.8 (13.1) _ _ _ 0.41 (0.09) _ 10.72 12.10 76.7 Soybean 882.8 (5.12) 4.29 (0.37) 40,020 (671) 37,750 (944) -3.6 (3.4) -3.0 (2.7) 0.1 (2.0) 158.8 (19.5) 51.8 (3.93) 5.0 (2.59) 126.0 (6.9) 336.3 (6.1) 345.0 (6.6) 2.7 (3.8) 0.32 (0.28) 290.83 (2.17) 10.95 (0.52) 11.90 (0.31) 77.03 (0.50) Mahua 874.5 (7.54) 5.06 (0.54) 40,180 (879) 36,880 (53) _ 4.0 (2.2) 4.0 (1.0) 150.6 (24.1) 56.9 (6.07) 2.1 70.8 (13.1) _ _ _ 0.41 (0.09) 294.4 _ _ _ Rice bran 880.9 (5.71) 4.70 (0.44) 40,475 (1201) 38,045 (1335) 0.3 (2.4) -0.9 (2.7) 5.0 (4.8) 157.8 (23.3) 56.3 (5.49) _ 91.8 (13.0) 336.0 346.0 (1.7) 5.3 (4.7) 0.28 (0.18) _ 11.26 12.38 76.22 Rapeseed 882.2 (4.06) 4.63 (0.37) 40,335 (743) 37,625 (663) -11.3 (5.9) -9.7 (3.4) -3.3 (2.4) 164.4 (15.5) 54.1 (3.32) 7.4 (1.81) 111.7 (7.4) 340.7 (10.1) 344.3 (4.6) 4.6 (3.6) 0.24 (0.11) 294.80 10.93 (0.48) 11.84 (0.46) 77.07 (0.49) Peanut 882.9 (2.62) 4.77 (0.26) 39,930 (205) 38,050 16.3 (0.6) -2.7 (8.4) 4.3 (3.8) 174.5 (5.9) 54.9 (2.74) _ 80.5 (15.1) _ _ 5.9 (7.8) 0.19 (0.13) _ _ _ _ Waste cooking 880.6 (6.04) 4.75 (0.46) 39,805 (607) 37,880 (1088) -2.8 (3.3) -0.3 (5.5) 5.3 (5.1) 161.7 (19.0) 56.2 (3.91) 5.0 (3.28) 85.1 (17.1) 340.4 (6.7) 350.3 (8.6) 8.6 (14.6) 0.41 (0.14) 289.31 (3.86) 10.77 (0.47) 12.02 (0.44) 76.90 (0.55) Safflower 883.8 (5.21) 4.10 (0.12) 40,155 (644) 38,145 (862) -8.3 (2.1) -8.1 (2.9) -4.9 (1.7) 169.9 (17.6) 51.8 (2.53) _ 136.7 (5.7) _ _ _ 0.32 (0.05) _ _ _ _ Olive 881.2 (1.56) 5.05 (0.39) 40,280 (1048) 37,290 -5.3 (3.8) -5.0 (1.7) -2.0 (0) 171 (6.5) 58.9 (2.00) 1.7 (2.33) 80.3 (3.2) _ _ 7.8 (3.1) 0.37 (0.33) _ _ _ _ Linseed 891.5 (3.61) 4.06 (0.20) 40,410 (676) 37,830 (1713) -7.7 (1.7) -8.0 (4.2) -1.7 (2.0) 170.3 (5.6) 51.3 (4.24) 0.40 (0.25) 184.5 (17.9) 338 361 8.2 (8.5) 0.29 (0.15) 290.07 (3.49) 11.29 (0.31) 77.75 (0.37) Rubber seed 882.3 (4.37) 4.79 (0.63) 40,350 (1018) 37,820 (807) -1.0 -7.0 (1.3) 3.6 (1.5) 158.3 (24.8) 50.4 (1.14) 7.2 (0.93) 139.7 (6.2) _ _ _ 0.22 (0.10) _ _ _ _ Source: Evangelos 2013, Physical and chemical properties of various biodiesel.

40 2.9.1

Viscosity Viscosity is a measure of the fluid resistance which is being distorted by either tensile stress

or shear. In the case of liquid fuels, the less viscosity of fluid makes high easier movement (fluidity). The diesel engine has higher viscosity which contributes to minimum of exact operation for the fuel injectors, and the lesser atomization of the fuel spray has high viscosity which stands for

that cannot be used carefully as fuels in a C.I.

engine. However, without earlier heating (decreases viscosity exponentially with temperature increasing), and only for moderately small mixing ratios.

A European stipulation says that an adequate viscosity of biodiesel range is 3.5 to 5 mm 2 /s and that is excluded from B100 form. In the US, the specification of the range is (1.9-6mm 2 /s), much high and the concept depicts all 25 analyzed feedstock's acceptable by Giakoumis (2013). 2.9.2 Density As comparedto petroleum diesel,

bio-

diesel fuels have high viscosity, which intends that with respect to volume the

fuel pumps will inject high mass of methyl ester than petroleum diesel fuel. These have an effect upon the fuel air ratio thus the exhaust gas temperatures and NOx emissions.

In actual fact, there is a link among the NOx emissions and density,

lower density preferring lower NOx, Eventhough, no other investigators have not supported such an unambiguous trend. Investigation of 25 types of bio-diesels and again excluding of castor, the ranges are starting

from 870.8 to 891.5 kg/m 3 , with the total average value being 880.2 kg/m 3 (i.e. almost 5% higher than the corresponding

conventional diesel value). The average density of

caster

bio-diesel is 917.6kg/m 3 it is totally contradictory from other 25 bio-diesel

feedstocks. In US, there is no particular specification for density of

bio-diesel

but in the EU suitable range is little bit varied from 860-900kg/ m 3 . The average density of

caster biodiesel is

41 917.6kg/m 3 it is completely different from other 25 feedstocks of bio-diesel.

When the amount of double bonds increased the density

is

also increased,

which means that, the more unsaturated the originating oil, the higher the density of the derived bio-diesel, and the larger the fuel mass that will be injected

in a diesel-tuned engine is run on bio-diesel.

The correlation, however, among the cetane number and density

from all studied feedstock is represented with higher densities imparting lower cetane numbers by Giakoumis (2013). 2.9.3 Heating Value The higher heating value (HHV) and lower heating value (LHV) of fuel is measured based on the fuel’s combustion, with the difference among them constituting the water’s heat vaporization.

The average value of bio- diesel has 10 12% w/w oxygen, which ensues to a proportionally lower heating value and energy density. As a result it indicates that in order to reach the same engine power output more fuel is needed for injection compared with the diesel. In the most recent research work technology, using

bio-diesel

resulted in the decrease in PM/smoke emissions or from other

bio fuel

such as alcohols derived from the higher oxygen content of the

bio fuel-

diesel fuel blend that enhances the in-cylinder soot oxidation rate.

From another point of view,

the fact that larger mass of biodiesel requires to be injected in order

that

to achieve the same engine power as with conventional diesel fuel.

The resulting oil contained

the HHV, accordingly, it is 38,980 (coconut), 40,550 kJ/kg (fish) and the overall average value is 40,100 kJ/kg. The effect of unsaturation on the heating values shows that increasing the unsaturation or the number of double bonds results in a moderate increase in the FAME heating value.

Both heating values correlate rather well with the oxygen content, whereas LHV seems to have a significant inter-dependence with viscosity and cloud point

by Giakoumis (2013).

42 2.9.4 Flash Point The flash point

temperature of fuel can be measured by heating process.in this process the vapor and air mixtures make the fuel ignited. The value of flash point and fuel’s volatility are related reciprocally. The above reason clearly states that the storage of pure biodiesel is much safer than diesel. In detail, the present works represents

the average values of flash point for all 26 analyzed feed stocks, the ranges are from 127.7 ˚ C to 174.5 ˚ C and the mean value from all feedstocks constituting 163.3 ˚ C.

In us

specifications require biodiesel fuels to have at least 93 ˚ C flash point, whereas in the European the minimum essential level is 101 ˚ C;

both are intended to determine a lower purity limit in the final FAME, and are easily

coped with by all examined feedstocks of

biodiesel. It should be observed that some researchers have described about very low

flash point values.In general, the originating oil is not straightly linked to the

composition of fatty acid. the Relationship between flash point and unsaturation degree (excluded methyl ester of coconut oil), while with a little increasing trend is observed that the double bonds number is raised

by

Giakoumis (2013). 2.9.5 Cetane Number Cetane number plays a crucial role in diesel fuel and it is expressible by a pure number (

dimensionless cetane number (CN)), which denotes the fuel ignitability, primarily critical on cold starting conditions. Low cetane numbers makes long ignition delay that means

a long time among the start of combustion and fuel injection. In accordance with, the Cetane Number islower for sudden premixed combustion phase and the contribution of combustion noise radiation is high. As an alternative the higher CNs encourage lower NOx emissions and faster fuel auto-ignition. In general the

bio-diesel

CNs is higher

than that of the petroleum diesel. Excluding castor, the (average) cetane numbers of the 25 feed stocks range from 50.4 for rubber seed methyl ester to 61.2 for PME, with the mean value from all feedstocks

43 being 54.8; that is 8 10% higher than that of

petro-diesel. biodiesel Cetane Number

may be caused by i) differences in the fractional conversion of triglycerides to ester, and ii) by presence of residual methanol and glycerol in the final fuel; the second argument, however, has not been confirmed by the present statistical analysis, which included methanol and (free and total) glycerol effects. It has also been argued that the effect of blending biodiesel on the CN is approximately linear for mixtures of esters with diesel fuel

reported by Giakoumis (2013). 2.9.6 Iodine Number

The

iodine value (IV) or iodine number (IN) is a parameter used to resolve the unsaturation degree in a vegetable oil or animal fat. This number specifies the iodine mass (I2) in grams that is necessary for complete saturation by means of a stoichiometric reaction, the molecules of 100 g of given oil. The average values of

iodine for the examined feedstock’s ranges are

from 7.8 (for the most saturated, coconut) to 184.5 (for the most unsaturated one, linseed), with an overall average value of 98.4

reported by Giakoumis (2013). 2.9.7

Oxidative Stability One of the major issues that limit the use of bio-diesel as a fuel in compression ignition engines is its poor oxidative stability. A brief assessment of

this work reveals that only a few of the investigated bio-diesels

fulfill the European specification of 6 hours oxidation stability using the Rancimat method (EN 14112). The US specifications are also in this case more lenient, hence biodiesel produced from feedstocks such as corn, coconut, fish, jatropha and karanja could be approved.

Many researchers used additives or did not apply the same esterification clean-up or storage conditions, all of which influence considerably the oxidation values

reported by Giakoumis (2013).

44 2.9.8 Carbon Content and Stoichiometric Air-fuel Ratio This result provides that

the correlation between FAME carbon content and number of carbon atoms with the respective degree of unsaturation.

The number of carbon atoms for

the corresponding sample is narrower compared to

the previous properties. It seems that the higher the unsaturation of the originating feedstock, the higher the carbon content although the absolute differences are small. However, no such correlation could be established for the hydrogen content. Apart from the degree of unsaturation, the FAME carbon content correlates rather well with the

cetane number, density, viscosity and all cold flow temperatures. The FAME, AFst (Air- Fuel Stoichiometric) correlates well with the cetane number, density, viscosity, higher heating value

and interestingly, oxidation stability. In general, the stoichiometric air–fuel ratio is of the order of 12.5:1 for the neat

bio-diesel,

whereas it is approximately 15:1 for the neat diesel fuel. This means that the air–biodiesel mixtures can reach the stoichiometric conditions nearly 15% faster than their air–diesel counterparts. It is expected that this difference in the air–fuel ratio, caused by the presence of oxygen in the bio-diesel, must influence combustion. More specifically, if the combustion process is mixing controlled, the use of biodiesel injection has a beneficial effect, while if the combustion process is evaporation controlled, the effect is adverse

reported by Giakoumis (2013). 2.9.9

Distillation Temperature Distillation is a method of separating mixtures based on differences in volatilities of components in a boiling liquid mixture.

The diesel contains hundreds of different compounds. It was demonstrated that most of the bio-diesels

generally contain a few number of major fatty acid compounds; these boil at roughly the same temperature (approximately 330˚C

to 357˚

C at atmospheric pressure). The distillation temperature specification has been

45 incorporated to ensure that fuels have not been contaminated with high boiling materials such as used motor oil. Hence, the 20 distillation of biodiesel fuel is a method that actually provides a demonstration of the FAME’s quality.

however, indicate that increasing the number of double bonds increases also the distillation temperature, with highly unsaturated feedstocks, such as linseed and fish (Rich in tri- and higher unsaturated acids) exhibiting the highest values

reported by Giakoumis (2013). 2.9.10

Low-Temperature Flow Properties The input flow properties for winter fuel specification are cloud and pour point. Cloud point (CP) is the temperature at which wax form a cloudy appearance. It is measured as the temperature of first formation of wax as the fuel is cooled. Pour point (PP) is the lowest temperature at which the fuel becomes semi solid and loses its flow characteristics being no longer pumpable; hence it is a measure of the fuel gelling point. The pour point is always lower than the cloud point. The cloud point is not generally affected by additives called flow improvers. However, flow-improver additives can decrease the size, or extent of the formation, from the wax crystallites formed on

cooling the fuel, and so it lowers the temperature at which wax plugging becomes a problem. Poor cold flow properties may result in fuel line and pump blockage, ultimately leading to fuel starvation. These problems are particularly making better during cold starting particularly at low ambient temperatures.

There are no European or US specifications for low temperature properties (Each country is free to determine its own limits according to local weather conditions), but it is well known that biodiesel fuels suffer from cold flow properties way more (i.e. they are higher) than mineral diesel fuel. The (average) values of CP for the 26 examined feedstocks in the present work confirm this fact; they range from 15.1

o C (castor ME; however with 12.5

o C

standard deviation, which indicates contradicting results)

to 13.8˚C (Tallow

46

methyl ester (

ME)), with the mean value from all feedstocks being 2.1

o C (

almost 20 o C

higher than for conventional diesel fuel). For PP, it is 23.4

o C (again castor ME; standard deviation 3.9 o C) up to 11.8 o C (

palm ME), with an overall mean value of 2 o C (More than 30 o C

higher than conventional diesel fuel).

