Major Project -II

SYNTHESIS OF BIODIESEL USING SUPERCRITICAL TRANS-ESTERIFICATION AND PERFORMANCE EVALUATION OF A SINGLE CYLINDER DIESEL ENGINE USING LINSEED METHYL ESTER.

Submitted to **Delhi Technological University** in partial fulfillment of the requirement for the award of the Degree of

Master of Technology
In
Thermal Engineering
By

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UNDER THE SUPERVISION OF

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DECLARATION

I, hereby declare that the work embodied in the dissertation entitled "**SYNTHESIS OF BIODIESEL USING SUPERCRITICAL TRANS-ESTERIFICATION AND PERFORMANCE EVALUATION OF A SINGLE CYLINDER DIESEL ENGINE USING LINSEED METHYL ESTER.**" in partial fulfilment for the award of degree of MASTER of TECHNOLOGY in "THERMAL ENGINEERING", is an original piece of work carried out by me under the supervision of Prof. Naveen Kumar, Mechanical Engineering Department, Delhi Technological University. The matter of this work either full or in part have not been submitted to any other institution or University for the award of any other Diploma or Degree or any other purpose what so ever.

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CERTIFICATE

This is to certify that the work embodied in the dissertation entitled **"SYNTHESIS OF BIODIESEL USING SUPERCRITICAL TRANS-ESTERIFICATION AND PERFORMANCE EVALUATION OF A SINGLE CYLINDER DIESEL ENGINE USING LINSEED METHYL ESTER."** by **DILEEP KUMAR GUPTA, (**Roll No.- **2K12/THR/09)** in partial fulfilment of requirements for the award of **Degree of Master of Technology in Thermal Engineering**, is an authentic record of student's own work carried by him under my supervision.

This is also certified that this work has not been submitted to any other Institute or University for the award of any other diploma or degree.

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ABSTRACT

Due to high energy demand and limited availability of fossil fuels, the energy necessity becomes a point of apprehension as it results in hike of fuel prices. It is essential to develop renewable energy resources while considering the impact on environment. In the last decade, demand of alternative fuels has increased a lot. Therefore, researchers have already started working on the aim of developing a green fuel to overcome the future energy demand. And as we know that the biodiesel is generally prepared from the non-edible and renewable resources thus, it can be among the competitive alternative future fuels. Besides that, it does not require any prior engine modifications for its usual advantage among other alternative fuels while using it within certain boundaries. However, the process biodiesel production is in itself time consuming which increases the cost of production while decreasing the yield. Supercritical method is drawing major attention for its efficient means with overcoming the negatives of conventional production processes. This method generally results in a high conversion rate of triglycerides into the FAME's (fatty acid methyl ester). But, it is confined to vegetable oils having low FFA content. Hence, in the present study, a non-catalytic supercritical trans-esterification process was utilized to produce biodiesel from Linseed oil. The analysis for the influence of temperature and time of run was done on the yield of FAME's, DG and TG. Results revealed that FAME's content increases with increase in temperature and the time of run. However, the pattern was uniform for all initial range of temperatures though a slight decrement was observed at higher temperature due to the thermal degradation of esters.

And further linseed oil methyl ester that was prepared from the above described process of supercritical transesterification were blended with diesel and investigation were carried out to evaluate performance and emission characteristics of linseed oil methyl ester blends (LME) (5%,10%,15% and 20% by volume) on unmodified diesel engine. The properties of these blends were found to be comparable to diesel and confirming to both the American and European standards. Engine performance (brake specific energy consumption, brake thermal efficiency) and emissions (CO, HC, NO_x, and smoke density) were measured to evaluate the behavior of the diesel engine running on biodiesel and diesel. It has been found that at peak load condition the brake thermal efficiency of 20% blend of LME is higher than that diesel. Emissions of HC and CO were found to be reduced with increase in LME percentage compared to diesel confirming better combustion due to the oxygenated fuel. However, there was substantial increase in NO_X in case of LME compare to diesel. From overall analysis it may be concluded that linseed oil biodiesel showed better performance and emission characteristics than that of diesel and used as a fuel in unmodified single cylinder diesel engine.

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NOMENCLATURE

A/F	Air to Fuel
ATDC	After Top Dead Center
AVL-437	AVL-437 Smoke Meter
LME05 D95	Linseed oil 5% + Diesel 95%
LME10 D90	Linseed oil 10% + Diesel 90%
LME15 D85	Linseed oil 15% + Diesel 85%
LME20 D80	Linseed oil 20% + Diesel 80%
D100	Neat diesel
BMEP	Break Mean Effective Pressure
BSEC	Brake Specific Energy Consumption
BSFC	Brake Specific Fuel Consumption
BTE	Brake Thermal Efficiency
BTDC	Before Top Dead Center
^{o}C	Degree Celsius
CA	Crank Angle
cc	Cubic centimetre
CI	Compression Ignition
CN	Cetane Number
CO	Carbon monoxide
CO_2	Carbon Dioxide
cSt	Centi Stoke
CV	Calorific Value
DI	Direct Injection

DF	Diesel fuel
g	Gram
g/cc	Gram per cubic centimetre
HC	Hydrocarbon
HP	Horse Power
IC	Internal Combustion
IS	Indian standard
Kgoe	Kilogram of oil equivalent
Mm	Millimeter
Mt	Million Tonnes
Mtoe	Million Tonne of Oil Equivalent
Mboe	Million Barrels of Oil Equivalent
NO	Nitric Oxide
No.	Numbers
NO_2	Nitrogen Di-oxide
NO_x	Oxides of Nitrogen
PM	Particulate Matter
ppm	Parts per million
rpm	Revolutions Per Minute
sfc	Specific Fuel Consumption
TDC	Top Dead Center
UBHC	Unburnt Hydrocarbon
v/v	Volume/ Volume
ρ	Density
%	Percent
MG	Mono Glycerides

DG	Di Glycerides
TG	Tri Glycerides
FAME	Fatty Acid Methyl Ester

CHAPTER 1

INTRODUCTION

1.0 ENERGY CRISIS

Energy is the prime mover of economic growth and is vital to the sustenance of a modern economy. Future economic growth importantly depends on the long-term availability of energy from sources that are affordable, accessible and environmental friendly. In the past few decades there has been a serious focus on the strategies to reduce air pollution and its hazardous effects like global warming and ozone layer depletion.

In the case of the developing countries, the energy sector assumes a critical importance in view of the ever-increasing energy needs requiring huge investments to meet them.

The Internal combustion engines have revolutionized the world in last hundred years but also contributed significantly towards environmental degradation. Post Kyoto Protocol; there have been considerable efforts to reduce GHG emissions and more emphasis is now given on using clean source of energy.

Diesel Engines due to its higher efficiency and ruggedness play a very significant role in Indian economy as these are used in agriculture, transport and industrial sectors. However, diesel engines also emit harmful emissions and pollute environment. Hence, from the point of view of protecting the global environment and the concern for long-term supplies of conventional diesel fuel, it becomes necessary to develop alternative fuels that give engine performance at par with diesel.

In this scenario, non-edible oils and its derivative – biodiesel provides a sustainable solution to the problem. By replacing a substantial amount of diesel fuel with an oxygenated alternative fuel, a significant reduction in emissions of the internal combustion engine could be achieved.

1.1 ENERGY SCENARIO

Secure, reliable and affordable energy supplies are fundamental to global economic stability and growth. There has been an enormous increase in the global demand for energy in recent years as a result of industrial development and population growth. Many countries spent billions of rupees to meet their current energy demand. The largest reserve of fossil fuels that is available in India is that of coal. India ranks fifth in terms of reserves of global coal reserves and shares 7 % of the global coal reserve and this reserve are used to feed the thermal power plants throughout the country.

India is among the fastest growing economy of world and use energy extensively to sustain its growth. Together, coal and oil represent about two-thirds of total energy use. Natural gas accounts for a seven percent share, which is expected to grow with the discovery of new gas deposits. Combustible renewables and waste constitute about one fourth of Indian energy use. This share includes traditional biomass sources such as firewood and cow dung, which are used by more than 800 million Indian households for cooking [1].

From the table 1.1 it is clear that India accounts for 4.67% of world's primary energy consumption in 2013. The per capita electricity consumption is lower than many developing countries. The per capita energy consumption in 2009 was 585 kgoe (Kg of oil equivalent) and that of the world was 1802.5 kgoe [2]. Currently, India is one of the world's fastest-growing economy. During the period between 2006 and 2010, the country's gross domestic product (GDP) increased at a CAGR of 8.2%, while global GDP increased at a CAGR of 4.5% [3]. The rapid increase in economic activity has been accompanied by rising energy consumption. During the period between 2006 and 2010, India's primary energy consumption increased at a CAGR of 8.3%, from 381.4 million tons of oil equivalent (mtoe) to 524.2 mtoe. Coal, oil and natural gas are the major sources of primary energy in India, accounting for 52.9%, 29.6% and 10.6%, respectively, of the primary energy consumption. Although the country has the world's fourth-largest coal reserves, the demand-supply gap of coal has been consistently increasing, with domestic production unable to keep pace with the demand. The deficit, in case of oil and gas, is even more. India holds just 0.7% of the world's proven oil reserves while accounting for 3.9% of the global oil consumption, thus importing 73% of its oil consumed. The country has 0.8% of the world's proven natural gas reserves accounting for 1.9% of the worldwide gas consumption, which results in importing nearly 20% of its natural gas consumed through LNG [2].

Table 1.1: The primary energy requirements of different countries
(In million tonnes of oil Equivalent) [2]

Commodity → **↓Country**	**Oil**	**Natural Gas**	**Coal**	**Nuclear Energy**	**Hydro electric**	**Renewable**	**Total**
CANADA	103.5	93.1	20.3	23.1	88.6	4.3	332.9
CHINA	507.4	145.5	1925.3	25.0	206.3	42.9	2852.4
FRANCE	80.3	38.6	12.2	95.9	15.5	5.9	248.4
INDIA	175.2	46.3	324.3	7.5	29.8	11.7	294.8
JAPAN	208.9	105.2	128.5	3.3	18.6	9.4	473.9
RUSSIAN FEDERATION	153.1	372.1	93.5	39.1	41.0	0.1	698.8
UNITED KINGDOM	69.8	65.8	36.5	16.0	1.1	10.9	200.1
USA	831.0	671.0	455.7	187.9	61.5	58.6	2265.7
WORLD	4185.1	3020.4	3826.7	563.2	855.8	279.3	12730.5

India does not have huge reserves of crude petroleum and it is heavily dependent upon imports to meet its requirement for automobiles and other sectors.

In the fiscal year 2012-13, indigenous crude petroleum production in India stood at 42.0 million tonnes, whereas consumption was nearly 175.2 million tonnes resulting in a humongous forex outflow of $144.3 billion for crude oil import. A major culprit in the arena of trade deficit witnessed by an emerging economy like India is its excessive dependence on overseas crude oil for domestic requirements.

To improve the present energy crisis, future energy conversion in India should be sustainable which include increase share of renewable fuel, increase efficiency of fuel conversion, reduce environmental impacts, and increase knowledge. In this context, efforts must be put to develop and promote the use of renewable sources of energy to meet the ever increasing energy requirement.

Oil constitutes over 35% of the primary energy consumption in India. It is expected that this would rise both in terms of absolute amount and proportion. The demand projections placed over 250 million metric tons by 2024-25 [4].

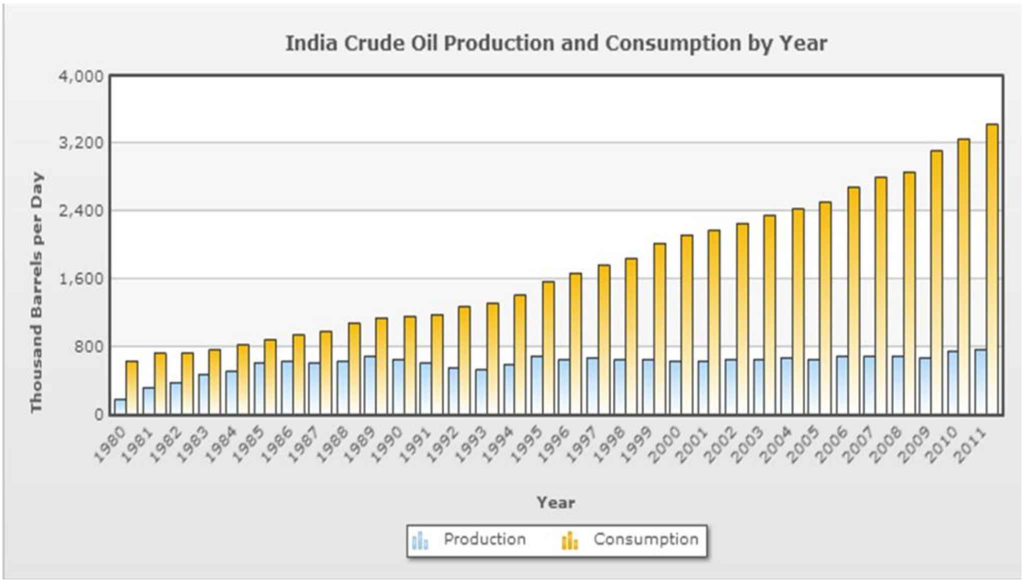


Fig. 1.1: India crude oil production and consumption by year

Table.1.2: Year wise oil production and consumption in India [2].

Year	Production(million ton)	Consumption(million ton)
2003	37.3	116.5
2004	38.2	119.5
2005	36.6	121.9
2006	37.8	128.3
2007	37.9	138.1
2008	37.9	144.7
2009	37.2	152.6
2010	40.8	155.4
2011	42.3	163.0
2012	42.0	173.6
2013	42.0	175.2

The fig.1.1 shows the production and consumption of crude oil over the past three decades and table 1.2 indicate the production and consumption of oil over past one decade. It can be seen from figure 1.1 that production of crude oil as compared to the consumption of crude oil is much lesser. Hence India has to heavily depend on imports to meet its crude oil requirement.

The country imported 163.59 million tonnes of crude oil in 2010-2011 to meet its oil requirement thereby spent nearly 4.559 trillion INR. The world's oil consumption pattern is shown in fig1.2 [2].

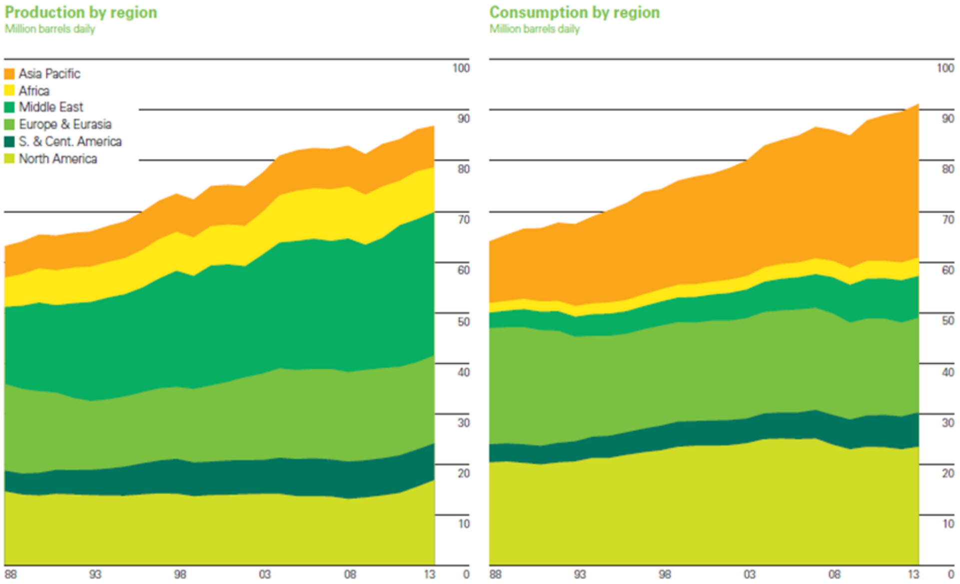


Fig. 1.2: Production and consumption of crude oil by region[2]

From the fig. 1.2 it can be seen that the production of oil in Asia pacific regions is much lower as compared to the consumption of oil products in the Asia pacific region. Hence, it triggers the need for the partial or complete replacement of oil products with the alternate fuels.

1.2 RENEWABLE ENERGY RESOURCES

The potential of renewable energy sources is enormous as they can meet the world's energy demand. Renewable energy sources such as biomass, wind, solar, hydropower, and geothermal can provide sustainable energy services, based on the use of routinely available, indigenous resources. A transition to renewables-based energy systems is looking increasingly likely as their costs decline while the price of oil and gas continue to fluctuate. In the past 30 years solar and wind power systems have experienced rapid sales growth, declining capital costs and costs of electricity generated, and have continued to improve their performance characteristics. In fact, fossil fuel and renewable energy prices, and social and environmental costs are heading in opposite directions and the economic and policy mechanisms needed to support the widespread dissemination and sustainable markets for renewable energy systems are rapidly evolving. It is becoming clear that future growth in the energy sector will be primarily in the new regime of renewable energy, and to some extent natural gas-based systems, not in conventional oil and coal sources. Because of these developments market opportunity now exists to both innovate and to take advantage of emerging markets to promote renewable energy technologies, with the additional assistance of governmental and popular sentiment. The development and use of renewable energy sources can enhance diversity in energy supply markets, contribute to securing long term sustainable energy supplies, help reduce local and global atmospheric emissions, and provide commercially attractive options to meet specific energy service needs, particularly in developing countries and rural areas helping to create new employment opportunities there [5].

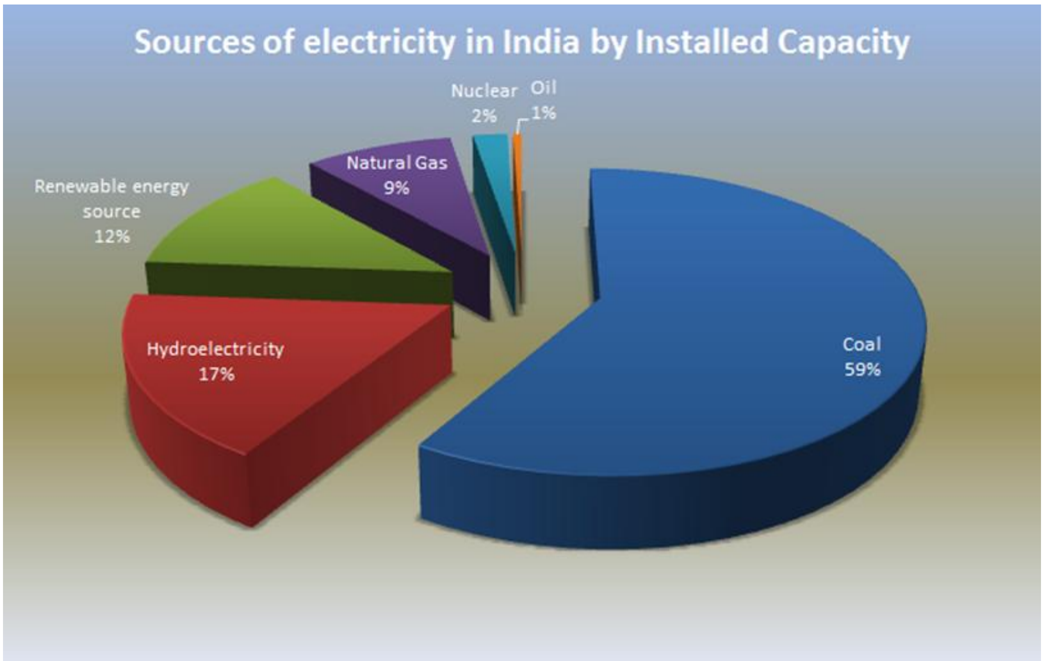


Fig.1.3: Share of various sources in power generation [6].

