OPTIMIZATION OF BIODIESEL PRODUCTION USING TAGUCHI AND SCREENING DESIGNS

Submitted in partial fulfillment for the Award of the Degree of

BACHELOR OF TECHNOLOGY

In

MECHANICAL ENGINEERING

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ABSTRACT

Biodiesels are known as ecofriendly and biodegradable fuels. The unique properties like high Cetane number and excess molecular oxygen helps the combustion complete with regulated emission. To mitigate the challenges of diesel engine exhaust emissions like NO_x, HC, CO, CO_2, PM and Smoke, biodiesel from different feed stocks of animal and plant oils are used. It has already proved experimentally that biodiesel can be used an alternative fuel without changing the basic design of the existing engines. Despite of their advantages their practical application was limited to experimental stage this is due to the fact its high production cost. In this research an attempt was made to reduce the initial raw oil cost by utilizing the low cost feedstock of used cooking oil which was available at free of cost. In order to maximize the biodiesel yield advanced optimization techniques of Taguchi and Definitive screening design was adopted. Different contributing parameters which influence the biodiesel yield were analyzed with these techniques. All the fuel samples were tested for different physicochemical properties by following the international standards. And it was concluded that by using the waste cooking oil the overall cost of the biodiesel was reduced and the optimization techniques help in the predicting the maximum yield.

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NOMENCLATURE

1. %: Percentage
2. 2^k: 2 to the power k
3. Å: Amstrong
4. ANOVA: Analysis of variance
5. Ar: Argon
6. ASTM: American society for testing and materials
7. B0: Neat biodiesel
8. BBL/Day: Barrels per day
9. BSES: Bharat stage emission standards
10. BSFC: Brake specific fuel consumption
11. BTE: Brake thermal efficiency
12. $C_4H_8O_2$: 1,4 Dioxene
13. CAGR: Compounded annual growth rate
14. CC: Catalyst concentration
15. CCD: Central composite design
16. CCRD: Central composite rotatable design
17. CH_3OK: Potassium methanolate
18. CH_3ONa: Sodium methoxide
19. CH_4: Methane
20. CN: Cetane number
21. CO_2: Carbon dioxide
22. COST: Changing one separate factor at a time
23. DCS: Direct injection combustion system
24. DI: Direct injection
25. DOE: Design of experiments
26. DSD: Definitive screening design
27. EGR: Exhaust gas recirculation
28. EGT: Exhaust gas temperature
29. EIA: Energy information administration
30. FAC: Fatty acid compostions
31. FAME: Fatty acid methyl ester
32. FBO: Food Business Operators

33. FFA: Free fatty acids
34. FFA: Free fatty acids
35. FSSAI: Food safety and standards authority of India
36. GBD: Global burden of disease
37. GDP: Gross domestic product
38. GISS: Goddard Institute for Space Studies
39. GT: Giga tonnes
40. HC: Hydrocarbons
41. He: Helium
42. IB: Iso-Butanol
43. ICS: Injection combustion system
44. ID: Ignition delay
45. IDI: Indirect injection
46. IEA: International energy agency
47. IEO2017: International energy outlook
48. JME: Jathropa methyl ester
49. Kg/m^3: Kg per meter cube
50. KOH: Potassium hydroxide
51. L9: Runs 9
52. LPG: Liquid Petroleum Gas
53. ml: milliliter
54. MMT: Million Metric Tons
55. MPa: Mega Pascal
56. MR: Molar ratio
57. MR: Molar ratio
58. MTs: Million Tons
59. MZME: Manikara zapota methyl ester
60. N_2: Nitrogen
61. NaOH: Sodium hydroxide
62. Ne: Neon
63. NO_x: Nitrous Oxides
64. O_2: Oxygen
65. OPEC: Organization of petroleum exporting countries
66. PBD: Palm biodiesel

67. PM: Particulate matter
68. PME: Palm methyl ester
69. Ppm: Parts per million
70. RPM: Revolutions per minute
71. RSM: Response surface method
72. R-sq (adj): Coefficient of determination (adjust)
73. R-sq(pred): Coefficient of determination (predicted)
74. R-sq: Coefficient of determination
75. RT: Reaction temperature
76. RTi: Reaction time
77. SO_2: Sulphur dioxide
78. WFPO: Waste frying palm oil
79. WFPOME: Waste frying palm oil methyl ester
80. WHO: World health organization
81. Wt%: weight percentage
82. WTI: West Texas Intermediate

CHAPTER-I

INTRODUCTION

1.1 Crude oil reserves

Crude oil exists as a liquid that rests in various formations deep within the Earth's crust. This liquid forms as a result of the decomposition of fossils that are trapped beneath the earth surface millions of years ago. Crude oil in both its unprocessed and refined states is broadly classified as petroleum. Petroleum satisfies a majority of the global energy demand. After crude oil has been extracted from the ground, it is generally transported to a refinery, where it is heated and distilled into more usable products. Most of these are various types of fuel i.e. Gasoline, Diesel, Kerosene, Liquefied petroleum gas (LPG) and Heating oil it also serves in other purposes like solvents, lubrications, Asphalt and coke. Crude oil is the major transportation fuel almost every road transport vehicle runs on crude oil petroleum products, it is estimated that around 1.2 billion vehicles are running worldwide and this figure is about to reach 2 billion by 2030 [W1].

Around the world 93 million barrels of crude oil is per day (bbl/day) on average in 2015 according to the International Energy Agency (IEA), this has now reached to 100 million barrels a day in early 2019 [W_2]. The Organization for Petroleum Exporting Countries (OPEC) reports that there are 1.5 trillion barrels of crude oil reserves left in the world, but the ever increasing demand and consumption rates are growing year by year. The main point is that crude oil is a non-renewable resource. So, regardless of the exact quantity, the fact remains that the supply is going to exhaust one day [W_3].

According to the U.S. Energy Information Administration's (EIA) International Energy Outlook 2017 (IEO2017), the global supply of crude oil is expected to be adequate to meet the world's demand for liquid fuels till 2050. Due to its increasing demand there's always been an increase crude oil price, crude oil prices back in 1947 was 17.37 dollars per barrel, it has risen now till 53$ per barrel as per Interactive charts of West Texas Intermediate (WTI) [W_4].

The oil and gas industry in India dates back in 1889 when the first oil deposits in the country were discovered in the state of Assam, As on March 2018, India had estimated crude oil reserves of 594.49 million tonnes and natural gas reserves of

1339.57 billion cubic meters but this reserves are just not enough to meet the growing need of India's crude oil consumption.

India is the third biggest oil importer after US and China. India is heavily dependent on crude oil and Natural Gas imports with 82.8% import dependence for crude oil and 45.3% for natural gas. India generated 35.2 million tons of petroleum products from indigenous crude oil production whereas the consumption of petroleum products is 204.9 million tons. India's crude oil production is not even close $1/4^{th}$ of its consumption and the demand is ever increasing, India accounted for 0.92% of world oil production in 2016-18, The production in India was 20,294 thousand tonne of crude oil in the first seven months of the financial year (April-October 2018), it is the lowest output recorded in the past seven years during the same period. [W_5]

India is highly dependent on import of crude oil. Net imports of crude oil rose from 121.67 MTs (Million tonnes) during 2007-08 to 213.93 MTs during 2016-17, more than 70% of its crude oil requirements and part of the petroleum product requirement is met from imports.

Exploring and drilling for oil may disturb land and marine ecosystems. Seismic techniques used to explore for oil under the ocean floor may harm fish and marine mammals. Drilling an oil well on land often requires clearing an area of vegetation which causes deforestation and oil spills most oil spills are the result of accidents at oil wells or on the pipelines, ships, containers, trains, and trucks that move oil from wells to refineries. Oil spills contaminate soil and water and may cause devastating explosions and fires. Construction activities associated with oil and gas drilling will leave behind radical impacts to the landscape and causes soil erosion which could lead to landslides, flooding and soil unsuitable for vegetation and also affect wildlife habitats. Extraction of crude oil causes a chemical process set in motion by the high temperatures created by the combustion of petroleum that sets off deadly smog of toxic gases. When these gases combine with the water in the air, the rain that falls is highly acidic, this acid rain cause dead tress soil erosion and spoiled buildings.

1.2 Pollution and climatic changes

Vehicular emissions are the major disadvantage of petroleum products, the tailpipe emissions that come out are Carbon dioxide (CO_2), Nitrogen oxides (NO_x),

Hydrocarbons (HC), Sulfur dioxide (SO_2), Particulate matter (PM) these emissions are toxic in nature and are degrading our environment day to day. According to a report released by the World Health Organization (WHO) nine out of 10 people around the world breathe polluted air and around 7 million people die each year from air pollution. Ambient air pollution alone caused some 4.2 million deaths in 2016, while household air pollution from cooking with polluting fuels caused an estimated 3.8 million deaths in the same period [W_7].

More than 90% of air pollution-related deaths occur in low and middle-income countries, mainly in Asia and Africa, followed by low and middle-income countries of the Eastern Mediterranean region, Europe and the America. Normal component of fresh air are consist of 78.084 percent Nitrogen (N_2), 21.946 percent Oxygen (O_2), 0.934 percent Argon (Ar), 0.0397% Carbon dioxide (CO_2), 0.00182% Neon (Ne), 0.0005% Helium (He) and 0.0002% Methane (CH_4).[W_6] Amount of CO_2 present in atmosphere in 2005 was 378ppm (parts per million) whereas now in 2019 it has reached 411ppm in January 2019 and the graph is increasing still, humans are emitting around 29 billion tonnes of carbon dioxide into the atmosphere per year increase in CO_2 is the main reason for global warming and causes suffocation of living organisms, high concentration of carbon dioxide gas causes narcosis[W_8].

Air pollution in India is a serious issue with the major sources being vehicle emission and traffic congestion because of a population of 1.33 billion and total number of vehicles being 210 million in 2015[W_9]. India was the third largest emitter of carbon dioxide in 2009 at 1.65 Gt (Giga tonnes) per year, after China (6.9 Gt per year) and the United States (5.2 Gt per year) sources of greenhouse gas emissions from India is from black carbon, NO_x, methane and other air pollutants. These pollutants are emitted in large quantities in India every day from incomplete and inefficient combustion of biomass and gasoline. India's poorly managed solid waste, inadequate sewage treatment plant, water pollution and agriculture are other sources of greenhouse gas emissions.

Outdoor air pollution has become the fifth largest killer in India after high blood pressure, indoor air pollution, tobacco smoking, and poor nutrition, Global Burden of Disease (GBD) report. To curb air pollution in India Bharat stage emission standards (BSES) were implemented , emission standards were instituted by the Government of India to regulate the output of air pollutants from internal combustion engines and Spark-ignition engines equipment, including motor vehicles.

The standards are based European regulations (EURO) and were first introduced in 2000, currently Bharat Stage IV emission norms are in place since April 2010 and it has been enforced for entire country since April 2017. The Indian government announced that the country would skip the BS-V norms altogether and adopt BS-VI norms by 2020. BS-IV engines have sulfur emissions of 50ppm but for BS-VI engines it has been cut down 10ppm.

Table 1.1 Diesel emission standards for passenger cars, (gm/Km)

S No.	Bharat Stages	Reference	Year	CO	NO_x	HC+NO_x	PM
1	India 2000	EURO-I	2000	2.72	-	0.97	0.14
2	BS-II	EURO-II	2001-2005	1.0	-	0.7	0.08
3	BS-III	EURO-III	2005-5010	0.60	0.50	0.56	0.05
4	BS-IV	EURO-IV	2010-Present	0.50	0.25	0.30	0.025
5	BS-V	EURO-V	Skipped	0.50	0.180	0.230	0.005
6	BS-VI	EURO-VI	2020	0.50	0.080	0.170	0.005

Table 1.2 Petrol emission standards for passenger cars, (gm/km)

S No.	Bharat Stages	Reference	Year	CO	THC	NHMC	NO_x	HC + NO_x	PM
1	India 2000	EURO-I	2000	2.72	-	-	-	0.97	-
2	BS-II	EURO-II	2001-2005	2.2	-	-	-	0.5	-
3	BS-III	EURO-III	2005-5010	2.3	0.20	-	0.15	-	-
4	BS-IV	EURO-IV	2010-Present	1.0	0.10	-	0.08	-	-
5	BS-V	EURO-V	Skipped	1.0	0.10	0.068	0.060	-	0.005
6	BS-VI	EURO-VI	2020 (Proposed)	1.0	0.10	0.068	0.060	-	0.005

Climate change has always happened on Earth, it is the rapid rate and the magnitude of climate change occurring now that is of great concern worldwide. Greenhouse gases in the atmosphere absorb heat radiation which tend to increase the temperature of earth ,land-use changes, such as deforestation have led to changes in the amount of sunlight reflected from the ground back into space.

The concentration of CO_2 in the atmosphere has reached a record high relative to more than the past half-million years, and has done so at an exceptionally fast rate. Current global temperatures are warmer than they have ever been during at least the past five centuries, probably even for more than a millennium. According to an ongoing temperature analysis conducted by scientists at NASA's Goddard Institute for Space Studies (GISS), the average global temperature on Earth has increased by about 0.8° Celsius (1.4° Fahrenheit) since 1880. Two-thirds of the warming has occurred since 1975, at a rate of roughly 0.15-0.20°C per decade [W_{10}].

This increase in global temperature and climatic changes is the cause of emissions from crude oil due to its high carbon and NO_x emissions to reduce these emissions and pollution concerns especially for a country like India switching to alternate fuels is the best available option.

1.2 Alternate for crude oils

Gasoline and diesel are still fossil fuel kings of the fuel supply, due to high demand of crude oil and their scarcity and increasing of prices alternate sources of energy are getting much popular these days. Alternative fuels generally have lower vehicle emissions, carbon emission is undoubted the primary advantage of alternative energy over fossil fuels that contribute to smog, air pollution and global warming. As fossil fuels are unequally distributed around the world, global economy is dependent on a few countries that export them. With the use of alternative energy, this dependence would be significantly reduced.

Most alternative fuels don't come from finite fossil-fuel resources and are renewable. Alternative fuels can help nations become more energy independent and will increase the economy of country by cutting its imports on crude oil.

The alternative fuels available are,

1.3.1 Alcohol fuels

Alcohols have been used as a fuel. The alcohols that can be used as fuels are (methanol, ethanol, propanol and butanol) because they can be synthesized chemically and have characteristics which allow them to be used in IC Engines, One advantage that alcohol fuels have over other alternative fuels is high octane rating with helps in fuel efficiency.

Methanol and ethanol fuel are primary sources of energy; they have better shelf life and can be transported easily. Methanol and ethanol both have advantages over

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Methanol and ethanol fuel are primary sources of energy; they have better shelf life and can be transported easily. Methanol and ethanol both have advantages over

fuels such as petrol and diesel fuel. In SI engines, both alcohols can run at a much higher exhaust gas recirculation rates and with higher compression ratios and have the potential to reduce exhaust emissions like NO_x , CO, HC etc.

1.3.2 Biomass

Biomass fuels are organic materials produced in a renewable manner. Two categories of biomass fuels are woody fuels and animal wastes. Municipal solid waste is also a source of biomass fuel. Biomass fuels have low energy densities compared to fossil fuels Biomass fuels are organic materials produced in a renewable manner. Two categories of biomass fuels are woody fuels and animal wastes. Municipal solid waste is also a source of biomass fuel. Biomass fuels have low energy densities compared to fossil fuels.