In order to enhance the poor cold flow properties of biodiesel, various measures have been suggested, such as the use of cold-flow improvers, ethyl or iso-propyl esterification (Higher cost than the use of methanol), removal of those components that have high melting points (Stearic and palmitic), or even modification of the fatty acid composition of the originating oil in order to produce an ester with optimum characteristics (concerns arise in this case over genetic modification issues

reported by Giakoumis (2013). 2.10 OPTIMIZATION PROCESS In general, there are three categories of catalysts used for

bio-diesel

production: alkalis, acids and enzymes. Enzyme catalysts have become more attractive recently since it can avoid soap formation and the purification process is simple to accomplish. However, they are less often used commercially because of the longer reaction times and higher cost. To reduce the cost, some researchers developed new

bio catalysts

in recent years. Compare with enzyme catalysts, the alkali and acid catalysts are more commonly used in biodiesel production. The alkali and acid catalysts include homogeneous and heterogeneous catalysts. Due to the low cost of the materials, sodium hydroxide and potassium hydroxide are usually used as alkali homogeneous catalysts and alkali-catalyzed transesterification is most commonly used commercially. These materials are the most economic because the alkali-catalyzed transesterification process is carried out under a low temperature and pressure environment, and the conversion rate is high with no intermediate steps. However, the alkali homogeneous catalysts are highly hygroscopic and absorb water from air during storage. They also from

47 water when dissolved in the alcohol reactant and effect the yield. Therefore, they should be properly handled. On the other hand, some heterogeneous catalysts are solid and it could be rapidly separated from the product by filtration, which reduces the requirement. In addition, solid heterogeneous catalysts can stimulatingly catalyze the transesterification and eaterification reaction that can avoid the pre-esterification step, thus the catalysts are particularly useful for those feed stocks with high free fatty acid content. However, using a solid catalysts, the reaction proceeds at a slower rate because the reaction mixture constitutes a three-phase system, which due to diffusion reasons, inhibits the reaction. The three categories of catalysts with their advantages and disadvantages shown in Table 2.5 (Leung et al. 2010). Table 2.5 Advantages and disadvantages at different types of catalysts in the biodiesel production Type Advantages Disadvantages Example Alkai Homogeneous Favorable kinetics, High catalytic activity, modest operation conditions,low cost. Emulsion formation, low FFA requirments,more wastewater from purification,

anhydrous conditions, disposable. NaOH,KOH. Heterogeneous Enviormentally benign, non coressive, easily sepration, fewer disposal problems, longer catalyst life times, recyclable, higher selectivity. More waste water from purification, Low FFA requirment, high molar ratio of alcohol to oil requirment, anhydrous conditions,diffusion limitations, high pressure, reaction temperature and high cost. CaO, CaTiO 3 ,CaZrO 3 , Cao-CeO2,

Ca 2 Fe 2 O 5 , CaMnO3, KOH/Al 2 O 3 , Al2O3/KI, KOH/NaY, alumina/silica supported, K 2 CO 3, .

ETS-10 Zeolite.

48

Table 2.5 (Continued) Type Advantages disadvantages Example

Acid Homogeneous Catalyze esterification and transesterification simultaneously, avoid soap formation. Equipment corrosion,

long reaction time,waste water from neutrlization, higher reaction temperature, weak catalytic activity,difficult to recycle. Concentracted sulphuric acid Heterogeneous Catalyze esterification and transesterification simultaneously recyclable, eco- friendly. Low microporosity, low acid site concentrations,high cost, diffusion limitations. TiO 2 /SO 2 4 ,

ZrO 2 /SO 2 4 , ZnO/I 2 , , carbon-based solid acid catalysts, carbohydrate-derived catalysts, Vanadyl phosphate, Nafion-NR50, Sulphated zirconia, Vanadyl phosphate, Amberlyst-15.

Enzymes Avoid soap formation, non polluting, easier purification. Expansive and denaturation. Rhizomucor mieher lipase, candida antarctical fraction B lipase.

Atapour and Kariminia (2011)

prepared biodiesel from bitter almond oil (BAO). In their investigation 0.9% w/w of KOH concentration produced maximum yield of 90.8% w/w of bio-diesel. Karnwal et al. (2010) prepared bio-diesel from Thumba oil by transesterification in the presence of potassium hydroxide as catalyst. In their study for 0.75% of KOH concentration maximum ester conversion of 97.8% was obtained.

49 2.11 QUALITATIVE ANALYSIS (

GC-MS) An effective and proven combination for fatty acid and bio diesel analysis is Gas chromatography (GC) and mass spectrometry (MS), to identify and quantify the various components of the sample mixture simultaneously. Due to the high sensitivity and high specific detection of this technique, it allows a much finer degree of substance detection than either unit used one by one. The capacity of GC-MS to confirm identity and separate by mass spectral structure elucidation has facilitated researchers to learn the influence of fatty acids on the quality of

bio-diesel

fuel. For example, methyloleate was proposed as a appropriate major

bio-diesel

component. Knothe also noted that, due to low-temperature properties methyl palmitoleic acid can be used an alternative to methyloleate. The identification of components by GC-MS has continued to gain importance in establishing the oil composition and quality reported by Iko et al. (2015). Koria & Nithya (2012) reviewed that, fatty acid composition of the bio-diesel samples synthesized from beef tallow oil and soybean has influence on density, viscosity, and cloud point. Physical and fuel properties of

bio- diesel

for several oils have been tested and compared with conventional diesel fuel. Daturastramonium (

Tamil name is umathan phu) bio-diesel

is analyzed for its fatty acid composition using GC-MS and important properties such as density, kinematic viscosity, cloudpoint, heat of combustion and cetane index (ignition quality) are studied. 2.12

USE OF BIO DIESEL IN COMPRESSION IGITION ENGINE Performance parameters like Brake Thermal Efficiency and Brake Specific Fuel Consumption (BTE & BSFC) and emission parameters of

50 Carbon monoxide (CO), Hydrocarbon (HC), Nitrogen Oxide (

NO x)

and smoke density of various bio diesel blends have been reviewed briefly for different bio diesel and blends with diesel in the subsequent sections. 2.12.1 Soyabean Oil Qi et al. (2010) reported the performance and combustion characteristics of a direct injection C.I engine fueled with

bio-diesel

produced from soya bean oil and its various blends (B0, B30, B50, B80, B100). The test result showed a small increase in Break Specific Fuel Consumption (BSFC) for

bio-diesel

and its blends are slightly lower than that of diesel keeps the same trend for low engine loads and higher engine load. The carbon monoxide (CO) and smoke emissions are significant improvement in decrease of biodiesel and its blends and it have little variance from diesel fuel. Bio- diesel and its blends emit slightly higher nitrogen oxides (Nox). This may be due to higher oxygen content in

bio-diesel.

The authors concluded that the excess oxygen contents present in the biodiesel plays a vital role in engine performance and

bio-diesel

is proved to be a potential fuel for partially (or) complete alternative of diesel fuel. 2.12.2 Sunflower Oil Ulusoy et al. (2009) reported that the emission characterisics of adirect injection, four cylinders, four stroke turbo diesel engine with using sunflower oil methyl ester at different speeds and full load condition. The result showed that, for all engine speeds the smoke, CO and HC emissions of sunflower oil methyl ester are generally lower than diesel fuel. The oxygen content of

bio-diesel

fuel and the test engines turbo charger mechanisms have caused lower emission values by increasing the combustion quality. The author’s concluded that exhaust emissions of

bio-diesel

are lower that of base

51 diesel fuel, which shows that sun flower oil methyl esters has more favorable effects on air quality. 2.12.3 Mahuan (Madhuca indica) Godiganur et al. (2009) reported that the performance and emission characteristics of a cummins 6BTA 5.9 G2-1, 158 HP rated power, water cooled, direct injection,

turbo charged,

constant speed (1500 rpm) diesel engine using diesel fuel, mahua bio-diesel and its blends at different load conditions. The test results showed that increased biodiesel blends of proportion significantly reduces the HC and CO emissions due to the

bio- diesel

complete combustion. Though, slightly increased Nox emissions (11.6%) as compared with that diesel. This could be attributed to the exhaust gas temperature increasing and the fact of some oxygen content present in the

bio-diesel

which facilitated NOx formation. It was observed that as compare with diesel the BSEC for B20 was lower but in case of B40, B60 and B100, the BSEC was higher. This reverse trend was observed due to lower heating value with increased

bio-diesel

percentage blends. At B20 (32.5%) the maximum brake thermal efficiency was higher than that of pure diesel. This may be due to calorific value reduction and increased BSFC as compared with B20 . There is a slight increase in BTE (brake thermal efficiency) and small reduction in BSEC (brake specific energy consumption) of B20 percentage blend in engine as compared to diesel fuel. When the concentration of

bio-diesel

blends increased the exhaust gas temperature (EGTs) also increased. Among those the mean EGTs of 7%, 9%, 10% and 12% are with respectable to B20 B40 B60 and B100 and it was higher than the mean EGT of diesel. This may be due to heat loss increased for higher bio-diesel blends and also it is evident from the lower BTE as compared to diesel. As of these findings, it is concluded that without significantly moving the engine performance (BSFC, BSEC, EGT) and emissions (CO, HC, and

52 NOx) the up to 20% of mahua biodiesel could be safely blended with diesel and it could be suitable substitutable fuel for heavy duty engines. 2.12.4 Mahuan Oil Puhan et al., (2005) reported that the performance and emission of a constant speed, direct injection ,single cylinder, four stroke compression ignition diesel engine (kirloskar) usingmahua biodiesel. The result exposed that diesel engineperformance with biodiesel neither does not vary significantly. The brake thermal efficiency is 13% lower than that of diesel and brake specific fuel consumption is 20% higher than that of diesel. Exhaust emission pollutant are reduced when compared to diesel. Carbon monoxide, Hydro carbon, smoke and NOx are compared with diesel and it is decreased by 30%, 35%, 11% and 4% respectively. This experiment study observed by several researchers that NOx emission was increased with

bio- diesel. 2.12.5

Karanja Oil Baiju et al. (2009) reported that the performance and exhaust emission characteristics using several blends of diesel and

bio-diesel

from karanja oil and by using base line fuel of petro diesel in a diesel engine. The result depicted that performance of engine does not vary greatly than diesel fuel. A little power loss, higher BSFC and lower BTE was observed due to lower calorific value of esters slightly produced higher power and emissions was lower than ethyl esters. They also found that at part loads when compare with conventional diesel fuel the emissions of NOx is increased by 10-25% when fuelled with diesel-biodiesel fuel blends. At maximum load, diesel emitted more NOx than esters and most of the major exhaust pollutants such as CO, HC and smoke were reduced with the use of neat

bio-diesel

and the blends. The study concluded that both methyl and ethyl ester of karanja oil

53 can be used as a fuel in compression ignition engine without any engine modification. 2.12.6 Cotton Seed Oil Nabi et al. (2009) studied the effect of cotton seed oil

bio-diesel

on engine performance and emission. It was found that thermal efficiency of

bio- diesel

fuel was lower than that of diesel fuel due to the poor spray characteristics, higher density, viscosity and lower heating values of

bio- diesel

fuel. The emissions of CO, PM and smoke from biodiesel was lesser than that of neat diesel fuel. But, NOx emission with

bio-diesel

mixtures was higher compared to neat diesel fuel. For instance, with 10%

bio-diesel

mixtures (B10) PM and smoke emission reduced by 24% and 14% respectively. While

bio-diesel

mixture of 30% (B30) reduced CO emissions by 24% and NOx emissions increased by 10%. The authors attributed the reduction in PM, smoke and CO emissions and increased in NOx emission with biodiesel mixtures to the presence of oxygen in their molecular structure as well as low aromatics in the

bio-diesel

blend. 2.12.7

Jatropha Oil Chauhan et al. (2012) evaluated the performance and exhaust emissions using 5%, 10%, 20% and 30% jatropha

bio-diesel blended

with diesel fuel on an unmodified diesel engine. The experimental results showed the engine performance with

bio-diesel

of jatropha and its blends were comparable to the performance of diesel fuel. In case of all fuel blends, brake thermal efficiency, HC, CO, CO 2 and smoke density were lower while BSFC and NOx were higher than that of diesel. The authors concluded that biodiesel derived from jatropha and its blends could be used in a conventional diesel engine without any modification. However, there were various parameters which

could

be evaluated in future such as the prediction of best blend with

54 respect to the various engine parameters by varying spray time of fuel using common rail fuel injection. 2.12.8 Tobacco Seed Oil Usta et al. (2005) evaluated the performance and exhaust emissions of a turbo charged indirect injection diesel engine using 10%, 17.5% and 25% tobacco seed oil methyl ester blend (TSOME). The authors found that the addition of up to 25% in volume TSOME did not cause any significant variation in the engine torque and power. Although the heating value of the TSOME is 10% less than that of the diesel fuel. Moreover, the blending of tobacco seed oil methyl ester to the diesel fuel reduced CO due to the fact that TSOME contained about 11.4% oxygen by weight and SO 2 emissions due to low sulphur content while causing slightly higher NOx emissions due to higher combustion temperature. 2.12.9 Polanga Oil Sahoo et al. (2007) evaluated the performance and emission of polanga

oil methyl

ester (POME) blends in a single cylinder diesel engine at different loads. Tha main findings of the study showed that the performance of

bio-diesel

fuelled engine was marginally better than the diesel fuelled engine in terms of thermal efficiency, brake specific energy consumption, smoke opacity, and exhaust emissions including NOx emission for entire range of operations. The 100%

bio-diesel

was found to be the best, which improved the thermal efficiency of the engine by 0.1%. Similar trend was shown by the brake specific energy consumption and the exhaust emissions were reduced. Smoke emissions also reduced by 35% for B60 as compared to neat petro diesel. Decrease in the exhaust temperature of a bio-diesel fuelled engine led to approximately 4% decrease in NOx emissions for B100

bio- diesel

at full load. It was conclusively proved that excess oxygen content of

55 bio-diesel played a key role in engine performance. However, long term endurance test and other tribological studies need to be carried out before suggesting long term application of polanga oil based

bio-diesel. 2.12.10 Jojoba Oil In an experimental investigation conducted by Shehata

and

Razek (2011) on the performance and emission characteristics using jojoba blend indicated BTE for pure diesel higher than B20 due to the higher heating value of diesel and higher BSFC due to the same reason. An increase in CO and CO 2 concentration due to higher carbon / hydrogen ratio and reduction in NOx due to lower gas temperature were also observed. 2.13.11 Koroch Oil Gogoi & Baruah (2011) used koroch seed oil methyl ester (KSOME) in a single cylinder diesel engine and reported that the engine BTE at full load for Numaligarh refinery limited (NRL) diesel, B10, B20, B30 and B40 were found to be 25.63%, 24.86%, 24.34%, 24.09% and 22.32% respectively . The BTE of various fuel blends were found to be lesser than NRL diesel due to its lower calorific value. Due to higher viscosity and density of KSOME blends, the rate of fuel consumption was found to be more for the methyl esters. Their study revealed the use of KSOME blends up to B30 as fuel for diesel engine without any significant drop in the performance characteristics. 2.12.12 Rubber Seed Oil Ramadhas et al. (2005

a)

evaluated the emissions and performance of a diesel engine fuelled with

bio-diesel

of rubber seed oil. Their results showed that the maximum brake thermal efficiency obtained is about 28% for

56 B10, which was quite higher than that of diesel (25%). Moreover, it was observed that using lower percentage of

bio-diesel in bio-diesel-

diesel blends, the brake specific fuel consumption of the engine is lower than that of diesel for all loads. Under all loading conditions, it was observed that the engine emits more CO using diesel as compared to that of

bio-diesel blends.