India, which has to build up its energy infrastructure to keep pace with the economic and social changes, faces a formidable challenge. Energy and electricity requirements have risen sharply in recent years, and this trend is likely to continue in the foreseeable future. As on October 2012, India has a total installed capacity of 209.28 GW, with coal being the principal source of electricity, followed by large-scale hydroelectric power. Renewable energy takes third place at 12%, having jumped nearly four times in the last decade [7].

1.3 FUTURE OUTLOOK

World primary energy consumption is projected to grow by 1.6% p.a. over the period 2010 to 2030, adding 39% to global consumption by 2030. The growth rate declines, from 2.5% p.a. over the past decade, to 2.0% p.a. over the next decade, and 1.3% p.a. from 2020 to 2030 Almost all (96%) of the growth is in non-OECD countries. By 2030 non-OECD energy consumption is 69% above the 2010 level, with growth averaging 2.7% p.a. (or 1.6% p.a. per capita), and it accounts for 65% of world consumption (compared to 54% in 2010). OECD energy consumption in 2030 is just 4% higher than in 2010, with growth averaging 0.2% p.a. to 2030. OECD energy consumption per capita is on a declining trend (-0.2% p.a. 2010-30).

The renewable energy technologies, including biofuels, wind, solar, geothermal, wave and tidal energy, see the fastest increase in demand, but their share of total energy use still reaches only 5.2% in 2030 up from 1.3 percent in 2010 [8].

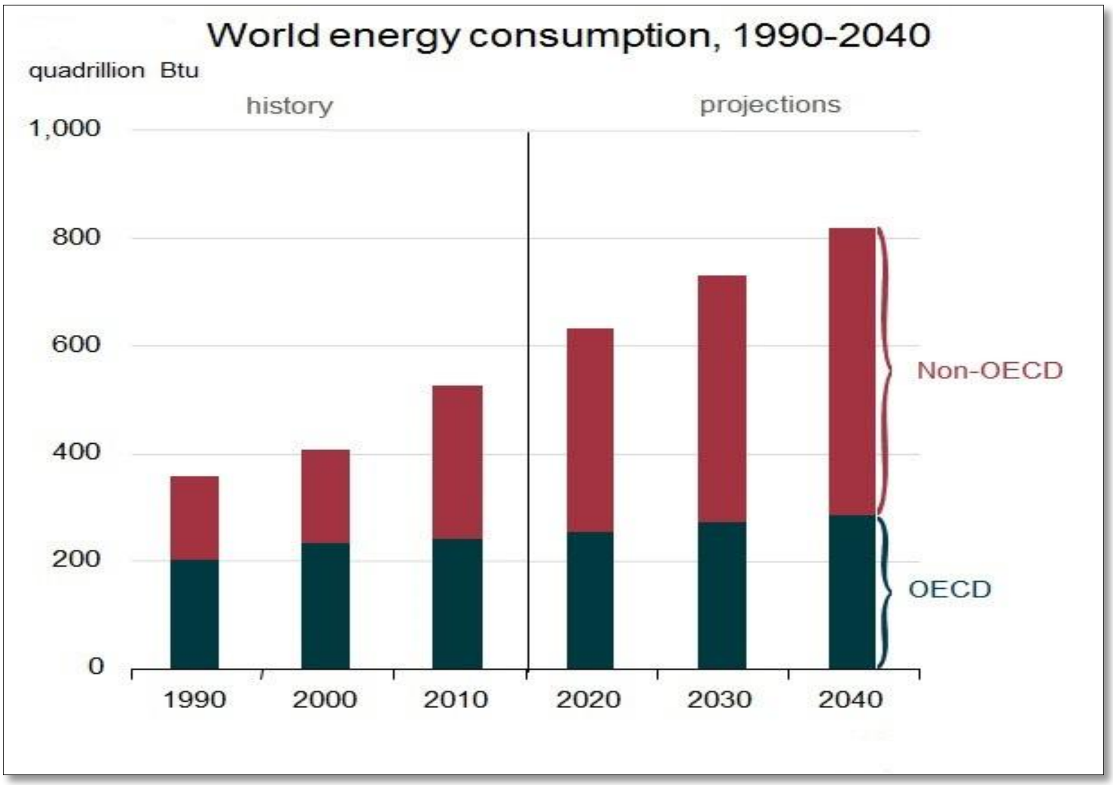


Fig.1.4: World energy consumption by International Energy Outlook 2012.

From the figure 1.4, it can be concluded that the world energy consumption of the Non-OCED countries will be greater than twice the energy consumption for the OCED countries in 2040.

And the demand for the energy consumption for the Non-OECD countries also doubles in the next two decades.

1.4 ENVIRONMENT DEGRADATION AND ITS EFFECT GLOBALLY

The indiscriminate use of fossil fuels has increased the air pollution comprising of the emissions of unburnt Hydrocarbons, NO_X emissions, CO emissions and CO_2 emissions. All these emissions tend to degrade the environment and cause Global Warming and ozone layer depletion.
The ozone layer is a layer in Earth's atmosphere which absorbs most of the Sun's UV radiation. It contains relatively high concentrations of ozone (O_3), although it is still very small with regard to ordinary oxygen, and is less than ten parts per million, the average ozone concentration in Earth's atmosphere being only about 0.6 parts per million. The ozone layer is mainly found in the lower portion of the stratosphere from approximately 20 to 30 kilometres (12 to 19 mi) above Earth, though the thickness varies seasonally and geographically. The ozone layer absorbs 97–99% of the Sun's medium-frequency ultraviolet light (from about 200 nm to 315 nm wavelength), which potentially damages exposed life forms on Earth. The ozone layer can be depleted by free radical catalysts, including nitric oxide (NO), nitrous oxide (N_2O), hydroxyl (OH), atomic chlorine (Cl), and atomic bromine (Br). Hence the emissions from the internal combustion engines tend to increase the NO_X emission thereby indirectly contributing to ozone layer depletion [9].

In 1985, a British scientist working in Antarctica discovered a 40 percent loss in the ozone layer over the continent. When Goddard Space Flight Centre researchers reviewed their data, they confirmed the ozone loss. Since then, scientists have relied on instrumentation developed by Goddard to keep track of the environmental phenomenon, which in the 1990s prompted a worldwide ban on chlorofluorocarbons (CFCs), a chemical used for refrigeration and other industrial uses. In this Fig 1.5, the blue/purple areas show low ozone, while the red areas indicate higher ozone levels.

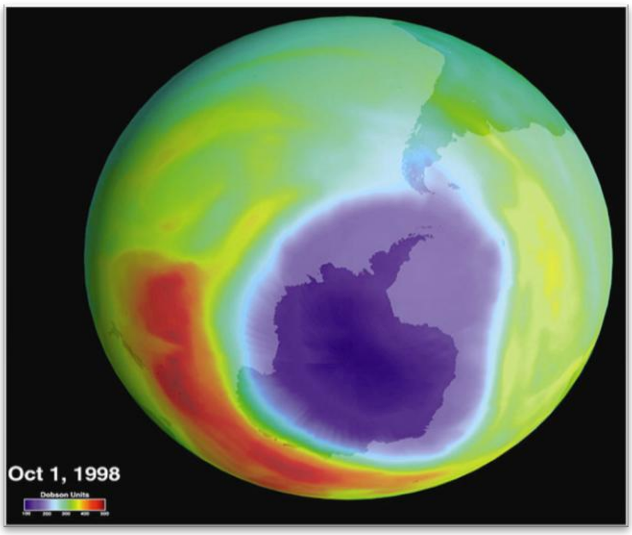


Fig.1.5: Hole in the Ozone Layer over Antarctica

Although ozone is considered a pollutant in the troposphere the atmospheric layer that contains the air we breathe in higher altitudes, notably in the stratosphere, ozone is considered vital. Stratospheric ozone blocks harmful ultraviolet radiation produced by the Sun. Scientists worry that the large ozone opening over the poles generally deplete ozone levels around the globe, which could cause a health risk to animals and plants.

1.5 GLOBAL WARMING

Global warming is the rise in the average temperature of Earth's atmosphere and oceans since the late 19th century and its projected continuation. It is one of the major threats in the present scenario. One of the prominent reasons that contribute towards global warming is the increase in CO_2 concentration in the atmosphere. The other factors which tend to increase Global warming are nitrous oxide emissions and methane.

Detailed researches of climatic events of the past 150 years have revealed that the temperatures have risen all over the globe, with the warming occurring in two phases. The first phase was from 1919 to 1940, with an average temperature gain of 0.35°C, and the second phase was from 1970 to the present, exhibiting temperature gains of 0.55°C.

Records show that the past 25 years have been the warmest time of the past 5 centuries. The global warming has resulted in the warming of the oceans, rising of the sea levels, melting of glaciers, and diminished snow cover in the Northern Hemisphere.

The recent catastrophic climatic events like the massive floods in Pakistan and India, the Hurricane Katrina in the United States, the prolonged droughts in Australia, China, Pakistan, India and Texas, are all the results of increased temperatures due to global warming. The increase in global CO_2 concentration in the atmosphere is shown in fig.1.6.

The last four complete years of the Mauna Loa CO_2 record plus the current year are shown. Data are reported as a dry air mole fraction defined as the number of molecules of carbon dioxide divided by the number of all molecules in air, including CO_2 itself, after water vapour has been removed.

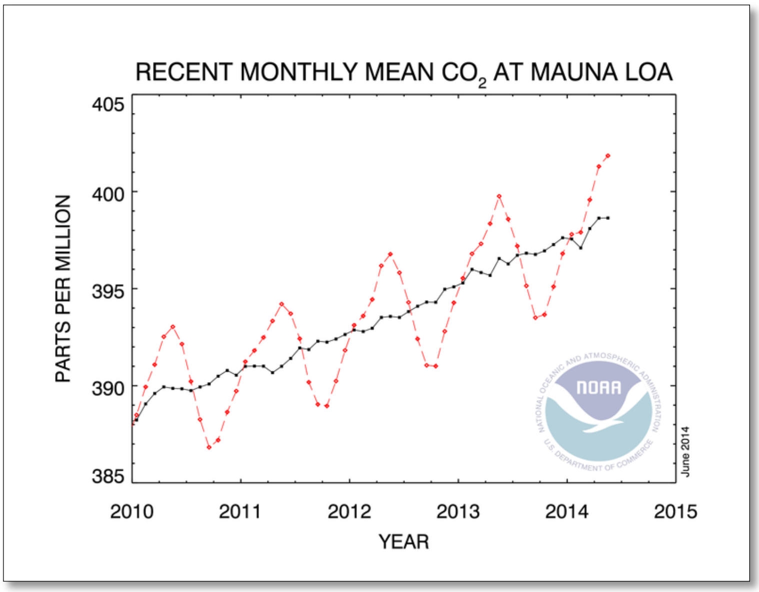


Fig. 1.6: Global CO2 concentration recorded at Mauna Loa Observatory

In the figure 1.6, the dashed red line with diamond symbols represents the monthly mean values, cantered on the middle of each month. The black line with the square symbols represents the same, after correction for the average seasonal cycle. It can be concluded that the global CO_2 emissions are increasing yearly. In April 2012, the CO_2 emissions level at Mauno Lao observatory was estimated at as 396.18 ppm.

Scientists have high confidence that global temperatures will continue to rise for decades to come, largely due to greenhouse gasses produced by human activities. The Intergovernmental Panel on Climate Change (IPCC), which includes more than 1,300 scientists from the United States and other countries, forecasts a temperature rise of 2.5 to 10 degrees Fahrenheit over the next century.

According to the IPCC, the extent of climate change effects on individual regions will vary over time and with the ability of different societal and environmental systems to mitigate or adapt to change.

The IPCC predicts that increases in global mean temperature of less than 1.8 to 5.4 degrees Fahrenheit (1 to 3 degrees Celsius) above 1990 levels will produce beneficial impacts in some regions and harmful ones in others. Net annual costs will increase over time as global temperatures increase.

"Taken as a whole," the IPCC states, "the range of published evidence indicates that the net damage costs of climate change are likely to be significant and to increase over time."[10]

Since the beginning of the heavily researched period of global warming, more certainties have been known, and some new connections have been made that are likely. Scientists in the mid and late 1980's, understood a good deal about greenhouse gases, but could not make an appropriate connection with the increase of the global mean temperature. Today, scientists have discovered that greenhouse gases trap heat in the Earth's atmosphere and tend to warm the planet.

By increasing the levels of greenhouse gases in the atmosphere, human activities are strengthening Earth's natural greenhouse effect. The key greenhouse gases emitted by human activities remain in the atmosphere for periods ranging from decades to centuries.

The effects of the warming are in some cases as broad as some of the hypothesis of the cause of the warming. Rising global temperatures are expected to raise sea level, and change precipitation and other local climate conditions. Changing regional climate could alter forests, crop yields, and water supplies. It could also affect human health, animals, and many types of ecosystems. Deserts may expand into existing rangelands, and features of some of our National Parks may be permanently altered. Most of the United States is expected to warm, although sulphates may limit warming in some areas. Scientists have made a connection between greenhouse gases and the increase in global mean temperature. Some greenhouse gases occur naturally in the atmosphere, while others result from human activities. Once, all climate changes occurred naturally. However, during the Industrial Revolution, we began altering our climate and environment through changing agricultural and industrial practices. Before the Industrial Revolution, human activity released very few gases into the atmosphere, but now through population growth, fossil fuel burning, and deforestation, we are affecting the mixture of gases in the atmosphere [11].

1.6 CO_2 EMISSIONS OUTLOOK

Since the beginning of the Industrial Revolution, the burning of fossil fuels has contributed to a 40% increase in the concentration of carbon dioxide in the atmosphere from 280 ppm to 400 ppm. Global CO_2 emissions rose by 4.6% in 2010, after having declined in 2009 due to the impact of the financial crisis, in particular on Western economies. In absolute terms, global CO_2 emissions increased by 1.3 $GtCO_2$ between 2009 and 2010. In 2010, 43% of CO_2 emissions from fuel combustion were produced from coal, 36% from oil and 20% from gas. Between 2009 and 2010, CO_2 emissions from the combustion of coal increased by 4.9% and represented 13.1 $GtCO_2$. Currently, coal fills much of the growing energy demand of those developing countries (such as China and India) where energy-intensive industrial production is growing rapidly and large coal reserves exist with limited reserves of other energy sources.

Without additional abatement measures, the WEO 2012 projects that emissions from coal will grow to 15.3 $GtCO_2$ in 2035. However, adopting a pathway towards limiting the long term temperature increase to 2°C as in the WEO 2012 450 Scenario – through use of more efficient plants and end-use technologies as well as increased use of renewables, nuclear and carbon capture and storage (CCS) technologies – could see coal consumption drop and CO_2 emissions from coal reduced to 5.6 Gt by 2035. Energy Technology Perspectives 2012 (ETP 2012) also shows that intensified use of coal would substantially increase CO_2 emissions unless there was a very widespread deployment of CCS.

Traditionally, industrialised countries have emitted the large majority of anthropogenic greenhouse gases (GHGs). More recently, however, shares of developing country emissions have been rising very rapidly and are projected to continue to do so.

Table: 1.3 CO2 emissions in 2012 (million tonnes CO2) and CO2/capita emissions, 1990–2012 (tonnes CO2/person) [13]

Country	**Emissions 2012**	**Per capita emissions**				
		1990	**2000**	**2010**	**2011**	**2012**
Annex I* United States	5,200	19.6	20.6	17.6	17.1	16.4
European Union	3,700	9.1	8.4	7.8	7.5	7.4
Germany	810	12.7	10.4	9.9	9.6	9.7
United Kingdom	490	10.3	9.2	8.2	7.5	7.7
Italy	390	7.5	8.1	6.9	6.7	6.3
France	370	6.9	6.9	6.2	5.8	5.8
Poland	320	8.2	7.5	8.7	8.7	5.8
Spain	290	5.9	7.6	6.1	6.2	6.1
Netherlands	160	10.8	10.9	10.7	10.0	9.8
Russian Federation	1,770	16.5	11.3	11.9	12.4	12.4
Japan	1,320	9.5	10.2	9.7	9.8	10.4
Canada	560	16.2	17.9	16.2	16.3	16.0
Australia	430	16.0	18.5	19.4	19.4	18.8
Ukraine	320	14.9	7.2	6.6	7.0	7.1
Non-Annex I China	9,900	2.1	2.8	6.4	6.9	7.1
India	1,970	0.8	1.0	1.5	1.5	1.6
South Korea	640	5.9	9.8	12.2	12.9	13.0
Indonesia	490	0.9	1.4	1.9	2.0	2.0
Saudi Arabia	460	10.2	12.9	15.6	15.6	16.2
Brazil	460	1.5	2.0	2.2	2.3	2.3
Mexico	490	3.6	3.6	3.9	3.9	4.0
Iran	410	3.6	5.2	5.2	5.3	5.3
South Africa	330	7.3	6.9	6.4	6.3	6.3
Taiwan	280	6.2	10.5	11.2	11.9	11.8
Thailand	260	1.6	2.8	3.6	3.8	3.9

** Annex I countries: industrialised countries with annual reporting obligations under the UN Framework Convention on Climate Change (UNFCCC) and emission targets under the Kyoto Protocol. The United States signed but not ratified the protocol, and thus the US emission target in the protocol has no legal status.*

The Kyoto Protocol commits industrialised countries (as a group) to curb domestic emissions by about 5% relative to 1990 by the 2008-12 first commitment period. Alongside the agreement to negotiate a new climate agreement by 2015, certain countries have agreed to take commitments under a second commitment period of the Kyoto Protocol to begin in 2013. The Kyoto Protocol also creates “flexible mechanisms” by which industrialised countries can transfer emission allowances among themselves and earn emission credits from emissions reduction projects in participating developing countries and economies in transition (EITs). Despite its extensive coverage (192 countries), the Protocol is limited in its potential to address global emissions since not all major emitters are included in reduction commitments. The United States remains outside of the Protocol’s jurisdiction and though most developing countries have signed, they do not face emissions target [12].

It can conclude from the Table 1.3 that maximum CO_2 emissions were from china followed by United States. The CO_2 emissions in India have doubled in the past two decades.

1.7 DIESEL ENGINE AND INDIAN ECONOMY

Diesel engines are the backbone of Indian economy. In India, the consumption of diesel fuel is nearly five times higher as consumption of gasoline. Diesel Engines due to its higher efficiency and ruggedness play a very significant role in Indian economy as these are used in agriculture, transport and industrial sectors. However, diesel engines also emit harmful emissions and pollute environment [14]. In the financial year 2012-13 the consumption of diesel in India was 69.59 million tones and the consumption of petroleum in India was 15.65 million tonnes [15].