Biomass is considered to be carbon neutral because the quantity of CO_2 is very less and the amount that is released during combustion is the same as that absorbed by the plant during photosynthesis. This differs from the conventional fossil fuels because although both originate from organic matter, the carbon in fossil fuels has been locked away for millions of years, and when released during combustion, disrupts CO_2 levels in the atmosphere.

Biomass in the energy production industry is increasing it is most often referred to plants or plant-based materials that are not used for food and are generally dumped because they are of no use. As an energy source, biomass can either be used directly via combustion to produce heat, or indirectly after converting it to various forms of bio-fuel.

1.3.3 Algae-based fuels

Algae fuel is an alternative to liquid fossil fuels that uses algae as its source of energy, Like fossil fuel, algae fuel releases CO_2 when burnt, but unlike fossil fuels, algae fuel only release CO_2 recently removed from the atmosphere which works like a cycle of CO_2 emission ,Among algal fuels' advantages are that they can be grown with minimal impact on fresh water resources, and can be produced using wastewater, Algae could yield more than 2000 gallons of fuel per acre per year of production. Algae bases fuels have a high flash point, and are completely biodegradable and harmless to the environment if spilled.

1.3.4 Hydrogen fuel

A hydrogen fuel cell is an electrochemical cell that converts the potential energy from a fuel into electricity through an electrochemical reaction of hydrogen fuel with hydrogen fuel is a zero-emission fuel when burned with oxygen very small amounts of nitrogen dioxide and other gasses are emitted. It can be used in internal combustion engines to power vehicles. It has begun to be used in commercial fuel cell vehicles and has been used in fuel cell buses for many years. It is also used as a fuel for the propulsion of spacecraft and liquid propellant rockets

The problems of using hydrogen fuel in cars arise from the fact that hydrogen is difficult to store in either a high pressure tank or a cryogenic tank

1.3.5 Ammonia

Anhydrous ammonia (ammonia without water) can be a substitute for crude oil. It has the potential to make the hydrogen fuel a reality at an affordable cost. Ammonia can be used in internal combustion engines, gas turbines and planes, ammonia fuel cells are also being developed which can be used in electric vehicles. Ammonia exposure limits are very low, less than 50 parts per million in air, and the consequences of exposure includes light caustic burns. Although there are safety issues with ammonia, the issues are no more severe than those with gasoline and diesel fuel. Its high octane rating and low flame temperature allows the use of high compression ratios without a penalty of high NO_x emission. Since ammonia contains no carbon, its combustion cannot produce carbon dioxide, carbon monoxide and hydrocarbons. Compared to hydrogen as a fuel, ammonia is much more energy efficient, and could be produced, stored, and delivered at a much lower cost than hydrogen.

1.3.6 Biodiesel

Bio-diesel is a clean diesel substitute fuel produced from animal fats or vegetable oils and oils that come from plants such as soybean, sunflowers, corn, olive, peanut, palm, coconut, safflower etc. These oils are filtered from their hydrocarbons and then combined with alcohol like methanol or ethanol to form methyl esters (Biodiesel). These methyl esters can either be mixed with pure diesel to make various proportions or used alone. Bio diesel can be used in diesel engines without any modifications; bio-diesel will release a

smaller number of pollutants (carbon monoxide particulates and hydrocarbons) than conventional diesel because bio-diesel burns both cleanly and more efficiently.

1.4 Benefits of biodiesel

Biodiesel has many environmentally beneficial properties. The main benefit of biodiesel is that it is carbon neutral i.e. the fuel produces no net output of carbon in the form of carbon dioxide (CO_2). This effect occurs because when the oil crop grows it absorbs the same amount of CO_2 as is released when the fuel is combusted. Biodiesel is completely biodegradable and completely non-toxic, Biodiesel has a higher flash point than fossil diesel and so is safer in the event of a crash, Biodiesel reduces emission of CO_2 by 15% and using biodiesel instead of petroleum diesel can reduce greenhouse gases up to 78%, Vehicles that run on biodiesel achieve 30% fuel economy than petroleum based diesel engines, Biodiesel can be locally produced which decrease the dependence on imports for diesel and improve the economy of a country It can be distributed through existing diesel fuel pumps, which is another biodiesel fuel advantage over other alternative fuels.

Most biodiesel produced at present is produced from waste vegetable oil sourced from restaurants, chip shops, industrial food producers, Though oil straight from the agricultural industry represents the greatest potential source it is not being produced because the raw oil is too expensive. After the cost of converting it to biodiesel (cost of methanol, catalysts) has been added on it is simply too expensive to compete with fossil diesel. Waste vegetable oil which is often dumped citing of no use can often be sourced for free from restaurant and can be made into biodiesel easily.

1.5 Oils for biodiesel production

We can make biodiesel from any plant or animal derived oils, but the oils suitable for production of biodiesel must be cheaper, easily available and less viscous because if the cost biodiesel is more than standard diesel it will not be a better alternative for diesel. The list of plant oils that can be converted into biodiesel are shown below:

1.5.1 Edible oil

Edible oils are most often plant extracted oils, Plant produced edible oils consist of carboxylic acids with long hydrocarbon chains compared to petroleum-based oils which lack the carboxyl group on the end.

Vegetable oils are triglycerides extracted from oil seeds and fruits such as olive, soy, rapeseed, sunflower, palm, cocoa and peanuts. These seed extracts are used in the food and feed industry as ingredient or as cooking oils. Almost every edible plant oils can be converted into biodiesel

Most of these plant oils need to be refined through chemicals agents i.e. bases like NaOH and acids like sulphuric acid or via physical processes (high temperature, high pressure, cold pressing or solvent extraction). Refined oils have usually soft tastes and a clear and transparent presentation.

Table 1.3 Edible oils

S No.	Commonly used edible oil	Nut oils	Oils from melon and citrus oils
1	Coconut oil	Almond oil	Bitter gourd oil
2	Cottonseed oil	Beech nut oil	Bottle gourd oil
3	Olive oil	Brazil nut oil	Buffalo gourd oil
4	Palm oil	Cashew oil	Egusi seed oil
5	Peanut oil	Hazelnut oil	Butternut squash seed oil
6	Rapeseed oil	Macadamia oil	Pumpkin seed oil
7	Safflower oil	Pumpkin seed oil	Watermelon seed oil
8	Sesame oil	Pecan oil	Grapefruit seed oil
9	Soyabean oil	Pine nut oil	Lemon oil
10	Sunflower oil	Pistachio oil	Orange oil
11	Corn oil	Walnut oil	

1.5.2 Non edible oils

Oils which cannot be added in food are called as Non edible oils such as engine oils, grease and lubricants and seed oils etc.

A large number of plants produce non edible oils such as Neem, Rubber seed, mahua etc. Extracting oils from edible for production of biodiesel can increase their demand and can lead to deforestation for their plantation to overcome this problem oils are extracted from plants that are cultivated for multipurpose usage not solely for producing oil-based biofuel (ex Neem, Rubber).

Few examples of Non edible oils are Copaiba, Jatropha oil, Jojoba oil, Nahor oil, Petroleum nut oil, Pongamia oil, Dammar oil, Linseed oil, Poppyseed oil, Stillingia oil, Tung oil and Vernonia oil.

Out of all edible and non-edible oils palm oil is the king of all oil plants because of its high production and high consumption rate.

1.6 Palm oil

Palm oil is produced from the fruit of the oil palm (Elaeis guineensis).Palm oil has been in use for more than thousand years the main plantation areas are in Indonesia and Malaysia. Palm oil is the most consumed edible oil in India with a share of 40 per cent, followed distantly by soya bean and mustard oil. However, domestic production of palm oil is limited. Nearly 95 per cent of palm oil consumed is imported. Palm oil is imported from the world's top two producers, Indonesia and Malaysia. India is the largest consumer of palm oil

A large amount of waste palm oil will be generated which is generally dumped and it's of no use, this waste cooking palm oil can be converted into Bio-diesel. Biofuels from palm oil are taking on renewed global importance as countries seek to substitute the soaring price of conventional oil and also cut hazardous emissions. Palm oil's emergence in the market comes quite some time after the introduction of ethanol, additives made from sugarcane.

1.7 Waste cooking oils

Around the globe 198 million metric tons of edible oil is consumed for cooking and other purposes and the consumption is increasing and expected to cross 200 million by the end 2019 India is the world's biggest vegetable oil importer and primarily imports palm oil from Indonesia and Malaysia. More than 70% of India's edible oil demand is met from imports. India's vegetable oil imports will rise to 25 million tonne in 2030 from 15.5 million tonne in 2017 due to increasing demand and it is estimated that it grows with a CAGR of 3% annually.

Increasing income, urbanization, changing food habits and growing consumption of processed foods are the indicators of future consumption growth of edible oil in the country The country's vegetable oil consumption was at 23 million tonne in 2017, market for Palm oil, soy oil and sunflower oil are expected to increase further in the future[W_{11}]. Indore-based GGN International, forecasts total edible oil production in the country was 7.66 million tonnes for the oil year (November–October) 2017-18, compared to 7.05 million tonnes in the previous year. With an increase in opening stock of a record 2.42 million tonnes, India's edible oil availability from domestic sources is estimated at a record high of over 10 million tonnes.

The Food Safety and Standards Authority of India (FSSAI) has taken action against the reuse of cooking oil and has asked restaurants and Food Business Operators (FBO) to dispose of the used cooking oil after using it for three times for

cooking. Polar Compounds that are formed in the oil after cooking have adverse effects on health and must be minimized, it has taken an initiative that polar compound beyond the limit of 25 per cent then the vegetable oil becomes unfit for use [W12]. With increasing use of edible oils and implementation of these rules by FSSAI a lot of waste cooking oil which is unfit for further use will be generated this waste cooking oil has to be dumped in oceans which is harmful and leads to pollution. With a lot of waste cooking oil generally, big food businesses involved in the manufacturing of fried foods dispose of their used cooking oil for industrial purposes like soap manufacturing still there will be a lot of waste cooking oil left out, this waste cooking oil can be used in the production of biodiesel

Annually, about 23-million tonnes cooking oil is consumed in India. There is potential to recover and use about 3 million tonnes of this for production of biodiesel, this 3 million tonnes can contribute to the scarcity of crude oil and reduce harmful emissions.

CHAPTER-II

LITERATURE REVIEW

The world's energy challenge may not abate soon due to increasing population and urbanization, and a lack of positive policies. However, efforts through the introduction of renewable energy, such as biofuel, are receiving attention via intensive research. The most promising biofuel touted as an alternative to the conventional petro-diesel or fossil fuel is biodiesel. Biodiesel is the mono-alkyl (methyl, ethyl, or propyl) esters of long-chain fatty acids obtained from vegetable oils, microalgae oils, waste oils and animal fats. Transesterification of oil with alcohol in the presence of a catalyst remains the most commonly used method for the production of biodiesel.

The most common way to produce biodiesel is transesterification. In this reaction, triglycerides, as the main components of vegetable oils, react with an alcohol to produce fatty acid mono-alkyl esters and glycerol as a by-product. Methanol is the most commonly used alcohol because of its low price compared to other alcohols. In this case, the reaction is referred to as methanolysis. Generally, this reaction is catalyzed by a basic or an acid catalyst. The alkali catalysts are the most commonly used, because they make the process faster and the reaction conditions are more moderated. The stoichiometry of methanolysis reaction requires 3 moles of methanol and 1 mole of triglyceride to give 3 moles of fatty acid methyl ester (FAME) and 1 mole of glycerol. This is the general transesterification reaction, because it consists of a number of consecutive reversible reactions. The first step is the conversion of triglycerides to diglycerides, which is followed by the conversion of diglycerides to monoglycerides and of monoglycerides to glycerol, yielding one methyl ester molecule from each glyceride at each step, (Murat Kılıç et al. 2013).

There are many parameters affecting the transesterification reaction. The ones known as greatly influencing the reaction are: temperature, methanol/oil molar ratio, catalyst type and amount of catalyst. The optimization of the production process should take into account such parameters. Considering homogeneous alkali catalytic systems:

(i) Optimum temperature tends to be the one which is the closest to the boiling point of the alcohol used;

(ii) Excess alcohol is necessary to promote a good conversion (6:1 is considered as the best methanol/ oil molar ratio by many authors);

(iii) Catalysts normally used are sodium and potassium hydroxides, sodium and potassium methoxides as well as sodium and potassium carbonates;

(iv) Amount of catalyst used might vary from 0.2 to 2 (wt%).

Most of the studies on the transesterification for optimization of reaction parameters were based on changing one separate factor at a time (COST). However, reaction system was influenced simultaneously by more than one factor, due to this fact it is important to investigate the effects of interactions between reaction parameters. For instance, Cavalcante et al (2013), studied transesterification of castor oil with ethanol using a central composite rotatable design. They determined the optimum reaction conditions as oil: ethanol molar ratio of 1:11, catalyst amount of 1.75% KOH, and reaction time of 90 min and obtained ≈86.0% of biodiesel yield. Ramezani et al, Murat Kılıç et al (2013) used Taguchi method for optimization of castor oil transesterification (Murat Kılıç et al. 2013). Optimum reaction conditions were determined as methanol: oil molar ratio of 8:1, catalyst amount of 0.5% CH_3OK, reaction time of 120 min, reaction temperature of 65°C and mixing intensity of 400 rpm. According to the optimum reaction conditions they obtained the biodiesel yield as ≈87.0% by using the Taguchi method.

Silva et al. Murat Kılıç, et al. (2013) studied alkali ethanolysis of castor oil and optimized the process parameters by using factorial design adding central points and axial points as star points. They used bioethanol obtained from sugar cane as alcohol for transesterification reaction. 99.0% of ethyl ester and 93.0% of biodiesel yield were obtained by using optimum reaction conditions which were determined as ethanol: castor oil molar ratio of 16:1, catalyst amount of 1.0% C_2H_5ONa, reaction time of 30 min, and reaction temperature of 30°C with mechanic stirrer. Jeong and Park Murat Kılıç, Başak Burcu Uzun, et al (2013) optimized the biodiesel production from castor oil using response surface methodology. They determined the optimum process parameters as the reaction time of 40 min, reaction temperature of 35.5 °C, oil: methanol molar ratio of 1:8.24, and catalyst concentration of 1.45% KOH. 92.0% biodiesel yield was obtained by using the determined conditions.

For strengthening the quality control requirements of engine and equipment manufacturers, and allowing further companies to issue biodiesel engine warranties

for the use of biodiesel fuels, a series of biodiesel standards were issued in succession such as DIN 51606 (Germany), EN 14214 (Europe), ASTM D6751 (USA and Canada)52,53. Other countries have also established or are planning to adopt similar standards for the use of biodiesel as a motor fuel. Those standards have been periodically revised and updated.

Now, biodiesel blend fuel is available at many normal service stations across Europe and US. With the quick development of biodiesel industry, biodiesel is playing a more and more important role in globe primary energy. Recently a British plane (Thomson Airways Flight) carrying 232 passengers and crew members created aviation history by flying from Birmingham to Lanzarote using used cooking oil that was collected from the kitchens of hotels and restaurants which was processed through a special processing treatment. This was the first commercial bio-fuels flight ever run from a UK airport. One of the engines on the twin-engined Boeing 757 flight was operated on a 50 percent blend of "Hydro processed Esters and Fatty Acids" produced from used cooking oil, and 50 percent Jet A1 fuel.