However, using higher concentration of biodiesel blends increase CO 2 and NOx emission. Smoke density for biodiesel blend is noticed to be generally lower than that of diesel. For instance, B20 blends gave smoke density of 20% as compared to 45% in the case of diesel. The experimental results proved that the use of biodiesel produced from unrefined rubber seed oil in compression ignition engines is a sustainable alternative to diesel. 2.12.13 Rice Bran Oil Saravanan et al. (2009) studied the feasibility of crude rice bran oil methyl ester (CRBME) in diesel engine. The results showed that, with CRBME blend the brake thermal efficiency decreases only marginally due to the lower heating value of the methyl ester. Moreover, a significant reduction in CO, HC and particulate emission were observed with a marginal increase in NOx emission than that of diesel due to higher oxygen contents of methyl ester. 2.12.14

Mahua

Oil

Saravanan

et al. (2010) investigated the performance and emission of a diesel engine fuelled with

Madhuca indicabiodiesel. Experiments were conducted on

a single cylinder, air cooled, four stroke, direct injection, compression ignition engine

using mahua oil methyl ester and diesel as a fuel. The result showed that at full load, the power loss was around 13% combined with 20% increase in fuel consumption with mahua oil methyl ester due to the lower heating value and higher viscosity of biodiesel fuel.

57 Emissions such as carbonmonoxide, hydrocarbons were lesser for mahua ester compared to diesel by 26% and 20% reapectively, due to the higher oxygen contents which promoted combustion. Oxides of nitrogen were lesser by 4% for the ester compared to diesel due to the lower in cylinder temperature. A smoke intensity reduction of 15% for MOME (Mahua oil methyl ester) was also observed at full load. It was also observed that the exhaust gas temperature for MOME operation is lower compared to that of diesel. 2.12.15

Turpentine Oil Anand et al. (2010)

reported that the performance emission of diesel engine operated using turpentine oil. The result showed that the higher brake power and heat release rate produced for 30% of TPOF with 70% of diesel fuel. Nox, CO, HC and smoke emission are reduced. Further increased 40% and 50% of TPOF blends, produced lower heat releasr rate and brake power. 2.12.16 Pine

Oil

Vallinayagam et al. (2013) evaluated the combustion, performance and emission of a diesel engine fuelled with

pine oil.

From the resin of pine tree the pine oil was extracted. It could be directly used in diesel engine without any modification.

The result showed that the pure pine oil (100%) at full load condition, the brake thermal efficiency and heat release rate increased by 5% and 27% respectively, and reduced hydrocarbon, carbon monoxide and smoke emissions by 30%, 65% and 70% respectively. However, slightly increased NOx emissions.

58 2.12.17

Camphor Oil and Cashew Nut Shell Oil Kasiraman et al. (2012) studied the effect of cashew nut shell oil blended with camphor oil. The experimental results at full load condition shows the camphor oil 30% and 70% cashew nut shell oil blends (CMPRO30) produced good performance and heat release rate compared with diesel.CMPRO30 blend brake thermal efficiency was 29.1% compared to diesel of 30.14% and slightly reduced NOx emission compared to diesel. 2.12.18

Ceiba Pentandra Oil Kathirvelu et al. (2014) reported that bio-diesel was

produced from curde ceiba pentandra seed oil through transesterification processes, using H_2SO_4 as a catalyst (1%v/v) methanol as a solvent, 45min reaction time and 60˚C reaction temperature. The maximum methyl ester yield obtained was 93%. The property of methyl ester viscosity was nearly close to diesel. BTE of B100 fuel at maximum load 6.26% reduced compared with diesel. The emissions of CO, CO_2 , HC and NOx were reduced. In compared

to

diesel similar cylinder pressure and heat release rate are observed. 2.12.19

Watermelon Oil Santhosh Kumar et al. (2016) reported that bio-diesel

was produced from watermelon seed oil through transesterification process. B20 biodiesel blend at maximum load 9kg, standard injection timing (23˚BTDC) with compression ratio of 18 has optimum engine performance of brake thermal efficiency was 33.55%. CO, HC, CO_2 and NOx were 0.02% vol., 7ppm, 1.1%vol. and 123ppm-vol. respectively.

59 2.13

SUMMARY The key points from the above discussion are, The four-stroke

DI-CI engine

are indispensable in major areas of transport, agriculture, etc. and it will be very difficult to replace it with any other type of power plant. The present situation for four-stroke DI-CI engines is a difficult one and the future of the engine depends on meeting the emission norms that are being imposed. Modification of the fuel is one of the best ways to reduce emissions without any modification on the existing engines

and exhaust gas treatment. Raw oils are not suitable for diesel

engines. Preheating, direct use blending, micro emulsion, pyrolysis (Thermal cracking) of straight vegetable oils (SVO) are used only for short term experimental purpose only. For long term application of non-edible oils chemically modified into bio diesel is known as transesterification process. Aalkaline catalyzed transesterification is the best way and most accepted method which have been used to prepare bio diesel from high free fatty acid content vegetable oils like Jojoba oil, Rice bran oil pungamia, soyabean oil etc.,

Very few research publications on production

of

bio-diesel from ceiba pentandra seed oil and watermelon seed oil.

No publication in CG-MS analysis of watermelon methyl ester.

60

Increased bio-diesel blends ratio with decrease the brake thermal efficiency of bio-diesel operated engine. This may be due to the more specific fuel consumption of bio-diesel due to heavier oxygen molecule structure present in the fuel. The increased bio-diesel percentage of blends significantly reduced CO, HC and smoke due to the presence of oxygen in the fuel. However, slightly increased NOx emission due to the higher combustion chamber temperature and Exhaust Gas Temperature (EGT) It is obvious in few of the researches that the neat orange oil, Pine oil, karanja oil, and turpentine oil brake thermal efficiency were slightly higher than diesel fuel due to less viscous and better atomization of those oils.

From the above literature review, it is pointed out that more work is needed for optimize the parameters of bio diesel production towards maximum yield, to analyze the blend properties and to find the best blend in an engine. The research problem for the current work is selected based on the above literature review. The following chapter briefly explains the problem definition, its objectives and scope of present work.

61

CHAPTER 3

OBJECTIVE AND METHODOLOGY 3.1

PROBLEM DEFINITION The chapter one and two discussed the serious description of the review of the literature. The study and investigation is based on the problems, production of methyl esters from non-edible oils such as alcohols, catalyst, reaction temperature and reaction time of different vegetable oils. The physic- chemical properties of the diesel, vegetable oil, methyl esters and its blends are to be analyzed. The performance, combustion and emission characteristics of different methyl esters, pine oil and diesel blends are to be analyzed. 3.2 OBJECTIVES OF THE STUDY By under viewing the review of literature, the following objectives were outlined for the present work:

To investigate the potential suitability of ceiba pentandra oil, watermelon seed oil and pine oil as a substitute fuel for DI-CI engine. To produce the bio diesel from ceiba pentandra

seed oil and watermelon seed

oil using transesterification process. To optimize the stirrer speed, amount of catalyst and solvent required for maximum yield of biodiesel production.

62 To study the quantitative analysis for methyl esters of ceiba pentandra and watermelon . To study

and compare the physical and chemical

fuel properties of pine oil, CPME and WME with its diesel blends

as per ASTM standards. To study the DI-CI engine performance and emission characteristics fuelled with

pine oil-CPME-WME and its diesel blends. To study the DI-CI engine

combustion

characteristics of fuelled with pine oil-CPME- WME and its diesel blends

The

current work focus on experimental investigations to achieve the above objectives. The experimental investigation of methyl ester production is made and the properties, performance, combustion, and emission characteristics were analyzed. 3.3 SCOPE OF THE PRESENT WORK The scope of the present investigation includes: Increase the conversion efficiency of

bio diesel production ceiba pentandra seed oil and watermelon seed oil by using

homogeneous catalyst. Qualitative analysis of GC-MS for methyl

esters of bio-diesel from ceiba pentandra oil and watermelon oil.

Comparing CPME, WME and PO blends with diesel tested in DI-CI engine.

63 Performance and emission analysis of PO-biodiesel-diesel. Combustion analysis of PO - bio diesel-diesel blends in DI-C.I engine. The current work focus involves experimental investigations to achieve the above objectives. 3.4

METHODOLOGY The methodology used in the present work is outlined below in a step-by- step manner as shown in Figure 3.1. Optimization of the important parameters like speed of the stirrer, catalyst concentration like KOH, NaOH and reaction time for producing higher yields of methyl esters are discussed. Ceiba pentandra seed oil, watermelon seed oil have been converted into biodiesel of the concerned oil. The important fuel properties like specific gravity, viscosity, flash point, cloud point, pour point, and calorific value of SVO and methyl esters of bio diesel – diesel blends were studied using ASTM specified equipment. The qualitative analysis of diesel and

two

esters were done using gas chromatography and mass spectrum analysis. A single cylinder,

direct injection

diesel engine

experimental setup is fabricated with the necessary instrumentation to study the performance, emission and combustion characteristics of the diesel engine

fueled by CPME, WME & pine oil and its blends with diesel.

64 Figure 3.1 Research methodologies for bio diesel production, study of fuel properties, engine performance, emission and combustion analysis

65 3.5

SALIENT FEATURES OF THE PRESENT WORK Following are some of the significant features of the present work: Bio-diesel is produced

from ceiba pentandra seed oil and watermelon seed oil using

electronically controlled batch type production unit. The present investigation has been carried out on a production model

DI-CI engine. Pines, bio-diesel from ceiba pentandra seed oil, watermelon seed oil

and diesel oil have been used as fuel. The physical and chemical properties of bio-diesel – diesel blends like flash point, fire point,relative density, viscosity, calorific value, cloud and pour point were studied. Exhaust emissions such as carbon monoxide, carbon dioxide, unburned hydrocarbon

(UBHC), Nitrogen oxides (NO x), smoke, cylinder pressure and combustion characteristics have been measured at each operating point. To achieve the above stated objectives and methodology, a detailed experimental work has been carried out. The details of the experiments and equipment's used are explained in Chapter 4.

66 CHAPTER 4

EXPERIMENTAL SETUP AND PROCEDURE 4.1

INTRODUCTION This chapter discusses

the experimental setup for bio diesel production unit, measurement of fuel physical chemical properties instruments and finally experimental engine setup along with necessary instrumentation to evaluate the engine performance, emission and combustion analysis of the engine with two types of bio diesel blends

with pine oil operating conditions. 4.2 BIO DIESEL PRODUCTION The vegetable oil usually contains free fatty acids, phospholipids, sterols, water, odorants and other impurities. Because of their presence, the oil cannot be used as fuel directly. To overcome these problems, the oil requires chemical modifications like transesterification, emulsification and pyrolysis. Among these, the transesterification is an significant process to produce clean and environment friendly fuel from vegetable oil and it seems to be more suitable because the by product (glycerol) has got commercial value. 4.2.1 Process of Bio Diesel Production The process flow chart for bio diesel production

from ceiba pentanndra seed oil and water melon seed oil

is shown in Figure 4.1.

67 Figure 4.1 Process flow chart for bio diesel production 4.2.2 Experimental Setup for Bio Diesel Production Unit A small batch type unit is designed and fabricated keeping in mind the objective of the present work for producing bio diesel from non-edible

ceiba pentandra seed oil and watermelon seed oil. It is very compact type and can maximum produce five

liter bio diesel from non-edible oils in a period of 1 1 / 2 hours. The production of bio diesel is approximately 70 to 80 liters per day. It has the following advantages Cost is low Easy to operate

68 Electronic temperature control monitor system Digital Speed monitoring system. According to the requirements the variable and constant speed can be maintained. The container and blades are made up of stainless steel. It can able to produce the bio diesel about 80 to 96% of yield based on the nature of the oils and reactants. 4.2.2.1 Description of bio diesel production unit

The transesterification unit used for bio diesel production is planned and fabricated in the laboratory. It consists of stainless steel container, with a capacity of six liters and a variable speed D.C motor

is

fixed on the top of the container. The motor is coupled with a mechanical stirrer. The operating speed of the mechanical stirrer range from 200 rpm to 900 rpm. The stirrer speed can be easily varied with the help of an electronic speed regulator and it

is

monitored using digital speed sensor along with a digital speed indicator. The stirrer consists of eight stainless steel blades at an angle of 45 oriented to the base. At bottom of the container a capacity of

1.5 kW is fixed to heat the oil. A temperature control unit is used to vary the temperature of oil from 30 C to 250 C. The oil temperature is measured by using Chromel Alumel (k-type) thermocouple along with digital temperature indicator. Control the motor speed and temperature an electronic microprocessor is used and the cooling fan which is inside the electronic kit is used to absorb the heat from the transformer and integrated circuits. The bottom of the container has one drain valve and the top of the container has one inlet valve. The line diagram and

photographic view of

the bio diesel production unit are shown in Figures 4.2 and 4.3.