Diesel (HSD), largely a transport fuel, forms the biggest chunk (35 per cent) of total petroleum product consumption in India. Diesel is mainly used in the road transport, rail transport, and agriculture and power generation sectors. Road transport and agriculture account for 73-75 percent of total diesel consumption. The balance is accounted for by the railways 4-5 per cent, the manufacturing industry (captive power generation) and power utilities 13-14 per cent and other end users. The transport fuel demand was met with by considerable imports in past six years [16]. From the point of view of protecting the global environment and the concern for long-term supplies of conventional diesel fuel, it becomes necessary to develop alternative fuels that give engine performance at par with diesel [17].

1.8 NEED FOR ALTERNATE FUELS

The world is facing crisis due to the dwindling sources of fossil fuels. Rapid depletion of conventional energy is a matter of serious concern for the mankind. It is clear from the above data that the large portion of the primary energy requirement is derived from oil (a fossil fuel) and country spent trillions of rupees to meet the oil energy requirement. Hence all this mandate the need to look for options such as substitute fuels, fuel conversion technologies, renewable energy and efficient energy systems etc. There is a necessity to find alternate fuels for different industrial needs. Alternate fuels can be suitably used in automobiles and aircrafts. Different researchers have conducted experiments on vegetables oils and alcohols as alternative fuels for I.C engines.

The selection of alternate fuels should be done so as to ensure:-

- No or minimum modification in the design of engine.
- Use of same storage and transportation infrastructure.
- No toxicity and bio degradability providing safe transportation and handling.
- Capability of being produced locally and low investment cost.

These fuels are essentially non-petroleum and result in energy security and environmental benefits. The alternate fuels tend to solve the problem of depleting fossil fuels and environmental degradation too.

According to the IEA, improvements in Internal Combustion Engine Technology and uptake of next generation vehicles as well as alternatives will lead to a 56% reduction in new car emission intensity by 2030.

1.9 POTENTIAL FUELS AVAILABLE

Performance tests have shown suitability of variety of alternative fuels such as hydrogen, alcohols, biogas, producer gas and various types of edible and non-edible oils. The economics of the fuels like vegetable oils, ethanol, and methanol etc. compared to the traditional petroleum resources are marginal.

The fuels available as alternate fuels are:-

1.9.1 Algae based fuels

Algae based biofuels have been hyped in the media as a potential panacea to our Crude Oil based Transportation problems. Algae could yield more than 2000 gallons of fuel per acre per year of production. Algae based fuels that were successfully tested by the U.S. Navy Algae based plastics show potential to reduce waste and the cost per pound of algae plastic is expected to be cheaper than traditional plastic prices.

1.9.2 Biodiesel

Biodiesel is made from animal fats or vegetable oils, renewable resources that come from plants such as, soybean, sunflowers, corn, olive, peanut, palm, coconut, safflower, canola, sesame, cottonseed, linseed etc. Once these fats or oils are filtered from their hydrocarbons and then combined with alcohol like methanol, biodiesel is brought to life from this chemical reaction. These raw materials can either be mixed with pure diesel to make various proportions, or used alone. Despite one's mixture preference, biodiesel will release a smaller number of its pollutants (carbon monoxide particulates and hydrocarbons) than conventional diesel, because biodiesel burns both cleaner and more efficiently. Even with regular diesel's reduced quantity of sulphur from the ULSD (ultra-low sulphur diesel) invention, biodiesel exceeds those levels because it is sulphur-free.

1.9.3 Alcohol fuels

Methanol and Ethanol fuel are primary sources of energy; they are convenient fuels for storing and transporting energy. These alcohols can be used in "internal combustion engines as alternative fuels", with butanol and Iso propyl alcohol also having known advantages, such as being the only alcohol-based motor fuel that can be transported readily by existing petroleum-product pipeline networks, instead of only by tanker trucks and railroad cars.

1.9.4 Hydrogen

Hydrogen is an emission less fuel. The by-product of hydrogen burning is water, although some mono-nitrogen oxides NOx are produced when hydrogen is burned with air [18].

1.10 BIODIESEL BLENDS

Biodiesel is a domestically produced, renewable fuel that can be manufactured from new and used vegetable oils, animal fats, and recycled restaurant grease. Biodiesel's physical properties are similar to those of petroleum diesel, but the fuel significantly reduces greenhouse gas emissions and toxic air pollutants. It is a biodegradable and cleaner-burning alternative to petroleum diesel.

Biodiesel can be blended and used in many different concentrations. They include B100 (pure biodiesel), B20 (20% biodiesel, 80% petroleum diesel), B5 (5% biodiesel, 95% petroleum diesel), and B2 (2% biodiesel, 98% petroleum diesel). The most common biodiesel blend is B20, which qualifies for fleet compliance under the Energy Policy Act (EPAct) of 1992.

As long as the vehicle was manufactured after 1993, biodiesel can be used in diesel engines and fuel injection equipment with little impact on operating performance. Regardless of your vehicle's age, it's a good idea to check original engine manufacturer (OEM) recommendations before using biodiesel. Most OEMs approve blends up to B5 in their vehicles. Some approve blends up to B20 if the fuel meets certain specifications and standards. One even approves B100 in certain types of farm equipment.

Testing results of engines operating on B20 show similar fuel consumption, horsepower, and torque to those operating on conventional diesel. Biodiesel has some additional desirable characteristics, including a higher cetane number (a measure of the ignition value of diesel fuel) and higher lubricity (the capacity for reducing friction) than U.S. diesel fuel [19].

The use of biodiesel in conventional diesel engines substantially reduces emissions of unburned hydrocarbons (HC), carbon monoxide (CO), sulphates, polycyclic aromatic HCs, nitrated polycyclic aromatic HCs, and particulate matter (PM). The reductions of these compounds increase as the amount of biodiesel blended into diesel fuel increases. B100 provides the best emission reductions, but lower-level blends also provide benefits. B20 has been shown to reduce PM emissions by 10%, CO by 11%, and unburned HCs by 21% (see Fig. 1.7) [19].

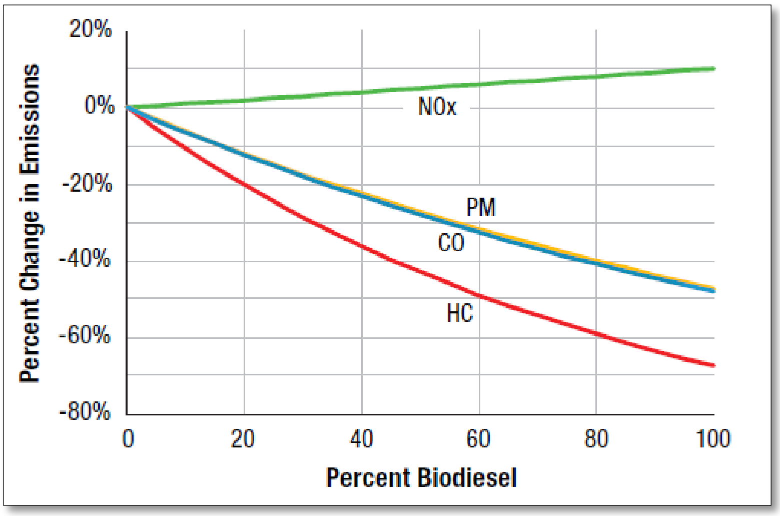


Fig. 1.7: Average Emission Impacts of Biodiesel for Heavy-Duty Highway Engines

Biodiesel use also reduces greenhouse gas emissions because the carbon dioxide released in biodiesel combustion is offset by the carbon dioxide sequestered while growing the feedstock. B100 use reduces carbon dioxide emissions by more than 75% compared to diesel. Using B20 reduces carbon dioxide emissions by 15%.

1.11 PRESENT WORK

In context to present work, a more elaborate discussion on adaptation of blends of Linseed bio-diesel and diesel is made. The use of vegetable oils in the diesel engines was known since 1920s. Biodiesel is an alternative fuel that can be made from SVO (Straight Vegetable Oil) in a chemical process called transesterification or sometimes two step transesterification process depending on the amount of free fatty acid content of the fuel. Biodiesel has substantially different properties than SVO and results in better engine performance. In particular, biodiesel has a lower boiling point and viscosity than SVO. Because of its improved qualities, vehicle and engine manufacturers generally approve the use of biodiesel blends in their products, though not all approve blend levels as high as 20% [20].

The biodiesel can be made from different edible and non-edible oils. In United States soybean oil is of prime interest because of its easy availability. Similarly in European countries more focus is given producing bio-diesel from rapeseed oil. Countries with tropical climate prefer to utilize coconut oil or palm oil. Other vegetable oils, including sunflower, safflower, etc., have also been investigated. Furthermore, other sources of biodiesel studied include animal fats and used or waste cooking oils [21]. In India, a variety of non-edible oils are used to produce bio-diesel. The price of this non-edible oil is higher as compared to petroleum products. But the motivation should be given and sincere efforts are to be made to increase the yield and oil extraction from the oil seeds to meet the current energy demand of crude oil.

In the present work Linseed Oil was taken as a feed stock for production of biodiesel. Bio-diesel can be prepared by various methods such as Acid catalyzed, base catalyzed, supercritical trans-esterification etc. out of which the supercritical trans-esterification process is considered as a best from the production point of view [22].

Trans-esterification of vegetable oils using alcohol (Methanol) in a catalytic environment is a chemical process to produce diesel-like liquid, biodiesel. Chemically, biodiesel is mono-alkyl esters (fatty esters) of long chain fatty acids. This method has however, some drawbacks such as difficulties in the recovery of glycerol, production process to be longer and reduces the conversion yield. The main factors affecting trans-esterification are the amount of alcohol, free fatty acid content, presence of water, catalyst reaction, temperature, pressure and time [23-25]. Furthermore, oils containing free fatty acids tend to form soapy products with alkaline catalyst. This problem has been greatly reduced by applying a novel method which is catalyst free and the alcohol (Methanol) is used at its supercritical conditions.

An alternative, catalyst-free process for trans-esterification uses supercritical methanol. The purification becomes much simpler, and it produces higher yield [26-27]. In this process, free fatty acids are converted to methyl esters instead of soap, so a wide variety of feed stocks can be used.

Also, the catalyst removal step is eliminated. Energy costs of production are similar or less than catalytic production routes [28].

Due to the limitations of homogeneous and heterogeneous catalytic reactions, an ideal technology that can produce biodiesel in a sustainable and environmental-friendly method has been the motivation of many researchers. For instance, it must be energy-efficient and does not produce unnecessary wastes in the processing steps. Besides, it must have high tolerance towards impurities (FFA and water) to allow wider range of feedstock application.

The biodiesel produce from supercritical technology (catalyst free reaction) blends with diesel and made a performance test on single cylinder diesel engine.

CHAPTER 2

LITERATURE REVIEW

2.0 INTRODUCTION

As elaborated in the previous section, rising crude oil prices and rapid decrease in fossil fuel reserves have driven the focus of the researchers to look for the sustainable alternative fuels for crude oil that may give the performance at par with the mineral diesel. Today there are many gasoline and diesel fuel additives available in the market to increase horsepower and improve engine operation, engine life, and fuel economy. They were also claimed to reduce emissions. There additives are the by-product of the refining process and the businessmen make it more profitable.

Biodiesel, as an alternative fuel of diesel, is described as fatty acid methyl or ethyl esters from vegetable oils or animal fats. It is renewable, biodegradable and oxygenated. Many researches pointed out that it might help to reduce greenhouse gas emissions, promote sustainable rural development, and improve income distribution. The biodiesel can be made from edible as well as non-edible oils, but because of the food security, in general it is made from non-edible oils. In the present scenario many researchers made sincere efforts on the use of biodiesel in diesel engines.

With respect to the biodiesel production researchers have shown an increased interest towards an emerging technology of producing biodiesel which is supercritical transesterification. Supercritical technique of production of biodiesel is one of the advanced technologies, where no catalysts are require for the reaction. The use of catalysts limits feedstock’s to those with low water and FFA content and requires difficult separation steps at the end of the process. Heterogeneous catalysts solve the separation problem, but still require purified feedstock.

Transesterification under supercritical reaction conditions has been used for catalyst-free biodiesel synthesis. Transesterification of triglycerides (nonpolar molecules) with an alcohol (polar molecule) is usually a heterogeneous (two liquid phases) reaction at conventional processing temperatures because of the incomplete miscibility of the nonpolar and polar components. Under supercritical conditions, however, the mixture becomes a single homogeneous phase, which will accelerate the reaction because there is no interphase mass transfer to limit the reaction rate. In addition, the non-catalytic supercritical process potentially has environmental advantages because no waste is generated from catalyst treatment and separation from the final product.

There are several problems associated with the use of vegetable oil. They can be categorized as operational and durability problems. The former included the ignition quality characteristics, e.g. poor cold engine start-up, misfire, and ignition delay, and the latter include characteristics demonstrating incomplete combustion, e.g. nozzle coking, deposit formation, carbonization of injector tips, ring sticking and lubricating oil dilution and degradation [29].

Problems faced on using the neat vegetable oil are explained below.

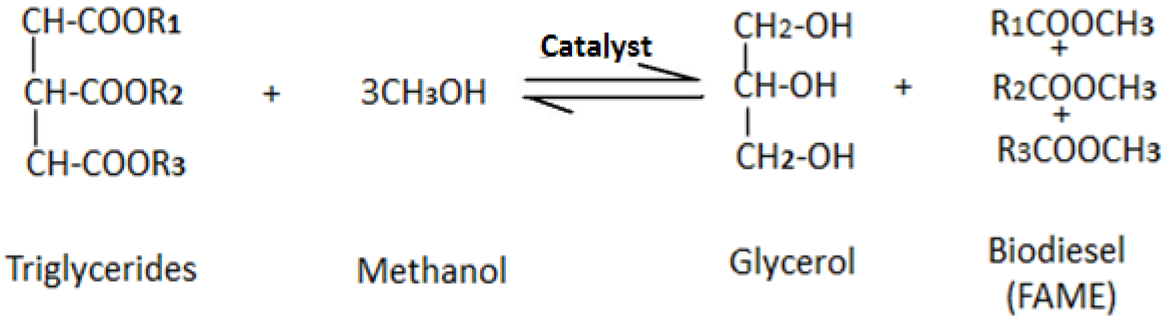
- The increased viscosity of the neat vegetable oils leads to poor atomization and incomplete combustion with an unmodified fuel injection system.
- The clogging of the fuel system.
- Polymerization during storage.
- Thickening and Gelling of the lubricating oil as a result of contamination by the vegetable oil.
- Oil ring sticking.
- Carbon deposits around the nozzle orifice, the upper piston ring grooves and on the piston rings [30].

Because of the above mentioned problems the use of straight vegetable oil in the diesel engines is avoided. Hence the properties of vegetable oil have to be modified to make them suitable for diesel engines. The major problem that is associated with vegetable oils is their higher viscosities which cause poor atomization. Four techniques can be used to lower the viscosity of straight vegetable oil:

1) Heating/Pyrolysis.
2) Dilution/blending.
3) Micro-emulsion.
4) Transesterification.

2.1 CATALYTIC BIODIESEL PRODUCTION

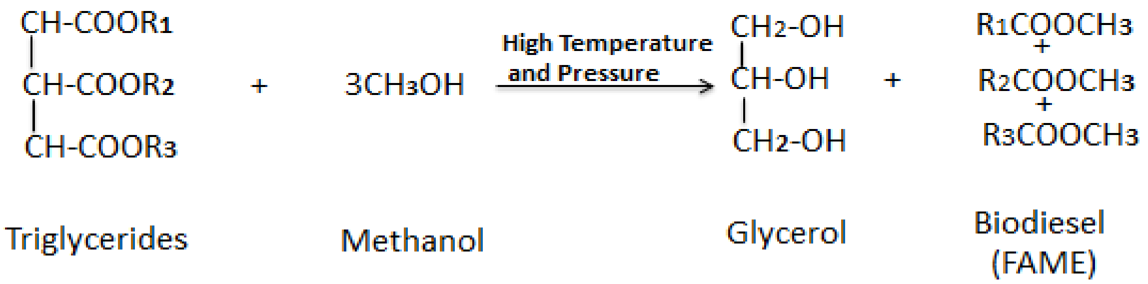
The conversion of vegetable oil into biodiesel through transesterification process is commonly known without significant operational and durability issues. However, this adds extra cost of processing because of the transesterification reaction involving chemical and process heat inputs. Transesterification is an equilibrium reaction in which excess alcohol is required to drive the reaction of straight vegetable oil very close to completion. The vegetable oil is chemically reacted with an alcohol in presence of a catalyst to produce methyl esters. Glycerol is produced as a by-product of transesterification reaction.



Where R1, R2, & R3 are long chain hydrocarbons and –COO- is the carboxyl group. The mixture is stirred continuously and then allowed to settle under gravity in a separating funnel. Two distinct layers form after gravity settling for 24 h. The upper layer is of ester and lower layer was of glycerol. The lower layer is separated out. The separated ester is mixed with some warm water (around 10 % volume of ester) to remove the catalyst present in ester and is allowed to settle under gravity for another 24 h. The catalyst gets dissolved in water, which is separated and the moisture is removed by heating [31].

2.2 NONCATALYTIC BIODIESEL PRODUCTION

The transesterification or biodiesel production under supercritical conditions (supercritical transesterification) is a catalyst-free chemical reaction between triglycerides, the major component in vegetable oils and/or animal fats, and low molecular weight alcohols, such as methanol and ethanol, at a temperature and pressure over the critical point of the mixture.



The reaction parameters that were typically investigated in supercritical transesterification reactions are the temperature, pressure, alcohol to oil molar ratio and reaction time in batch and continuous reactors. Supercritical transesterification reaction has many intermediate products such as mono-glycerides, di-glycerides, tri-glycerides. The final major product of this reaction is fatty acid methyl ester (FAME) followed by glycerol as a byproduct.

2.3 BIODIESEL AS A POTENTIAL C. I. ENGINE FUEL

A large number of alternate fuels are available for the replacing the mineral diesel partially or completely. Among those different alternate fuels the most popular ones are the biodiesel, vegetable oils, alcohols, dimethyl ethers and other related fuels. Biodiesel is produced from vegetable oils by transesterification using an alkali or acid or enzyme as catalyst, and also with non-catalytic supercritical transesterification.

The properties of transesterified vegetable oil are much better as compared to straight vegetable oil. After the transesterification of straight vegetable oils, the kinematic viscosity, and specific gravity is reduced, the viscosity of straight vegetable oil is 3-4 times more than transesterified oils. The properties of transesterified oils are comparable with that of diesel [32].

Table 2.1: ASTM Standards for Various properties of diesel and biodiesel

Fuel Properties	**Diesel ASTM D975**	**Biodiesel ASTM D6751**
Lower Heating Value, BTU/gal	129,050	118,170
Kinematic Viscosity @ 40 °C	1.3-4.1	4.0-6.0
Specific Gravity @ 60 °C, g/cm^3	0.85	0.88
Carbon , wt.%	87	77
Hydrogen, wt.%	13	12
Oxygen, by dif. Wt.%	0	11
Sulphur, ppm	500	0
Boiling Point, °C	180 to 340	315 to 350
Flash Point, °C	60 to 80	100 to 170
Cloud Point °C	~15 to 5	~3 to 12
Pour Point °C	~35 to 15	~15 to 10
Cetane Number	40-55	48-65
Lubricity (HFRR), µm	300-600	<300

Since the properties of transesterified vegetable oil are closer to diesel fuel, hence it is possible to replace the mineral diesel partially or completely with the biodiesel fuel. Petroleum based diesel fuels have different chemical structure than vegetable oil. The former contain only carbon and hydrogen atoms which are arranged in normal (straight chain) or branched chain structures as well as aromatic configurations. The normal structure is preferred for better ignition quality. Diesel fuel can contain both saturated and straight or un-branched chain unsaturated hydrocarbons, but the later are not present in large amounts to make oxidation a problem [33]. The conversion of triglycerides in the straight vegetable oil through transesterification process reduces the molecular weight to one-third that of triglycerides, the viscosity by a factor of eight and reduces the volatility marginally. The biodiesel contains 15 20% oxygen which may enhance the combustion process inside the cylinder than the hydrocarbon fuel.