Studies from the National Biodiesel Board (association representing the biodiesel industry in the United States) show that the biodiesel burning can emit, on average, 48% less carbon-monoxide; 47% less particulate material (absorbed by lungs) and 67% less hydro-carbon. A great economic challenge for the biodiesel commercialization is the high cost of pure vegetable oils, representing between 70% and 85% of the overall production cost of this energetic input. Among the sources of feed stock for the biodiesel production are rapeseed, palm, sunflower, and soybean oils. However, their availability to be used in the biodiesel production is limited due to competition with edible oil markets and consequent price increase victor et.al (2016). Moreover, biodiesel production from edible oil causes a negative impact in the environment, as it requires large amounts of available farm land. Therefore, this arch of new sources of biodiesel production is necessary, such as waste frying oil that can be acquired at no charge. Such waste is generated in several locations, mainly when we consider urban areas with high consumption refined vegetable oils. There are basically two possible destinations for waste frying oil: sewage systems, causing water pollution and encumber in its treatment, and processing equipment that can transform waste into a new product.

The main advantages of using biodiesel are its renewability better-quality exhaust gas emissions, its biodegradability and the organic carbon present in it is photosynthetic in origin. It does not contribute to emissions of carbon dioxide to the atmosphere, and consequently, to the greenhouse effect. Biodiesel contributes to a reduction of the main emissions resulted from petroleum derivatives, with considerable exception to nitrogen oxides (NO_x). Several studies have confirmed the increase of NO_x emissions. Its attenuation has been suggested with the use of additives and engine modifications. In the case of sulfur oxides (SO_x), as biodiesel do not contain sulfur; the emissions of these oxides are minimized with its use (A.kolakoti 2015 & 2017).

Literature on biodiesel application in internal combustion engines revels that:

S. L. Cheah (2004) stated that biodiesel has a major advantage over petroleum diesel, since; it is derived from renewable sources. It is a clean burning fuel that does not contribute to the increase of carbon dioxide, being environmentally friendly.

A. Aziz et al. (2005) mentioned that biodiesel is an oxygenate, sulfur-free and biodegradable fuel, and its content of oxygen helps improve its combustion efficiency. Therefore, fewer greenhouse gases such as carbon dioxide are released into the atmosphere. Biodiesel has positive performance attributes such as increased cetane, high fuel lubricity, and high oxygen content. Since, biodiesel is more lubricating fuel than diesel fuel, it increases engine life.

Y. Basiron (2004) concluded that the direct use of crude palm oil has been shown feasible in the Elsbett engine. However, a problem of clogging of the filter by impurities is observed, which can be eliminated by using processed liquid palm oil (PLPO) directly or in the blends with petroleum diesel to overcome this problem (Y Barson).

Nwafor et al. (2000) conducted test with rapeseed oil with advancing injection timings on four stroke IDI diesel engine. The test results showed that plant oil fuels exhibited longer ignition delay with slower burning rates. The test results also showed that each alternative fuel requires injection advance appropriate to its delay period. The delay period was noted to be influenced by the engine load, speed and the system temperature. At the engine speed of 2400 RPM, there seems to be a significant increase in brake thermal efficiency when running on rapeseed oil fuel with standard

injection timing. Mechanical efficiency was observed to be reduced with advanced timing compared to the standard timing test results at 2400 RPM. The engine ran smoothly with advance of 3.58^0 as compared to the standard timing. A further 1.58^0 advance tended to produce erratic behavior of the engine. There seems to be a significant reduction in CO and CO_2 emissions with advanced timing for the speeds tested. A moderate injection advance is recommended for operations at low engine speeds. The overall results indicate that vegetable oils exhibit longer combustion duration with moderate rates of pressure rise, unlike petroleum derived fuels.

Yousef et al. (2011) conducted an experimental study on the use of raw Algae oil and its methyl esters in an Indirect Injection (IDI) diesel engine. (Ricardo E6 engine) Effects of engine speed, engine load output, injection timing of the algae biofuel and engine compression ratio on the engine output torque, combustion noise (maximum pressure rise rate), maximum pressure and maximum heat release rate have been studied. It has been shown that the algae oil methyl ester's properties are similar to diesel fuel and its use has been successful in running the diesel engine smoothly using less methanol (10%) in producing algae oil methyl ester resulted in a better performance of the engine. However micro algae production and extraction of raw oil is more expensive than the seeds which give oil by merely squeezing them.

Kalam et al. (2011) experimental study carried out to evaluate emission and performance characteristics of a multi-cylinder IDI diesel engine operating on waste cooking oil such as 5% palm oil with 95% ordinary diesel fuel (P5) and 5% coconut oil with 95% ordinary diesel fuel (C5). Neat biodiesel (B0) was used for comparison purposes. The results show that there are reductions in brake power of 1.2% and 0.7% for P5 and C5 respectively compared with B0. In addition, reduction of exhaust emissions such as unburned Hydrocarbon (HC), smoke, Carbon monoxide (CO) and Nitrogen Oxides (NO_x) is achieved by the blended fuels.

Ali Turkcan and Canakci M (2011) found that IDI engines do not depend upon the fuel quality, have a lower Ignition Delay (ID) and combust faster than Direct Injection (DI) diesel engines. The combustion characteristics of IDI diesel engines are different from the DI diesel engines. IDI diesel engines hold a simple fuel injection system and lower injection pressure level because of higher air velocity and rapidly occurring air–fuel mixture formation in both combustion chambers of the IDI diesel engines. In addition, they do not depend upon the fuel quality and produce lower

exhaust emissions than the DI diesel engines. This advantage is being made use of in our present work.

K.Prasad Rao and B.V.Appa Rao (2014) conducted experiments on IDI engine fuelled with Mahua Methyl Ester (MME) along with Methanol additive blends with an attempt to reduce NOx, HC, CO and smoke emissions. Mahua methyl ester was used with additive methanol in different proportions such as 1%, 2%,3%, 4% and 5% and was tested at different loads in an IDI diesel engine. It is observed that a 3% additive in biodiesel performed better and could be a replacement for diesel fuel. The research on the IDI engine indicates lower emissions in the exhaust. Additive mixing further reduced the HC and CO emissions to a large extent. NOx emission was also reduced at higher loads, especially with the additive.CO_2 emission increased because of combustion improvement. Thermal efficiency and specific fuel consumption improved with the 3% additive in biodiesel.

Y.Ashok kumar reddy & B V Appa Rao (2014) experimental investigation is carried out on a four stroke single cylinder IDI engine to find out the performance and emission characteristics with the preheated Jatropha Methyl Ester (JME) with the viscosity 4.36 cSt. JME is preheated at 60,70,80,90 and 100^{0}C temperatures using online electronic preheating system. Experiments were done using diesel, biodiesel and biodiesel at different preheated temperatures and for different engine loading conditions keeping speed constant at 1500rpm. Improvement in performance and emission characteristics is obtained with biodiesel preheated to 60^{0}C and showed increase in break thermal efficiency over unheated biodiesel at 2.7kW load. Average smoke level is lesser than the unheated biodiesel with reduced CO& HC levels in exhaust.

Victor et al. (2014) conducted experiments on 4stroke IDI Diesel engine with conventional petro-diesel and biodiesel along with biodiesel and Isopropanol (C_3H_8O) as an additive in various percentages viz. 2%, 3%, 4% and 5% is injected. Biodiesel (Rice Bran Methyl Easter) is injected through the regular nozzle in a dual fuel mode. Isopropanol is injected into the air inlet at 3bar without sacrificing the volumetric efficiency. The main idea in introducing the additive is to dilute the combustion and thereby to reduce the combustion temperatures to limit the NOx formation. Higher latent of isopropanol and volatility of the additive has its effect in reducing the

combustion temperatures. Results envisage hike in thermal efficiency at maximum load with respect to diesel & biodiesel. HC reduction is observed as 5ppm at maximum load with respect to diesel fuel.CO reduction is nearly 14% with respect to diesel at maximum load. CO_2 has increased by 7.6% with respect to neat diesel. There is a reduction of 36.5% of NO with respect to diesel fuel. Reduction of 27.5 HSU is observed at maximum load with respect to diesel.

Naidu et al. (2015) Examines experimentally the performance and emissions of IDI engine fuelled with neat Palm Methyl Ester(PME) along with 1,4-Dioxane($C_4H_8O_2$) as an additive. Since 1, 4-Dioxane possesses higher latent heat [413kJ/kg] than that of diesel [280kJ/kg], its blending creates low temperature combustion and reduces the formation of NOx. The results show reduction in brake specific fuel consumption (BSFC) by 2.64 percent compared with neat PME, an increase of Brake thermal efficiency (BTE) by 9.89 percent, Exhaust gas temperature (EGT) and smoke readings are decreased by 4^0C for 3 percent. Engine emissions are decreased by 2ppm for HC, 40 percent for CO, 10.89 percent for CO_2 and 56ppm for NOx with 3 percent additive in biodiesel with respect to diesel fuel.

Raju et al. (2015) In this study, experimental investigations have been carried out to examine the combustion, exhaust gas emission and cylinder vibration characteristics of an indirect injection (IDI) diesel engine. The engine was operated with five fuel samples of Pongamia Methyl Ester (PME) with Isobutanol (IB) as an additive. Engine tests were performed at five different engine loads ranging from no load to maximum load at fixed engine speed 1500 rpm. The results obtained with the fuel samples were compared to those obtained for the neat biodiesel and conventional diesel as a baseline fuel. The exhaust gas emissions of the engine using 6 percent isobutanol in biodiesel were compared with conventional diesel fuel operation. The reduction percentages quantified are by 71 for HC, 0.02 for CO, 0.5 for CO_2 and 31 for NO. The Fast Fourier Transformation signatures recorded vertical on the cylinder head and foundation indicate uniform amplitude modulation revealing smoother combustion for the 6 percent of additive in biodiesel. The time waves recorded on the cylinder head envisage better combustion in the main combustion chamber and judicious combustion sharing in between chambers.

Jincheng Huang et al. (2014) Conducted experiments on direct and indirect injection combustion systems of a same model of two diesel engines fuelled with diesel and the blend of diesel and Chinese pistache biodiesel. The results show that the NOx emissions from the indirect injection combustion system (ICS) fuelled with diesel are reduced by around two thirds, compared to that from direct injection combustion system (DCS). Smoke emissions from the engine using ICS are all significantly lower than that of DCS.

Gomaa et al. (2010) investigated the engine performance and soot emission by using blended Jatropha biodiesel with different EGR rates. A CI engine that is water-cooled, turbocharged, using indirect injection system was used. Soot emission, NO_x, CO_2, Carbon monoxide (CO) were recorded and various engine performance parameters were also evaluated. On the basis of experimental data, it was found that blended biodiesel and EGR both can be used in compression ignition engine to simultaneously reduce NO_x and soot emissions. The Jatropha biodiesel together with 15%EGR was found to be useful in improving both of brake thermal efficiency, brake specific energy consumption and reduces exhaust emissions.

Shahabuddin et al. (2012) investigated the experimental data analysis of different parameter of a Turbocharged (IDI) Diesel engine, exhaust emissions while running with POME blended anti-corrosion additive as fuels. This investigation focused on the significant effects of additive on POME-Diesel blended fuels especially for B_2O +1% (20% biodiesel + 80% diesel + 1% additive). The additive used in this experiment is IRGANOR NPA (Product name) as a corrosion inhibitor for fuels. Fuel B20+1% produce lowest level of CO emissions which is on average of 0.141%, B_2O is 0.213% and OD is 0.296%. The difference between B20+1% and B20 shows the effect of additive in Biodiesel fuel especially with B20 fuel. The blended fuel with additive reduces friction between the cylinder wall and piston thus the heat loss is controlled in the cylinder which result in considerable reduction in NO_x. Over the entire speed range, B_2O +1% fuel produces an average of 95 ppm and fuel B_2O produces 123 ppm. Hence the 28 ppm reduction is due to the effect of 1% additive in B_2O fuel. It can be seen that additive mixed with biodiesel fuel produces comparatively lower level of HC as compared to OD fuel. This is mainly due to complete combustion in the combustion

process. But the corrosion aspect of eth elements in the engine is not being correctly defined.

Sahin et al. (2012) examines experimentally the effects of gasoline fumigation (GF) on performance and exhaust emissions of a turbocharged indirect injection diesel engine. Results showed that effective power generally reduced and effective efficiency increased. Brake specific fuel consumption reduced and its decrement ratios are approximately at the levels of 5%. GF becomes economic for this engine and averagely 5% reduction in fuel cost was attained. NO_x concentration decreased approximately at the levels of (5–10%). Smoke index 'K' reduced up to (8–12) % GF and after this it began to increase. Its maximum reduction ratio was 20% for 8% GF at 2500 rpm.

T. Leevijit et al. (2012) Conducted experiments on indirect injection (IDI)-turbo automobile diesel engine operated with diesel and blends of degummed-de acidified mixed crude palm oil in diesel at portions of 20, 30, and 40 vol.% are examined and compared at various loads and speeds. Results show that all blends produce the same maximum brake torque and power. A higher blending portion results in a little higher brake specific fuel consumption (+4.3% to +7.6%), a slightly lower brake thermal efficiency (−3.0% to −5.2%), a slightly lower exhaust gas temperature (2.7% to 3.4%), and a significantly lower amount of black smoke (30% to 45%). The level of carbon monoxide from the 20 vol.% blend is significantly lower (70%), and the levels of nitrogen oxides from all blends are little higher. Anyhow, the use of crude oil is not being encouraged because of the reason blending becomes increasingly difficult.

Sukumar Puhan et al. (2010) Conducted experiments on a constant speed diesel engine, which develops 4.4 kW of power, was run with biodiesels and it performance was compared with diesel fuel. The results show that Linsee oil methyl ester with high linolenic (unsaturated fatty acid ester) does not suit best for diesel engine due to high oxides of nitrogen emission and low thermal efficiency.

Despite the benefits of biodiesel (viz. renewability, biodegradability, environmental friendliness, intrinsic lubricity, higher flash point, and miscibility with petro-diesel), the greatest challenge militating against its full acceptance remains its higher cost than the petro-diesel. This problem has been addressed in many ways for

example the use of waste cooking oils and non-edible oils as sources of cheap feedstock(A Kolakoti et al.2015).

Another plausible way of dealing with the high cost of biodiesel is by modeling and optimizing the processes involved using tools such as response surface methodology (RSM), Taguchi, Definitive Screening Design (DSD), Factorial Design and Mixture design etc.

R. Satish Kumar et al. (2014) studied the transesterification process parameters for the production of Manilkara Zapota Methyl Ester (MZME) i.e. Molar ratio of methanol to oil, time of reaction, temperature of reaction, and concentration of catalyst by using Taguchi experimental design with the optimization of the above mentioned four process parameters of transesterification. The physicochemical properties and fatty acid methyl ester concentrations were experimentally analyzed. Results shows that 50^0C temperature of reaction, 90 min of time of reaction, 6:1 ratio of methanol to oil and 1 wt% of concentration of catalyst are the optimal process parameters for maximum yield, also the study revealed that out of the four parameters considered, methanol to oil molar ratio was most effective in controlling the optimal biodiesel production. The optimal conditions yielded 94.83% of biodiesel. The biodiesel MZME produced with the optimized process parameters meets the global standards for biodiesel EN 14214 and hence could be considered as a suitable substitute for fossil diesel in unmodified diesel engine applications.

