RESPONSE SURFACE OPTIMIZATION OF BIODIESEL FROM USED COOKING OIL FOR THE APPLICATION IN CI ENGINES

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CERTIFICATE

This is to certify that the Project Report entitled " **RESPONSE SURFACE OPTIMIZATION OF BIODIESEL FROM USED COOKING OIL FOR THE APPLICATION IN CI ENGINES** " being submitted by **K. Revanth (319126520016), M. Vinay (319126520023), T. Jyothi (320126520L04), S. Jaya Chandra(319126520042), T. Likith (319126520052)** to the Department of Mechanical Engineering, ANITS is a record of the bonafide work carried out by them under the esteemed guidance of **Dr. M. Vinod Babu.** The results embodied in the report have not been submitted to any other University or Institute for the award of any degree or diploma.

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ABSTRACT

Many nations have been looking for more affordable alternative fuels due to the growing demand for quickly depleting diesel in transportation applications and the rising levels of vehicle exhaust pollutants. One of the most economical alternative fuels on the market today is biofuel derived from used cooking oil. This study attempted to analyse the yield of biodiesel created from used cooking oil, a low cost and high potential source. Responsive Surface Methodology (RSM) approach was utilised to optimise the process parameters and determine the best conditions for the synthesis of biodiesel from wasted cooking oil.

The concentration of the catalyst, the methanol ratio, and the reaction duration are tested for their optimal values while maintaining a steady stir speed (400rpm) to generate the highest yield. Influence of process parameters, including reaction duration of transesterification process(60-120 min), mass percentage of catalyst with respect to oil (0.9-1.3%), and volumetric percentage of methanol to oil (18-28%) were studied. The significant physico-chemical characteristics of biodiesel obtained were assessed and compared with the standards ASTM-D6751 and EN-14214. Using a single-cylinder, four-stroke diesel engine, performance tests were run for pure diesel, B5, B10, B15, B20, and B25, and the emissions were analysed.

With NaOH as the catalyst (0.957996 %wt), a biodiesel yield of 232.387 ml, or 93%, with a methanol ratio of 25.8382 %vol was achieved in a short period of time. Due to the R-Squared, adjusted R-Squared, and predicted R-Squared correlation coefficients being so near to 1, the regression model was deemed to be highly significant at the 95% level of confidence. This suggests that the model has strong co-relational and predictive capabilities. Decrease in CO and HC emissions were observed at all engine loads compared to diesel fuel. It was found that CO_2 emissions were higher for biodiesel and its blends than for diesel fuel. It is observed that with the increase in the percentage of biodiesel in the blends, brake specific fuel consumption increases and brake thermal efficiency of engine decreases.

Keywords*: Response Surface Optimization, Used Cooking oil, Transesterification, Volumetric ratio, NaOH, biodiesel blends, performance, emissions.*

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NOMENCLATURE

ANN	Artificial Neural Network
ANOVA	Analysis of variance
ASTM	American Society for Testing and Materials
BDC	Bottom dead centre
CH_3OH	Methanol
CI	Compression ignition
CO	Carbon Monoxide
CO_2	Carbon Dioxide
DOE	Design of experiments
EPA	Environmental Protection Agency
FAME	Fatty Acid Methyl Esters
FBOs	Food business operators
FFA	Free fatty acid
FSSAI	Food Safety and Standards Authority of India
HC	Hydrocarbons
IEA	International Energy Agency
LPG	Liquefied Petroleum Gas
MCDA	Multi-Criteria Decision Analysis
NaOH	Sodium Hydroxide
No_x	Nitrogen oxides
PUFA	Poly Unsaturated Fatty Acids
RSM	Response Surface Methodology
TDC	Top dead centre
TPC	total polar compound
UCO/WCO	Used cooking oil / waste cooking oil
WHO	World Health Organization

Chapter-1

INTRODUCTION

1.1. Present day Oil Resources:

Nearly all road transport vehicles run on petroleum products such as gasoline, diesel, liquefied petroleum gas (LPG). It is projected that there are currently 1.2 billion automobiles on the road in the globe, and that number is expected to rise to 2.5 billion by 2050 with most of them powered by gasoline or diesel. As of 2016, the world's proven oil reserves totaled 1.65 trillion barrels. The global stock of proved reserves is 46.6 times that of yearly usage. This indicates that it has roughly 47 years of oil left (at current consumption levels and excluding unproven reserves). So, regardless of the precise number, the supply will eventually run out. India is heavily reliant on crude oil imports. According to the IEA, India accounted for 40 per cent of Russia's crude oil imports in February 2023.

1.2. Problems Associated with use of Petroleum Products:

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. 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.

Vehicular emissions are the major disadvantages of petroleum products. Tailpipe emissions that come out of vehicles are Carbon dioxide (CO_2), Nitrogen oxides (NO_x), Hydrocarbons (HC), Sulfur dioxide(SO_2) and Particulate Matter (PM).These emissions are

toxic in nature and degrading our environment day to day. According to a report released by the World Health Organization (WHO), ninety present of people around the world breathe polluted air and around 7 million people die each year due to air pollution.