The cetane number of biodiesel is close to 50. Biodiesel has lower volumetric heating values (about 10%) than mineral diesel but has a high cetane number and flash point. The esters have cloud point and pour points that are 15–25 C higher than those of mineral diesel [34].

The American Society for Testing and Materials (ASTM) standards for commercial diesel (ASTM D975) and biodiesel (ASTM D6751) can be found in Table 2.1[35]

Table 2.2:- Chemical structure of some common fatty acids present in biodiesel.

Common Name	Carbon Atoms	Double Bonds	Scientific Name
Butyric acid	4	0	butanoic acid
Caproic Acid	6	0	hexanoic acid
Caprylic Acid	8	0	octanoic acid
Capric Acid	10	0	decanoic acid
Lauric Acid	12	0	dodecanoic acid
Myristic Acid	14	0	tetradecanoic acid
Palmitic Acid	16	0	hexadecanoic acid
Palmitoleic Acid	16	1	9-hexadecenoic acid
Stearic Acid	18	0	octadecanoic acid
Oleic Acid	18	1	9-octadecenoic acid
Ricinoleic acid	18	1	12-hydroxy-9-octadecenoic acid
Vaccenic Acid	18	1	11-octadecenoic acid
Linoleic Acid	18	2	9,12-octadecadienoic acid
Alpha-Linolenic Acid (ALA)	18	3	9,12,15-octadecatrienoic acid
Gamma-Linolenic Acid (GLA)	18	3	6,9,12-octadecatrienoic acid
Arachidic Acid	20	0	eicosanoic acid
Gadoleic Acid	20	1	9-eicosenoic acid
Arachidonic Acid (AA)	20	4	5,8,11,14-eicosatetraenoic acid
EPA	20	5	5,8,11,14,17-eicosapentaenoic acid
Behenic acid	22	0	docosanoic acid
Erucic acid	22	1	13-docosenoic acid
DHA	22	6	4,7,10,13,16,19-docosahexaenoic acid
Lignoceric acid	24	0	tetracosanoic acid

Various vegetable oils are distinguished by their fatty acid compositions. Triglyceride molecules have molecular weights between 800 and 900 and are thus nearly four times larger than typical diesel fuel molecules [36]. Chemical structure of common fatty acids is given in table 2.2.

Advantages of the biodiesel as engine fuels lie in their renewable nature and wide availability from the variety of sources. This is particularly attractive to countries lacking sources of liquid fossil fuels. They can also be produced on small scale, for on-farm operation to run tractors, pumps and small engines for power generation. There is a potential for a lower contribution, on combustion, to the atmospheric concentration of "Greenhouse gas" carbon dioxide, than from the fixed carbon in fossil fuels.

It is clear that the use of biodiesel as fuels for diesel engines depends on their physical and chemical properties, and on their combustion characteristics as well as the type of engine use and the conditions of operation. They also have a lower Kinematic viscosity and density and higher cetane number, stoichiometric mixture ratio, and specific enthalpy of combustion than the diesel fuel. Though the properties vary from one type of oil to another, this general comparison with the diesel fuel is valid for all [37].

2.4 LITERATURE REVIEW

A pool of good quality recent research works on the potential of production of biodiesel using supercritical trans-esterification for internal combustion engines applications have been thoroughly reviewed. Some of these works have been described below:-

Ching-Hung Chen et. al. [38] showed in their research that supercritical carbon dioxide extraction of triglycerides from Jatropha kernels followed by subcritical hydrolysis and supercritical methylation to obtain biodiesel. The activation energies of hydrolysis and trans-esterification reactions were 68.5 and 45.2 kJ/mole, respectively. The activation energy necessary for hydrolysis was higher than that for methylation which indicated hydrolysis was the rate determining step for biodiesel production. Experimental data indicated that the two-step process of hydrolysis and subsequent methylation was a more suitable pathway for producing biodiesel from Jatropha kernel. The effect of carbon dioxide addition on the accelerated reaction rate of supercritical methylation of the hydrolyzed Jatropha oil requires future investigation.

Joaquin Quesada-Medina et. al. [39] conducted an experiment in which the highest yield of biodiesel (84%) for a methanol-to-oil molar ratio of 43 (optimal ratio) was achieved at a temperature of 325°C (which corresponded to a pressure of 35 MPa) for a reaction time of 60min, conditions in which thermal decomposition of the fatty acid chains was observed, it can be affirmed that even the best reaction conditions leading to the highest yield of biodiesel during the transesterification of soybean oil with supercritical methanol and not using catalysts, involve a certain degree of thermal decomposition of the unsaturated fatty acid chains with two or more double bonds.

Kok Tat Tan et. al. [40] has discussed SCM and SCE reactions involving palm oil by RSM analysis and the effect of alcohol in supercritical transesterification reaction was investigated and elaborated thoroughly. Apart from that, the effects of variables interaction on the yield of biodiesel in SCM and SCE processes were studied and compared. It was found that for SCM reaction, temperature is the most important parameter whereas for SCE reaction, reaction time seems to be the most influencing variable. In addition, optimization studies of SCM and SCE were successfully carried out and it was revealed that SCM has higher optimum yield of 81.5% compared to 79.2% of SCE reaction.

Pilar et. al. [41] has indicated that in the one step supercritical methanolysis of refined soybean oil, the methyl ester content of the product can be maximized at different reaction temperatures and times. The best reaction conditions leading to the highest yield of FAME (84%, equivalent to an 85.8 wt% in the ester phase) were at temperature of 325°C (which corresponds to a pressure of 35 MPa) and a reaction time of 60 min. In these conditions, practically total conversion of TG was reached. However, 8.1 wt% of MG and DG remained in the medium and a 7.9 wt% decomposition of fatty acid chains was observed.

The only way to reduce the content of these glycerides in this catalyst-free one-step supercritical process, and thus increasing the yield of biodiesel, is to raise the temperature, which would inevitably lead to an appreciable increase in the thermal decomposition products.

P. Valle et. al. [42] has examined that the non-catalytic transesterification of fodder radish oil with supercritical alcohols can be performed with molar ratios of alcohols to oil in the range of 30:1 to 40:1 and temperatures of 583–590 K. A homogenous phase with the advance of the reaction can be obtained at pressures of 11 and 14 MPa, for supercritical ethanol and methanol, respectively, depending on the global density of the initial reactants used in the batch reaction process. Reaction times higher than 20 min are required to obtain high ester contents (greater than 96% by weigh fraction) even when the reaction takes place in homogeneous phase conditions. Present results are important regarding the continuous process because in this case the residence time is directly related to the global density of the reaction mixture.

Cheong-Song Choi et. al. [43] has determined that preheating at 245°C and 10 MPa brings methanol and palm olein oil into a homogeneous mixture which defines a clear initial condition for reaction. Under these conditions, palm olein oil is thermally stable and prevents feed loss. The maximum FAME yield (94.96%) was obtained at a reaction temperature of 350°C, a pressure of 35 MPa, a residence time of 20 min, and a methanol-to-oil molar ratio of 40:1.

Hee-Yong Shin et. al. [44] has stated that trans-esterification of refined lard in supercritical methanol was performed to determine the optimum reaction conditions. The highest FAME(fatty acid methyl ester) content of 89.91% was obtained from refined lard at a temperature of 335°C, a molar ratio of 45:1 of methanol to oil, a pressure of 20 MPa, a reaction time of 15 min, and an agitation speed of 500 rpm. Production of biodiesel from waste lard under the optimal reaction conditions was also carried out to validate the use of waste lard as a feedstock. Even though waste lard samples contain various FFA (Free fatty acid) and water contents, FAME contents from waste lard with no pre-treatment were found to be comparable to those from refined lard. Though it is concluded that waste lard can be utilized as an alternative feedstock for biodiesel production, thus replacing high cost refined vegetable oil feedstock, and supercritical trans-esterification is verified to be a suitable process for converting waste lard to valuable biodiesel.

Sandra B. Glisic et. al. [45] has explained that non-catalytic triglycerides trans-esterification is carried out at elevated temperatures and pressures. The phase distribution of reacting species, mass transfer of reacting species and simplified procedure for the determination of kinetic constants were used in the model. The model assumes that all consecutive steps in this complex reaction are reversible. Experimental investigation of the biodiesel synthesis was performed in batch reactor at 150°C and 1.1MPa; at 210°C and 4.5MPa. Concentrations of reacting species were monitored during reaction, as well as the existence of multiple liquid phases and distribution of reacting species between the phases. The predictions of triglycerides conversion and FAME yield were very good, as well as predicted concentrations of intermediates during reaction. In the initial phase of reaction, at subcritical conditions of methanol, the mass transfer of reacting species plays a significant role in limitation of the overall reaction rate. At conditions of supercritical methanol, corresponding to the single reaction phase and the absence of mass transfer limitations, the model predicted yields of FAME were in excellent agreement with previously reported experimental data.

Freedman et. al. [46] has developed a process for alkali-catalyzed transesterification is widely referred for experimental details and protocols for batch-scale transesterification process. The molar ratio (MR) is associated with the type of catalyst used. An acid catalyzed reaction requires higher molar ratio (30:1) than alkali catalyzed reaction (6:1) to achieve the same ester yield for a given reaction time.

Labeckas et. al. (2006) [47] studied an unmodified, naturally aspirated Diesel engine operating on neat rape seed methyl ester (upto 35 %) and diesel fuel blends. The maximum brake thermal efficiency varied from 0.356 to 0.398 for RME and from 0.373 to 0.383 for Diesel fuel. The maximum NOx emissions increased proportionally with the increase of mass percent of oxygen in the biofuels and also with engine speed. CO emissions and visible smoke emerging from the biodiesel over all loads and speed ranges were lower by up to 51.6% and 60.3%, respectively. The emissions of unburned hydrocarbons, HC, for all biofuels were low, ranging at 5-21 ppm levels.

Kumar et. al. (2008) [48] conducted engine tests to get the comparative measures of brake specific fuel consumption (BSFC), brake specific energy consumption (BSEC) and emissions such as CO, CO_2, HC, NOx to evaluate the behaviour of PPME and diesel in varying proportions. The results reveal that blends of PPME with diesel up to 40% by volume (B40) provide better engine performance (BSFC and BSEC) and improved emission characteristics.

Lapuerta et. al. (2008) [49] compared the diesel engine emissions of biodiesel fuels as opposed to conventional diesel fuels. Special attention was paid to nitric oxides and particulate matter and it was observed that sharp reduction in particulate emissions in case of biodiesel fuel in comparison to diesel fuel.

Yang et. al. (2012) [50] investigated the performance, combustion and emission characteristics of diesel engine fuelled by biodiesel at partial load conditions. Experiments were conducted on a common-rail fuel injection diesel engine using ultra low sulphur diesel, biodiesel (B100) and their blend fuels of 10%, 20%, and 50% under various loads. The results showed that biodiesel/blend fuels had significant impacts on the engine's brake specific fuel consumption (BSFC) and brake thermal efficiency (BTE) at partial load conditions.

Nabi et. al. (2009) [51] investigated different parameters for the optimization of biodiesel production and carried out the performance test of a diesel engine with neat diesel fuel and biodiesel mixtures. The engine experimental results showed that exhaust emissions including carbon monoxide (CO) particulate matter (PM) and smoke emissions were reduced for all biodiesel mixtures. However, a slight increase in oxides of nitrogen (NOx) emission was experienced for biodiesel mixtures.

Hoekman et. al. (2012) [52] has studied “biodiesel NOx effect, and theories to explain this effect. The biodiesel NOx effect can be mitigated by modifying engine control settings particularly by retarding injection timing and increasing exhaust gas recirculation (EGR). The absolute magnitude of the biodiesel NOx effect appears to be reduced with modern engines, although there are cases where the percentage change is still substantial.

Yanowitz et. al. (2009) [53] found that the use of a common biodiesel blend (B20) consistently reduces emissions of particulate matter, hydrocarbons, and carbon monoxide by 10-20% and B20 show varying effects on oxides of nitrogen (NOx). The emissions effect of B20 on heavy-duty diesel truck emissions did not show any correlation with model year or type of fuel injection equipment as per their study.

Muralidharan et. al. (2011) [54] found the performance, emission and combustion characteristics of a single cylinder four stroke variable compression ratio multi fuel engine fuelled with waste cooking oil methyl ester and its 20%, 40%, 60%and 80% blends with diesel (on a volume basis) and compared with standard diesel. Experiment has been conducted at a fixed engine speed of 1500 rpm, 50% load and at compression ratios of18:1, 19:1, 20:1, 21:1 and 22:1. The results indicate longer ignition delay, maximum rate of pressure rise, lower heat release rate and higher mass fraction burnt at higher compression ratio for waste cooking oil methyl ester compared to that of diesel. B40 is found to give maximum thermal efficiency at 50 % loading condition. The blends when used as fuel results in reduction of carbon monoxide, hydrocarbon and increase in nitrogen oxides emissions.

Devan et. al. (2009) [55] studied on methyl ester of paradise oil and eucalyptus oil to study the performance and emission characteristics of these fuels. Various proportions of paradise oil and eucalyptus oil are prepared on a volume basis and used as fuels in a single cylinder, four-stroke DI diesel engine, methyl ester derived from paradise oil was considered as an ignition improver. The results show a 49% reduction in smoke, 34.5% reduction in HC emissions and a 37% reduction in CO emissions for the Me50–Eu50 blend with a 2.7% increase in NOx emission at full load. There was a 2.4% increase in brake thermal efficiency for the Me50–Eu50 blend at full load. The combustion characteristics of Me50–Eu50 blend are comparable with those of diesel.

Ren et. al. (2008*)* [56] evaluate combustion and emissions of a DI diesel engine fuelled with diesel-oxygenate blends. The results showed that there exist the different behaviours in the combustion between the diesel-diglyme blends and the other five diesels-oxygenate blends. The smoke concentration decreases regardless of the types of oxygenate additives, and the smoke decreases with the increase of the oxygen mass fraction in the blends without increasing the NOx and engine thermal efficiency. CO and HC concentrations were found to decrease with the increase of oxygen mass fraction in the blends. Unlike conventional diesel engines fuelled with pure diesel fuel, engine operating on the diesel-oxygenate blends presents a flat NOx/Smoke trade off curve versus oxygen mass fraction.

Hoekman et. al. (2012) [57] have found that differences in chemical and physical properties among biodiesel fuels can be explained largely by the fuels' FA profiles. Two features that are especially influential are the size distribution and the degree of unsaturation within the FA structures. For the biodiesel types reviewed, it was found that several fuel properties - including viscosity, specific gravity, cetane number, iodine value, and low temperature performance metrics - are highly correlated with the average unsaturation of the FAME profile**s.**

Gill et. al. (2011) [58] found condensed overview of Gas-to-Liquid (GTL), Biomass-to-Liquid (BTL) and Coal-to- Liquid (CTL) theory and technology by the use of Fischere-Tropsch (FeT) processes. FeT diesel fuels typically have a number of very desirable properties, including a very high cetane number. This review focused on how fuel properties impact pollutant emissions and draws together data from various studies that have been carried out over the past few years. Reduced emission levels as demonstrated in several publications have been attributed to several chemical and physical characteristics of the FeT diesel fuels including reduced density, ultra-low sulphur levels, low aromatic content and high cetane rating, but not all of them contribute to the same extent to the emissions reduction.

Selim et. al. (2003) [59] carried out investigation to examine for the first time the performance and combustion noise of an indirect injection diesel engine running with new fuel derived from pure jojoba oil, jojoba methyl ester, and its blends with gas oil. A Ricardo E6 compression swirl diesel engine was fully instrumented for the measurement of combustion pressure and its rise rate and other operating parameters. Results showed that the new fuel derived from jojoba is generally comparable and good replacement to gas oil in diesel engine at most engine operating conditions, in terms of performance parameters and combustion noise produced.

Ramesh et. al. [60] investigated the performance and emission characteristics of Diesel engine with jatropha bio-diesel and its blends. It was observed that in the case of Jatropha biodiesel alone, the fuel consumption in the diesel engine was about 14 per cent higher than that of diesel. The percent increase in specific fuel consumption ranged from 3 to 14 for B20 to B100 fuels. The brake thermal efficiency for biodiesel and its blends was found to be slightly higher than that of diesel fuel at tested load conditions and there was no difference between the biodiesel and its blended fuels efficiencies. For jatropha biodiesel and its blended fuels, the exhaust gas temperature increased with increase in load and amount of biodiesel.

Puhan et. al. [61] studied the effect of biodiesel fatty acid composition specifically unsaturation for the engine combustion by comparing five Biodiesel produced from different 34 vegetable oils those were tested on a diesel engine. Biodiesel (93% unsaturated fatty acid ester composition) with 55% linolenic ester emitted more oxides of nitrogen of 16.83 g/kWh and exhibited higher thermal efficiency of 29.76% compared to diesel of 12.83 g/kWh and 28.41% respectively. No significant differences in unburnt hydrocarbon (UBHC), carbon monoxide (CO) and smoke emissions among the biodiesel fuels were noticed except linseed oil methyl ester.

Aydin et. al. [62] performed investigation on performance and emission characteristics of a single cylinder, four strokes, air cooled diesel engine fuelled with the blends of cottonseed methyl ester. For the study, cottonseed oil methyl ester (CSOME) was added to diesel fuel, numbered D2, by volume of 5% (B5), 20% (B20), 50% (B50) and 75% (B75) as well as pure CSOME (B100). The experimental results showed that the use of the lower blends (B5) slightly increases the engine torque at medium and higher speeds in compression ignition engines. However, there were no significant differences in performance values of B5, B20 and diesel fuel. Also with the increase of the biodiesel in blends, the exhaust emissions were reduced. They concluded that the lower contents of CSOME in the blends can partially be substituted for the diesel fuel without any modifications in diesel engines.

Karabektas et. al. [63] studied the effects of preheated cottonseed oil methyl ester on the performance and exhaust emissions of a single cylinder, four stroke, and naturally aspirated diesel engine. In their investigation, before supplying to the engine, COME was preheated to four different temperatures, namely 30, 60, 90 and 1200°C. The test data were used for evaluating the brake power and brake thermal efficiency (BTE) together with CO and NOx emissions. The results revealed that preheating COME up to 900°C leads to favorable effects on the BTE and CO emissions but causes higher NOx emissions. Moreover, the brake power increases slightly with the preheating temperature up to 900°C. When the COME is preheated to 1200°C, a considerable decrease in the brake power was observed due to the excessive fuel leakage caused by decreased fuel viscosity. The results suggest that COME preheated up to 900°C can be used as a substitute for diesel fuel without any significant modification in expense of increased NOx emissions.