Murat Kılıç et al (2013) studied the optimization of biodiesel production from castor oil using full factorial design. The biodiesel production was carried out in a batch laboratory scale reactor by alkaline-catalyzed transesterification process. Effects of temperature, methanol/oil molar ratio and catalyst concentration were optimized according to the 2^3 full factorial central composite design (CCD). For determining the influence of purification methods on biodiesel yield, different purification methods were applied to the product after transesterification reaction. Second order model was obtained to predict biodiesel yield as a function of these variables. According to the experimental results this process gave an average yield of biodiesel more than 90%.

Prafulla D.Patil et al. (2009) found out that non-edible vegetable oils such as Jatropha curcas and Pongamia glabra(karanja) and edible oils such as corn and canola were found to be good viable sources for producing biodiesel. Biodiesel production from different edible and non-edible vegetable oils was compared in order to optimize the biodiesel production process. The analysis of different oil properties,

fuel properties and process parameter optimization of non-edible and edible vegetable oils were investigated in detail.

Shashikant et al (2006) used central composite rotatable design to study the effect of methanol quantity, acid concentration and reaction time on the reduction of free fatty acids content of mahua oil during its pretreatment for making biodiesel. All the three variables significantly affected the acid value of the product, methanol being the most effective followed by reaction time and acid catalyst concentration. Using response surface methodology, a quadratic polynomial equation was obtained for acid value by multiple regression analysis.

Alok Kumar Tiwari (2007) used Response surface methodology (RSM) based on central composite rotatable design (CCRD) to optimize the three important reaction variables—methanol quantity , acid concentration and reaction time for reduction of free fatty acid (FFA) content of the oil to around 1% as compared to methanol quantity and reaction time and for carrying out transesterification of the pretreated oil. Using RSM, quadratic polynomial equations were obtained for predicting acid value and transesterification. Verification experiments confirmed the validity of both the predicted models.

XuanWu Dennis et al (2011) performed optimization based on sixteen well-planned orthogonal experiments (OA_{16} matrix). Four main process conditions in the transesterification reaction for obtaining the maximum biodiesel production yield (i.e. methanol quantity, reaction time, reaction temperature and catalyst concentration) were investigated. It was found that the order of significant factors for biodiesel production was CC, RTi, RT and MR. Based on the results of the range analysis and analysis of variance (ANOVA), the maximum biodiesel yield was found at a molar ratio of methanol to oil of 8:1, a reaction time of 70 min, a reaction temperature of 50^0C, and a catalyst concentration of 1 wt.%. The product and FAME yields of biodiesel under optimal conditions reached 95.8% and 98.4%, respectively. The properties of the optimized biodiesel, including density, kinematic viscosity, acid value, etc., were determined and compared with those produced from other oil feed stocks. The optimized biodiesel from camelina oil meets the relevant ASTM D6571 and EN 14214 biodiesel standards and can be used as a qualified fuel for diesel engines.

In order to achieve the maximum biodiesel yield with limited raw material cost and time, optimization techniques are most suitable (Wu et al., 1999; Vicente et

al., 2007; Cavalcante et al. 2010). The main aim of optimization is to produce the maximum possible output without having to depend on experimental trial and error methods which saves a lot of time and cost, Optimization techniques also reveal crucial information about the factors which influence biodiesel yield and individual contribution of each factor to the biodiesel yield, Optimization techniques also generate graphs and plot for better understanding of the generated model and whether the provided data is correct and its feasibility. Due to wide range of advantages and efficiency optimization techniques are most suitable for improvisation of biodiesel yield.

CHAPTER-III

BIODIESEL PRODUCTION PROCESS

3.1 Introduction

Using the vegetable oils directly into the engine will lead to many problems like fuel injector clogging, fuel filter clogging, poor atomization and incomplete burning due to high density, high viscosity and poor volatility. To reduced the high viscosity problem there are different methods that are available. Some of them were:

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

From the above processes transesterification is widely accepted method for reducing the viscosity, density of the oil. (Barnwal and Sharma 2005).

3.2 Pyrolysis

The pyrolysis refers to a change of chemical state by the application of thermal energy in the absence of air. In this process the thermally decomposed liquid will tend to covert as diesel fuels. The obtained fuel from the pyrolysis, have low viscosity, flash point, pour point compared to diesel fuel but will have similar calorific values. Generally the pyrolyzed vegetable oil contains limited amount of water, sulfur, copper corrosion and other quantities at acceptable level. But the ash, carbon residual, pour point are at unacceptable level. The pyrolysis can be implemented depending upon the existing conditions.

3.3 Micro-emulsification

The micro-emulsification is one of the most promising techniques for reducing viscosity of the vegetable oil. These Micro-emulsions are transparent, thermodynamically stable colloidal dispersion droplets with diameter from 100 to 1000 Å (Angstrom). Microemulsion can be made of vegetable oils with an ester and dispersant, or of vegetable oils, and alcohol and a surfactant and a cetane improver, with or without diesel fuels. All Micro-emulsions with butanol, hexanol and octanol reached the maximum viscosity standard for diesel fuel. The 2-octanol was found to be an important amphiphile in the micellar solubilization of methanol in triolein and soybean oil.

3.4 Dilution

The dilution of vegetable oils can be made using diesel fuels, ethanol and solvent. The dilution process reduces the viscosity and density of vegetable oils. Upon addition of 4% ethanol to diesel fuel increases the brake thermal efficiency, brake torque and brake power, while decreasing the brake specific fuel consumption. As the boiling point of ethanol is low compared to diesel fuel, it helps in development of the combustion process through an unburned blend spray.

3.5 Transesterification

Transesterification is one of the most appropriate methods for the production of biodiesel from the oils and fats by following any one of the process:

(a) Catalytic Transesterification.

(b) Supercritical Methanol Transesterification.

3.5.1 Catalytic transesterification

The Catalytic Transesterification is a process in which the triglycerides of fat or oil react with an alcohol with any of the catalysts to produce ester and glycerol. A triglyceride has a glycerin molecule with three long chain fatty acids attached at its base. In organic chemistry, transesterification is the process of exchanging the organic group R″ of an ester with the organic group R′ of an alcohol. These reactions are often catalyzed by the addition of an acid or base catalyst. The reaction can also be accomplished with the help of enzymes (biocatalysts) particularly lipases. In the transesterification mechanism, the carbonyl carbon of the starting ester react to give a tetrahedral intermediate, which either reverts to the starting material, or proceeds to the transesterified product ($RCOOR^2$). The various species exist in equilibrium, and the product distribution depends on the relative energies of the reactant and product as shown in Fig 3.1.

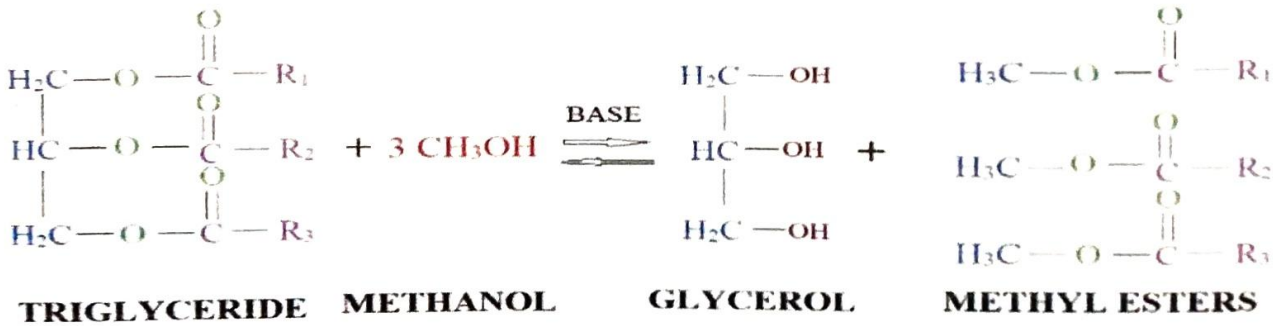

Figure 3.1 Transesterification reaction

The oil or fat can be characterized by the nature of fatty acids attached to the glycerin, which will affect the physicochemical properties of the biodiesel. The transesterification reaction is the separation of ester and glycerol when the triglyceride reacts with alcohol. Thus obtained glycerin due to its heavier density settles down and can be separated and used for various applications in many industries like soaps production, pharmaceuticals, cosmetics etc.

3.5.2 Acid catalyzed transesterification

The acid catalyzed transesterification process involves the reaction of triglyceride with an alcohol in the presence of acid catalyst, preferably sulphonic and sulphuric acids to produce esters (biodiesel) and glycerol. These catalysts results in very high yields in alkyl esters, but the reactions are slow, requiring, typically, temperatures above 100°C. The acid-catalyzed transesterification is performed in the absence of water, so as to avoid the formation of carboxylic acids which results in low biodiesel yield.

3.5.3 Alkaline catalyzed transesterification

The alkaline catalyzed transesterification process involves the reaction of triglyceride (fat/ oil) with an alcohol in the presence of an alkaline catalyst such as alkaline metal alkoxides and hydroxides, sodium or potassium carbonates to produce esters and glycerol. The alkaline catalyzed transesterification of vegetable oil proceeds faster than the acid catalyzed reaction. So the alkaline catalysts are less corrosive than acidic compounds. The industrial processes usually choose alkaline catalysts, such as alkaline metal alkoxides and hydroxides, sodium or potassium carbonates. But due to the presence of water and high amount of free acid leads to saponification of oil and so the reaction will be incomplete with the formation of emulsion and difficulty in separation of glycerol.

3.5.4 Lipase catalyzed transesterification

The lipase catalyzed transesterification process involves the reaction of triglyceride (fat/oil) with an alcohol in the presence of lipase enzyme as a catalyst to form esters and glycerol. This type of transesterification process eliminates the difficulty in recovery of glycerol and also in elimination of catalyst and soap. This process is environment friendly, attractive although the reaction time and yields are poor when compared to alkaline catalyzed reaction system

3.5.5 Super critical transesterification

The above mentioned transesterification processes comprises of two difficulties, i.e. The simple transesterification processes discussed above are confronted with two problems, i.e. the processes are relatively time consuming and needs separations of the catalyst and saponified impurities. Due to the phase separation of vegetable oil/alcohol mixture, the reaction time increases which can be eliminated by stirring vigorously. In the supercritical method of transesterification these problems are eliminated due to the presence of single phase formation in dielectric constant of alcohol in supercritical state that is at 340°C and 43 MPa. This reduces the reaction time drastically to 2-4 minutes. Further, the purification of biodiesel becomes easy because of absence of catalyst. The process becomes trouble free and environmental friendly (Demirbas, 2005).

3.6 Catalyst availability

Catalysts are the main source to speed up the chemical reaction by minimizing the activation energy, which is primary for initiating the reaction. These catalysts are divided into two types of systems namely, heterogeneous and homogeneous systems.

The heterogeneous catalyst system includes:

- Enzymes
- Titanium silicates
- Alkaline-earth metal compounds
- Anion exchange resins
- Guanidines heterogenized on organic polymers

Heterogeneous base catalysts like Mg/Al based sulphate and nitrate compound can be used, because of Heterogeneous catalysts' recycling is very easy one and they can be used for about 5 times and helps in increasing the pH value of biodiesel. At present, heterogeneous catalysts are not that emerging because of its high cost and its inability to accomplish the degree of chemical reaction to ASTM standards (Gerpen et al 2004).

The homogeneous system of catalysts consists of acids and bases. In general the acid catalysts are not preferred than base catalysts because of slower reaction to form fatty acids methyl ester from transesterification process. The acid catalysts are used for the pre treatment of high free fatty acid composition feed stocks, where these fatty acids are converted to fatty acid ester (Gerpen et al 2004). Although different

kinds of base and acid catalysts are available for transesterification processes, most of the commercial biodiesel production firms use the base catalyst.

The most common alkali catalysts are

- Sodium hydroxide (NaOH)
- Potassium hydroxide (KOH)
- Sodium Methoxide (CH_3ONa)
- Potassium Methoxide (CH_3OK)

Methoxide ion is the most preferable catalyst for the production of biodiesel. These Methoxide ions are processed by various methods. Methoxide ion has been described as the preferred catalyst for the transesterification process of biodiesel production. Methoxide ions can be obtained via several different methods. The very traditional method of preparing catalyst solution is by mixing either sodium hydroxide or potassium with methanol, within the biodiesel production plant.

In general both the homogeneous and heterogeneous catalysts are used for biodiesel production from waste cooking palm oil.

3.7 Materials

The biodiesel production involves various chemicals for different treatments and also many apparatus which will be used for different purposes.

- The different apparatus required for the production of biodiesel are an electric heater with magnetic stirring support, 1000 ml beaker, 1000 ml conical flask with cork stopper fitted with thermometer (0-150^{0}C range), magnetic stirrer, measuring jar, weighing machine, 1000ml separating funnel, stand for separating funnel, strainer and funnel.
- The chemicals required for the production of biodiesel are methanol, sodium hydroxide pellets, sulfuric acid.

3.8 Production of biodiesel by transesterification process

After acquiring the required apparatus and chemicals the biodiesel can be prepared by following the steps given below.

- The fried palm oil was taken and filtered using strainer to remove the unwanted particles.

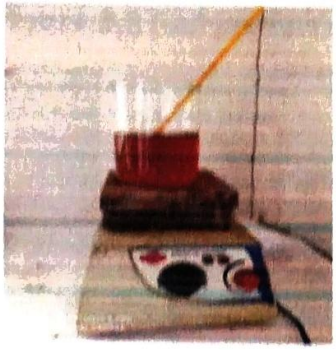

Figure 3.2 Heating of oil.

Figure 3.3 Filtered oil.

Figure 3.4 Setup for acid treatment.

Figure 3.5 Glycerin formation after acid treatment.

Figure 3.6 Glycerin formation after base treatment.

Figure 3.7 Clear separation of ester and water.

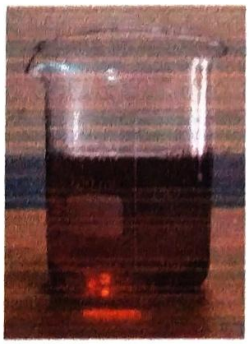

Figure 3.8 End product of transesterification process (Biodiesel).