69

Figure 4.2 Line diagram of the bio diesel production unit 1. Container 2. DC motor 3. Heater 4. Thermocouple 5. Blades 6. Speed sensor 7. Inlet valve 8. Outlet valve 9. Microprocessor kit 10. Electrical connection for motor, eater and thermocouple 11. Cooling fan for microprocessor kit 12. Temperature indicator 13. Speed (rpm) indicator 14. ON/OFF switch 15. Speed regulator for DCMotor 16. Transformer 17. Sensor controller 18. Shaft

70 Figure 4.3 Photographic view of bio diesel production unit 4.2.2.2

Features of the bio diesel production unit

a)

The automatic temperature controller is provided in the unit to vary the temperature of oil from 30˚C to 250˚C. This feature is highly useful for pre-treatment, transesterification and evaporating of water molecules present in the

bio

diesel after washing.

b)

It consists of eight numbers of stainless steel blades with an angle of 45˚ and rotating in clockwise direction to produce the centrifugal effects to separate the fatty acids from the oil more effectively. c) The volume of the tank is six liters and it is capable to produce maximum of five liters or minimum of two liters of oil

depending upon the requirements.

71 4.2.3

Procedure for Transesterification Process Mixing of reactants is found to be an important factor which effects the transesterification and yield of bio diesel. Operation is carried out at a speed of 600 rpm in the first stage of reaction (10 min) and a lower stirring speed of 550 rpm for the remaining period. 2.5 litre of ceiba pentandra oil is filtered using cloth to remove the impurities in the oil and poured into a bio diesel production unit and 500 ml of methanol is used as a solvent for 2.5 litre oil based on literature review in first phase of work. Under agitation, the raw oil is heated up to 60ºC (nearer to boiling point of methanol) for production of methyl ester by using heating coil. The amount required for catalyst pellets (based on literature) KOH is quickly weighed, protecting it as much as possible from atmospheric moisture and carbon di-oxide. The pellets are quickly moved to the dry mixture grinder to change into powder form. The powdered form of catalyst and solvents (methanol) solution is then vigorously shaken in a conical flask for homogeneous mixing. At this point dissolved catalyst (KOH) is presumed to have been converted into potassium/ sodium/

methoxide. A fixed amount of solvent and catalyst solution is poured into the oil to mark the beginning of the reaction.

72 When the reaction reached the preset reaction time, the process of heating and stirring are stopped. The oil has taken 60 minutes for obtaining reaction products. Once the reaction period is over, then the product is allowed to settle for 24 hours. In the output two distinct liquid phases are identified. At the top phase is Crude ester and the bottom phase is glycerol. That is bio diesel process turns the oil into esters, separating out the glycerol. The glycerol sinks at the bottom and the bio diesel floats on top and can be syphoned off. The crude ester phase is separated and glycerol phase is then washed by warm de-ionized (double distilled) water for four times to remove un-reacted oil, catalyst and methanol until the water becomes clear. The solution mixture was again allowed to settle under gravity and the impurities were drained out along with water at the bottom of the funnel. So there is no need for further wash. Heating the oil in an open container nearer to 110˚C to evaporate the water content in the washed ester is done. The excess methanol and water in the ester phase is removed (or recycled) by evaporation under atmospheric condition after being measured for product yield calculation. The fuel properties analysis like flash point, pour point, cloud point, density, calorific value, viscosity, acid value and cetane number of ester are as per the ASTM standard.

The final product is analyzed by gas chromatography and mass spectrum to determine its constituents.

73 4.2.4

Production of Mthyl Esters of Bio Diesel from Ceiba pentandra Oil

and watermelon seed oil

Methanolysis of

triglycerides is represented in Equation (4.1).

OH - CH CH- COO -R OH CH R- COO - CH | | OH - CH CH- COO -R OH CH R- COO - CH | | OH-

CH CH- COO -R OH CH R- COO - CH 2 3 3 2 3 Catalyst 3 2 3 3 2

triglyceride + 3

methanol 3 bio diesel + glycerol (4.1) Tranesterification

is

one of the reversible reactions and proceeds essentially by mixing the reactants. however, the presence of a catalyst (a strong acid or base) accelerates the conversion

of esters (Adewale et al. 2015). 4.2.5 Optimization

The increase bio diesel production requires optimize production processes that are compatible with high production capacity, feature simplified operations, high yields, quantity of catalyst, solvent requirements and waste streams reaction are investigated systematically. The optimal value of each parameter in the process is determine while the rest of parameters are kept constant. After each optimal value is attained, this value is adopted for the optimization of the next parameter. It should be noted that, once the reaction takes place, it would continue even at room temperature, as long as the catalyst exist in the reactant mixture, which is favorable for bio diesel production. In the present experiment, when heating is finished, no acid is added into the product mixture to neutralize the catalyst and to stop the

74 reaction at once, but rather the catalyst is kept in the mixture and experiences a long settling time together with the mixture. Better conversion is obtained by extended reaction time. The reaction will stop completely when water washing is finished, because the catalyst in product mixture would have been removed during this process. The PH value of both the product and washed water was '5' at that time. Therefore, in order to compare the product yield under different reaction conditions, the product mixture is allowed settle for a period of 15 hours and washed for three to four times in an interval of one hour each. 4.2.6

Washing of Biodiesel When the reaction is completed the bio diesel and glycerol are separated. Consequently, with hot distilled water the bio diesel is washed, to remove entrained glycerol, catalyst, soap and excess methanol. The excess methanol in bio diesel corrodes the fuel injection system and hence it should be separated from the bio diesel (Naik et al. 2008). 4.2.7 Drying of Bio Diesel By heating the washed fuel approximately to 110 C in an open container the drying is determined up to that there is no more steam from the fuel, which should be a clear, amber- colored liquid as shown in Figure 4.4. This heating process will also drive off any traces of remaining alcohol as well. Then it is allowed to cool to room temperatures, it can be pumped directly into vehicles, or into storage containers. If the fuel still appears somewhat cloudy after drying, the drying cycle should be repeated but the likely culprit is probably the presence of non-water soluble contaminant in the fuel (such as mono and di-glycerides). The pure bio diesel is collected and stored in separating funnel as shown in Figure 4.5.

75 Figure 4.4 Photographic view of drying the bio diesel in open container Figure 4.5 Photographic view of pure bio diesel 4.3 FUEL PROPERTIES The Fuel properties of vegetable oil, methyl esters (Bio diesel) can be grouped conveniently into physical, chemical and thermal properties. The physical properties have flash point, pour point, cloud point, viscosity,

76 density, boiling range, freezing point and refractory index. The chemical properties contains chemical structure, saponification value, hydroxyl value, peroxide value, acid value, iodine, acetyl value, overall heating value, ash and sulphur contents, sulphur and copper corrosions and ignitability of products. Thermal properties are distillation temperature, thermal degradation point, carbon residue, specific heat value, thermal conductivity etc (Demirbas 2008;

Verma et al. 2016).

The present work gives the details about the following properties. Relative density Cloud and Pour point Calorific value Viscosity Flash point Water and sediments GC-MS Analysis 4.3.1 Specific Gravity Specific gravity is also one of the most important fuel properties, because injection system, pumps and injectors must deliver the correct amount of fuel precisely adjusted to provide proper combustion (Dzida & Prusakiewic (2008);

Verma et al. (2016)).

Specific gravity or else relative density refer

the ratio of the density of a fuel to the density of

water at the same temperature. Specific gravity of methyl esters and diesel blends was measured by means of hydrometer (ASTM D1298 method), which is shown in Figure 4.6.

77 Figure 4.6 Photographic view of hydrometer 4.3.2 Viscosity Viscosity is

one of the major factors for establishing their suitability to the metering requirements of engine operation. Figure 4.7 shows the redwood viscometer (ASTM D 445 method) (Verma et al. 2016). The redwood viscometer is mainly for the measurement of dynamic viscosity of pine oil, bio diesel and diesel blends. The experiments were performed at 40ºC and the apparatus works on the principle of measuring the time of gravity flow in seconds of the sample through a specified hole. The following Equations (4.3) and Equation (4.4) represent dynamic viscosity calculation (4.2) and (4.3). $\mu = 0.0026\ t-1.79/t$ for t upto 100 second (4.2) $\mu = 0.00247\ t-0.5/t$ for $t < 100$ second (4.3) where μ = dynamic viscosity in centistokes t = flow time in second

78 Figure 4.7 Photographic view of Red wood viscometer 4.3.3 Flash Point

It plays a crucial role for determining the fire hazard of the fuel.

Flash point is the lowest temperature at which the vapor given off by a fuel will flash with a test flame held above the surface without continuous burning.

The fire point is the minimum temperature at which the inflammable vapours will continue to form and steadily burn once ignited.

Depends upon the original crude oil nature the flash and fire points may vary. Figure 4.8 shown the flash apparatus of

Pensky Martens (ASTM D93 method). It is used to measure the flash point of the pine oil, bio diesel and diesel blends (Verma et al. 2016).

79 Figure 4.8 Photographic view of pensky Martens Apparatus 4.3.4 Cloud and Pour Point

Cloud points are one of the main guides to evaluate the wax precipitation potential of a fluid.

Petroleum oil when cooled may become plastic solids as a result of partial separation of wax congealing of the hydro carbons, with some oils. The separation of wax becomes visible at temperature slightly above the solidification point and this temperature is known as the cloud point (ASTM D-2500). The pour point (ASTM D-97) indicates the temperature at which oil stops flowing to the pump, bearings or cylinder walls. It is particularly important for immediate oil circulation with respect to starting of engines in very cold climates with gravity lubricating system. The fluidity is a factor of pour point and viscosity of the cold oil. Pour point depressants may be added to wax containing oils to lower the pour points instead of de-waxing the oil. The cloud and pour point temperatures have much importance in knowing the behavior of fuels in cold weather. The apparatus used for measurement of cloud and pour point of bio diesel and diesel blends, which is shown in Figure 4.9.

80 Figure 4.9 Photographic views of cloud and pour point apparatus 4.3.5 Higher Calorific Value The

calorific value or heat of combustion can be defined as

the standard measure of the energy content of a fuel

and its heating value (H.V).

The heating values are obtained by the complete combustion of a unit quantity of liquid fuel in an oxygen-bomb calorimeter under carefully defined conditions. The gross heat of combustion or heating value (GHC or HV) is obtained by oxygen-bomb calorimeter method as the latent heat of moisture in the combustion product is recovered. The heating value is one of the most important properties of a fuel.

Figure 4.10has shown the “Advance (ARICO) bomb calorimeter”. It is used to measure the heating values of bio diesel- diesel blends. As per the

ASTM D2015 standard method, an oxygen-bomb was pressurized to 25 kg/cm 2 with oxygen. The bomb is fired automatically after the jacket and bucket temperatures equilibrated within acceptable accuracy of each other. The calibration of the bomb calorimeter has been carried out by using the standard benzoic acid samples which were obtained from Advance Research Instrument Company (ARICO) New Delhi.

81 Figure 4.10 Photographic view of Bomb Calorimeter 4.3.6 Qualitative Analysis The chemical composition is very important to determine the quality of bio diesel like the amount of conversion in fatty acid alkyl esters, and presence glycerol, free fatty acid, some metal alkyl hydrocarbons, presence of unreacted alkyl alcohol and KOH/NaOH

reported by

Khan (2002). GC-MS is a method that combines the features of gas-liquid chromatography and mass spectrometry to identify different substances within a test sample and identification of unknown samples. Additionally, it can identify trace elements in materials that were previously thought to have disintegrated beyond identification. The schematic diagram of GC-MS is shown in Figure 4.11. The GC-MS is composed of two major building blocks namely Gas chromatograph and Mass spectrometer.

82 The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties (example 5% phenyl) polysiloxane. The difference in the chemical properties between different molecules in a mixture will separate the molecules as the sample travels through the length of the column. The molecules take different period of time (called the retention time) to come out of (elute from) the gas chromatograph and this allows the mass spectrometer downstream to capture, ionize, accelerate, deflect and detect the ionized molecules separately. The mass spectrometer does this by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio. The qualitative analysis of GC-MS is performed for the observed compounds present in every step and also the nature of compounds change during the various processes during bio diesel formation. It also identifies the components in the compounds, molecular weight and purity of compounds by this analysis it

is known

that conversion or interchange in the compound.By the purification and transesterificationprocess http://en.wikipedia.org/wiki/ Gas_chromatography%E2%80%93mass_spectrometry-2014). Transesterification process is the conversion of triglycerides into fatty acid alkyl esters (bio diesel) that needs to be separated from several impurities and compounds. The conversion of triglyceride to FAME takes place in different phases and is determine by knowing the compound and its quality. Hence, it is required to perform the GC-MS to identify unknown compounds in the samples at each phase. The compounds were identified mainly by comparing their mass spectra with those of the standard compounds in the NIST and Wiley library, together with the comparison of their GC retention times with those of standard compounds and were confirmed by the standard addition technique. Tentatively the components are identified based only on

83 comparison of the mass spectra with the reference spectra of the NIST and WILEY library that yielded more than 90% matches (Plonjarean et al. 2007). The GC-MS analysis was performed on a FISON (GC) 8000 series/ MD 800 which is shown in Figure 4.12. Separation was achieved using helium as the carrier gas (1ml / min) with a fused silica capillary column 30m long, inner diameter 5 micron and 0.5 micron film thickness. The GC injector was in a split less mode. Injector and detector temperature were 250˚C and 260˚C respectively. The oven temperature was ranges from 100˚C to 250˚C. The effluent from the capillary column went directly into mass spectrometer. This instrument was operated is the full scan and electron impact ionization mode with an ionizitionation voltage 70eV and an acceleration voltage of 70V the iron source temperature was 200ºC and the GC-MS

transfer line was set to 250 ˚C. The sample was diluted with petroleum ether the data is compared with NIST and WILEY library to know the peak compounds. Figure 4.11 Schematic diagrams of GC-MS

84 Figure 4.12 Photographic view of gas chromatography mass spectrum analyzer. FISON (GC) 8000 SERIES / MD 800 4.4

ENGINE EXPERIMENTAL INVESTIGATIONS An experimental set up

is

made to evaluate the performance, emission and combustion parameters of diesel engine with essential instrumentation at different operating conditions. 4.4.1 Description of Engine Experimental Set up for Comparative of Different Methyl Esters Naturally aspirated (NA- DI) engine

is selected for evaluation of bio diesel, pine oil fuels based on two specific reasons. First, a NA-DI

engine represents a larger population of engines sold in agricultural and construction equipment for the last 55 years. Secondly, a naturally aspirated engine is more sensitive due to longer ignition delays and lower pressure injection equipment. The Kirloskar engine is one of the widely used engines in agricultural, pump sets, farm machinery, transport-vehicles, small and

85 medium scale commercial purposes. The engine can withstand higher pressures encountered during tests because of its rugged construction. Further, the necessary modification on the cylinder head and piston crown can be easily carried out in this type of engine. Hence this engine is selected for the present research work.