Sahoo et. al. [64] investigated the combustion characteristics of Jatropha, Karanja and Polanga based biodiesel in a diesel engine. The investigations on the combustion characteristics were conducted on a small size 6 kW air-cooled single cylinder four-stroke diesel engine (Kirloskar Oil Engines Ltd., India) fueled with the different biodiesel fuel blends. They conclude that that biodiesel from unrefined Jatropha, Karanja and Polanga seed oil was quite suitable as an alternative to diesel. They also concluded that neat Polanga biodiesel (PB100) which resulted in maximum peak cylinder pressure (6.61 bars higher than that of diesel) was the optimum fuel blend as far as the peak cylinder pressure is concerned. The ignition delays were consistently shorter for JB100, varying between 5.90 and 4.20 crank angle lower than diesel with the difference increasing with the load. Similarly, ignition delays were shorter for KB100 (varying between 6.30 and 4.50 crank angle) and PB100 (varying between 5.70 and 4.20 crank angle) lower than diesel.

Gogoi et. al. [65] analyzed the performance and combustion characteristics of 10%, 20%, 30% and 40% blending of Koroch Seed Oil Methyl Ester (KSOME) and Jatropha Methyl Ester (JME) with diesel as fuels in a diesel engine. They found that the brake specific fuel consumption (BSFC) was more for the methyl ester blends and particularly for the JME blends. The brake thermal efficiency (BTE) was slightly lower for the biodiesel blends and for the JME blends it was less compared to that of the KSOME blends. The indicated power was more in case of the blends; however it reduced significantly for the 40% blend of KSOME. Both the KSOME and JME blends exhibited similar combustion trend with that of diesel, however, the blends showed an earlier start of combustion with shorter ignition delay. The ignition delay was less and the combustion duration was more for the JME blends as compared to the KSOME blends. They observed that the KSOME blending up to 30% showed an acceptable performance and combustion trend whereas the JME blends showed favorable combustion trend but due its comparatively higher fuel consumption characteristics and the engine BTE was less with the JME fuel blends.

2.5 STATEMENT OF THE PROBLEM

After literature review it can be concluded that supercritical transesterification is an attractive methods of production of biodiesel. And also biodiesel is an enchanting replacement of diesel fuel. But some researchers have reported difficulties encountered with use of vegetable oil in diesel engine. These difficulties are mainly due to high viscosity of vegetable oil. Mahua oil is a sustainable replacement of diesel in India. However its high viscosity issue is to be resolved for its long term utilization in diesel engine. Blending of linseed oil with diesel is one of the methods to decrease the viscosity of the linseed oil which produce methyl esters through supercritical transesterification process that could be used straight instead of diesel.

Therefore, the following objectives were envisaged for the present research work.

1. Comprehensive literature survey.
2. Identification of non-edible oil for biodiesel production.
3. Preparation of biodiesel.
4. Development of dual fuel mode experimental diesel engine test rig.
5. Determination of important Physico-chemical properties of linseed oil biodiesel.
6. Conducting exhaustive experiments on the test rig to evaluate performance and emission characteristics of pure diesel and linseed oil methyl ester blend with different proportion in crude diesel.
7. Analysis of Result.

CHAPTER 3

SYSTEM DEVELOPMENT & EXPERIEMNTAL PROCEDURE

3.0 INTRODUCTION

Diesel engines are amongst the most efficient prime mover amongst all the power producing machines. Also keeping the fact of depletion of petroleum based fuels and environmental degradation into consideration it becomes necessary to develop an environment friendly fuel having properties similar to the diesel which should meet the future energy demand.

In this context bio-origin fuels are getting attention due to its renewable property and comparable properties. These fuels also have fewer tendencies to pollute the environment. The bio-origin fuels may be alcohol, vegetable oil, animal fats and biogas.

The diesel engine finds its application in the heavy-duty field, for example, industrial power plants, marine propulsion, railroad locomotives, trucks, tractors, buses and farm equipment. Due to high thermal efficiency leading to low fuel consumption and industrial growth, the use of diesel engine has increased exponentially in the last decade. The oil crisis and the emphasis to conserve petroleum- based fuels have provided the motivation for several studies on the developments and testing of alternate fuels. In recent years many countries have taken initiatives in this field and re-forestation has a very important role to play in meeting the challenge of Climate Change. Several initiatives have been taken indifferent parts of the country to promote large scale cultivation of oilseed bearing plants. Amongst the various plant species, oil extracted from seeds of Linseed plant has been found very suitable as a substitute to diesel fuel.

3.1 LINSEED PLANT DESCRIPTION

Linseed oil, its scientific name is *Linum Usitatissimum*, belongs to the family *linaceae* and the genus *Linum* which has 100 species. It originates from Mediterranean coastal and is cultivated in Canada, Argentina, India and USA. It is growing to a height of 3.0-1m, which is cultivated for the production of textile fiber, seed and linseed oil. Linseed is cultivated in Araucaria region for oil extraction. Studies have shown the crop yield is higher in this region, because of its soil and climate characteristics. Linseed is the best suited for fertile, fine textured and loamy soil: an important factor is the amount of rain fall during the growing period. Adequate moisture and relatively cool temperature, particularly during the period from flowering to maturity, seem to favor both oil content and oil quality. The seed is located in the extremities of the branches in round capsules, each of which contains from one to ten seeds. In India, it is grown for seeds, used for extracting oil.

Under optimum conditions the oil content of linseed seed varies from 33% to 47%. In India various states have sufficient forest area for the plantation of linseed plant. Madhya Pradesh leads in yield and acreage, followed by Uttar Pradesh and Maharashtra, Bihar, Rajasthan, Karnataka and West Bengal also grow linseed in large areas. Madhya Pradesh and Uttar Pradesh together contribute to the national linseed oil production to the extent of about 70%. India accounts for about 1.9 million hectares, with a seed production of 4.98 lakhs of tones. The crop in northern India generally gives higher yield than in central and peninsular India. The irrigated crop may yield 1200 to 1500 kg per ha. and occupies the third rank among the linseed production countries Australia and Canada [66]. Linseed oil is the most commonly used carrier in oil plant. It is available in Asian countries. It is an important oil seed in the world. Linseed is a source of high quality proteins, soluble fiber and a high content of polyunsaturated fatty acids. They present values of 30-40% lipids, 20-15% proteins, 4-8% moisture, 3-4% ash and 20-25%

fietary fiber. Common names of linseed are flax, alsi, tisi, kshuma, lin, llion, liner, linum, line, linen, lein.

3.1.1 Cultivation of linseed crop

Flax is a broadleaf with small, narrow leaves that are less than an inch long, stem are slender, flexible, branches, near base of plan, with heights 30-36 inches, dividing at their tips into inflorescences bearing attractive blue flowers. Flowers are mostly self-pollinated with some cross pollination by insects. New flower will emerge for week, each developing into a round seed capsule or boll about one-third inch in diameter. Each capsule contains 4- 10 seeds, Glossy in appearance, traditionally brown color. However, a new variety of flex, Omega, is golden colored to make it more acceptable in the food maker. If exposed to water, flex seed will become sticky due to mucilage in the coat [67].

Climate

Crop, grown for seed, performs well in moderately cold climate, but the crop for fiber purpose, requires cool and moist climate. Linseed requires moderate or cool temperature during vegetative stage and dry weather during the maturity of the crop. It requires temperature of 25-39°C during germination, 15-20°C during seed formation. It requires high humidity. Temperature above 32°C along with the drought during flowering reduces yield, oil content and oil quality of linseed. Plants are susceptible to frost and cause injury to blossom. It is resistant to drought and grows well in areas receiving an annual rainfall of 450-750mm [67].

(a) Linseed plants

(b) Flower *(c) Capsules* *(d) Seeds*

Fig.3.1 Linseed crop

Soils

Linseed can be grown on a range of soil types, although heavier clay loam soils are preferred over lighter sands. Heavier soils have a higher water holding capacity and hence less irrigation water is required later in the season. Surface crusting can reduce germination considerably due to low seedling vigour. Linseed prefers high fertility soils. Linseed can tolerate acidic soils, however a pH in water of >6 is required for optimum yields.

Linseed prefers well drained soils. It will tolerate some waterlogging but not during establishment. Linseed can be grown on raised beds in wetter lying areas. Poorly drained soils should be avoided. Alternatively, spring sowing may be preferred in areas likely to lie wet over winter [68].

3.1.2 Uses of linseed plant

Flex is roughly 40% oil by weight, about 55% of which is alpha linolenic acid (also called omega-3 fatty acid). On a small scale seed is directly used for edible purpose and 80% of oil goes to industries. Oil is rich in linolenic acid (66% and above) and is a perfect drying oil and used in plant and varnishes. Its use is limited to animal fee because of undesirable compounds like phytic acid, cyanogenic glucoside and goitoren. It is used in the manufacture of lithographic inks and soaps. Oil cake is good feed for milch cattle and also as manure having microbial activity. Linseed stem yields fiber of good quality having strength and durability. The fibers are lustrous and blend with wool and silk. The fiber is used in gloves, foot wear, netting spots, paper and textile industry, cigarette wrapping paper, strong canvas, suiting and shirting. Woody matter and sort fiber used as raw pulp in paper industries. One hundred kg retted flax yields he following co-product.

Table 3.1 Uses of linseed plant

S. No.	Products	Utility
1.	Sketched flax	Spinning
2.	Tow broken fiber	High grade paper
3.	Woody parts	Chip board panels
4.	seeds	Oil and Meal
5.	straw	For Folder
6.	Dust	Compost

Cake/meal utilization

After extraction of the linseed oil a large quantity of cake remains. The linseed cake is mainly used as a cattle feed. It is very good manure and animal feed. Linseed cake/meal is used as additives in baking products. Linseed meal is used normally for ground un-extracted seed (35%), ground linseed cake (10%) and linseed meal (3%) from a solvent plant. Rich oil content can affect texture and flavor of meat and butter obtained.

Protein, gluten and carbohydrates

Amino acids are the building blocks of protein (20-25%). Pattern of linseed protein is similar to that of soybean protein. Linseed is gluten-free and it is low in carbohydrates.

Fibers

Linseed straw products fiber of good quality and fiber accounts for about 28% of the weight of linseed fat. It was most popular plant used to produce fibers for linen in Egypt. There are two main varieties of linseed; one gives a high yield of seed and other fiber. Fiber occurs as structural material in the cell wall of plants and has impotent health benefits for human and it is also used in paper making.

3.1.3 Properties and chemical composition of linseed oil

The chemical composition of linseed oil varies with geographical location and verity. Oils from different sources have different fatty acid compositions.

Linseed oil consists chiefly of three glycerides linolein, linolenim, and olein. A small amount of free fatty acids such as palmitic and arachidic, is also present. The triglycerides of linseed oil contain five major fatty acids. The main fatty acids are palmatic, stearic, oleic, linoleic and linolenic. It was observed that percentage of FFAs in linseed is 1.94%. linolein, which is present in linseed oil to the extent of about 20%, is the glyceride of linoleic acid, and has forula $C_{57}H_{98}O_6$.

Table 3.2 Properties of linseed oil [69].

S. No.	Properties	Values
1.	Flash Point	222°C
2.	Auto Ignition Temperature	343°C
3.	Melting Point	-19°C
4.	Specific Gravity	0.93
5.	Solubility	Insoluble in cold water

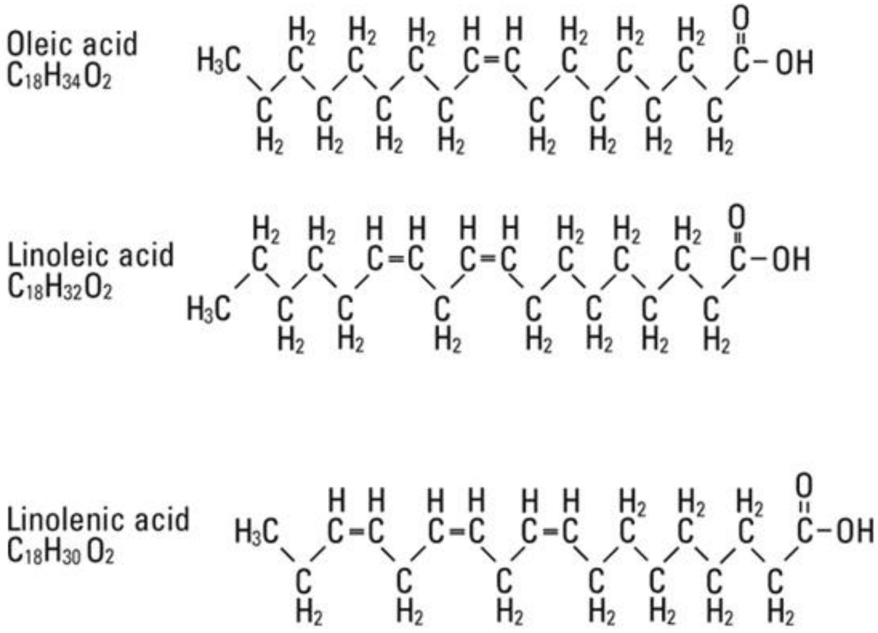


Fig. 3.2 Structure of different acid of linseed oil

3.1.4 Extraction of crude linseed oil

Flaxseed, also known as linseed, originates from an annual herb called the flax plant. Oil is extracted from the seed to produce flaxseed oil. According to the University of Maryland Medical Center, flaxseed contains properties that appear to be beneficial for a variety of health problems. It is simple to integrate flaxseed oil into your diet. Many companies manufacture flaxseed oil in capsule or liquid form. Alternately, you can create your very own flaxseed oil. Just combine flaxseed with water and follow the following steps [70].

- Pour 2 cups of water into a pan and boil it.
- Add 30gm flaxseed to the boiling water. Place the temperature on medium-low. Leave the pan uncovered. Boil for 10 minutes. The oil will start to accumulate at the top of the water.
- Remove the pan from the heat. Set aside and allow the pan to cool.
- Cover a bowl with a piece of cheesecloth. Pour the contents through the cheesecloth to strain the flaxseed oil. Use your hands to bunch up the cheesecloth to form a pouch. Grab the top of the pouch and squeeze. Slide your hand down the cheesecloth, while squeezing, to release the flaxseed oil into the bowl.
- Pour the flaxseed oil into a plastic bottle or a sealed glass container. Store the container in the refrigerator for up to 10 days. Ensure the lid is tightly sealed. Flaxseed oil can turn rancid very quickly. After 10 days, discard the flaxseed oil and make a fresh batch.

3.2 EXPERIMENTAL DESIGN

The experimental set-up as shown in the fig. 3.3 constitutes of a reactor in which the solution of linseed oil and methanol is kept and stirred with the help of a stirrer connected to a motor. Pressure sensors and thermocouples are used to measure the pressure and the temperature respectively inside the reactor. Two pipes one for the inlet of Nitrogen gas and the other for the outlet of FAME which is the end product are provided as shown.

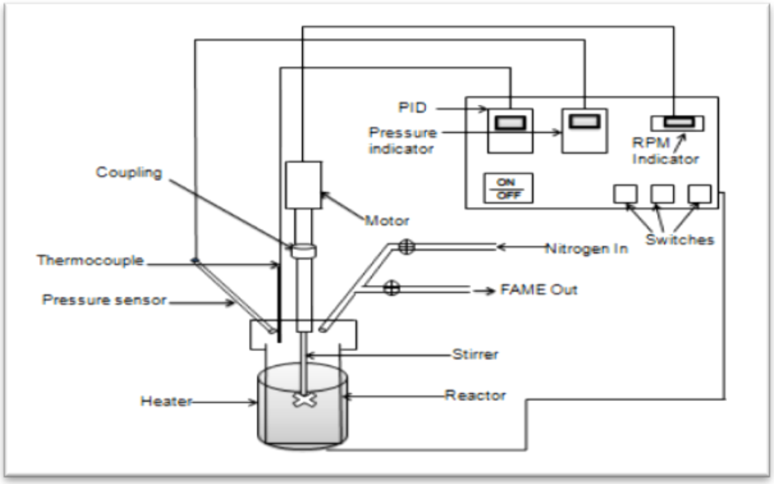


Fig.3.3 Schematic Diagram of Experimental Set-up

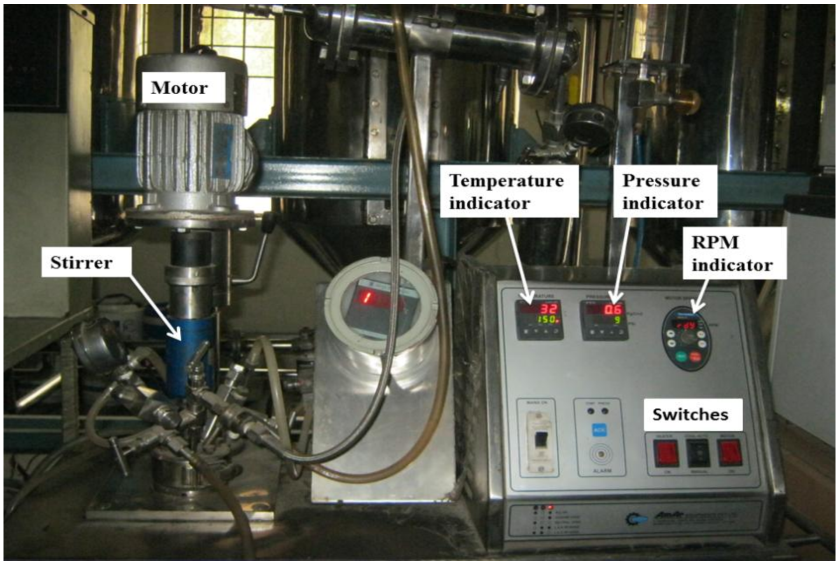


Fig. 3.4 Experimental Set-up

3.3 BIODIESEL PRODUCTION FROM LINSEED OIL USING SUPERCRITICAL TRANSESTERIFICATION

Under optimum conditions linseed seed can yield up to 33-47% oil. Climatic and soil condition of our country is convenient for the production of linseed crop. Major problems encountered with linseed oil as biodiesel used in CI engine are its low volatility and high viscosity due to long chain structure. Methyl ester of linseed oil production from supercritical transesterification process by using methanol. The viscosity of linseed oil biodiesel highly decreases after transesterification process. Properties of linseed oil biodiesel produces by transesterification are comparable with conventional diesel fuels.

In the current research, linseed oil was bought from an oil supplier. Then its acid number and molar mass was calculated with the help of triglyceride composition of raw oil. After this a solution of methanol and oil was taken in the reactor according to molar weight of 42:1. After this, the nitrogen was passed into the reactor from the nitrogen cylinder through a pipe. After filling the nitrogen gas in the reactor the tap is closed. The main reason behind the use of nitrogen gas is that it acts like an inert gas even at a high temperature and pressure during the supercritical transesterification reaction and thus doesn't react with the chemicals. Therefore, it is just used for maintaining the reaction pressure inside the reactor. At the end the stirrer motor, heater and chiller were turned on and the reaction was started.

Chiller is used to circulate cold water to the area where unwanted temperature increase is there, which may cause failure of equipment. Heater is used to increase the temperature of the sample inside the reactor which causes increase of pressure inside the reactor. As the previous studies indicated that there is almost no reaction at temperatures below 250°C [40-41], so the temperature range that was selected for experiment is 250^0C – 350^0C which causes the pressure to rise in between the range of 13Mpa-45Mpa. The Stirrer is connected with motor which is continuously used to stir the sample.