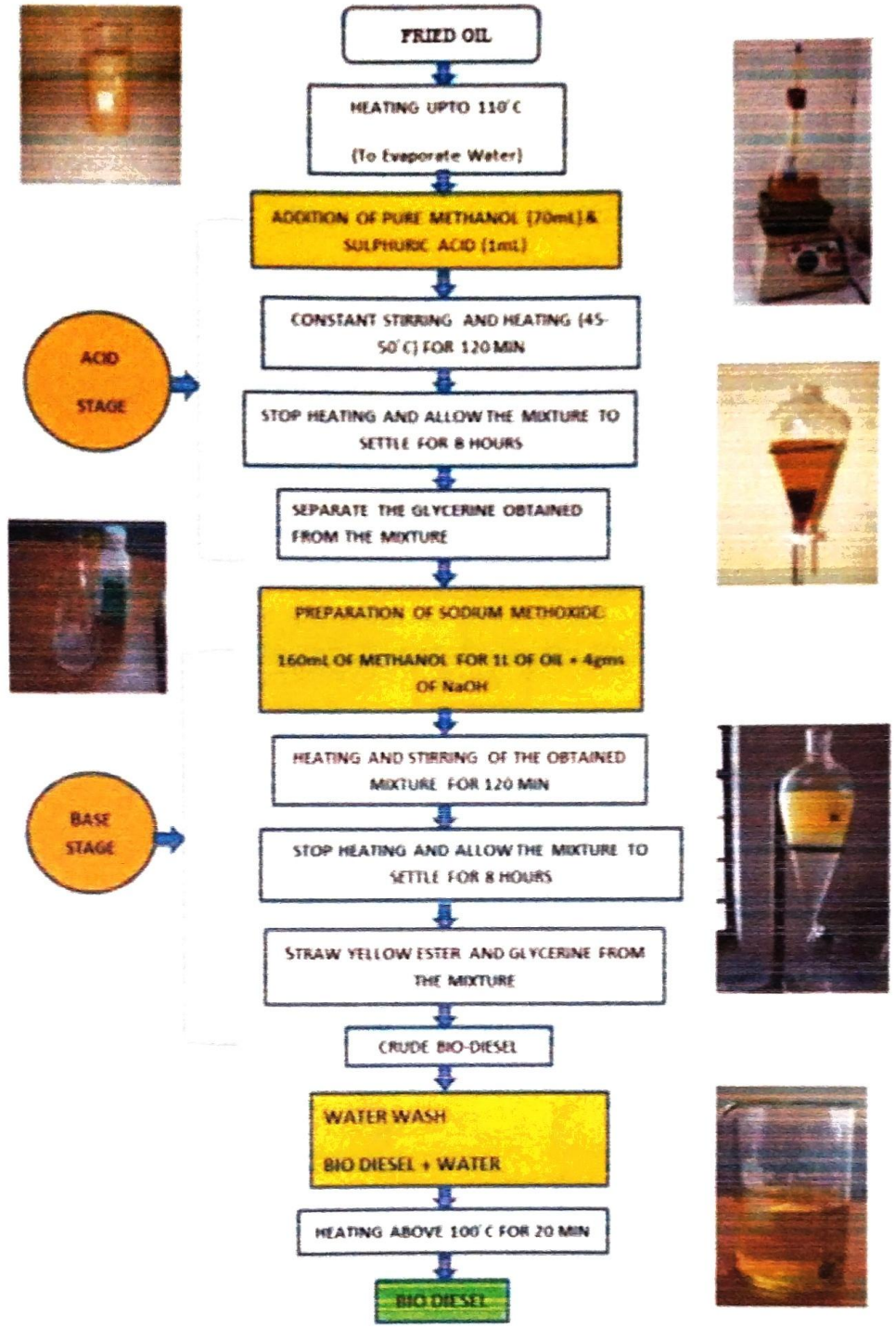

Figure 3.9 Flow chart of biodiesel production

3.9 Production of soap from glycerin

The byproduct glycerin can be converted into soap by performing some chemical processes; these can be performed by following the steps given below.

- The byproduct glycerin was heated to 70^0C to remove any excess methanol and until it reaches molten state.

- Sodium hydroxide (NaOH) was mixed with hot water and stirred.
- The NaOH mixture was poured into liquid glycerin and stirred at slow speed.
- Essential oil and liquid dye were added to the mixture for fragrance and color.
- This molten mixture was poured into mould and allowed to cool.
- After the molten mixture solidifies soap is ready as shown in the fig 3.10.

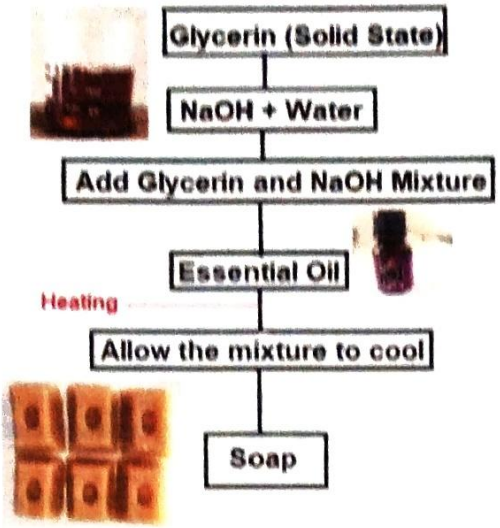

Figure 3.10 Flow chart of soap production

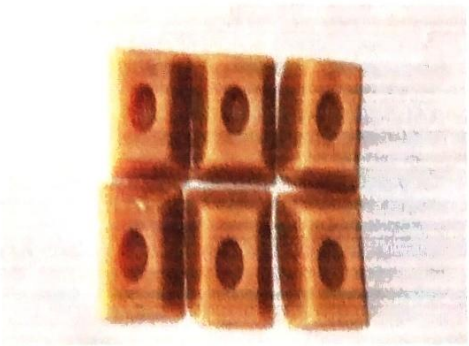

Figure 3.11 Produced soap

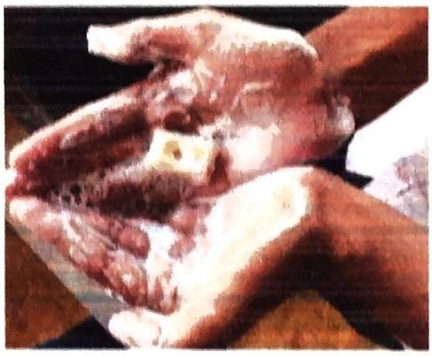

Figure 3.12 Lather produced by soap

CHAPTER-IV

OPTIMIZATION OF BIODIESEL

Biodiesel produced from transesterification process may not give the highest possible yield, to improve the output yield produced from transesterification process optimization techniques are used which indicate the factors which contribute to the yield and their required proportions to be added. There are number of statistical software like Rstudio, Number analytics and IBM SPSS statistics which are available in the market which can be used for optimization, of all these statistical software Minitab is easy to learn, affordable and has a student friendly interface.

4.1 Minitab 18

Minitab is a statistics package developed at the Pennsylvania State University by researchers Barbara F. Ryan, Thomas A. Ryan, Jr., and Brian L. Joiner in 1972. Minitab 18 Statistical Software provides the tools you need to analyze data and predict factors affecting the data. Minitab statistical software is also used for quality analysis and improvement of products & services. Huge number of companies from manufacturing to service industries from Pharmacy to healthcare industries uses Minitab for their data analysis Minitab provides a simple, effective way to input the statistical data, manipulate that data and lets you know if entered data is correct and also helps you change the data, identify trends and patterns, and then extrapolate answers to the current data Minitab can generate various types' graphs and plots which can help variations and data trends to be understood easily.

4.2 Optimization using Minitab 18

Optimization helps you identify the combination of variable settings that jointly optimize a single response or a set of responses. This is useful when you need to evaluate the impact of multiple variables on a response. Optimization in Minitab is done using response optimizer in DOE. Response optimization helps to identify the combination of variables that jointly optimize a response. This is useful when you need to evaluate the impact of multiple variables on a response. Minitab calculates an individual desirability for each response and weights each by the importance you assigned to it. An optimal solution occurs where composite desirability obtains its maximum. Using an optimization plot, you can adjust the variable settings and determine how the changes affect the response.

4.3 Optimization Techniques in Minitab 18

Optimization techniques in Minitab 18 are performed through design of experiments (DOE) in Minitab 18. The objective of a design of experiments (DOE) is

to optimize the value of a response variable by changing the levels, of the factors that affect the response. The DOE methodology ensures that all factors and their interactions are systematically investigated. Therefore, information obtained from a DOE analysis is much more reliable and complete. There are five types of optimization techniques in DOE, definitive screening design is a new technique added in DOE in Minitab 18.

4.3.1 Screening Design

Definitive Screening Design in DOE identifies the most important factors early in the experimentation process. Definitive screening designs can fit 2–48 factors. Categorical factors can have only 2 levels. Continuous factors have 3 levels in the designs. Definitive screening design is used when you are starting with 6 or more factors and want to identify the most critical factors to study in subsequent experiments. Definitive screening is useful when you want to consider both linear and quadratic terms when you identify the most critical factors. There are two designs in Screening

I. 2-level fractional factorial designs

II. Plackett-Burman designs

4.3.2 Factorial design

Full Factorial Design in DOE can study factors that can have any number of levels. You can use a general full factorial design to create full resolution, 2-level designs for 8 or more factors. A factorial design lets you study of the effects that several factors can have on a response. When conducting an experiment, varying the levels of all factors at the same time instead of one at a time lets you study the interactions between the factors and contribution of each factor to the response. The factorial designs are:

I. 2-level full factorial designs that contain only 2-level factors.

II. General full factorial designs that contain factors with more than two levels.

The number of runs necessary for a 2-level full factorial design is 2^k where k is the number of factors. As the number of factors in 2-level factorial design increases, the number of runs necessary to do a full factorial design also increases.

4.3.3 Response Surface Design

Response Surface Design in DOE is used to create a designed experiment for 2–10 factors to model curvature in your data and identify the factor settings that optimize the response. Response surface design methodology is often used to refine models after you have determined important factors using screening designs or factorial designs. In the response surface Central composite designs enables you to build on factorial or fractional factorial design by adding center points, augmented with axial points. There are two main types of response surface designs:

I. Central Composite designs

II. Box-Behnken designs

4.3.4 Mixture Design

Mixture Design in DOE is used to create a designed experiment when the response depends on the relative proportions of mixture components, such as the ingredients in making tea. Mixture designs are useful because many product design and development activities in industrial situations involve formulations or mixtures. In these situations, the response is a function of the proportions of the different ingredients in the mixture, for example the ingredients in making tea i.e. tea leaves, milk, sugar, response is assumed to only depend on the proportions of these three components in the mixture. Minitab provides three design types, including simplex centroid, simplex lattice, and extreme vertices designs. You can also include process variables or an amount variable in the design

4.3.5 Taguchi Design

Taguchi based optimization technique makes use of specially constructed tables known as "orthogonal arrays" to perform computational analysis to achieve the targeted value. Taguchi Design is used to create a robust parameter design to identify controllable factors in your process that can minimize response variation and make your product insensitive to changes in noise factors. Taguchi method contains system design, parameter design, and tolerance design procedures to achieve a robust process and result for the best product quality . Taguchi designs include control factors, which are factors in the process that you can control, and noise factors, which are factors that you cannot control when the process is in use

4.4 Optimization Using Definitive Screening

In this research biodiesel was obtained using transesterification process and definitive screening design was used for optimization of biodiesel yield. A two-level fractional factorial design with three factors was chosen. The three contributing factors were molar ratio (A), catalyst concentration (B) and reaction temperature (C) and their ranges are shown in the table 4.1(Murat Kılıç et al. 2013).

Table 4.1 Factors and their ranges

S. No	Variable	Range Minimum	Range Maximum
1	Reaction Temperature (^{0}C)	27	55
2	Catalyst Concentration (grams)	3.50	5.0
3	Molar Ratio	4:1	6:1

For the above three factors and their ranges, an experimental matrix containing different combinations of maximum, minimum and median values was generated for the factors and their ranges containing 13 runs as shown in table 4.2.

Table.4.2 Matrix of factors and their response yield

Run No	Reaction Temperature	Catalyst Concentration	Molar Ratio	Yield (%)
1	55	5	5:1	93.24
2	27	4.25	4:1	84.97
3	41	3.5	4:1	84.85
4	41	4.25	5:1	88.32
5	55	3.5	4:1	86.55
6	27	3.5	5:1	86.72
7	41	5	6:1	93.95
8	27	5	6:1	93.15
9	27	3.5	6:1	90.10
10	27	5	4:1	86.79
11	55	5	4:1	90.61
12	55	3.5	6:1	93.82
13	55	4.25	6:1	94.40

The yield of the biodiesel is the amount of output biodiesel obtained after transesterification process and separation of byproducts and other chemicals through water wash, biodiesel yield can be calculated from the Eq.4.1.

$$\text{Yield (\%)} = \frac{\text{Weight of Biodiesel}}{\text{Weight of Raw oil}} \quad (4.1)$$

From the generated experimental matrix containing 13 runs of different combinations of factors, 13 experiments of producing biodiesel from transesterification process were conducted for the given ranges and the biodiesel yield was obtained for each experiment. A new row was added into the experimental matrix under the name yield and obtained biodiesel yield for the 13 experiments were filled accordingly.

The definitive screening design generated matrix was analyzed using ANOVA, Analysis of variance (ANOVA) is based on an approach, in which the procedure uses a variance to predict whether the means are differentiable. It also measures the importance of contributing factors by comparing the response (Yield) variable at different levels. Based on the experimental yield matrix the response yield was analyzed and the ANOVA tables.4.3 & 4.4 were generated for the contributing factors.

Table 4.3. Analysis of Variance

S.No	Source	DF	Adj SS	Adj MS	F- Value	P-Value
1	Model	3	153.333	51.111	111.41	0.000
2	Linear	3	153.333	51.111	111.41	0.000
3	Reaction Temperature	1	28.527	28.527	62.18	0.000
4	Catalyst Concentration	1	24.665	24.665	53.76	0.000
5	Molar Ratio	1	100.141	100.141	218.27	0.000
6	Error	9	4.129	0.459		
7	Total	12	157.462			

Table 4.4. Definitive screening analysis

S.No	Term	Coef	SE Coef	T-Value	P-Value	VIF
1	Constant	89.806	0.188	478.05	0.000	
2	Reaction Temperature	1.689	0.214	7.89	0.000	1.00
3	Catalyst Concentration	1.571	0.214	7.33	0.000	1.00
4	Molar Ratio	3.165	0.214	14.77	0.000	1.00

From the analysis of variance table contribution of each factor can be found out by using p and t values, p values tests the null hypothesis of the give data the

lower the p value better the significance of the contributing factor, from the table.4.4 the value of p can be found 0.000 in all the cases which indicates the design was very suitable, t value indicates the individual contribution of each factor to the response. From the table.4.4 it is clear that molar ratio has the highest contribution in the yield, the t value of molar ratio is (14.77).

In addition, table 4.5 represents the Standard deviation (S) Coefficient of determination(R-sq) and adjust coefficient of determination(R-sq(adj)). S represents the standard deviation between data values and fitted values. Lower the S values better the model describes the response. The R-sq represents the percentage of variation in the responses and it also determines how well the model fits the experimental data. Higher R-sq indicates that most of the variations in response can be explained; here R-sq (97.38%) indicates that the model fits the data.