A single cylinder, water cooled, four stroke direct injection compression ignition engine

with compression ratio of 16.5: 1, developing 3.7 kW at

a rated speed 1500 rpm is

used for the present study. The specifications of the engine setup are tabulated in the appendix 1. The layout of the experimental setup is shown in Figure 4.13. The photographic view of the experimental setup is shown in Figure 4.14. Variable load tests are conducted for no load, 0.74, 1.48, 2.22, 2.96 and 3.7 kW power output at a constant rated speed of 1500 rpm, with fuel injection pressure of 200 bar and cooling water exit temperature at around 60ºC. The injection timing recommended by the manufacturer is 23˚ BTDC. The governor was used to maintain constant speed under varying load conditions, which control the fuel flow as load changes over engine. The engine had an open combustion chamber with

over head valves operated through push rods. A provision is

made in the cylinder head surface to mount an Optrand make pressure transducer for measuring the cylinder pressure. AVL-444 DI Gas analyzer

is

used to measure the exhaust tail pipe emissions such as HC, CO, CO 2 , and NOx. The smoke opacity is measure with the aid of AVL 437 standard smoke meter. Initially, before starting experimental tests, the engine is made to run under ideal condition as warm up phase and then the tests were conducted. The engine is started and allowed to warm-up for about 10 minutes. The engine is tested under five discrete part load conditions Viz; No load, 20%, 40%, 60%, 80% and 100%. The combustion characteristic varies in terms of pressure traces, maximum pressure and heat release. These performance and emission characteristics are compared with the results of base line diesel.

86 Figure 4.13 The layout of the experimental setup Figure 4.14 shows the

experimental setup photographic view

87 4.4.2 Fuel Supply Measurement The fuel from the tank is connected to a solenoid valve, the outlet of the solenoid valve is connected to a glass burette and the same is connected to the engine through a manual ball valve. The fuel solenoid of the tank remain open, until the burette is filled to the high level sensor, during this time the fuel is flowing to the top level, the fuel solenoid closes fuel tank outlet line now the fuel in the burette gets discharged to the engine. When the fuel level reached the high level optical slot sensor, the sequence running in the computer records the time of this event. Likewise when the fuel level crossed the low level optical slot sensor, the sequence running in the computer records the time of this event and immediately the fuel solenoid opens filling up the burette and the cycle is repeated. The volume of the fuel between the high level and low level sensors is 20 CC. Figure 4.15 shows the photographic view of fuel supply measurements.

Figure 4.15

Fuel Supply measurement photographic view

88 4.4.3 Temperature Measurement Figure 4.16 shows the photographic views of temperature measurements. The thermocouples are a widely used as a temperature sensor and can also be used as to convert thermal potential difference into electric potential difference. It can be measured a wide range of temperatures and system error of less than 1˚C is difficult to achieve. K-Type thermocouples are located at the following points in the engine test bed. (i) Engine exhaust (ii) Engine cooling water inlet (iii) Engine cooling water outlet

Figure 4.16 Temperature measurements photographic views

The output temperature transmitters are connected to the Data Acquisition Card.

89 4.4.4

Air Flow Measurements Figure 4.17 Air flow measurements photographic views The

air flow to the engine is routed through cubical air tank. The air tank fulfills the purpose of regulating the flow of air to the tank. The inlet of the air tank is provided with an orifice, the airflow rate is measured using the mass air flow sensor. Figure 4.17 shows the photographic views of air flow measurements. Mass air flow sensor is used to measure the airflow rate. A hot wire mass airflow sensor determined the mass of air flowing into the engine’s air intake systems. This is achieved by heating a wire with an electric current that is suspended in the engine’s air stream, not unlike a toaster wire. The wire’s electrical resistance increase with the wire’s temperature, which limits electrical current flowing through the circuit. When air flows through the wire, the wire cools decreasing its resistance, which in turn allows more current to flow through the circuit. As more current flows, the wire’s temperature increases until the resistance reaches equilibrium again. The amount of current required to maintain the wire’s electrical resistance is directly proportional to the mass of air flowing past the wire. The integrated electronic circuit converts the measurement of current into a voltage signal when is sent to the computer.

90 4.4.5 Speed and Load Measurements Figure 4.18 shows the photographic views of speed and load measurements. The engine was set to run at a constant speed of 1500 rpm. The load of the engine is obtained from load cell reading. The speed of the engine is monitored using photo sensor along with digital speed indicator.

Figure 4.18 Speed and load measurements Photographic views 4.4.6

Eddy Current Dynamometer The test engine is directly coupled by using a tyre coupling to an eddy current dynamometer with suitable switching and control facility for loading the engine. It is air cooled and the length of torque arm of the dynamometer is 0.195 m. The output shaft of the Eddy Current Dynamometer is fixed to a strain gauge type load cell for measuring applied load to the engine. Figure 4.19 shows the photographic view of Eddy Current Dynamometer and specifications are given in Appendix.2.

91

Figure 4.19 Eddy Current Dynamometer Photographic view 4.4.7

Combustion Analysis with Data Acquisition System

The combustion analysis of diesel and bio diesel-diesel

pine oil blends is

carried out with the assistance of combustion pressure sensor (Optrand make

combustion pressure sensor range 0-200 bar), Combustion parameters were analyzed using a combustion analyzer. The cylinder pressure and maximum rate of pressure rise were analyzed. The combustion analyzer used for the present research work is shown in Figure 4.20

and specifications are given in an appendix 3.

Figure 4.20 Photographic views of Encoder and combustion pressure sensor

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It is easy to operate, it contains menu driven parameter editor for setting up the system, utilized for TDC detection, the numerical monitor for on-line display of calculated results like Indicated Mean Effective Pressure (IMEP) or mass burn fractions as well as monitor program for oscilloscope like graph display. This versatile software designed by legion brothers written in “Visual C” was used for on-line data acquisition from the pressure transducer and crank angle Encoder (Make Kubler, 360 PPR coupled to crank shaft). 4.4.8 Exhaust Emission Measurements The intensity of smoke is measured by light obscuration method, in which the intensity of a light beam is reduced by smoke, which is a measure of smoke intensity. The oxides of nitrogen (NOx) are measured by chemical reaction method. The carbon monoxide emission is measured by Non- Dispersive Infra-Red (NDIR) analyzer and unburned hydrocarbons (UBHC) by Flame Ionization Detector which is a well-established and accepted method for measuring unburned hydrocarbons. The AVL five-gas analyzer and smoke meter are shown in Figure 4.21 and specifications are given in an Appendix 4 &

Appendix 5. Figure 4.21 AVL 437 standard smoke meter and AVL 444 digas analyzer photographic views

93 4.4.9

Experimental Procedure Initially the engine is allowed to run with diesel at a constant speed of 1500 rpm for nearly 30 minutes to attain the steady state conditions at the lowest possible load. During the investigation, the temperature of lubricating oil and temperature of the engine cooling water were held constant to eliminate their influence on the results. The speed of the engine was stabilized with injected fuel to attain the temperature of lubricating oil as 65˚C and temperature of cooling water as 60˚C. Then the following observations were made twice for concordance: The exhaust gas analyzer and smoke meter is switched on quite early so that all its systems will get stabilized before the commencement of the experiment and the following observations were documented. Time for 20 cc of fuel consumption. (s) Exhaust gas temperature. (˚C) Measurement of smoke using AVL

437 smoke meter Measurement of CO, CO 2 , HC, O 2 , and NOx using AVL 444 digas analyzer. Combustion parameters were analyzed using Optrand make combustion pressure sensor. Bio diesel is the pure or 100% bio diesel fuel. It is referred to as B100 or neat fuel. Bio diesel-diesel blend means, pure bio diesel blended with diesel. Bio diesel blends are referred to as Bxx. The xx indicates the amount of bio diesel blend i.e. B25 blend is 25% bio diesel and 75% diesel.

94 After completing the experiments with neat diesel, the same experimental procedure was repeated with B25 blend of the WME. The experiments and similar observations were repeated with other four blends in order B50, B75, and B100 for CPME, WME

and, B25CPME pine oil

and

diesel.

After completing the experiment with the bio diesel, the engine was allowed to run for about half an hour with diesel to eliminate the interference of the previous bio diesel fuel. The entire bio diesel, diesel pine oil blends were tested by the similar procedure. The diesel fuel is then retested to check repeatability. The results tended to confirm the result of the first run with diesel fuel. 4.4.10 Experimental Uncertainty This work, several measurements are taken only one sample type i.e., the

one instruments made used for measurements.

Necessary care has been taken to maintain the original accuracy by frequent calibration wherever possible. The uncertainties for the basic measurements liketime, barometer pressure,temperature,weight, length, etc., are equal to the least count of individual instruments. The errors on measures likeviscosity, specific gravity, characteristic gas constant, density etc., are taken from hand books and tables. The uncertainty on the derived quantity is calculated by using the method suggested by Holman (2012), which is based on the works of Kline and McClintock. The uncertainties in the measured values like engine power, fuel flow,

pressure andairflow

are calculated and the results are presented in Table 4.1.

95 Table 4.1 Uncertainties

of experimental S.No Variable Units Uncertainties 01 Engine power kW ± 0.9434 % 02 Airflow kg/hr ± 0.6481 % 03 Fuel flow kg/hr ± 0.7319 % 04

Cylinder pressure Bar ± 0.61644 05

Viscosity centiStokes ± 0.7 % 06 Smoke HSU ± 1 % 07 HC Ppm ± 0.2 It can be found from Table 4.1, except for measurement

of

emission, that the uncertainties involved in the various parameters are less than 1%. The emission calculation involves many parameters such as

engine

power, temperature and emission level. So the uncertainty rises in particular emission values. However, the uncertainty of 1.5% is still a minor value and may not affect the accuracy of results.

Chapter 5 presents the results and discussion of various methods of bio diesel production techniques, properties of raw oil, bio diesel-diesel

blends with pine oil. Performance, combustion and emission studies were studied.

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CHAPTER 5 RESULTS AND DISCUSSION PHASE 1 5.1 BIO DIESEL PRODUCTION FROM RAW OIL Methyl esters of watermelom seed oil and ceiba pentandra seed oil were produced. The parameters influencing bio diesel production such as speed of the stirrer, reaction time catalyst concentration and methanol to oil ratio were studied and presented in the following sections. 5.1.1 Production of Bio Diesel from Ceiba Pentandra Seed Oil 5.1.1.1 Effect on speed of the stirrer

Mixing is very important in the transesterification reaction, as an oil and fat are immiscible with potassium hydroxide-methanol solution.

Once the two phase are mixed the reaction is started.

Methanolysis is conducted with different stirrer rates such as 350, 400, 450, 500, 550,600, 650 and 700 revolutions per minutes (rpm).

The variation of methyl esters yield with different rate of stirrer is shown in Figure 5.1. It is observed that

the reaction is incomplete up to 500 rpm, and rate of mixing is insufficient for methanolysis. Further increasing the speed of 550 rpm

the yield of 92% obtained and again further increase in the speed of stirrer to 600 rpm, the yield of methyl esters was reduced to 88%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel for this batch type production unit.

This occurrence was reported with experimental work

97 performed by (Meher et al. 2006, Rashidand Anwar 2008, Ong et al. 2011, and Venkanna & Venkataramana 2011). Figure 5.1 Variation of different stirrer speed with methyl esters yield for transesterification of ceiba pentandra oil 5.1.1.2 Effect of reaction time The reaction time is required for the completion of alkaline-catalyst transesterification depends not only on the reaction temperature, but also on the degree of mixing in the process. The reaction rate of transesterification increases with increase in temperature and degree of mixing. The reaction time of transesterifaction process is conducted at 60 *H C ie. Nearer to the boiling point of the methanol.The variation of methyl ester yield with different reaction time as shown in Figure 5.2.The figure

shows that

yield of bio-diesel

is less at the beginning and reaches a maximum at the reaction time of 60 minutes (min) at 60 *

H

C and relatively decrease afterwards. The reaction time of the product yield after more than 65 minute to 75 minute has no

98 significant effect on the conversion triglycerides, but leads to a reduction in the product yield. This is because, longer reaction time enhances the hydrolysis of esters (reverse reaction of transesterification) which results in the

loss of esters as well as causing more fatty acids to form

soap. The gradual increase in reaction time shows the negative effect on the productyield. It is clear that the reaction time is an important controlling factor for the product yields. The similar observation is reported by Eevera et al. (2009), Karavalakis et al. (2011), Sbihi et al. (2014). Figure 5.2 Variation of different reaction time with methyl esters yield for transesterification of ceiba pentandra oil 5.1.1.3

Quantity of catalyst The effect of two catalyst used in the transesterification were compared through examining the esters content in the bio diesel product obtained. Two catalyst exhibited similar trends on the conversion of triglycerides to esters but different quantity of catalyst were required for 76 78 80 82 84 86 88 90 92 94 30 40 50 60 70 80

Yield (%) Reaction time (min.) CPME Reaction time : 60 min Reaction temp : 60 ˚C Speed : 550 rpm No. of wash : 5 Amount of KOH : 14 g Amount of Methanol : 500 ml

99

achieving the same conversion. The effect of NaOH and KOH quantity on the transesterification of the ceiba pentandra oil was investigated with its quantity varying from 8 g to 16g. The operating conditions during the reaction process were fixed as reaction temperature is 60ºC, reaction time is 60 minute and amount of methanol used for the study is 500ml.

Figure 5.3 present the result of variation of yield with different quantities of NaOH and KOH as catalysts.