After completion of a fixed reaction period, the sample was collected in a round bottom flask by opening the tap and was subjected to 70min in rotary evaporator for complete evaporation of methanol.

3.3.1 Fuel properties of linseed oil biodiesel

Table 3.3 Fatty acid composition of linseed oil biodiesel

Common Name of fatty acid	Chemical name of fatty acids	Structure	Formula
Palmitic	Hexadecanoic	C16:0	$C_{16}H_{32}O_2$
Stearic	Octadecanoic	C18:0	$C_{18}H_{36}O_2$
Oleic	Octadecenoic	C18:1	$C_{18}H_{34}O_2$
Linoleic cis-9, cis-12	Octadecadienoic	C18:2	$C_{18}H_{32}O_2$
Linolenic	Cis-9, cis-12, cis-15-octadecatrienoic	C18:3	$C_{18}H_{30}O_2$

Table 3.4 Fuel properties of linseed oil biodiesel

Properties	Unit	Value
Density (15^0C)	gm/cm^3	0.893
Kinematic viscosity (40^0C)	mm^2/sec	26.24
Flash point	(°C)	265
Cloud point	(°C)	-2
Calorific value	KJ/kg	40950
Pour point	(°C)	-6

3.4 BLEND PREPARATION

Linseed methyl ester was obtained from the above described process of biodiesel production from production from supercritical methanol technique was blended with diesel in different composition.

The blends (Diesel in biodiesel vol. /vol.) were made of different composition by mixing the constituents. The blends (100ml) were designated as follows:

LME5 D95 - Linseed oil methyl ester 5ml + Diesel 95ml

LME10 D90 - Linseed oil methyl ester 10ml + Diesel 90ml

LME15 D95 - Linseed oil methyl ester 15ml + Diesel 85ml

LME20 D80 - Linseed oil methyl ester 20ml + Diesel 80ml

LME100 - Linseed oil methyl ester 100ml

D100 - Diesel 100ml

3.5 DETERMINATION OF PHYSICO-CHEMICAL PROPERTIES

3.5.0 EQUIPMENTS

The following equipment's were used in the laboratory for determination of Physico-chemical properties of the fuel for the specified project. These are used in CASRAE of Delhi Technological University, Delhi.

3.5.1 DENSITY METER

Density meter used in laboratory is DMA 4500AntoParr model. This density meter works on the principle of oscillating U-tube. The oscillating U-tube is a technique to determine the density of liquids and gases based on an electronic measurement of the frequency of oscillation, from which the density value is calculated. This measuring principle is based on the Mass-Spring Model.

The sample is filled into a container with oscillation capacity. The Eigen frequency of this container is influenced by the sample's mass. This container with oscillation capacity is a hollow, U-shaped glass tube (oscillating U-tube) which is electronically excited into undamped oscillation (at the lowest possible amplitude). The two branches of the U-shaped oscillator function as its spring elements.

The direction of oscillation is normal to the level of the two branches. The oscillator's Eigen frequency is only influenced by the part of the sample that is actually involved in the oscillation. The volume involved in the oscillation is limited by the stationary oscillation knots at the bearing points of the oscillator. If the oscillator is at least filled up to its bearing points, the same precisely defined volume always participates in the oscillation, thus the measured value of the sample's mass can be used to calculate its density.

Plate 3.1 Density Meter

3.5.2 KINEMATIC VISCOMETER

Viscosity is an important property of fuel and it can be defined as measure of the resistance of a fluid which is being deformed by either shear stress or tensile stress. Viscosity describes a fluid's internal resistance to flow and may be thought of as a measure of fluid friction. In general too viscous fuel tends to form scum and deposits on cylinder walls, piston head etc., and cause atomization problems. So it is desirable that viscosity of fuel should be low. The different blend samples are prepared are investigated for viscosity at 400 C using a kinematic viscometer as per the specification given in ASTM D445. It consists of a capillary tube in which sample to be test is filled. The capillary tube has two marks engraved on it. The time for flow of fuel sample from upper mark to lower mark is measured and kinematic viscosity is calculated using time taken by each sample. The plate of the kinematic viscometer apparatus is shown below.

The kinematic viscosity of different fuel blends can be calculated as:

$$\upsilon = k \times t$$

Where,

υ= kinematic viscosity of sample;

k = constant for viscometer;

t = time taken by the fluid to flow through capillary tube

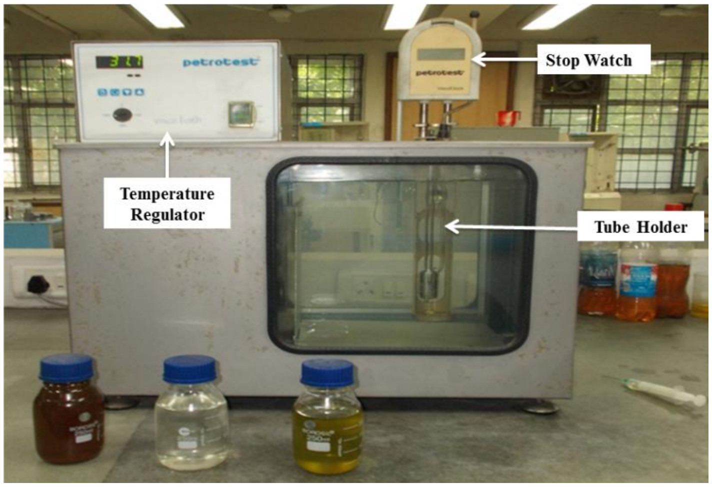


Plate 3.2 Kinematic Viscometer

3.5.3 BOMB CALORIMETER

The calorific value is defined in terms of the number of heat units liberated when unit mass of fuel is completely burnt in a calorimeter under specified conditions. Higher calorific value of fuel is the total heat liberated in kJ per kg or m^3. All fuels containing hydrogen in the available form will combine with oxygen and form steam during the process of combustion. If the products of combustion are cooled to it initial temperature, the steam formed as a result will condense. Thus maximum heat is abstracted. This heat value is called the higher calorific value.

The calorific value of the fuel was determined with the Isothermal Bomb Calorimeter as per the specification given in ASTM D240. The combustion of fuel takes place at constant volume in a totally enclosed vessel in the presence of oxygen. The sample of fuel was ignited electrically. Then the fuel samples were burnt in bomb calorimeter and the calorific value of all samples were calculated. Parr Model 6100EF was used in laboratory for measuring calorific value of biodiesel. The Bomb Calorimeter used for determination of Calorific value is shown in plate 3.3

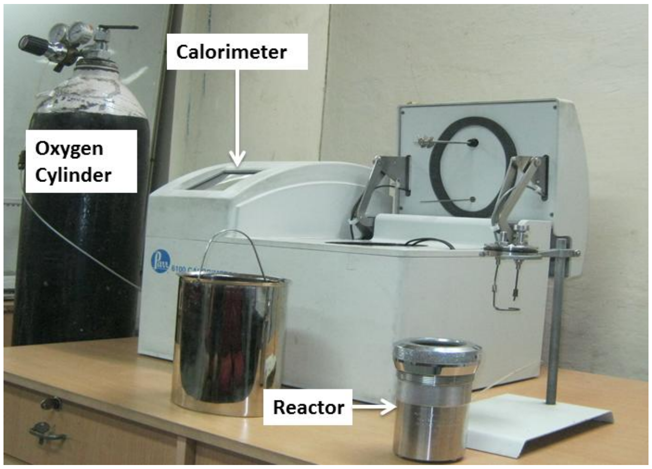


Plate 3.3 Parr 6100 calorimeter

3.5.4 COLD FILTER PLUGGING POINT (CFPP)

Cold Filter Plugging Point (CFPP) is defined as the minimum temperature at the fuel filter does not allow the fuel to pass through it. At low operating temperature fuel may thicken and does not flow properly affecting the performance of fuel lines, fuel pumps and injectors. Cold filter plugging point of vegetable oils reflects its cold weather performance. It defines the fuels limit of filterability. The apparatus for CFPP measurement is shown in plate 3.4

Plate 3.4 Cold Filter Plugging Point Apparatus

3.5.5 FLASH AND FIRE POINT

Flash point is the minimum temperature at which the oil vapour, which when mixed with air forms an ignitable mixture and gives a momentary flash on application of a small pilot flame. The flash and fire point of the test fuels were measured as per the standard of ASTM D 93. The sample was heated in a test cup at a slow and constant rate of stirring for proper and uniform heating. A small pilot flame was directed into the cup though the opening provided at the top cover at the regular intervals. The temperature at which these vapour catches flash is observed and called as the flash point of that liquid. Fire point is an extension of flash point in a way that it reflects the condition at which vapour burns continuously for at least for 5 seconds. Fire point is regularly higher than the flash point by 5-8°C.

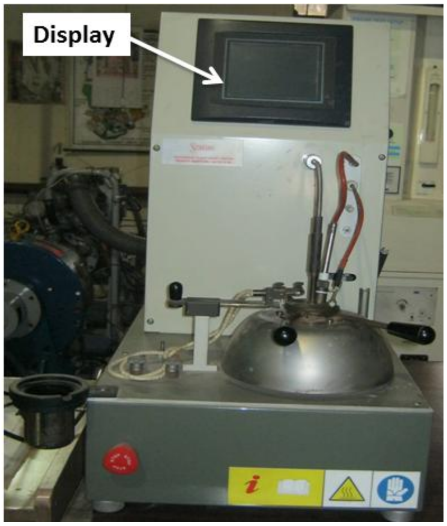


Plate 3.5 Pensky merten flash point apparatus

3.5.6 Gas Chromatograph

A gas chromatograph is a chemical analysis instrument for separating chemicals in a complex sample. A gas chromatograph uses a flow-through narrow tube known as the column, through which different chemical constituents of a sample pass in a gas stream (carrier gas, mobile phase) at different rates depending on their various chemical and physical properties and their interaction with a specific column filling, called the stationary phase. As the chemicals exit the end of the column, they are detected and identified electronically. The function of the stationary phase in the column is to separate different components, causing each one to exit the column at a different time (retention time). Other parameters that can be used to alter the order or time of retention are the carrier gas flow rate, column length and the temperature.

In a GC analysis, a known volume of gaseous or liquid analyte is injected into the "entrance" (head) of the column, using a micro syringe (or, solid phase micro extraction fibers, or a gas source switching system). As the carrier gas sweeps the analyte molecules through the column, this motion is inhibited by the adsorption of the analyte molecules either onto the column walls or onto packing materials in the column. The rate at which the molecules progress along the column depends on the strength of adsorption, which in turn depends on the type of molecule and on the stationary phase materials. Since each type of molecule has a different rate of progression, the various components of the analyte mixture are separated as they progress along the column and reach the end of the column at different times (retention time). A detector is used to monitor the outlet stream from the column; thus, the time at which each component reaches the outlet and the amount of that component can be determined. Generally, substances are identified (qualitatively) by the order in which they emerge (elute) from the column and by the retention time of the analyte in the column.

The instrument present here is equipped with a FID (flame ionization detector), enabling us to quantify the quantities of various organic compounds present in the sample being tested. Hence, we can find out precisely the percentage of different fatty acid present in the biodiesel.

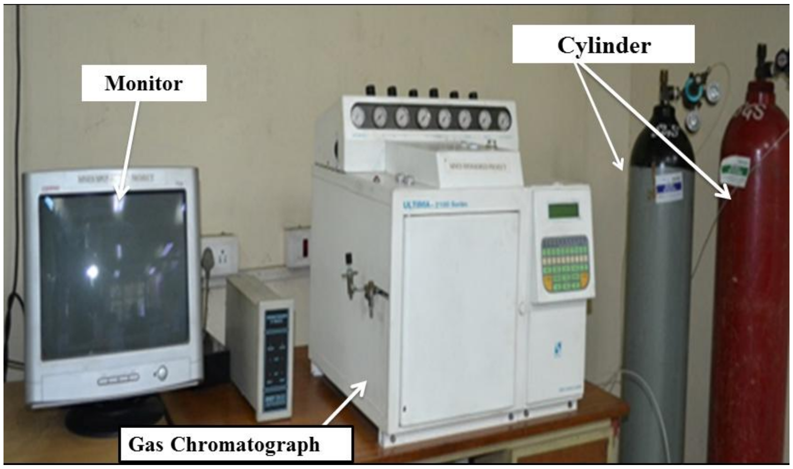


Plate 3.6 Gas Chromatograph

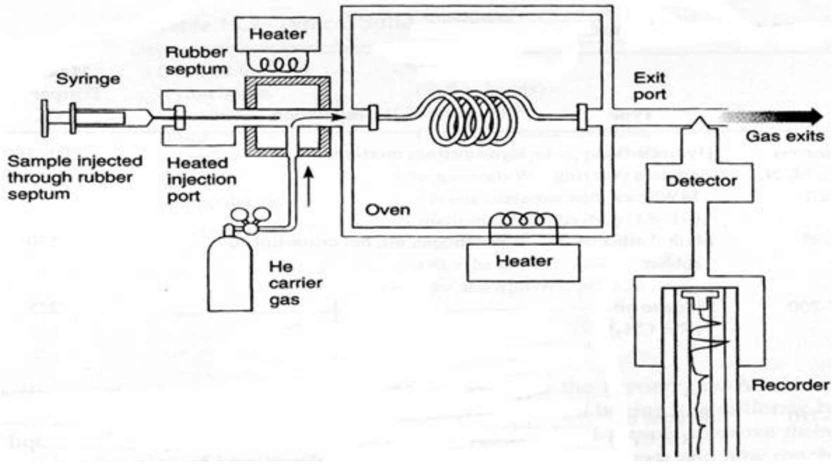


Fig.3.5 Schematic diagram of gas chromatograph

3.5.7 Rotary evaporator

A rotary evaporator is a device used in chemical laboratories for the efficient and gentle removal of solvents from samples by evaporation.

Rotary evaporators (also called "rotavaps") are used to remove solvents from reaction mixtures and can accommodate volumes as large as 3 liters. They are found in almost every organic laboratory, since they allow performing this task very quickly. A typical rotary evaporator has a water bath that can be heated in either a metal container or crystallization dish. This keeps the solvent from freezing during the evaporation process. The solvent is removed under vacuum, is trapped by a condenser and is collected for easy reuse or disposal. Most labs use a simple water aspirator vacuum on their rotavaps, so a rotavap cannot be used for air and water-sensitive materials unless special precautions are taken i.e. additional traps are used. In the lab, the house vacuum line, a circulation bath or a membrane pump are used as source for the vacuum (40-50 torr). The fact that a vacuum is usually applied to the setup means that the boiling points of the solvents are going to be significantly lower than at ambient pressure.

Since the flask is rotated during the evaporation process, the surface area is larger than normal which increases the evaporation rate significantly. The solvent is collected in a flask and can properly be disposed off afterwards (organic solvent waste). In addition, this method also avoids overheating of the target compound i.e. oxidation because lower temperatures are used. The same rules like for vacuum filtrations apply here in terms of the glassware and other precautions i.e. no cracks on the flask, etc.

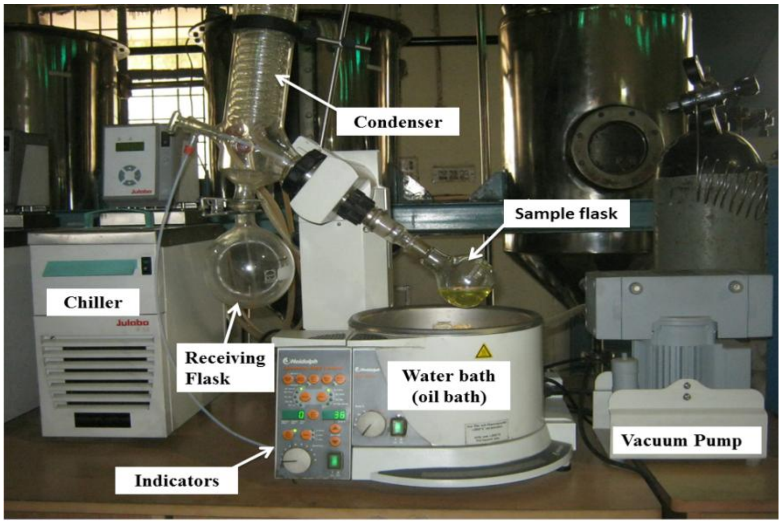


Plate 3.7 Rotary evaporator

3.5.8 CENTRIFUGE

A centrifuge is a piece of equipment, generally driven by an electric motor (or, in some older models, by hand), that puts an object in rotation around a fixed axis, applying a force perpendicular to the axis. A centrifuge is also used to separate the components of blood in blood banks. The centrifuge works using the sedimentation principle, where the centripetal acceleration causes denser substances to separate out along the radial direction (the bottom of the tube).

Plate 3.8 Centrifuge

3.6 SELECTION OF DIESEL ENGINE

Due to the robustness and high load carrying capacity diesel engine are preferred more than the patrol engine in agriculture, marine and other load carrying locomotives like trucks. Also the fuel economy and cheap diesel fuel than petrol has attracted the manufacturers to make vehicles having diesel engine.

Air pollution created by diesel engine is also more severe than the petrol engine. Also due to bulkiness in terms of more storage capacity of engine for moving more goods at the same time they consume more fuel and so create more air pollution. Due to this reason by changing some trends in the decrease of air pollution or harmful emissions by changing the fuel can bring considerable changes in the environment. Keeping the specific feature of diesel engine in mind, a typical engine system, which is actually used widely in the Indian agricultural sector, has been selected for the present experimental investigations.

3.7 DEVELOPMENT OF EXPERIMENTAL TEST RIG

A Kirloskar model number CAF 8 make, single cylinder, direct injection, diesel engine was selected for present research work, which is ma inly used for agricultural activities and for household electricity generator.

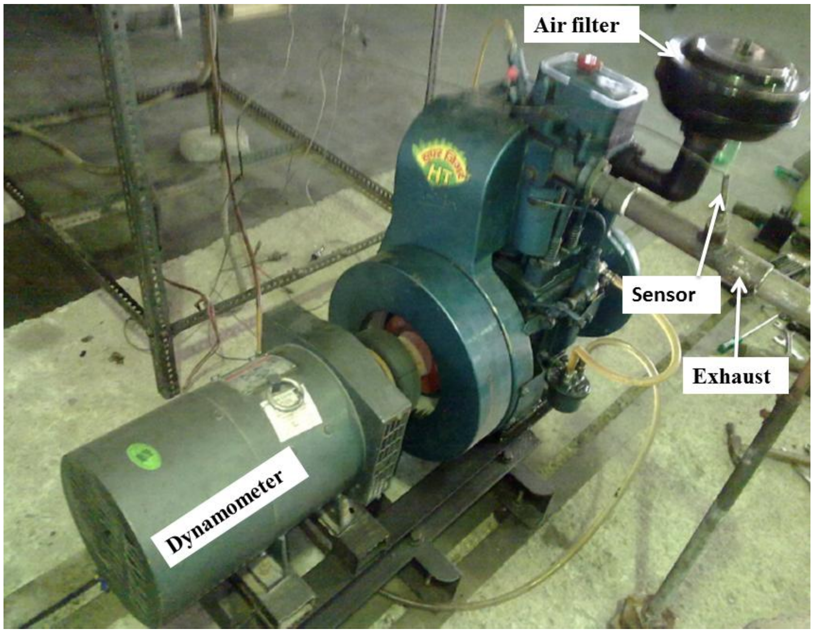


Plate 3.9 Test Engine

Table 3.5 describes the specifications of the diesel used for the present work.