Table 4.5. R-sq values for the optimized model

S. No	S	R-sq	R-sq (adj)	R-sq(pred)
1	0.677335	97.38%	96.50%	95.43%

4.5 Optimization by Taguchi

In this research biodiesel was obtained using transesterification process and Taguchi optimization method was used for optimization of biodiesel yield. The main objective of the analysis was to maximize the methyl ester yield, for this purpose Taguchi based optimization technique was adapted. Taguchi method uses orthogonal array for designing the experiments and it can predict the important parameters which influence the quality of the product and thereby reducing the experimental time and valuable resources. In the present analysis L9 orthogonal array was chosen for optimization of biodiesel yield. Equation 4.2 represents the number of experimentations that has to be carried out and it is clear that number of experimental runs depend upon chosen level and number of factors. From the equation it is found that there are nine set of experiments that has to conduct in order to calculate response parameters (R. Sathish Kumar et al 2015).

$$N = (L-1)\,F+1 \text{ ------Eq.4.2}$$

Where: L represents the chosen level

F represents the number of factors

A 3-level design with four factors and 9 runs was chosen, the corresponding factors were molar ratio, catalyst concentration, reaction time and reaction temperature,3 level of values for each factor were given, Table 4.6 represents the different factors which are varied at three levels

Table.4.6 Factors at three different levels

S.No	Parameters	Level-1	Level-2	Level-3
1	Molar ratio (Methanol to oil ratio)	4	5	6
2	Catalyst concentration	3.5	4	4.5
3	Reaction Time	120	150	180
4	Reaction Temperature	44	48	54

A 9 run experimental matrix was generated for the four factors as shown in table 4.7

Table.4.7 Orthogonal array for DOE with different factors

S. No	Molar Ratio	Catalyst concentration (Wt %)	Reaction time (min)	Reaction temperature (^{0}C)	Yield (Wt %)	SNRL
1	4	3.5	120	44	83.64	38.44
2	4	4.0	150	48	84.25	38.67
3	4	4.5	180	54	89.60	38.70
4	5	3.5	150	54	81.49	38.89
5	5	4.0	180	44	88.90	38.19
6	5	4.5	120	48	87.98	38.70
7	6	3.5	180	48	85.20	38.58
8	6	4.0	120	54	80.88	39.01
9	6	4.5	150	44	90.21	38.49

In this analysis 9 experiments were conducted according to the combination of factors and their ranges and biodiesel yield was found, the obtained biodiesel yield for the 9 experiments were filled in a new column in the experimental matrix After the yield column is filled and the design was analyzed and yield was chosen as response.

Table.4.7 represents the biodiesel yield and signal to noise ratio level (SNRL) for nine set of experiments. The SNRL is the algebraic mean of particular parameter at that level. From the table.4.7 it is evident that experiment no 9 give the maximum yield of (90.21%) and experiment no 8 results in low yield (80.88%).

From the experimental results optimization was done for larger the better (Eq.4.3)

Nominal the best $-\ SNR_i = 10\log\left(\frac{\bar{y}_i^2}{s_i^2}\right)$

Smaller the better $-SNR_i = -10\log\left(\sum_{j=1}^{n}\frac{y_j^2}{n}\right)$

Larger the better $-SNR_i = -10\log\frac{1}{n}\left(\sum_{j=1}^{n}\frac{1}{y_j^2}\right)$ Eq(4.3)

Where

$$y_i = \frac{1}{n}\left(\sum_{j=1}^{n} y_{ij}\right) \text{ (mean value of response)}$$

$$s_i^2 = \frac{1}{n-1}\left(\sum_{j=1}^{n} y_{ij} - \bar{y}_i\right) \text{ (variance)}$$

The experimental matrix was analyzed and analysis of variance was generated. The signal to noise ratio (SNR) represents the ratio of mean value of biodiesel yield to standard deviation. SNR helps in predicting the optimum level of parameters with respect to optimum conditions to maximize the biodiesel yield. The exact parameter which influences the biodiesel yield at that particular condition is unable to predict by using SNR. Hence, a statistical analysis of variation is used for the biodiesel yield (ANOVA). In the present analysis ANOVA test is used for the prediction of PME and also the different parameters that influence the biodiesel yield. The percentage of contribution was also calculated by the contribution factor formula as shown in the equation 4.4 The Contribution factor will also revel the significant process parameter but this F value generated by ANOVA give the reconfirmation.

% Contribution factor $= \frac{SS_f}{SS_T} \times 100$ -------Eq (4.4)

Where $SS_f = \sum_{j=1}^{3} n\left[(SNR_L)_{fj} - SNR_T\right]^2$

$$SS_T = \sum_{i=1}^{9}\left[SNR_i - SNR_T\right]^2$$

SS_f represents the sum of the squares for the f^{th} parameter

SS_t represents the total sum of the squares of all parameters

N is the number of experiments at level J of factor f

CHAPTER V

RESULTS AND DISCUSSIONS

5.1 Physicochemical properties

The obtained methyl esters from transesterification process were tested for different physio chemical analysis. Biodiesel from different sources of edible and non edible oils are gaining more importance, due to the fact that, its increasing demand in worldwide on the other hand it is a clean burning and eco friendly fuel. The properties of the biodiesel depend on the chemical compositions, fatty acid compositions and these properties are also varies from oil to oil. The different physio chemical properties were measured by following international standards.

Table 5.1 Comparing properties of Palm Methyl Ester, WFPOME with Diesel

S. No	Property	Diesel	PME	WFPOME	Standards
1	Density (kg/m^3)	853.97	864.42	870	ASTM-D1298
2	Viscosity (mm^2/s)	4.33	4.71	3.6	ASTM-D445
3	Flash point (^0C)	64	196	145	ASTM-D93
4	Pour point (^0C)	-4	11	-5 to 7	ASTM-D97
5	Calorific Value (MJ/kg)	42 – 46	37.27	38	ASTM-D240
6	Cetane Number	47.5	55.38	53	ASTM-D613
7	Cloud Point (^0C)	-5.0	16.0	-3 to 10	ASME-D2500
8	Cold Filter Plugging Point (^0C)	-6.0	12.0	0	ASME-D6371

5.2 Chromatogram for different oils by Gas Chromatography

There are many conventional methods to study the different oils for their fatty acid analysis which states the quality of the fuel. These methods are time taking, costly and laborious when compared to the newly developed technologies. So the Gas Chromatography analysis is newly developed and widely accepted method for the fatty acid analysis. This method will give accurate results in less time and makes the analysis easy. The following graphs were obtained for different types of oils when analyzed using Gas chromatography.

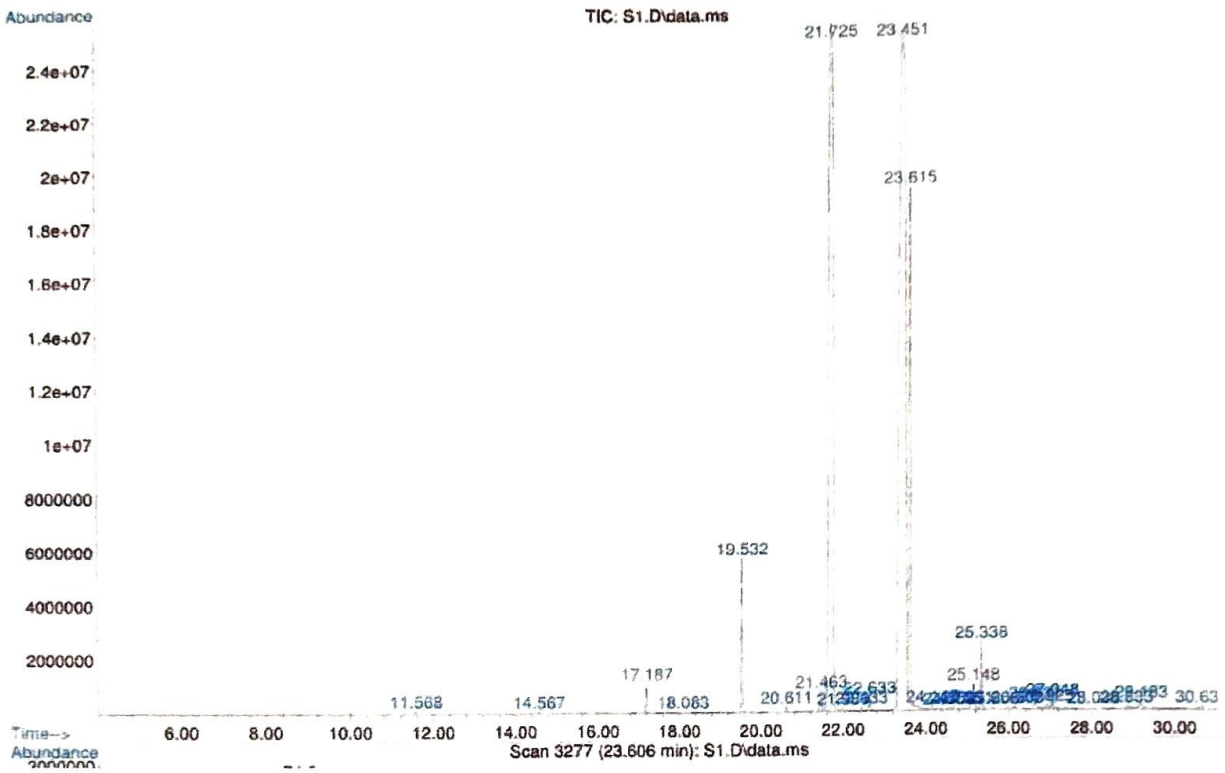

Figure 5.1 Gas chromatographic analysis of fatty acid methyl ester

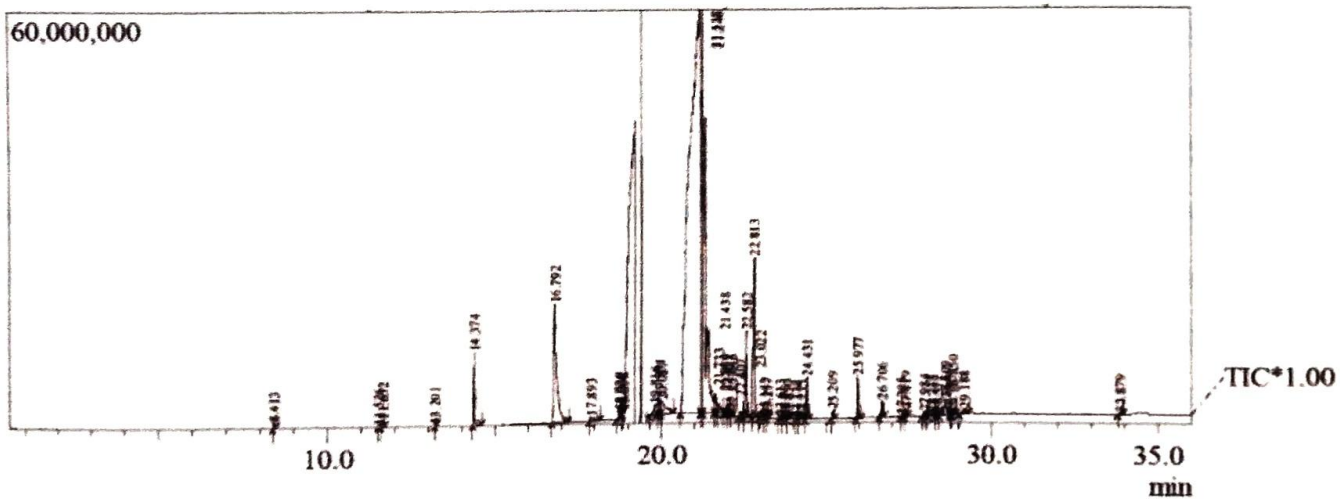

Figure 5.2 Gas chromatographic analysis of PME.

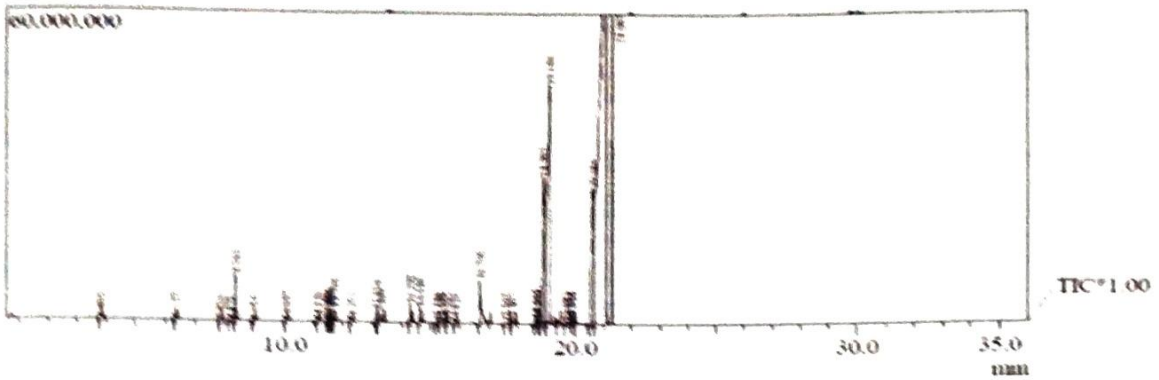

Figure 5.3 Gas chromatography analysis of WFPOME

5.3 Fatty acid composition of Palm Methyl Ester

Fatty acid compositions play a vital role during the combustion in the combustion chamber. The different types of analysis are carried out to study the fatty acid composition. These studies for the biodiesel showed that the major types of fatty acids are saturated and unsaturated fatty acids, these unsaturated fatty acids further divided as mono and poly unsaturated fatty acids. Biodiesel Characterization by using GC-MS (Gas Chromatography and Mass Spectrometry) analysis also showed that major fatty acid components in all esters are saturated and unsaturated fatty acid. The retention time and peak height/area were used in qualitative and quantitative determination of the biodiesel components. The presence of saturated fatty acid in the obtained biodiesel leads to high viscosity, high cetane number and better biodiesel stability. The dielectric constant values of biodiesels were mainly affected by unsaturated fatty acids Further- more, the dielectric constant of biodiesels increased with increasing the degree of unsaturation or the number increasing of double bonds of unsaturated fatty acids in biodiesel. The higher level of unsaturated fatty acid reduces thermal efficency fuel quality and emits NOx because of its easy oxidation (Aditya kolakoti et al. 2017: Puhan S et al. 2010). It is also difficult to convert the raw oil into methyl ester if the presence of free fatty acids (FFA) is more than 2.5%.

Table 5.2 Fatty acid composition of crude palm oil

S. No	Fatty acid	Saturated/ Unsaturated	Wt%
1	Lauric	Saturated	0.1
2	Myristic	Saturated	0.7
3	Palmitic	Saturated	36.7
4	Stearic	Saturated	6.6
5	Oleic	Unsaturated	46.1
6	Linoleic	Unsaturated	8.6
7	Linolenic	Saturated	0.3
8	Archidic	Unsaturated	0.4

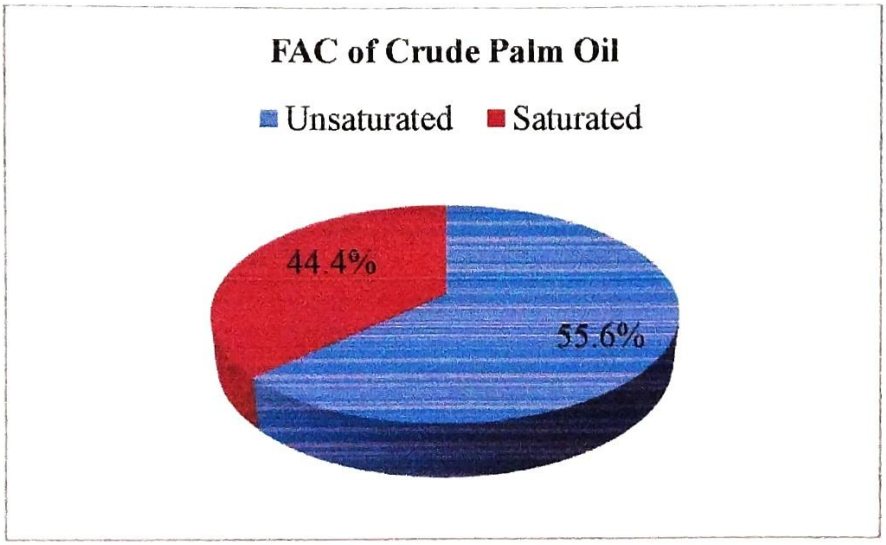

Figure 5.4 FAC for crude palm oil.