Two catalyst exhibited similar trends on the conversion of triglycerides to esters but different amount yields were obtained for the same

amount of catalyst used. Figure 5.3 Variation of different catalyst quantity with methyl esters yield for transesterification of ceiba pentandra oil

Maximum esters content of the bio diesel product reached at 92% and 88% yield for catalyst quantity for 14g of KOH and NaOH respectively. The amount of KOH used was less than that of NaOH for same mass of feedstock oil. Hence, KOH is the best catalyst when compared to NaOH.

The

100 similar observation is reported by Karmee and Chadha 2005, Meher et al. (2006); Rashid & Anwar (2008); Fahad Ahmed (2016). It can be analyzed that from Figure 5.3, increase in NaOH/KOH concentration beyond the optimal value which results drop in yield of bio diesel production and soap formation. This

is

observed during the experimental study. This is because of addition of excess of alkaline catalyst (NaOH/KOH) caused more triglycerides participating in the saponification reaction with NaOH/KOH producing more soap there by reducing the yield of esters.

The another possible reason for the rise is formation of emulsion may be the

addition of excess amount of catalyst gave rise in the formation of an emulsion, which increased the viscosity and led to the formation of gels. The similar results were observed by Karmee & Anju Chadha (2005); Ramadhas et al. (2005 b) & Meher et al. (2006);

Nur Syakirah Talha & Sarina Sulaiman (2016). 5.1.1.4 Production of bio diesel from watermelon oil The above procedure is followed by production of biodiesel from watermelon seed oil. It is observed from Figure 5.4

the reaction is incomplete up to 500 rpm, and rate of mixing was insufficient for methanolysis. Further increasing the speed of 550 rpm

is yield 88% is obtained

and again further increase in the speed of stirrer to 600 rpm. The yield of methyl esters is reduced to 86%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel

from watermelon seed oil for this batch type production unit.

101

Figure 5.4 Variation of different catalyst quantity with methyl esters yield for transesterification of

watermelon oil. PHASE II 5.2

FUEL PROPERTIES Laboratory tests were carried out to determine the properties such as relative density, viscosity, flash point, cloud & pour point and calorific values for two different blend ratios ofesters in varying proportions viz., 25%, 50%, 75%, with diesel and 100% of esters (bio diesel). Gas chromatography

and mass spectrum

studies were also employed for qualitative analysis. The result of this study is presented below. 5.2.1 Specific Gravity The variation of specific gravity of ceiba pentandra oil methyl ester (CPME) and watermelon oil methyl ester (WME) and diesel blends with different percentage in volume are shown in Figure 5.5.

102 It can be observed from

the figure

specific gravity increases linearly with increasing concentrations of CPME, WME in diesel blends. The higher specific gravity of CPME and WME

compared to diesel may be attributed to the higher molecular weight of triglyceride molecules present in the oil. Slightly higher specific gravity of the

ester blends compared to base diesel makes their energy content on volume basis nearly closer to diesel than the energy content on mass basis. The fuel injection equipment operates on a volume metering system hence a higher specific gravity for esters and its blends with diesel results in the delivery of a slightly higher mass of fuel to engine.

As compared with WME specific gravity of CPME is slightly low for the same blend ratio.

These results are in agreement with work done by Alamu et al. (2007), Raheman and Ghadge 2007, Demirbas 2008,Srivastava, PK and Verma,

M 2008, Ijaz et al.., 2016).

Figure 5.5 Variation of percentage of bio diesel in diesel blends with specific gravity

103 5.2.2 Viscosity The variation of viscosity of the of ceiba pentandra oil methyl ester and watermelon oil methyl ester and diesel blends with different percentage in volume is shown

in

Figure 5.6. After the transesterification process,

the viscosity of the FAME is reduced considerably to levels comparable to (But still higher than) that of

diesel. All of the biodiesel blended with diesel have a slightly higher viscosity; particularly B100 of CPME and WME have higher viscosity than diesel and other blends. The viscosity rises with the increasing concentrations of methyl ester with diesel blends.

As compared to WME the values of viscosity is less for CPME for the same blend ratio at a temperature of 40˚ C.

This is due to the higher molecular mass in the structure of esters (Mittel 1996; Karmee & Chadha 2005; Joshi & Pegg 2007; Demirbas 2008; Rao 2011& Kaisan et al. 2013). Figure 5.6 Variation of percentage of bio diesel in diesel blends with viscosity

104 5.2.3 Flash Point Figure 5.7 shows the variation of flash point with percentage of

two types

of methyl ester and bio diesel in diesel blends.With reference to the figure, all blends have the flash point, which is higher than that of diesel fuel. Flash point increases linearly with increasing concentration of methyl ester and bio diesel-diesel in the blends. The flash point of diesel is 54ºC, the flash point of CPME and WME are 148ºC, and 154ºC respectively.It is also higher than that of diesel but lower than that of before transesterification of the oil. This is due to the larger molecular mass in the structure of oil and methyl ester companied with unsaturated and saturated fatty acids present in their structure. . This leads to rises

in

the flash point. This higher flash point of methyl esters and its blends with diesel makes storage safer. It also called as “safe fuel”. These results are in accordance with work done by Alamu et al. (2007); Srivastava & Verma (2008); Naik et al. (2008); Suresh Kumar et al. (2008) & Pradeesh et al. (2016). Figure 5.7 Variation of percentage of bio diesel in diesel blends with flash point

105 5.2.4 Cloud and Pour Point Figure 5.8 and Figure 5.9 shows the variation of cloud and pour point with percentage of bio diesel in diesel blends. From the

Figure 5.8 and Figure 5.9,

it was found that, cloud and pour points of diesel fuel are 2º C and 1º C respectively, whereas biodiesel at 100% volume (B100) becomes a gel at approximately 7ºC. In such a state it becomes problematic to use in CI engine, it may clog filters and causes difficulties in pumping from the fuel tank to the engine. This is due to presence of unsaturated and saturated fatty acids present in the structure. The cloud and pour points of oils and bio diesel can be reduced by blending it with diesel fuel. In general, the pour and cloud points of diesel were lower than that of bio diesel. Both pour point and cloud point of bio diesel and its blends with diesel fuel increase with increasing the concentrations of bio diesel. Three trials were conducted for each (biodiesel- diesel blends) sample to check the consistency of results. The difference among all the three measurements are never more than 1ºC. The presence of alcohol made the pour and cloud points of bio diesel slightly lower than that of raw oil. Upto B25 blend, these properties were found to be almost closer to that of diesel and as the blend ratio increases, the value of cloud point is also increases. This might be due to the presence of wax in oil, which begins to crystallize with the reduction in temperature. This difficult can be overcome by using lower blends up to 25% (Alamu et al. 2007;

Joshi & Pegg 2007; Srivastava & Verma 2008; Sajjad et al. 2014).

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Figure 5.8 Variation of percentage of bio diesel in diesel blends with cloud point Figure 5.9 Variation of percentage of bio diesel in diesel blends with pour point

107 5.2.5 Calorific Value Figure 5.10 Variation of percentage of bio diesel in diesel blends with calorific value Figure 5.10 shows the variation of calorific value with percentage of bio diesel in diesel blends. It is clearly observed from the

figure the calorific values of blends uniformly decrease with the increase in bio diesel percentage in blends. The calorific values of diesel and B100 of CPME and WME were 42,800 kJ/kg, 40016 kJ/kg and 39,850 kJ/kg respectively. Measured calorific values of diesel were found to be higher than that of bio diesel blends, due to the difference in their chemical composition from that of diesel or the difference in the percentage of carbon and hydrogen content of oil and esters (bio diesel) or due to the oxygen content as suggested (Naik et al. 2008). Tranesterification process improves the calorific value of raw ceibea pentandra seed oil compared to esters (bio diesel). This is due to unwanted fats presents in the raw oil were removed in

108 the tranesterification process. These results are in accordance with the work done by (Srivastava & Verma 2008; Suresh Kumar et al. 2008;

Giakoumis 2013) 5.2.6 Fuel Properties of Bio-Diesel-

Pine Oil-Diesel Blends In general, when high viscous fuels are blended with low viscous fuels such as

bio-diesel (

or) diesel the overall viscosity of the resulting blend could be lower. In the same way, the blend of high viscous CPME, less viscous pine oil with diesel has brought a reduction in biodiesel and diesel viscosity, increasing in pine oil viscosity

as shown in Table 5.1

which in- turns has improved the its atomization and mixing with air.

Table 5.1 Fuel properties of bio diesel, pine oil and diesel blends

Properties DIESEL PI B25 CPME+PI75 B25 CPME +PI50+DI25 B25 CPME +PI25+DI50 Kinematic viscosity (cSt) @ 40˚C 3.21 2.5 3.05 3.22 3.38 Specific gravity 0.838 0.841 0.852 0.851 0.85 (C.alorific value kJ/kg) 42800 42961 42227 42188 41147 Flash point ˚ C 52 49 73.5 74.5 75.5

109 PHASE III 5.3

QUALITATIVE ANALYSIS 5.3.1 Diesel The identified peaks and compounds in the diesel are shown in Figure 5.11 and Table 5.2 respectively. It is clearly shown from the above results it contains large peaks and high purities of –ane- group (octadecane, nondecane, furan etc.,) present, which leads to good combustion properties.

Figure 5.11 GC-MS analysis for diesel

110

Table 5.2 Major compounds in the diesel Peak No Compound Chemical formula M W (Mol. Weight) Retention time

R T (

min) 1. 2-Hydrobicyclo[3,1,1]heptan-6-one

C 7 H 10 O 2 126 6.262 2. 3-Methyl-2-(2-oxopropyl)furan C 8 H 10 O 2 138 8.067 3. Octadecane (cas)$$ N- Octadecane C 18 H 36 254 10.021 4. Octadecane(cas)$$ N- Octadecane C 18 H 36 254 11.968 5. 2-Hydrobicyclo[3,1,1]heptan-6-one C 7 H 10 O 2 126 12.525 6. Butyl 2,4- dimethyl-2-nitro-4- pentenoate C 11 H 19 O 4 N 229 13.535 7. Octadecane(cas)$$ N- Octadecane C 18 H 36 254 13.876 8. Butyl 2,4- dimethyl-2-nitro-4- pentenoate C 11 H 19 O 4 N 229 15.692 9. Nondecane (cas)$$ N- Nondecane C 19 H 40 268 17.450 10. Octadecane(cas)$$ N- Octadecane C 18 H 36 254 19.130 11. Butyl 2,4- dimethyl-2-nitro-4- pentenoate C 11 H 19 O 4 N 229 20.743 12. Nondecane(cas)$$ N- Nondecane C 19 H 4 0 268 22.291 13. Octadecane(cas)$$ N- Octadecane C 18 H 36 254 23.880 14. Nondecane(cas)$$ N- Nondecane C 19 H 40 268 25.600 15. Octadecane (cas)$$ N- Octadecane C 18 H 36 254 27.341 16. Docosane(cas)$$ N- Docosan$$C22H46 standard C 22 H 46 310 28.327 17. Nondecane(cas)$$ N- Nondecane C 19 H 40 268 29.270 18. Doctricontanol(cas)$$doctricontanol$$ N-Dot C 32 H 66 O 466 31.674 19.

Docosane(cas)$$ N- Docosane$$C22H46 standard C 22 H 46 310 34.731 20. 1-

Tetradecanol(cas)$$ alfol 14 $$ tetradecanol$$loxanol V $$ lanette K $$ lanette C 22 H 46 214 35.224

111 5.3.2 Methyl Esters of Ceiba Pentandra Oil Methanol (CH 3 OH) is used as the solvent alcohol and KOH as a catalyst for production of methyl esters (bio diesel) from ceiba pentandraoil. Some compounds are found in esters having good fuel properties of after transesterification. The identified peaks and compounds in the biodiesel of ceiba pentandra oil are shown in Figure 5.12 and Table 5.3 respectively. The majority of bio diesel (methyl ester) is in highly purified form and in large quantities. The compounds found in high quantities are shown in peak Nos. 1,2,3,7 as the esters compound (bio diesel), but some compound in

peak Nos. 4,5,6 and 8

remain as the impurities in less amount. These compounds of

peak Nos 6 and 8

are the fatty acid alcohols. Figure 5.12 GC-MS analysis for methyl esters of ceiba pentandra oil

112 Table 5.3 Major compounds in the methyl esters of ceiba pentandra

oil Peak No Compound Chemical formula M W (Molecular Weight) Retention time R T (min) 1. Tetradecanoic acid,10,13- dimethyl,methyl esters

C 17 H 34 O 2 270 19.499 2. Docasnedioic acid, dimethyl esters (cas) $$ methyl Docosane-1, 22-dioate. C 24 H 46 O 4 398 22.664 3. Heptacosanoic acid, 26- methyl,- methyl esters C 29 H 58 O 2 438 22.752 4. 3, 4 Epoxy-6, 9- octadecadiene. C 18 H 32 O 264 23.049 5. Pentane, 3-ethyl-(cas) $$ ethyl pentane. C 7 H 16 100 23.910 6. 1-Tetradecanol(cas)$$ 14 $$ tetradecanol $$ loxanol V $$ lanette K $$ C 14 H 30 O 214 26.022 7. Heptacosanoic acid, 26- methyl,- methyl esters C 29 H 58 O 2 438 26.161 8. 1-Dotricontanol. C 32 H 66 O 466 30.523 5.3.3

Methyl Esters of Watermelon Oil Methanol (CH 3 OH) is used as the solvent alcohol and KOH as a catalyst for production of methyl esters (bio diesel) from watermelon oil. The identified peaks and compounds in the methyl esters of watermelon oil are shown in Figure 5.13 and Table 5.4 respectively. According to the methyl esters results

as shown in

Table 5.4,

it is clearly seen that a high percentage of esters compound are formed as in peak Nos. 3, 4, 5, and 8. However, some compounds in peak Nos. 1, 2, 6, 7, 9, and 10 are in less percentage as impurities or supporting compound and may be found in dissolved form in esters or alcohol.