Table 3.5 Diesel Engine Specification

Make	**Kirloskar**
Model	CAF 8
Rated Break Power	5.9KW/8HP
Rated Speed (rpm)	1500
Number of Cylinder	One
Bore X Stroke (mm)	95 x 110
Compression Ratio	17.5:1
Cooling System	Air Cooled
Lubrication System	Forced Feed
Cubic Capacity	0.78 Lit
Inlet Valve Open (Degree)	4.5 BTDC
Inlet Valve Closed (Degree)	35.5 ABDC
Exhaust Valve Open (Degree)	35.5 BBDC
Exhaust Valve Closed (Degree)	4.5 ATDC
Fuel Injection Timing (Degree)	26 BTDC

This engine is coupled to an electrical alternator used to produce the electric current. This supply is given to the electrical loads (bulbs). The engine is to be started by hand lever. A decompression lever is used for combustion chamber to be vented and results in low compression ratio.

Various instruments and components are mounted and coupled with the engine to give us the desired experimental data required for the calculation purpose.

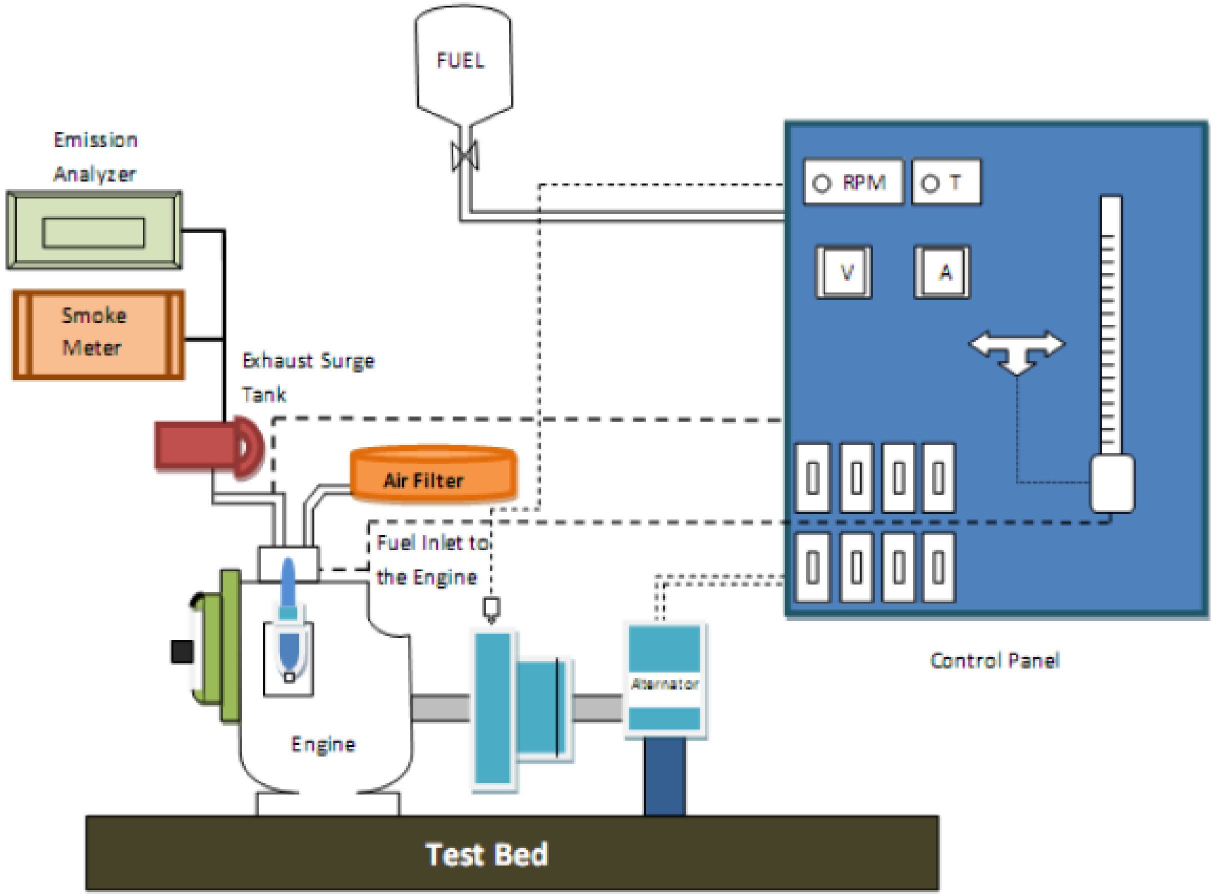


Fig 3.6 Schematic of test Rig

Fig. 3.6 shows the schematic of the test rig used in experiments for the determination of performance and emission characteristics for the engine working at different electrical loads. Plate 3.10 shows the actual test rig used for the experimental purpose

Plate 3.10 Test Rig

3.8 CONTROL PANEL INSTALLATION

After the test rig has been developed, the control panel is made and connected to the engine the control panel is mounted with the burette, electrical loads, ammeter, voltmeter, temperature indicator, rpm indicator, switches, MCB, fuel line and control valves as shown in the plate 3.12 and plates 3.11 . Fuel tank is also mounted to prove undisturbed supply of fuel to the engine.

Plate 3.11 Fuel Tank

Plate 3.12 Control Panel (Indicator & Meter)

The Engine is loaded by using electrical load bank comprising of twelve 500W tungsten electrode bulbs. The load on the engine is varied through a series of individual switches.

Plate 3.13. Burette & Valves

Plate 3.14 Electric Load Bank

Electric load bank, i.e., 12 bulbs each of 500 watts, were mounted on the rare side of the Bakelite sheet as shown in the plate 3.14 and their switches provided on the front side of the control panel.

3.9 RPM MEASUREMENT OF THE ENGINE

For the RPM measurement a digital tachometer of MTC company is being used is mounted. A nut was welded on the flywheel face of the engine and tachometer was mounted on a bracket near the flywheel at a distance less than 5mm. the display unit is mounted on the control panel board.

Plate 3.15 digital Tachometer

3.10 EXHAUST EMISSION ANALYSIS

The major pollutants appearing in the exhaust of a diesel engine are Unburned Hydrocarbons, Carbon Monoxide, smoke and NOx. For the measurement of UBHC, CO and NOx, AVL 4000 Di-Gas Analyzer was used. Smoke is also to be measured as smoke is a primary sign of incomplete combustion since it is mainly composed of very small unburnt carbon particles: therefor it is important to determine the smoke content of the flue gas. For measuring the smoke opacity, AVL 437 smoke analyzer was used. This instrument gave reading in terms of percentage opacity of the light beam projected across a flowing stream of exhaust gases, a certain portion of light is absorbed or scattered by the suspended soot particles in the exhaust. The remaining portion of the light falls on a photocell, generating a photoelectric current, which is a measure of smoke density.

The instruments that were used for the analysis of emissions are shown in plate 3.16 and plate 3.17.

Plate 3.16 AVL 437 Smoke meter

Plate 3.17 AVL Di-Gas Analyzer

In addition a simple surge tank is also mounted at the exhaust of the engine to receive consistent exhaust emissions.

3.11 DETERMINATION OF IMPORTANT PARAMETER

Various parameters are selected and determined with the help of experimental data for the analysis of the blends prepared.

These parameters are –

1. Break power produced by the engine.
2. Break specific energy consumption.
3. Break mean effective pressure.
4. Exhaust emission i.e. CO, HC, smoke count and $NO_{X.}$
5. Exhaust temperature.

With a view to calculate the parameters mentioned above, it was essential to pick the following signals from test bench:

1. Voltage generated by the alternator.
2. Current generated by the alternator.
3. Fuel consumption rate.
4. RPM of engine.

5. AVL 437 Smoke meter readings.
6. AVL DI Gas Analyzer readings.

Once the parameters were selected, the essential instruments required for sensing these parameters were installed at the appropriate points in the experimental set-up.

3.12 EXPERIMENTAL PROCEDURE

All electrical and flow connections are checked before starting the engine. If any problem is observed it is corrected. The engine to be used in the determination of performance characteristics i.e. Kirloskar CAF 8 is started by the hand cranking it at no load by the use of decompression lever. Then the flow valves are set to control the flow and the engine is allowed to run for nearly 30 minutes to attain steady conditions. With the help of stop watch the time elapsed for the consumption of 10, 20 and 30 ml of the fuel quantity is measured and the average of time is taken. Voltage, current, RPM, CO, HC, NOX, smoke density and exhaust gas temperature are measured by the various devices connected to the engine. Then the engine is loaded at different electrical loads gradually and all the parameters mentioned above are again determined and noted down. The performance characteristics and emission characteristics were evaluated for different blends and neat diesel. Comparison is made and studied after these characteristics are plotted in the graph. Before turning the engine off, the fuel was replaced with diesel oil and it was run on diesel till all the fuel in fuel filter and pipe line is consumed

CHAPTER 4

RESULTS AND DISCUSSION

4.0 INTRODUCTION

The present study was done on an unmodified diesel engine which was converted to run on a dual mode operation. The main objective of the study was to fuel the diesel engine with blends of diesel, Linseed oil methyl ester and performance and emission studies of the diesel engine when run on various blends and compares the results with baseline data of diesel engine.

4.1 GAS CHROMATOGRAPH ANALYSIS

Fatty acid profile of the biodiesel was determined by gas chromatography.Figure 4.1 shows the gas chromatogram of linseed oil methyl ester. The peak of the graph indicates retention time.

To determine the individual methyl esters the standard UNE-EN 14103:2003 was applied. For this, 250 mg of sample were dissolved in 5 ml of standard solution (prepared in a volumetric flask by dissolving 500 mg of methyl heptadecanoate in 50 ml of heptane) in a 10 ml vial. Then, the vial was closed with a Teflon cap and shaken vigorously. Finally, 2 _l of the solution obtained were injected into the gas chromatograph. The apparatus was fitted with a CP-Wax 52-CB capillary column (30 m X 0.32 mm, 0.25 _m film). Helium was used as carrier gas at a flow rate of 1 ml/min with a split ratio of 1:50. The temperature of the oven was kept at 210 ◦C and that of the injector and detector at 250 ◦C. Analyses were performed in duplicate and the errors in the determination of the FAME yield was ±2.6%.

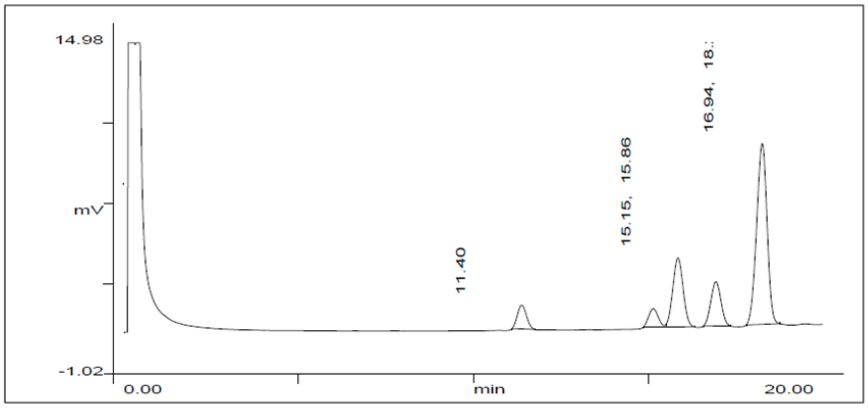


Fig. 4.1 Graph obtained from Gas Chromatograph for Linseed oil biodiesel

4.2 PROCESS OPTIMIZATION FOR THE PRODUCTION OF LINSEED BIODIESEL USING SUPERHEATED METHANOL

In the present investigation, linseed oil was mixed with methanol in the ratio of 1:42 (by weight) and five different temperatures were selected to study the triglycerides conversion i.e. 225, 250, 275, 300, 325^{0}C for the varying reaction time of 15, 30, 45, 60, 75 and 90 min. The prepared samples were tested for the mono, di and tri-glycerides. However, the FAME content also tested in the all different test fuel samples and the results obtained were categorised on the basis of reaction time and temperature. The results obtained are shown in fig. 4.2-4.5.

As we know that monoglyceride and diglyceride are the intermediate products generated during the process, and are the results of incomplete conversion of triglycerides into FAMEs. The evolutions of both are shown in Fig. 4.2 & 4.3. From fig. 4.2, it can be observed that at low temperature i.e. 225, 250 and 275^{0}C the monoglyceride are found more in the biodiesel. However, decrement was obtained for the higher temperature and longer period of time. The yield of MG passed through a maximum at 300^{0}C and above, which was reached in shorter reaction time as the temperature increased.

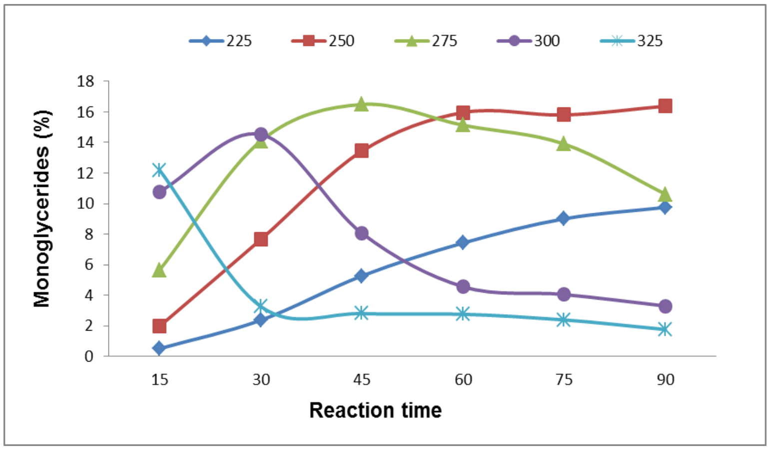


Fig. 4.2: Variation of monoglyceride content in the biodiesel obtained after supercritical trans-esterification.

After reaching a maximum, and for longer reaction time, the yield of MG seems to decrease very slowly and hence, higher the temperature, the lower the minimum yield reached. Thus, for a 90 min reaction time, the MG content was observed minimum. Therefore, from the above results it can be said that the MG can be reduced by increasing the temperature of the process.

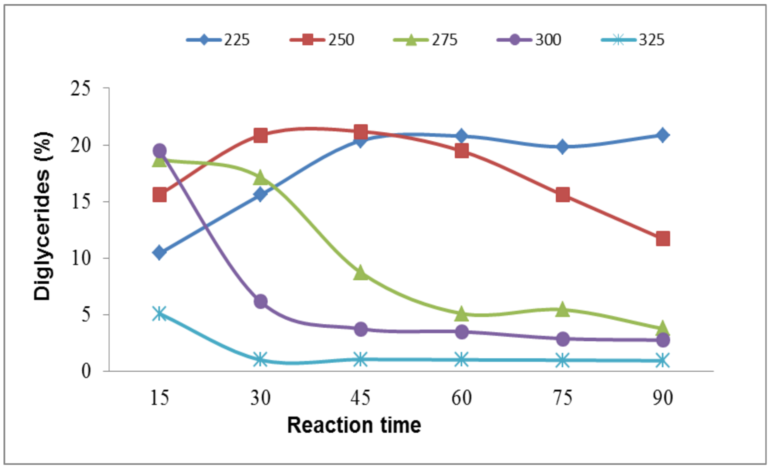


Fig. 4.3: Variation of diglyceride content in the biodiesel obtained after supercritical trans-esterification.

The fig. 4.3 & 4.4 shows the variation of diglyceride and triglyceride content in the biodiesel produced from the SCT. The yield of DG increases to a maximum value and then decreases to a minimum value after which it remains constant for a longer duration of reaction time. Higher the temperature, lower is the yield of DG. Thus, the minimum yield is attained at

325^{0}C. Therefore, and as indicated for MG, the only way to reduce the DG content is by increasing the temperature. As expected, the maximum yield of DG was reached at shorter reaction time than that of MG, a behavior that is typical in reactions that are developed in successive steps, such as the three-step trans-esterification of TG with methanol. Also, the same decrement is observed in triglyceride content as shown in fig. 4.4.

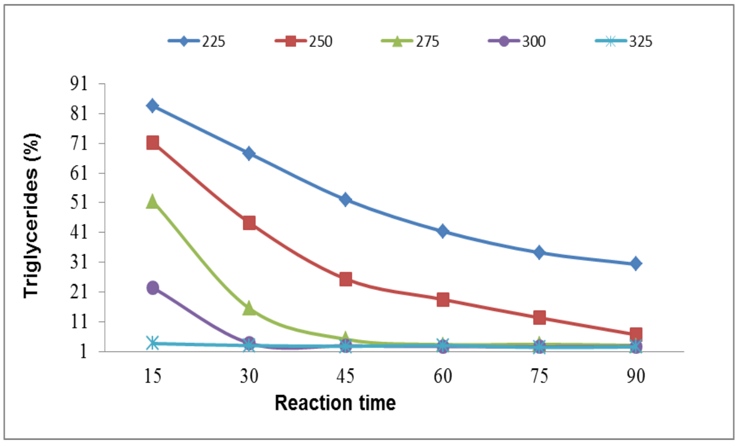


Fig. 4.4: Variation of triglyceride content in the biodiesel obtained after supercritical trans-esterification.

As expected, the TG conversion is higher for higher temperature; however this increment was observed only up-to a limit 300^{0}C in the present study. The fig. 4.5 shows the variation of FAME's with reaction time which was done on the basis of the process with appropriate EN Standard (EN 14103:2003).

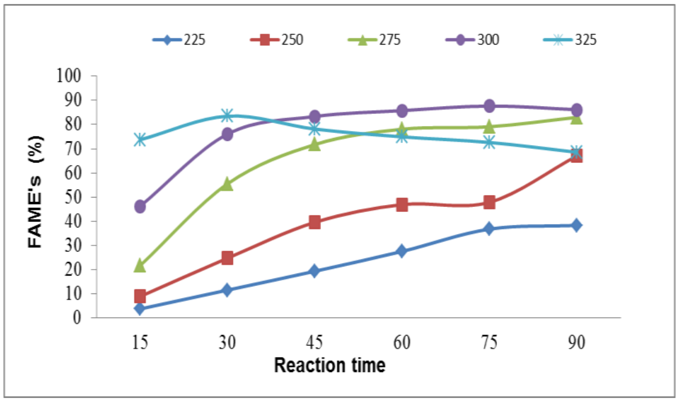


Fig. 4.5: Variation of Fatty acid methyl esters content in the biodiesel obtained after supercritical trans-esterification.

Further, increase in temperature results in the decrement in the FAME content in the produced biodiesel. The maximum reached at 300^0C (80% yield of FAME) was higher than that obtained at 325^0C (68% yield of FAME), and was reached at a longer reaction time (70 min at 300^0C vs. 30 min at 325^0C). The fact that the yield of FAME decreased at long reaction times was probably due, as discussed below, to the thermal degradation of the polyunsaturated fatty acid methyl esters generated [39].

4.3 PHYSICO CHEMICAL PROPERTIES

The Diesel fuel and various blends formed were analyzed for several physical, chemical properties. Density and viscosity of the various blends formed was found higher than diesel. Presence of oxygen in the blended fuel improves combustion properties and emissions but reduces the calorific value of the fuel. All the blends have approximately 96-98% calorific value compared to diesel. Nitrogen content of the fuel also affects the NOx emissions (by formation of fuel NOx).