1.3. Need for Biodiesel and its Sources:

The global energy demand continues to rise. The global energy demand in the year 2022 was 6.2 x 10^{20} J. Studies predict that the energy demand shall rise by a factor of 1.4 to reach 8.7 x 10^{20}J in 2040. The possible solution to address this energy demand is through the use of biofuels.

Greater efforts are being made to address climate change as it becomes an ever-increasing concern around the world. Biodiesel is one of the first biofuels. Rudolf Diesel, the inventor of the diesel engine in 1897, experimented with using vegetable oil as fuel in his engines. The fuel made from vegetable oils and animal fats that we call biodiesel today is named after him because it is mostly used in diesel engines. Biodiesel meets the American Society for Testing and Materials (ASTM) specification D6751 and is approved for blending with petroleum diesel.

Biodiesel can be produced 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 of biodiesel is more than petroleum diesel it will not be a viable alternative for diesel. The list of plant oils that can be converted into biodiesel are discussed below.

1.3.1. Edible oil sources:

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, peanuts etc. These seed extracts are used in the food and feed industry as ingredient or as cooking oils. Almost every edible plant oil 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).

1.3.2. Non-edible sources:

The main focus of feedstock selection was primarily on non-edible sources as edible sources doesn't seem to be an ideal feedstock due to food vs fuel problem. Also, extracting oils from edible sources for production of biodiesel can increase their demand and hence lead to deforestation for their plantation. To overcome this problem, oils are extracted from plants that are cultivated for multipurpose usage and not solely for producing oil-based biofuel. A considerable amount of research has been done on alternative feedstocks for biodiesel production all over the world. There are large number of edible and non-edible plant species for which engine tests and physico-chemical laboratory test have already been conducted. In contrast to edible oil, non-edible oils like jatropha, castor, karanja, rubber seed and sea mango are not suitable for human consumption due to the presence of toxic compound [1]. A large number of plants produce non-edible oils which are suitable candidates for the production of biodiesel. Few examples of non edible oils are Pongamia oil, Jatropha oil, Jojoba oil, Sterculia foetida oil, Linseed oil, Nahor oil, Petroleum nut oil, Dammar oil, Poppyseed oil. Copaiba oil, Stillingia oil. Tung oil,, Vernonia oil, waste cooking oil, waste animal fats, and microalgae [1-3].

1.4. Used Cooking Oil as Feedstock:

Used cooking oil (UCO) can be converted into biodiesel by a simple chemical reaction called transesterification which results in the production of fatty acid methyl esters with properties similar to diesel. Waste cooking oil can be a great source of biodiesel as it is available indigenously, at a low cost. Utilizing waste cooking oil helps reduce waste that would otherwise end up in a landfill or sewer pipes.

1.4.1. Necessity for disposing used cooking oil:

Food business operators (FBOs) are supposed to discard vegetable oils after four times of frying or when its total polar compound (TPC) levels reach 25 according to the Food Safety and Standards Authority of India (FSSAI) rules [4].

The oils tend to be used repeatedly to reduce expenses. Heating of oils to their boiling points repeatedly results in the formation of free reactive oxygen (free radicals) which is responsible for oxidative stress causing elevated levels of glucose, creatinine and cholesterol in the body [5]. Repeated frying also alters Poly Unsaturated Fatty Acids (PUFA) molecules present in the oil resulting in the formation of oxidized monomers, dimmers and polymers which further break down into toxic Malondialdehyde which is linked to coronary heart disease and cancers.

1.5. India's Biodiesel Target:

India's energy security will remain vulnerable until alternative fuels are developed based on renewable feedstocks. As per the Biofuel policy 2018, India aims to blend 5% biodiesel by 2030. It currently blends a dismal 0.16% biodiesel, as per the United States Department of Agriculture report, 2020 [6].

Ministry of Petroleum and Natural Gas claims India annually uses about 27 billion litres of cooking oil, of which 1.4 billion litres UCO can be collected from bulk food operators alone to make 1.1 billion litres of biodiesel [7].

India consumes 102 billion litres of diesel annually. Official statistics suggest that over 5 billion litres of biodiesel are needed per year to meet the blending targets. Of this, 1.1 billion litres can be obtained from UCO itself. However, Indian oil companies procured only 105 million litres of biodiesel in 2019-20 which is 10% of the potential.

1.6. Biodiesel Production Process:

Using the vegetable oils directly into the engine will lead to many problems like fuel clogging, poor atomization and incomplete burning due to high density, high viscosity and poor volatility [8]. Some of the methods adopted by research community to reduce the high viscosity problem are discussed below.