113

Figure 5.13 GC-MS analysis for methyl esters of watermelon oil

Table 5.4 Major compounds in the watermelon methyl esters Peak No Compound Chemical formula M W (Mol. Weight) Retention time

R T

in min 1 Heptanone, 4 Methyl (CAS) $$ 4- methyl – 3 Heptanone

C 8 H 16 O 128 4.927 2 3-Heptanone 4-methyl (Cas) $$ 4 methyl – 3- Heptano C 8 H 16 O 128 5.048 3 Hexadecanoic acid, ethyl esters (cas) $$ ethyl palm C 18 H 36 O 2 284 20.396 4 9-Octadecenic acid (z). Methyl esters (cas) $$ Met C 19 H 36 O 2 296 22.406 5 8.11.14 eicosatrienoic acid, methyl esters (cas) $$M C 21 H 36 O 2 320 22.522 6 2.5.8 Heptadecatrien – 1OL C 17 H 30 O 250 24.391 7 3.Methyl – 2 (2-Oxopropyl) furan C 8 H 10 O 2 138 25.935 8 Hexadecanoic Acid, Ethyl Esters (cas) $$ Ethylpalm C 18 H 36 O 2 284 33.019 9 4Isoxazolamine, 3 Phenyl C 9 H 80

N 2 160 34.068 10 3,4 Benzo – 6, 7-Dihydro – 6,7 Dimethyl (1H) Azepine – 2,5–Dio C 12 H 13 O 2

N 203 34.466

114 PHASE IV 5.4

PERFORMANCE AND EMISSION CHARACTERISTICS OF COMPRESSION IGNITION ENGINE FUELLED WITH CEIBA PENTANDRA AND WATERMALON METHYL ESTERS

A series of experiments has been conducted on a single cylinder four stroke 3.7kW direct injection diesel engine fuelled with diesel, pine oil, bio-diesel

from CPME, WME and

diesel blends. The tests were carried out in the standard injection timing (23º BTDC).

The important results of the experimental work are presented in the following sections. The configurations of different fuel blends are given in Table 5.5 Table 5.5 Configuration of the different fuels S.No Description Abbreviation 1 Diesel DI 2 Pine oil PO 3 Ceiba pentandra oil methyl ester CPME 4 Watermelon oil methyl ester WME 5.4.1 Brake Thermal Efficiency (Performance Parameters)

The following sections describe

the disparity of brake thermal efficiency with respect to brake power

for biodiesel-diesel blends of CPME, WME and diesel. Figure 5.14 and Figure 5.15 shows

the

variation of brake thermal efficiency with brake power for CPME, WME and its blends. The calorific value of

bio-diesel is

lower and the BSFC of biodiesel was much higher

115 compared to base diesel operation condition. Since, the brake thermal efficiency is inversely proportional to its BSFC and heating value of fuel. For all the blends, the brake thermal efficiency has the propensity to increase with increase in applied brake power.

At full load conditions, among the B25, B50, B75 and B100

bio-diesel

blends up to B25 has a maximum brake thermal efficiency of 30.69%, 30.35% and 31.88% respectively, for CPME, WME and diesel.At full load conditions,the brake thermal efficiency up to B25 is almost closer to that of base diesel

which is depicted in figure 5.14 and 5.15. As

comparedto B100 bio-diesel

operation

the brake thermal efficiency of B25 has increased by 3.76% and 3.28% for CPME and WME. It may be due to enhanced spray characteristics and dissolved oxygen

molecule structure in bio-diesel

of B25 blend of CPME and WME in the combustion chamber, which leads to effective utilization of air resulting in complete combustion of the fuel.

Figure 5.14 The variation of brake thermal efficiency for CPME

116

It is also evident from the plots that when the brake thermal efficiency is gradually decrease,

the percentage of bio-diesel (CPME and WME) from B50 to B100 is gradually increase. The reason for that is the supply of biodiesel fuel to the engine

is high as compare with

base diesel, in order to maintain the equal energy input to the engine. The high viscous of the blended fuels inhibits the fuel vaporization, proper atomization and combustion. This trend is also due to the combined effect of lower heating value, higher viscosity and density of the blended fuel.

In addition to this, studies conducted by pervious researchers Kumar et al. (2001), Nazar et al. 2004, Suresh Kumar et al. (2008), Dinesha and Mohanan (2015) and Abedin et al. (2016).

From the experimental results proved that performance of ceiba pentandra methyl ester (CPME) B25 blend is the best fuel compared to WME and other blends.

Figure 5.15 The variation of brake thermal efficiency for WME

117 5.4.2 Exhaust Emissions Diesel engine emits carbon monoxide, carbon dioxide, hydrocarbon, oxides of nitrogen

and smoke are considered to be the major emissions from exhaust. From the above emission stated NOx and smoke are most important emissions from a

C.I

engine that has to be efficiently controlled due to diffusive combustion and high flame temperature in the combustion chamber. The lesser amount of CO and HC emission produces

C.I engine than S.I engines. Moreover, in case of bio-diesel-

diesel blended fuelled engines, presence of airborne oxygen as well as its presence in the molecule structures of bio-diesel-diesel blended fuel aids nearly complete combustion of fuel. From the above study, CO, CO 2 , HC, NOx and smoke analysis presented in the following sections. 5.4.2.1 Carbon monoxide (CO) The variation of carbon monoxide (CO) emissions with respect to brake power presented in

Figure 5.16 and Figure 5.17

are discussed for CPME and WME and its blends with diesel. The characteristics of

DI-CI

engine shows when the load increases the air fuel ratio decreased. Usually, lower CO emissions emits CI engine due to excess air. CO is mainly dependent on the air-fuel ratio relatively to the chemically correct proportion and increase the air-fuel ratio becomes more than stoichiometric air-fuel requirements stated by (Kalam et al. 2003). It could be seen from Figure 5.16 and

Figure 5.17 that the CPME and WME fuelled engine emits less CO emission in compare with diesel fuelled. Because

bio-diesel

is an oxygenated fuel, which helps for complete combustion; reduction of CO emissions were observed in the experimental work. It is also clear from the plots the minimum CO emissions were observed with B100 for CPME and WME becomes maximum of 0.14 and 0.15 by %

in

vol. at full load condition. The combustion of biodiesel presence of extra oxygen molecule in the bio-diesel

118 chain might have converted some of the CO in to CO 2 and reduced CO formation. Similar finding were noted with CO in the experimental work conducted by Kumar et al. (2001), Sureshkumar et al. (2008), Datta and Mandal(2016). 5.4.2.2 Carbon dioxide Figure 5.18 and

Figure 5.19

show

the variation of carbon dioxide (CO 2) emissions in percentage in volume with brake power for diesel, CPME, WME and its blends. This measurements reveals that the brake power increases with increases in CO 2 emissions; it indicates the combustion chamber efficiency, When compared with diesel

the B25 percentage of blends emit almost same amount of CO 2 emission. This is because

bio-diesel

is generally a lower elemental carbon to hydrogen ratio and has a low carbon fuel than base diesel fuel.

The ratio of the blends increases

from B50 to B100, there is a slight increase in CO 2 emission which is due to more oxygen content of

methyl esters. In B100,

the value of CO 2 for CPME and WME were emits 6.7% and 6.9 % by vol. respectively and more CO 2 emissions in compression with base diesel. The insignificant differences were observed between

bio-diesel blends and diesel from B25 to B100. In general, bio diesel themselves are considered carbon neutral because, all the CO 2 released during combustion has been sequestered from the atmosphere during the process of photosynthesis for the growth of vegetable oil crops, which are later processed into fuel. Hence, bio diesel also helps to mitigate global warming, as carbon dioxide levels are kept in balance. These results are in accordance with experimental work done by Barsic and Humke (1981), Ramadhas et al. (2005 a), Alamu et al. (2007), Suresh Kumar et al. (2008) andKumar and Sharma (2016).

119 Figure 5.16 The Variation of COEmission for CPME Figure 5.17 The Variation of COEmission for WME

120 Figure 5.18 The Variation of CO 2 Emission for CPME Figure 5.19 The Variation of CO 2 Emission for WME

121 5.4.2.3 Hydrocarbon Figure 5.20 and

Figure 5.21show

the variation of hydrocarbon (HC) emissions in parts per million (ppm) with respect to brake power for diesel, CPME, WME and its blends. Experimental study exposed that the HC emission is reduced for all blended fuels (B25 to B100) as compare with diesel at full load. For all the blends, up to partial load the HC emission are lower, but increase at maximum load. This is due to the availability of relatively a lesser amount of oxygen content for the reaction, when more fuel is injected into the cylinder at higher load. The ratio

of the bio-diesel blends increases from B25 to B100, the

HC emission decreases. This is due to the fact that cetane number of diesel fuel is generally lower than that of

bio- diesel.

The HC emission for diesel at full load is 63

ppm where as for B100 of CPME and WME were 45ppm and 48ppm respectively. This is due to poor physical properties; high surface tension and viscosity affect the penetration rate, maximum penetration and droplet size which in turn affect the mixing of air-fuel. Apart from this the diesel has higher volatility compared to B100 which may also responsible for more HC emission; the engine operations are being affected. These results are good association with the previous researchers reported by (Kumar et al. 2001; Nazar et al. 2004; Srivastava & Verma 2008; Banapurmath et al. 2008; Agarwal et al. 2015). However, HC emission was lower for blended fuels (B25 to B75) as compared with B100 and diesel at maximum load.

The ignition delay period effects causes

in lower HC emission. In addition, the intrinsic oxygen contained in the biodiesel is responsible for the reduction in HC emission value for 22ppm which is less than the diesel. This is due to the sufficient oxygen presence in the biodiesel than base diesel. This phenomena also observed by the previous researchers (Sureshkumar et al. 2008 & Abedin et al. 2016).

122

Figure 5.20 The Variation of HC Emission for CPME

Figure 5.21 The Variation of HC Emission for WME

123 5.4.2.4

Oxides of nitrogen The oxide of nitrogen (NOx) in the exhaust emission

consists of

nitrogen dioxide (NO 2) and nitric oxide (NO).

The

NOx formation is highly dependent on engine technology. In general

concentration of

the NOx emissions varies linearly with the load, and when the engine load increases the overall air-fuel ratio increase resultant in an increase in the average temperature of the gas in the combustion chamber and more NOx formation.

The variation of NOx emissions (ppm) with respect to engine brake power

shown in Figure 5.22 and 5.23

for diesel, bio-diesel and its blends.The NOx emission of diesel at full load was 875ppm, whereas B100 of CPME and WME

is 768

ppm and 754ppm respectively. This is due to reduced premixed combustion rate and hence reduces in combustion temperatures. High viscosities of B100 bio-diesels resulted in poor atomization, decrease cone angle, reduce spray penetration and larger mean droplet size than base diesel, it resulting in lesser amount of air entrainment and to poor combustion characteristics leading to lower combustion temperatures. These results are in agreement with the experimental study done by (Patterson et al. 2006;

Agarwal & Agarwal 2007; Rashedul et al. 2015). The

bio-diesel

blends ratio increases (B25 to B50) with increase in NOx emissions. This is due to complete combustion of fuel increasing exhaust gas temperature and also by the presence of oxygen molecules in the

bio-diesel. when the emissions of NOx increased up to B50, bio-diesel blended fuel

is attributed to difference in the compressibility of thediesel andbio-diesel

blends fuel, which results in inadvertent advance in dynamic injection timing when

bio-diesel-

diesel blends are used in the engine. Apart from this, the bio-diesel-

diesel blends

has longer combustion duration compared to diesel resulting in higher exhaust gas temperature.

These results were in good association with the experimental results of previous researchers (Patterson et al. 2006; Nazer et al. 2004;

Prabakaran & Dinoop Viswanathan, 2016).

124 Figure 5.22 The Variation of

NO x Emission for CPME Figure 5.23 The Variation of NO x

Emission for WME

125 5.4.2.5

Smoke Smoke is nothing but solid soot particles suspended in the exhaust gas. The quantity of soot formed depends upon the type of fuel and fuel ratio. In general NOx and smoke are trade-off. The variation of smoke emissions with respect to brake power shown in Figures 5.24 and

Figure 5.25

discussed for diesel, CPME, WME and its blends. the smoke variations are given in Hartridge Smoke Unit (HSU). For all the bio-diesel blends, the brake power increase with increase in smoke emission. The more smoke emissions are forme is indicative of incomplete combustion of fuel. The smoke quantity present in the exhaust gas is measured to amount the particulate matter present in the

exhaust gas . The smoke emission of base diesel at full load is 55 HSU whereas B100 of CPME and WME is 67 HSU

and 71HSU respectively. This is due to lower volatility and higher viscosity can result in poor air-fuel mixture formation, resulting in higher smoke emissions. Another probable reason is B100 biodiesel has stronger molecule structures, which leads to poor atomization of fuel there by increasing smoke emission.

These results are in good agreement with the pervious researchers reported by (Hebbal 2006; Pradeep & Sharma 2007; Datta & Mandal 2016).

However at full load condition the smoke emission is reduced for lower blends (B25 and B50) as compared with the higher blends (B75 and B100) due to betterfuel combustion. The lower viscosity of blended fuels (B25 and B50) as well as oxygen present in the molecules lower the smoke emission

as reported by (Kalam et al. 2003; Banapurmath et al. 2008). When the bio-diesel blend ratio increase (B75 and B100)

the value of

smoke emission also increased. This is may be due to high viscosity

of B100,

which reduce the fuel jet Reynolds number, decrease in air fuel mixing rate and leading to larger mean fuel drop sizes at higher blends (B75 and B100)

as suggested by (Barsic & Humke 1981; Devan & Mahalakshmi 2009).

126

Figure 5.24 The variation of Smoke Opacity for CPME Figure 5.25 The variation of Smoke Opacity for WME

127 From Phase IV experimental results proved that B25 CPME is the best blend ratio for DI-CI engine as compared with other bio-diesel blends. Because brake thermal efficiency of the engine is not affected and emissions are reduced (Expect NOx). So, in this phase concludes B25 CPME is best blend ratio compared with WME and its blends.

In general, when high viscous fuels are blended with low viscous fuels such as

bio-diesel (

or) diesel the overall viscosity of the resulting blend could be lower. In the same way, the blend of high viscous CPME, less viscous pine oil with diesel has brought a reduction in

bio-diesel

and diesel viscosity, increasing in pine oil viscosity, which in-turns has improved the its atomization and mixing with air.