Table 5.3 Fatty acid composition of PME

S. No	PME	Saturated/ Unsaturated	Wt (%)
1	Lauric	Saturated	0.1
2	Myristic	Saturated	1.06
3	Palmitic	Saturated	35.52
4	Stearic	Saturated	0.04
5	Palmitoleic	Unsaturated	0.4
6	Oleic	Unsaturated	35.67
7	Linoleic	Unsaturated	14.52
8	Linolenic	Saturated	12.69
Total			100

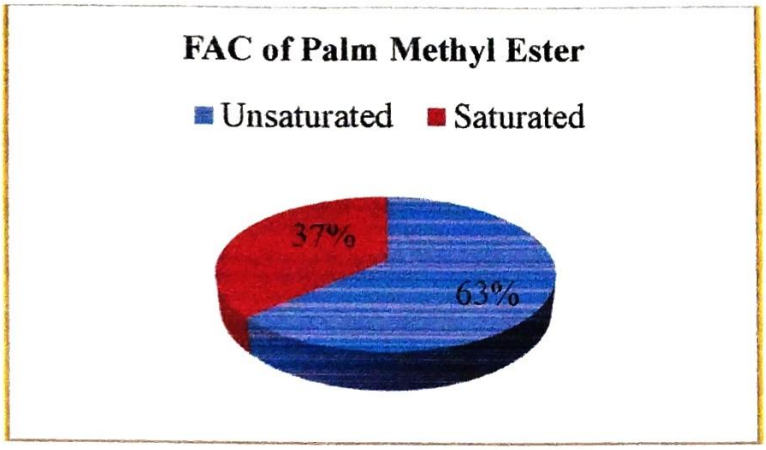

Figure 5.5 FAC for methyl ester.

Table 5.4 Fatty acid composition of WFPOME

S. No	Composition	Saturated/Unsaturated	Wt%
1	Palmitic acid	Saturated	22
2	Linoleic acid	Unsaturated	52
3	Oleic acid	Unsaturated	7
4	Myristic acid	Saturated	4
5	Caprylic acid	Saturated	3
6	Stearic acid	Saturated	12
		Total	100

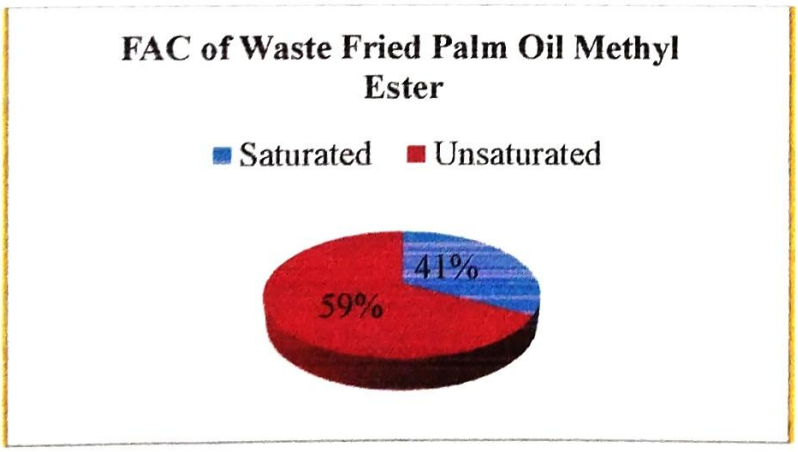

Figure 5.6 FAC for FPOME.

5.4 Prediction of cetane number (CN) using fatty acid compositions (FAC)

The quality of combustion depends on the quality of the fuel, Cetane number (CN) is known for the diesel quality index. The cost involved in determining the CN through experimental approach is high and less accurate due to the experimental errors. In order to overcome the experimental cost and errors different methods have been proposed to predict the CN. Van Gerpen tested the CN of soybean biodiesel and concluded that CN is depends on the distribution of fatty acid composition (FAC) in the oil (Van Gerpen 2017). The CN is more in saturated FAC, less in unsaturated FAC and moderate in mono unsaturated FAC [15]. A.I. Bamgboy suggested that, with the FAC it is possible to predict the CN. Equation (5.1) represents the relation between biodiesel FAC and CN. (A.I Bangboye). From the FAC of PME and WFPOME the predicted CN is 55.38, 53.32 respectively (Pischinger G.M et al.) and by experimental approach the CN is 54.

Cetane number $= 61.1 + 0.088X_2 + 0.133X_3 + 0.154X_4 - 0.101X_5 - 0.039X_6 - 0.243X_7 - 0.395X_8$ ---------Eq 5.1[16]

Where,

X_2 _ Myristic acid

X_3 _ Palmitic acid

X_4 _ Stearic acid

X_5 _ Palmitoleic acid

X_6 _ Oleic acid

X_7 _ Linoleic acid

X_8 _ Linolenic acid

For Palm Methyl Ester,

Cetane number $= 61.1 + 0.088x_2 + 0.133x_3 + 0.154x_4 - 0.101x_5 - 0.039x_6 - 0.243x_7 - 0.395x_8$

$=$ **55.38.**

For Waste Fried Palm Oil Methyl Ester (WFPOME),

Cetane number $= 61.1 + 0.352 + 2.926 + 1.848 - 0.273 - 12.636$

$=$ **53.32**

Table 5.5 Predicted Cetane number for PME and WFPOME

S. No	Type of oil	Cetane number
1	PME	55.38
2	WFPOME	53.32

5.5 Optimization by Taguchi method

The main objective of the present analysis is to maximize the methyl ester yield, for this purpose Taguchi based optimization technique was adapted. Initially a nine set of experiments were performed and yield percentages were calculated for each case by varying the four different parameters of molar ratio, catalyst concentration, time and temperature. Table.5.6 represents the PME yield and signal to noise ratio level (SNRL) for nine set of experiments. From the table 5.6 it is evident that experiment no 9 give the maximum yield of (90.21%) and experiment no 8 results in lowest yield (80.88%).

Table 5.6 Signal to noise ratios for 9 set of experiments

S. No	Molar Ratio	Catalyst concentration (Wt %)	Reaction time (min)	Reaction temperature (^{0}C)	Yield (Wt %)	SNRL
1	4	3.5	120	44	83.64	38.44
2	4	4.0	150	48	84.25	38.67
3	4	4.5	180	54	89.60	38.70
4	5	3.5	150	54	81.49	38.89
5	5	4.0	180	44	88.90	38.19
6	5	4.5	120	48	87.98	38.70
7	6	3.5	180	48	85.20	38.58
8	6	4.0	120	54	80.88	39.01
9	6	4.5	150	44	90.21	38.49

Table 5.7 represents the catalyst concentration, molar ratio and reaction time and temperatures for different levels. Here the SNRL represents the algebraic mean of particular parameter at that level. The delta represents the difference in maximum and minimum values of particular parameters, based on the higher and lower values of delta, the ranks were assigned. Catalyst concentration is ranked as one and it is determined as influencing parameter for the PME production and yield similarly.

Table.5.7 Response table for signal to noise ratios (larger is better)

Level	Reaction Time (RTi)	Catalyst Concentration (CC)	Molar Ratio (MR)	Reaction Temperature (RT)
1	38.43	38.65	38.50	38.84
2	38.55	38.70	38.61	38.67
3	38.99	38.62	38.86	38.46
Delta	0.57	0.07	0.36	0.39
Rank	1	4	3	2

5.5.1 Analysis of variance

The signal to noise ratio (SNR) represents the ratio of mean value of biodiesel yield to standard deviation. SNR helps in predicting the optimum level of parameters with respect to optimum conditions to maximize the biodiesel yield. The exact parameter which influences the biodiesel yield at that particular condition is unable to predict by using SNR. Hence, a statistical analysis of variation is used for the biodiesel yield (ANOVA). In the present analysis ANOVA test is used for the prediction of PME and also the different parameters that influence the PME yield. Table.5.8 represents the different process parameters for F and P values. Higher the F value represents the significant parameter in the preparation of methyl esters and its yield. The Contribution factor will also revel the significant process parameter but this F value gives the reconfirmation. From the table.6 the maximum F value (28.86) and the corresponding low P value (0.006) are significant.

Table.5.8 Analysis of variance for different process parameters

S.No	Source	DF	Adj SS	Adj MS	F Value	P Value
1	Regression	4	87.6484	21.9121	13.35	0.014
2	Molar Ratio	1	0.0600	0.0600	0.04	0.858
3	Catalyst Concentration	1	47.3766	47.3766	28.86	0.006
4	Reaction Time	1	18.7267	18.7267	11.41	0.028
5	Reaction Temperature	1	21.4851	21.4851	13.09	0.022

5.5.2 Signal to noise ratio (SNR)

Figure.5.4 represents variations of molar ratio, catalyst concentration, reaction time and temperature of the reaction in terms of signal to noise ratio. The maximum value in each parameter represents the optimum level for PME yield. Therefore, at molar ratio of 5:1, catalyst concentration of 4.0 grams, reaction time of 180 minutes and temperature of 44^0C gives the maximum yield for PME.

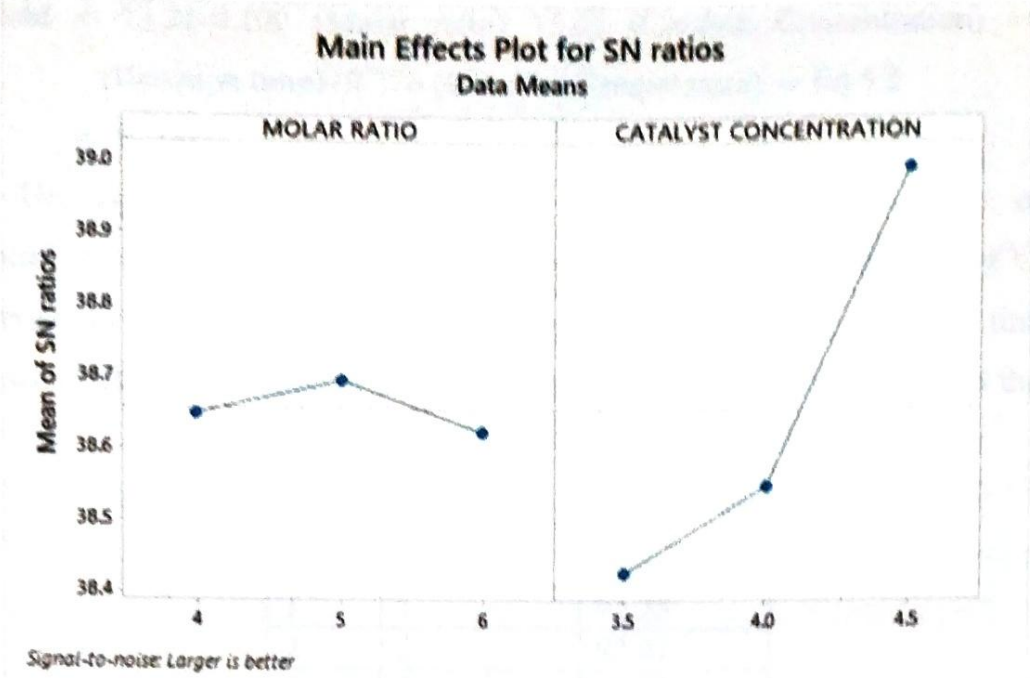

Figure 5.7 SNR plot for molar ratio and catalyst concentration

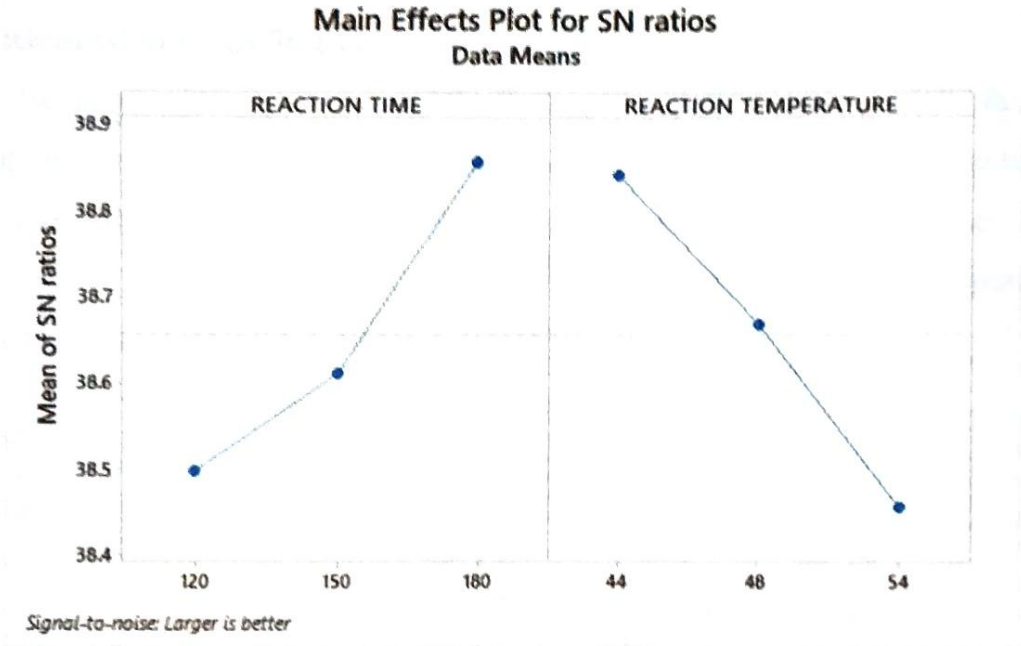

Figure 5.8 SNR plot for reaction time and temperature

5.5.3 Regression equation

The optimum yield of PME is calculated by using the regression equation. From the SNR graphs the optimum value of each parameter for the maximum yield are obtained. By substituting these optimum values in the regression equation, the optimum yield value is 92.06%.