Table 4.1 Physico-chemical properties of different blends of diesel and linseed biodiesel

Properties → Fuels ↓	**Density (g/mm^3) at 15°C**	**Calorific value (MJ/kg)**	**Kinematic Viscosity (mm^2/s) at 40°C**
LME5 D95	0.82791	45.26	3.2222
LME10 D90	0.83161	45.04	3.3210
LME15 D85	0.83532	44.81	3.4198
LME20 D80	0.83903	44.58	3.5187
LME100	0.89835	40.95	5.1000
D100	0.82420	45.49	3.1231

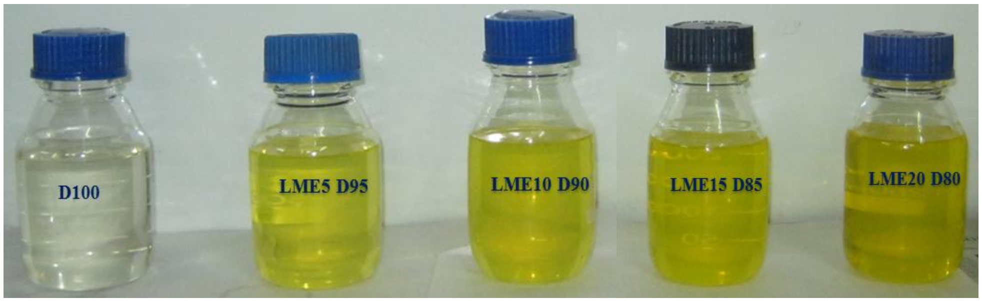


Plate 4.1 Blends of linseed oil biodiesel and diesel

4.4 PERFORMANCE CHARACTERISTICS

Various performance characteristics were analyzed for different test fuels and they are summarized in this section. The results obtained for different blends of linseed oil methyl ester and diesel are compared with baseline diesel fuel.

4.4.1 Brake thermal efficiency (BTE)

The variation in brake thermal efficiency (BTE) is shown in Fig 4.6. It can be observed that BTE of all blends of linseed oil methyl Ester with diesel is higher than that of mineral diesel. At full load condition, the BTE is 13.3% and 23 % higher for LME5, LME10, LME15, and LME20 respectively than diesel. This is due to the fact that biodiesel contains around 10-11% in-built oxygen and higher cetane number than diesel fuels. Due to this, better combustion take place hence there is improvement in the break thermal efficiency in case of biodiesel diesel blends. It can also be seen that the increase in biodiesel concentration in blends increases the BTE.

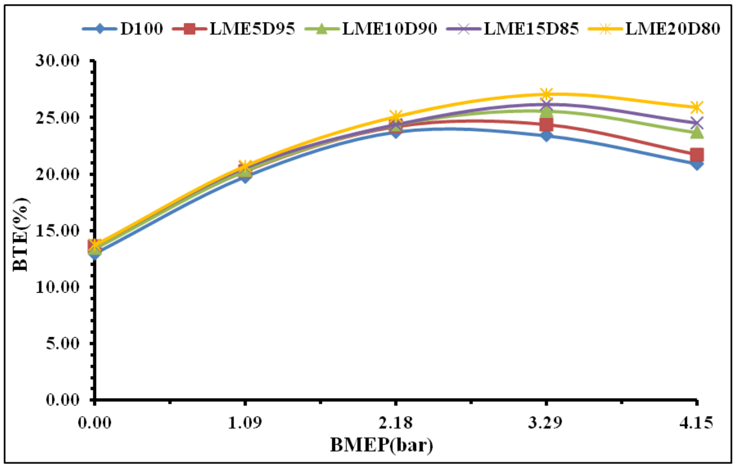


Fig: 4.6 Variation of BTE (%) with BMEP (bar)

4.4.2 Brake specific energy consumption (BSEC)

Brake specific energy consumption (BSEC) measures the amount of input energy required to develop one-kilowatt power. The BSEC is an important parameter of an engine because it accounts for both mass flow rate and heating value of the fuel. Basic specific energy consumption is an essential and ideal parameter for comparing engine performance of the fuels having different calorific value and density. Fig.4.7 shows the variation of BSEC for neat diesel and linseed oil biodiesel blends. It is observed that the BSEC is lower for all blends of linseed oil biodiesel as compared to the mineral diesel fuel. At full load condition, the BSEC for LME20 blend is 17% less than that of mineral diesel.

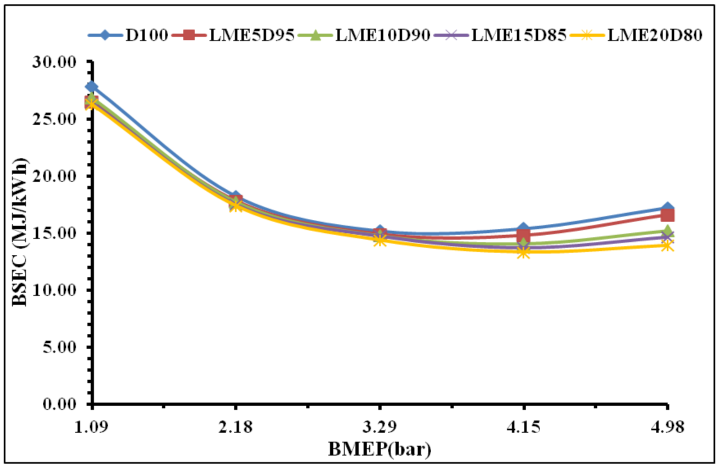


Fig. 4.7 variation of BSEC with BMEP

4.5 EMISSION CHARACTERISTICS

The emission characteristics of various blends of linseed oil biodiesel and diesel are summarized in this section. Main exhaust emissions are CO, UBHC, NO_X and smoke opacity.

4.5.1 Carbon monoxide (CO)

The variation of carbon monoxide (CO) emission of diesel and blends of Linseed oil biodiesel is shown in Fig 4.8. It has been observed that the CO emission is lower for all the blends of linseed oil methyl ester than that of neat diesel. The CO emissions are found to be increasing with increase in load since the air–fuel ratio decrease with increase in load in internal combustion engines [71]. The engine emits less CO using biodiesel blends as compared to that of diesel fuel under all loading conditions. With increasing biodiesel percentage, CO emission level decreases as amount of oxygen content in biodiesel improve combustion and result in better oxidation. The higher cetane number of blend as compared to that of mineral diesel is also one of the reasons of better combustion.

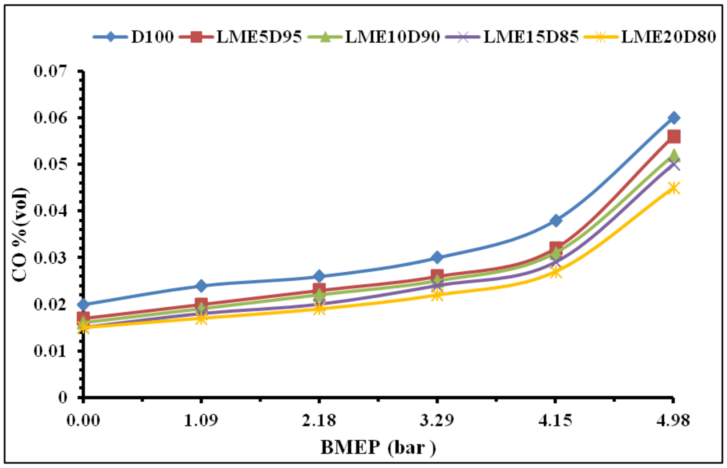


Fig: 4.8 variation of CO %(vol.) With BMEP

4.5.2 Hydrocarbon (HC)

The HC emissions of different blends are shown in Fig.4.9. It is found that the hydrocarbon (HC) emission is less for all the blends of linseed oil methyl ester as compared to neat diesel fuel. As percentage of biodiesel increase in the blend, HC emission decreases. LME20 has 14 % less hydrocarbon emission than that of mineral diesel fuel at full load condition but at part load condition HC emission of diesel is 20 -23 % more than that of biodiesel blends. The significant decrease in HC emission is due to the improved combustion of the fuels.

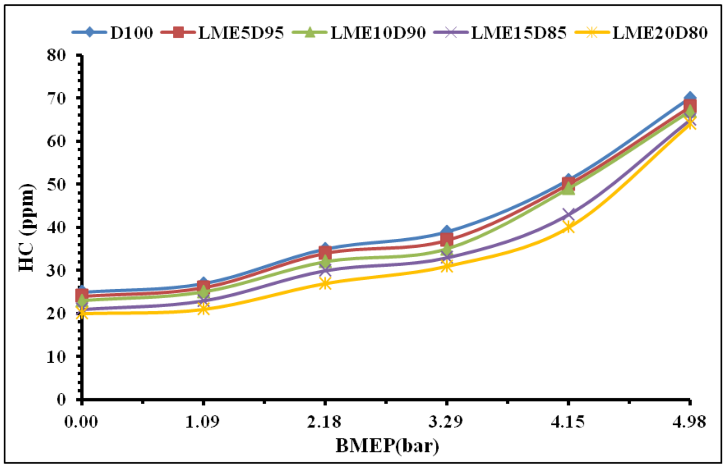


Fig. 4.9 variation of HC (ppm) with BMEP (bar)

4.5.2 Nitrogen oxide (NO_X)

The nitrogen oxide (NO_X) emissions for different fuels are shown in Fig 4.10. It has been found that the NO_X emission of biodiesel blends is quite high as compare to the neat diesel with maximum value for LME20 fuel. The emission of NO_X is determined by oxygen concentration, peak pressure, combustion temperature and time. The availability of oxygen in biodiesel results in increase in NO_X emission.

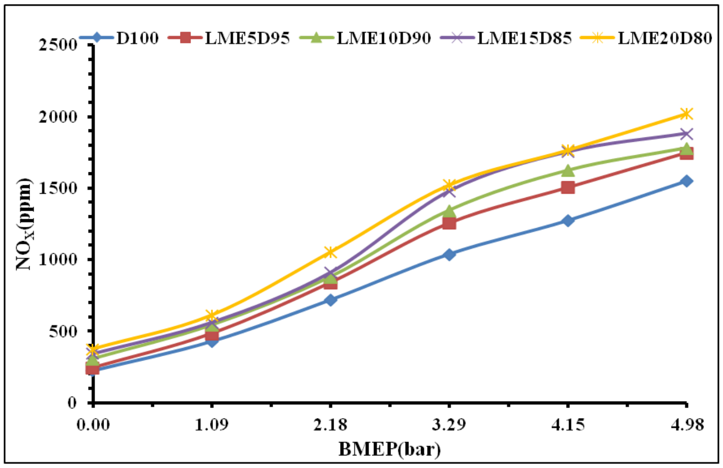


Fig. 4.10 variation of NO_X (ppm) with BMEP (bar)

4.5.4 Smoke opacity

Fig. 4.11 shows the variation of smoke opacity of linseed oil methyl ester blends and diesel fuel. From the test results, it was found that the smoke opacity of neat diesel fuel is higher to that of biodiesel blends. At lower loads, the opacity difference is comparable but for full load condition LME20 shows lower smoke opacity than that of diesel fuel. This occurs due to longer ignition delay period (keeping all parameters constant) because of more fuel injection before ignition, higher temperature in the cycle and earlier termination of combustion process.

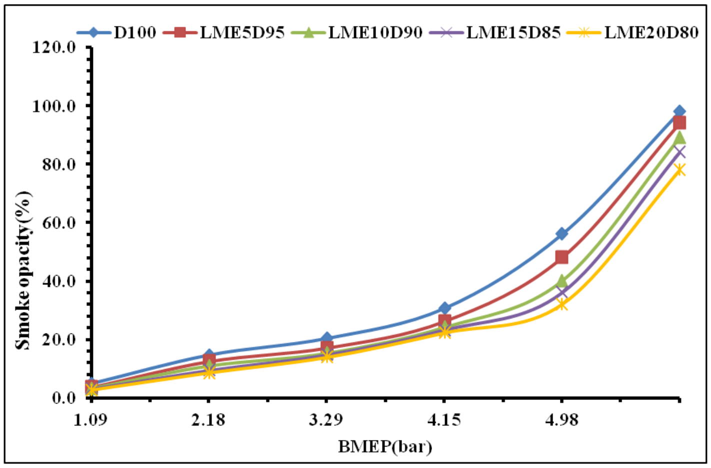


Fig. 4.11 variation of smoke opacity (in %) with BMEP (bar)

The residence time is therefore increased. All these factors have found to reduce smoke opacity in exhaust. However, earlier injection may results in more combustion noise and nitrogen oxide emission. . This result is in confirmation with Agrawal et.al. [72] And Sayin et.al.[73].

CHAPTER 5

CONCLUSION AND SCOPE FOR FUTURE WORK

1.0 CONCLUSION

As a definitive outcome for the series of trials performed for the biodiesel production using supercritical technology to study the temperature and run time effect on the FAMs, DG and TG, the following conclusions can be drawn:

- The Mono-glycerides were found to be more at lower temperature and they decreases with increase in temperature. The MG's were least for 325^0C and 90min run.
- At low temperature, the Di-glycerides followed an increasing curve with reaction time. However, it followed the opposite pattern for higher temperatures.
- The triglyceride decrease with the increase in reaction time and temperature and subsequently, least for the 325^0C temperature.

FAMEs curves show an increasing pattern for both reaction time and temperature but after 300^0C, a sudden decrement is observed.

Exhaustive engine trials conducted on a naturally aspirated, single cylinder diesel engine fuelled with various blends of LME and diesel. Several performance and emission characteristics of the engine trial and their comparative assessment with diesel baseline are described briefly below.

1. Full load brake thermal efficiency was found to increase with increase in LME percentage in the blend as a result of sufficiently good cetane rating of biodiesel. Full load BTE of LME10 was found to be nearly 4% higher than diesel baseline, where as that of LME20 exhibited an impressive 23% increase over baseline data of diesel.
2. Maximum peak load brake specific energy consumption of 17.23 MJ/kWh was observed at the baseline data of diesel. With increase in percentage of LME in the blend a steady reduction in BSEC was reported. LME10 exhibited BSEC of 16.58 MJ/kWh where as that of LME20 showed a minimum BSEC of 13.91 MJ/kWh.

3. Emission of carbon monoxide was found to reduce with increase in LME percentage in the blends. At part loads CO emission was found to be low for all the test fuels, however, substantial increase was observed after 60% load. Reduction in carbon monoxide emissions at higher blends may be attributed towards better combustion characteristics of sufficiently good cetane oxygenated LME.
4. LME10 exhibited parallel hydro carbon emission tendencies as that of diesel baseline. However, other higher blend of LME showed reduction in HC emission at all loads compared to that of diesel confirming better combustion characteristics.
5. Due to sufficiently good cetane rating of LME and improved combustion characteristics, the in-cylinder temperature was increased resulting in higher NO_X emission for blended fuels as compared to baseline data. Full load NO_X emission was steeply increased by 22% for LME20 as compared to diesel baseline. LME10 exhibited marginal increase in NO_X emissions.
6. Opacity of full load engine smoke was insignificant at lower loads for all the test fuels. However, at higher loads LME blends showed reduction in smoke opacity as compared to neat diesel operation.

From the above conclusion it can be said that up to 20 % blend of linseed oil methyl ester with neat diesel may be advised for direct injection diesel engine application.

Additional tests should be done to evaluate emissions impacts of linseed oil biodiesel, including particulate matter and other emission cycles as used in the homologation. Also some durability tests should be done to investigate the effects of this new fuel.

5.1 FUTURE WORK

On the basis of experience gained during the present experimental studies, the following diections are indicated for further investigation and development.

1. Optimizing the yield of FAME by varying the methanol concentration.
2. Use of different catalyst i.e. homozanious and hetrozanious for the complete conversion of triglyserides in to FAME.
3. Utilization of blends of biodiesel-alcohol-diesel is suggusted for improvement in performance characteristic and NO_x emission.
4. In the present investigation, an unmodified diesel engine was used havig compression ratio 17.5. It is recommended that further work may be carried at variable compression ratios and injection angles.
5. Some computational and simulation analysis may be carried by further researchers.

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APPENDIX-I

Technical Specifications of AVL Di-Gas Analyser

Measurement principle	CO, HC, CO2	Infrared measurement
	O2	Electrochemical measurement
	NO (option)	Electrochemical measurement
Operating temperature	+5 to +45ºC	Keeping measurement accuracy
	+1 to +50ºC	Ready for measurement
	+5 to +35ºC	with integral NO sensor (+40ºC)
Storage temperature	-20 to +60ºC	
	-20 to +50ºC	With integrated O2 sensor
	-10 to. +45ºC	With integrated NO sensor
	0 to +50ºC	With water in filter or pump
Air humidity	90% max,	non-condensing
Power drawn	150 VA	
Dimensions	432 x 230 x 470 mm	(w x h x l)
Weight	16 Kg	

Measurement Ranges of AVL Di-Gas Analyser

Parameter	**Measurement Range**	**Resolution**
CO	0-10% vol	0.01% vol
CO_2	0-20% vol	0.1% vol
HC	0-20000 ppm vol	1 ppm
NO_X	0-5000 ppm vol	1 ppm
O_2	0-25% vol	0.01% vol

APPENDIX-II

Technical Specifications of AVL 437 Smoke Meter

Accuracy and Reproducibility	± 1% full scale reading
Heating Time	Approx. 20 min
Light source	Halogen bulb 12 V / 5W
Colour temperature	3000 K ± 150 K
Detector	Selenium photocell dia. 45 mm, Max. Sensitivity in light In Frequency range: 550 to 570 nm. Below 430 nm and above 680 nm sensitivity is less than 4% related to the maximum sensitivity
Maximum Smoke	250ºC Temperature at entrance

APPENDIX-III

Accuracies and uncertainties of measurements

S.N.	**Measurements**	**Measurement Principle**	**Range**	**Accuracy**
1	Engine load	Strain gauge type load cell	0-25 Kg	±0.1Kg
2	Speed	Magnetic pick up type	0-2000 rpm	±20 rpm
3	Time	Stop watch	--	±0.5%
4	Exhaust Temperature	K-type thermocouple	0-1000°C	±1^{0}C
5	Carbon monoxide	Non-dispersive infrared	0-10% vol.	±0.2%
6	Carbon dioxide	Non-dispersive infrared	0-20% vol.	±0.2%
7	Total hydrocarbons	Non-dispersive infrared	0-20,000 ppm	±2 ppm
8	Oxides of nitrogen	Electrochemical	0-4000 ppm	±15ppm
9	Smoke	Photochemical	0-100%	± 2%
10	Crank angle encoder	Optical	0-720 °CA	± 0.2^{0}CA
11	Pressure	Piezoelectric	0-200 bar	± 1 bar
	Calculated results			**Uncertainty**
12	Engine power	--	0-8 kW	±1.0%
13	Fuel consumption	Level sensor	--	±2.0%
14	Air consumption	Turbine flow type	--	±1.0%
15	BTE	--	--	±1.0%
16	BSEC	--	--	±1.5%
17	Heat release	Sorenson model	--	±5.0%
18	In-cylinder temp.	Ideal gas equation	Up to 3000°K	±5.0%