In Phase V pine oil mixed with B25 CPME blend. Because pine oil has a less viscosity

compared to

diesel. On the other hand, B25 CPME is higher viscosity compare with diesel (Devan & Mahalakshmi et al. 2009; Vallinayagam et al. 2014). PHASE-V 5.5 PERFORMANCE AND EMISSIONS CHARACTERISTICS OF DI-CI ENGINE FUELLED WITH B25 CPME-PO- DIESEL

BLENDS

In this phase

same test engine were used without any modification, to study the, performance, emissions and combustion characteristics of PO, B25 + PO75 (B25%CPME and Pine oil 75%), B25+ PO50+DI25 (B25%CPME, Pine oil 50% and Diesel 25%), B25+PO25+DI50 (B25% CPME, Pine oil 25% and Diesel 50%) and diesel.

128 5.5.1

Brake Thermal Efficiency Figure 5.26 shows

the disparity of brake thermal efficiency (BTE) with respect to brake power. For diesel, pine oil,

bio-diesel-

pine oil-diesel and its blends. B25+PO50+DI25 blends brake thermal efficiency (30.71%) is close to diesel (31.88%) and PO (31.98%) due to viscosity and heating value close to diesel. The oxygen content in

bio-diesel

and pine oil slightly larger fuel droplets which contribute better atomization, leading to proper mixing, vaporization, ignition and as a result complete combustion. The engine brake thermal efficiency depends upon number of factors but the most meaningful property is viscosity, density and heating value

suggested by (Kasiraman et al. 2012).

Figure 5.26 The variation of brake thermal efficiency for biodiesel, PO, and its blends

129

PO brake thermal efficiency is higher than diesel, and PO75+DI25 blends brake thermal efficiency also nearly close to diesel. This is may be due to higher heating value of PO than diesel. From this experimental investigation with

bio-diesel-

PO-diesel blends in diesel engine, it is observed that 50% of PO is found to be optimize blend because for blends beyond 50% of PO, the engine suffered some vibration, particularly at full load condition. However, lesser cetane number of PO affected the fuel injection suggested by Vallinayagam et al. (2014). From the experimental results,

it is

proved that performance of B25+PO50+DI25 blend is the best fuel compared to other

blends. 5.5.2

Exhaust Emission The exhaust emissions such as CO, CO 2 , HC, NOx and smoke are measured and analyzed. For above mentioned emissions, NOx and smoke is the most significant emissions for diesel engine due to high flame temperature and diffusive combustion in the combustion chamber. Since NOx and smoke emissions from current diesel technologies are closer to the limits permitted by regulations and both limits will be even more stringent in the near future, these two emissions will be critical factors in the development of new diesel engine. Apart from this, diesel engine produces lesser amount of CO and HC emissions then spark ignition engine.From the above facts, emission from

B25 CPME-pine oil blended with diesel focused on NOx and smoke. 5.5.2.1

Oxides of nitrogen Figure 5.27 describes

the variations of NOx emissions with respect to brake power for diesel, pine oil, biodiesel-PO-diesel and its blends. The emissions of NOx for diesel at maximum load is 875ppm, where PO, B25 +PO75, B25+PO50+DI25 and B25+PO25+DI50 are 913, 899, 869 and

130 865ppm respectively. POand B25+PO75 blend NOx emission was higher than diesel. This is due to the sudden burning of PO air mixture by the auto- ignition of diesel, lower cetane number (longer ignition delay) of fuel is higher heat release rate, could have resulted higher in-cylinder temperature and presence of inbuilt oxygen with in the PO has produced more NOx formation

suggested by (Lee et al. 2011 & Zhang et al. 2012).

B25+PO50+DI25 blend NOx emission is lower than diesel, PO, and B25+PO75 blends. The blend of less viscous pine oil and high viscous CPME (lower calorific value) with diesel has brought a reduce in

bio-diesel

and diesel viscosity, increasing in pine oil viscosity, which in-turns has improved the atomization and mixing with air

suggested (Devan & Mahalakshmi et al. 2009; Vallinayagam et al. 2014).

Figure 5.27 The variation of

NOx for biodiesel, PO, and its blends.

131 5.5.3

Smoke Emission The variation of smoke emission with respect to brake power for diesel, PO, bio-diesel-PO-diesel blends as shown in Figure 5.28. Smoke is attributed to either mixture of fuel-air that are too lean to auto ignite (or) to support a propagating flame (or) mixture of air fuel that are also rich to ignite

is suggested by (yashiyuki et al. 2000).

PO, biodiesel-PO- diesel blends smoke emission was (51 HSU) lower compared to diesel (55

HSU).

This may be due to higher calorific value, lower viscosity and boiling point of PO is effectual mixing with air forming locally lean regions limiting the primary smoke formation, responsible for decreasing smoke emission reported by Anand et al. (2010); Vallinayagam et al. (2014).

Figure 5.28 The

variation of

smoke for biodiesel, PO, and its blends

132 PHASE- VI 5.6 ENGINE COMBUSTION ANALYSIS

FUELLED WITH B25 CPME - PINE OIL – DEISEL The combustion investigation of pine oil-bio-diesel blends with diesel is

carried out with the help of combustion pressure sensor (optrand make pressure range 0-200bar),

and software (Engine test express, legion brothers,written “visul C”), crank angle Encoder (Make kubler, 360 PPR coupled to crankshaft).

In a C.I engine, cylinder pressure depends on the burned fuel fraction during the premixed burning phase, ie, initial stage of combustion. This stage of combustion depends upon the ability of the fuel to mix well with air and burn. Heat release diagram is a quantitative description of timely burning fuel in engine. It has a major effect on cycle efficiency and maximum cylinder pressure. The heat release program is based on the first law of thermodynamics. 5.6.1 Cylinder Pressure Figures 5.29 depicts the variation of cylinder pressurewith respect to the crank angle

of diesel, B25+PO50+DI25 and B100

CPME at full load and standard injecton timing (23º BTDC).

It is proved that, at same operating condition as compared to diesel, the methylester percentage blends rises the peak cylinder pressure were reduced and also the peak pressures of diesel, B25 +PO50+DI25 and B100 CPME were recored as 65.16bar, 63.85 bar and 47.44 bar respectively. The major possible reason for decreased cylinder pressure when compared to diesel is greater molecular structure, lesser heating value and volatility slightly higher viscosity of the bio-diesel. Additional possible reason owing to slow burnning rates of the methyl ester

133 blends. The supplementary imaginable reason is during the combustion of diesel, it released more amount of energy.

Another possible explanation the combination of slow burning rates at a lower minimum pressure rise of the bio-diesel fuels, which might have caused lower mechanical stress in the blended fuel operation.

Therefore combustion noise is lower when bio diesel- diesel blends operation. Figure 5.29 The variation of cylinder pressure with crank angle for biodiesel, PO, and its blends. 5.6.2 Heat Release Rate Figure 5.30 shows the variation of maximum heat relase rate (kJ/m 3 degree) with respect to the crank angle fordiesel, B25+PO50+DI25 and B100 CPME.The combustion rate difference, start of combustion and the fraction of fuel burned in the premixed modes of fuel are identified by the heat release rate. At full load and standard injection timing (23 *

H

BTDC). The variation of

134 heat release rate with crank angle were recorded. The maximum and minimum heat release rate for diesel, B25+PO50+DI25 and B100 CPME were recorded as 62.57kg/m 3 deg., 59.58 kg/m 3 deg., 43.58 kg/m 3 deg. The bio- diesel blends had shorter ignition delay, When compared to diesel, Because of heat relase ratereduced, the shorter ignition delay is caused. Diesel is longer ignition delay to allowed for more air/fuel prepartion, which is ready to auto ignite and result a higher premixed peak (Ozener et al. 2014). Figure 5.30 The variation of heat release rate with crank angle for biodiesel, PO, and its blends

135 CHAPTER 6 CONCLUSIONS The following conclusions were drawn in the present experimental work 6.1

BIO-DIESEL PRODUCTION

Biodiesel is produced from ceiba pentandra seed oil and watermelon seed oil. The catalyst KOH is found to be superior than NaOH, So, KOH is the best suitable catalyst of methanolysis process. The maximum yields of 92% and 88% bio-diesel obtained from ceiba pentandra oil and watermelon oil. 6.2

PHYSICO-CHEMICAL PROPERTIES AND QUANTITIVE ANALYSIS The transesterification

process improved the fuel properties of the oil with respect to relative density,

viscosity, flash point, cloud point, pour point and calorific value.

The methyl esters properties of ceiba pentandra with pine oil blends physic- chemical characteristics are near to ASTM standards. When the

blends ratio of bio-diesel increased, increased kinematic viscosity, specific gravity, flash point and reduced

136 calorific value when compared to diesel.

The methyl esters properties of ceiba pentandra with pine oil blends physic- chemical characteristics are near to ASTM standards.

Quantitive analysis proved that produced CPME and WME have more ester content high purity and good fuel property. 6.3

PERFORMANCE AND EMISSION CHARACTERISTICS

The engine performance of B25 blend (30.69%) is nearly similar to that of diesel (31.88%) at the same time not affected the engine performance. Compared with diesel, bio-diesel-diesel blends emit lower CO, HC, smoke and slightly increased NOx emission.

From the experimental results proved that performance of ceiba pentandra methyl ester B25 best blended fuel compared to WME and other blends.

B 25

CPME +

PO50 + DI25 blends brake thermal efficiency (31.06) is close to diesel(31.88%) due to viscosity and heating value

are close to diesel.

Up to B25 CPME + PO50 + DI25 blends can be used in a diesel engine without any engine modification. It does not affect performance, emission and combustion characteristics. The ceiba pentandra with pine oil-diesel blends improved the fuel atomization and easily mixed with air result in increased in brake thermal efficiency compared with CPME-WME.

137 6.4

COMBUSTION ANALYSIS

The overall combustion characteristics of B 25

CPME +

PO 50 + DI 25 blend is proved that the cylinder pressure and heat release rate is (63.85bar and 59.58 kJ/m 3 deg.) higher than B100CPME (47.44 bar and 43.58kJ/m 3 deg.) and closer to diesel (65.16 bar and 62.57 kJ/m 3 deg.). 6.5 OVERALL CONCLUSION From the experimental result proved that 25% of ceiba pentandra methyl ester blended with 25% of diesel and 50% of pine oil blends ratio is a best alternative fuel for naturally aspirated direct injection diesel engine producing a 3.7 kW at a rated speed of 1500 rpm without any modification of the engine. 6.6

FUTURE WORK

Addition of

adding some anti-freezing additives is needed to improve the cold flow properties of bio diesel to use under severe winter conditions.

Engine performance characteristics on the

blends of bio diesel and pine oil can be investigated on the various injection timing

in DI-CI

engine. Measurement of aldehyde emissions can be carried out. Study the characteristics of LHR engine fuelled with the B25CPME+PO50+DI25 blend ratio.

Study the NOx emission on DI-CI

engine can be operated with the different percentage of EGR.

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APPENDIX 1 SPECIFICATIONS OF THE ENGINE MAKE Kirloskar AV1 Type Single cylinder, Direct Injection,Naturally Aspirated, 4 Stroke,water cooled Engine BHP 3.7 kW = 5HP @ 1500 rpm BORE 80 mm STROKE 110 mm COMPRESSION RATIO 16.5:1 TYPE OF IGNITION Compression Ignition INJECTION TIMING 23º bTDC METHOD OF LOADING Eddy Current Dynamometer AVL 444-5 GAS ANALYZER Emission (HC, CO, CO 2 , O 2, & NO X) AVL 437 SMOKE METER Smoke Density FUEL USED Diesel, Bio-diesel, pine oil Blends

139 APPENDIX 2 SPECIFICATIONS OF THE DYNAMOMETER Make PowerMag Type Air cooled eddy current dynamometer Loading Auto loading system, the engine loading is automatically done through the computer Coupling type Tyre Load measurement method Strain gauge Maximum Power 5hp @ 2000 rpm

140 APPENDIX 3 SPECIFICATION OF THE COMBUSTION ANALYSER Make Optrand make combustion pressure sensor Type AutoPSI-S AutoPSI-TC and Auto PSI-HT Sensors 1. Specifications Over Pressure : 2 x Pressure Range (typical) Non-Linearity & Hysteresis : ±0.5% FS under non-combustion Conditions, under constant temperature ±1% FS under combustion conditions, i.e., varying temperature within one Combustion cycle Frequency Range : 0.1 Hz to 20 kHz (Standard) Diaphragm Resonant Frequency : 120 kHz min. Cable Operating Temperature : -40˚C to 200˚C Sensor Housing Temperature Range: -40˚C to 350˚C

141 Fiber optic Cable Length : 1.5m (5') Fiber optic Cable Min. Sensor Type : Sealed Gauge Interface Unit : Integrated with Sensor Bending Radius : 5mm (3/16") (Analog) 5V DC input : 0.5 - 4.5 V Diagnostic Output Signal : 9-18V DC input: 0.5 – 2.5 V (Analog) 5V DC input : 0.5 – 2.5 V Pressure Output Signal : 9-18V DC input: 0.5 – 4.5 V Power Supply Voltage : 9-18V DC or 5V DC Interface Temperature Range : AutoPSI-S,A,TC:-20˚C to 60˚C Current Draw : 85 mA Max, 50 mA Typical AutoPSI-HT : -20˚C to 125˚C Vibration : 100G Pressure Media : Gaseous or Liquid Guaranteed Lifetime : 1, 2 or 3 Years depending on sensor package or application

142 APPENDIX 4 SPECIFICATIONS OF THE AVL DIGAS 444 ANALYSER Make AVL Type AVL Digas 444 Power supply 11…22 voltage 25 W Connector gas in 180 l/h, max. overpressure 450 hPa Warm up time 7 min. Storage temperature 0…50 C Operating temperature 5 …45 C Response time t 95 15s Inclination 0…90 Relative humidity 95%, non-condensing Weight 4.5 kg net weight without accessories Dimension (w x d x h) 270 x 320 x 85 mm 3 Interfaces RS 232 C, Pick up, Oil temperature probe

143 APPENDIX 5 SPECIFICATIONS OF THE AVL 437 SMOKE METER Make AVL 437 Smoke meter Type IP 52 Measuring range 0 to 100 opacity in % 0 to 99.99 absorption m -1 Measurement chamber Effective length 0.430 m 0.005m Accuracy and reproducibility 1 % full scale reading Light source Halogen bulb 12 V/5W Heating time 220 V approximately 20 min. Dimensions 570mm 500mm 1250mm Power supply 190 – 240 V AC, 50…60 Hz, 2.5 A Maximum smoke temperature 250 C Weight Approx. 50 kg net

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