Yield = 73.21-0.100 (Molar ratio) +5.62 (Catalyst Concentration) +0.0589 (Reaction time)- 0.376 (Reaction Temperature) -- Eq 5.2

= 92.06%

The optimum values from SNR plots of molar ratio (5:1), catalyst concentration (4), reaction time (180min) and reaction temperature of 44^0C were considered for experimentation, the experimentation is repeated for three times for better accuracy. The average of three experiments was 91.65% as shown in the table 5.9

Table 5.9 Experimental optimum yield

S.No	Trails	Yield (%)
1	1	91.25
2	2	92.01
3	3	91.69
4	Average	91.65

5.6 Optimization by Definitive screening method

Response optimizer was used for finding the optimum values of the factors to generate maximum yield, Response optimization helps you identify the combination of variable settings that jointly optimize a single response or a set of responses. This is useful when you need to evaluate the impact of multiple variables on the response; the response optimization plot is shown in figure 5.6

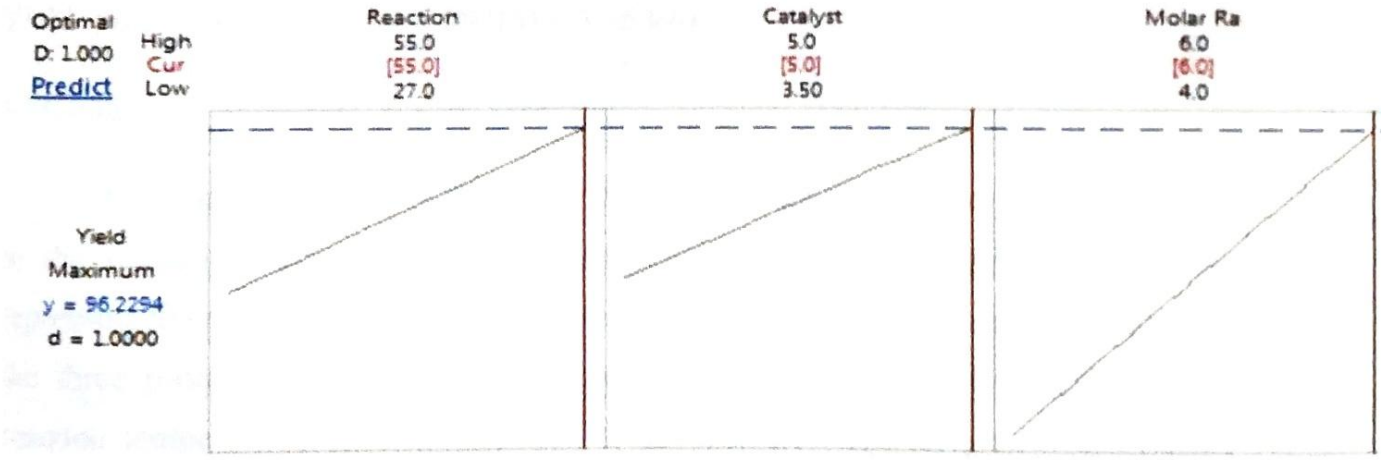

Figure 5.9 Main effect plots for yield.

The optimum values were found as reaction temperature 55^0C, catalyst concentration 5 grams and the molar ratio as 6:1 as shown in table.5.10

Table 5.10 Optimum contributing factors

S. No	Variable	Setting
1	Reaction Temperature	55
2	Catalyst Concentration	5
3	Molar Ratio	6

5.6.1 Regression equation

Regression equation was generated for the three factors. A regression equation is used to describe the relationship between the response and the factors in the model. The regression equation is an algebraic representation. The regression equation for the linear model takes the following form: $Y= b_0 + b_1x_1$. In the regression equation, Y is the response variable, b_0 is the constant or intercept, b_1 is the estimated coefficient for the linear factor. Regression equation is shown below in equation 5.3

Yield = 60.14 + 0.1206 R.T + 2.093 C.C+ 3.165 M.R - Eq 5.3

Where

R. T= Reaction Time

C.C= Catalyst Concentration

M.R= Molar Ratio

Optimum yield was found out from the regression equation by substituting the optimum values in the equation

Yield=60.14 + 0.1206(55) + 2.093(5) + 3.165(6)

= **96.230**

Maximum yield of **96.230** was obtained from substituting the optimum values in the regression equation. Equation (Eq 5.3) developed by regression analysis represents the theoretical yield. It is validated by conducting the experiments by using the three process parameters of methanol to oil ratio, catalyst concentration and reaction temperature. In order to achieve the accurate response yield, experiments were repeated for three time and the average yield is calculated as 95%. The experimental yield result was in a reasonable agreement with the predicted yield results with an error of 1.230% which may acclaim as human error and can be neglected. Thus, from the experimental results it can be said that the definitive

screening methods is effective in predicting the important response parameters for the biodiesel production.

5.6.2 Pareto chart of standardized effects

The Pareto chart gives the information about the absolute values of the standardized effects from maximum to minimum effect. The standardized effects test the null hypothesis. The chart also plots a reference line which indicates the effects that are statistically significant and it is denoted by alpha. From the figure 5.7 all the three contributing factors cross the reference line which indicates that the all the three factors statically significant with 95% confidence and the null hypothesis can be rejected.

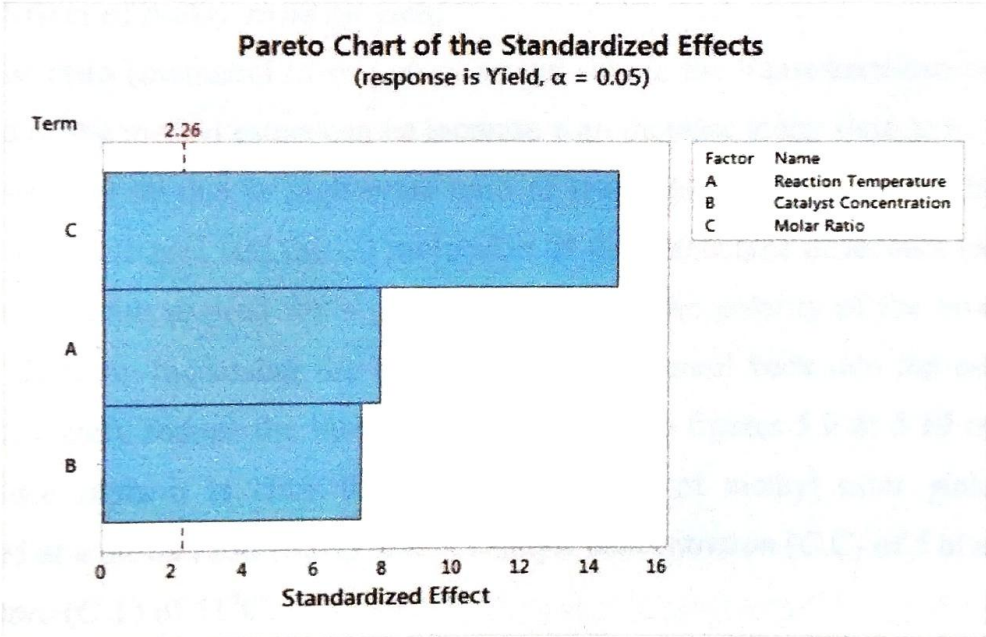

Figure 5.10 Pareto Chart

Mean affects plot shows the individual contribution of each factor at different levels with respect to the response yield figure shows the individual contribution of reaction temperature, catalyst concentration and molar ratio to the yield

Figure 5.12 Contour Plot for max yield

Figure 5.13 Surface Plot for max yield

5.6.4 Effect of reaction temperature on yield

To achieve a maximum yield, reaction temperature and the presence of alcohol in a given mixture are very important. At high temperatures (<62^{0}C) the rate of reaction in the mixture is more and the contributing yield will increase. If the reaction temperature exceeds the boiling point of methanol (64.5^{0}C) then the evaporation of methanol takes place there by decreasing the volume of the mixture will results in decrease in the biodiesel yield. From the figures 5.11 & 5.12 a maximum yield of 94% is achieved at a reaction temperature of 55^{0}C, molar ratio of 6 by maintaining a constant catalytic concentration of 4.25.

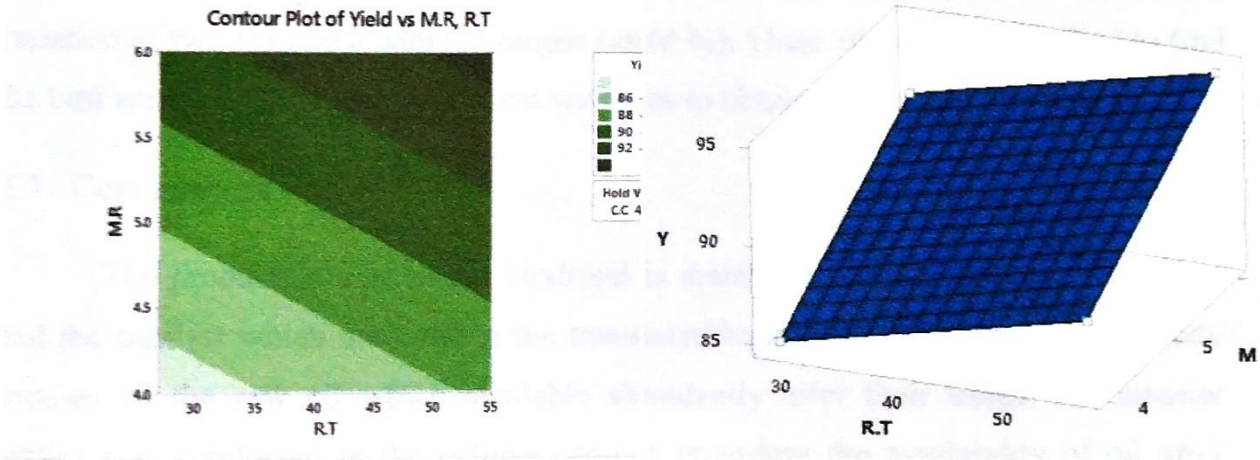

Figure 5.14 Contour Plot for max yield

Figure 5.15 Surface Plot for max yield

5.6.5 Effect of Catalyst concentration on yield

Different catalysts were used for the production of methyl esters some of them were sodium hydroxide (NaOH) and potassium hydroxide (KOH). Due to the wide availability and for better biodiesel yield sodium hydroxide is used as catalyst. In this present analysis 3.5 to 5 grams of NaOH is used in the transesterification process. Figures 5.13 & 5.14 represents the contour and surface plots for the WFPO biodiesel yield at different catalyst concentration and different reaction temperature. From the figures 10 & 11 it is clear that by increasing the CC (4.75 to 5 grams) and the RT (47 to 55^{0}C) simultaneously the maximum yield (93%) of the WFPO biodiesel is achieved by maintaining a constant molar ratio of 5.

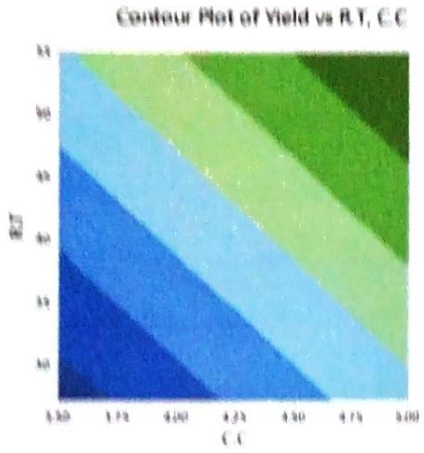

Figure 5.16 Contour Plot for max yield

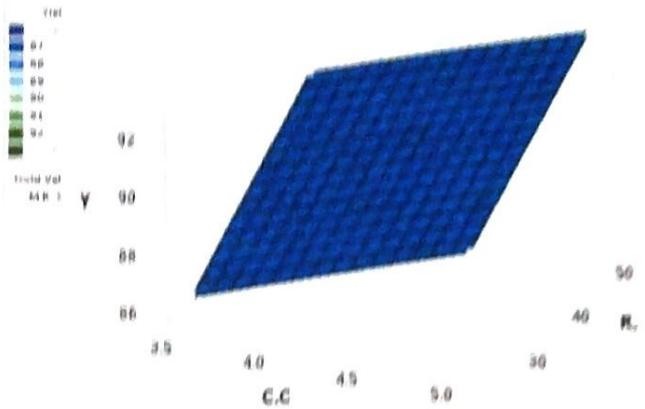

Figure 5.17 Surface Plot for max yield

The above obtained contour and surface plots were obtained by taking variation of two variables with the output (yield %). These plots can be studied to find the best suited combination of process variables to obtain the maximum yield.

5.7 Cost analysis

The production cost of the biodiesel is mainly dependent on the raw oil cost and the catalyst which are used in the transesterification process. The present study focuses on the raw oil which available abundantly after their usage. An internal survey was conducted in the college campus regarding the availability of oil after their usage. It is observed that around 85 liters of oil per day is dumped. Hence, the waste fried palm oil is used for the biodiesel production and the detail cost involved in the process is shown in the table.5.11. The cost of biodiesel is Rs 61.27/- which is lower than the present diesel cost of Rs 68/- (Sumit H. Dhawane et al.).

Table 5.11 Cost analysis

S. No	Material Used	Quantity Used	Price/Liter/Kg	Rs
1	Waste fried Palm oil	1 liter	0	0
2	Methanol	260 ml	160	41.6
3	H_2SO_4	2ml	35	0.07
4	NaOH	8 grams	450	3.6
5	Distilled water	1 liter	6	6
6				51.27
7	Heating and miscellaneous cost			10
8	Total Cost of the WFPME			61.27

CHAPTER VI

CONCLUSIONS

Biodiesel has a significant application in reducing the harmful exhaust emissions like NOx, CO, PM, HC and smoke etc. On the other hand their utilization was limited due to the fact of its high production cost. In this research an attempted was made to reduce its cost and to increase the biodiesel yield. For this purpose low cost feed stocks like used cooking oil (Palm) was used. To increase the biodiesel yield advanced optimization techniques of Taguchi and Definitive screening design were implemented. After an extensive experimental process the following conclusions are illustrated.

- To minimize the raw oil cost, in this research used palm oil (5 liters) which was available in our college food courts was collected.
- To reduce the viscosity of the raw oil a widely accepted method of transesterification was implemented.
- In order to verify the different physicochemical properties both used palm oil and neat palm oil were used for biodiesel production.
- The obtained fuel samples from the transesterification process were tested for different fuel properties analysis and it was observed all the properties were within the standards
- Different process parameters like molar ratio, catalyst concentration, reaction time and temperature and their influence on biodiesel yield was analyzed by optimization techniques.
- For neat palm oil Taguchi based optimization was implemented and it was observed that catalyst concentration and the molar ratio are the important parameters which show a significant role in the palm methyl ester yield. The maximum yield of 96.83% was achieved by maintaining the process variable

at 5:1 molar ratio, 4 grams of catalyst concentration, 220 minutes of reaction time and 45^0C of reaction temperature.

- For used cooking oil Definitive screening design was implemented. The optimum process parameters were determined using definitive screening design were: molar ratio, catalyst concentration and reaction temperature with the corresponding yield of 96.230%. The molar ratio was observed to be most influencing parameter with a contribution factor of 6:1 followed by reaction temperature of 55^0C and the least influencing parameter was catalyst concentration.
- Fatty acid compositions were analyzed by gas chromatography. It was found that for palm biodiesel 37.12% saturated and 62.88% unsaturated fatty acids were present. For used cooking oil 41% of saturated 59% of unsaturated fatty acids.
- In order to achieve the accurate response yield, experiments were repeated for three times and the average yield was calculated as 95% for both the biodiesels. The experimental yield results were in a reasonable agreement with the predicted yield results with an error of 1.5% which may acclaim as human error and it can be neglected.
- The byproduct of transesterification process was glycerin and it was converted to soap.
- The cost analysis of waste fried palm biodiesel was lower than the standard diesel cost.
- Therefore, waste fried palm oil could be considered as most suitable for biodiesel production and can be used as an alternative fuel in diesel engines without changing the basic engine design parameters.

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