1.6.1. Pyrolysis:

This process involves heating vegetable oil to a high temperature while breaking down the larger molecules into smaller ones in an oxygen-free atmosphere. Separations at C-C or CH bonds are the result of this mechanism. There are three stages to this process: hydro,

catalytic, and thermal cracking. The method employed and the reaction conditions affect how much product is produced. For instance, slow reaction rates at low temperatures yield solid products, but quick temperature increases and quick cracking activities yield more liquid products.

In the pyrolysis process, although the fuel properties of vegetable oils approach to diesel fuel properties, high energy consumption is the most important disadvantage. The pyrolysis method is a good method for the evaluation of industrial wastes and urban wastes besides obtaining fuel.

1.6.2. Dilution:

Dilution is the process of thinning waste and vegetable oils by combining them in a specific ratio with a solvent or diesel fuel. The mixing of oils and diesel fuel is the most typical of this procedure. This lessens oil viscosity and lowers the need for diesel fuel. Oils used in the dilution method of biodiesel production are peanut oil, rapeseed oil, sunflower oil and waste oils.

1.6.3. 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 A (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.

1.6.4. Transesterification:

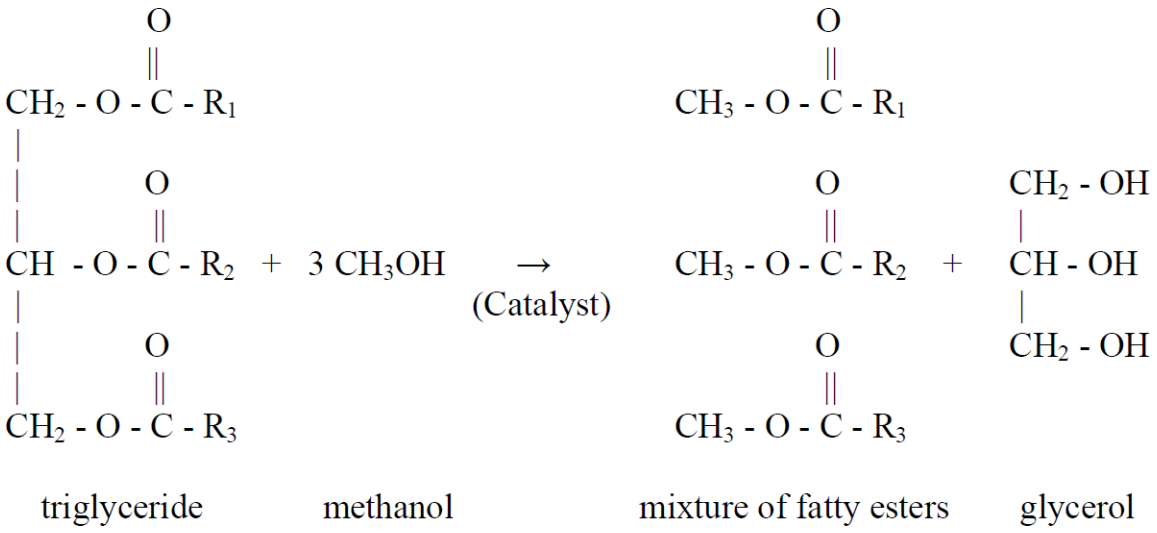
Transesterification or alcoholysis is defined as the process in which non-edible oil is allowed to chemically react with alcohol and catalyst to produce biodiesel and glycerol as by product. Transesterification is the most extensively used method for the production of biodiesel [9-11]. Various methods of transesterification are discussed below.

1.6.4.1. 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 [10].

1.6.4.2. 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. In this process, 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 [12]. The reaction equation of catalytic transesterification is shown below.



The alkaline catalyzed transesterification of vegetable oil proceeds faster than the acid catalyzed reaction [13]. 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. The presence of water and high amount of free fatty acid leads to saponification of oil and so the reaction will be incomplete with the formation of emulsion and difficulty in separation of glycerol.

1.6.4.3. 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.

1.7. Principle of Operation of 4-Stroke Diesel Engines:

Compression Ignition Engine is an Internal combustion engine also known as the Diesel engine named after Rudolf Diesel. One complete cycle of operation typically consists of series of events namely suction, compression, combustion, expansion and exhaust in 4 strokes or 720^0 of crank rotation. Fresh air from atmosphere enters the engine cylinder when piston moves from top dead center (TDC) to bottom dead center (BDC) during suction stroke and gets compressed from low pressure and low temperature to high pressure and high temperature during compression stroke as piston moves from BDC to TDC. Fuel injected at the later stages of compression is evaporated into compressed air utilizing the heat of compression. After attaining the self-ignition temperature (well before TDC), combustion takes place leading to further rise in pressure and temperature. These high-pressure gases push the piston from TDC to BDC during expansion/power stroke and power gets delivered to the crank shaft. Residual gases are then pushed into the atmosphere during exhaust stroke as piston moves from BDC to TDC, completing the cycle. Next working cycle starts with opening of intake valve(s).

Amount of fuel injected varies depending on speed and load on the engine against constant amount of air entering the engine cylinder at any given speed. As such, the value of λ (excess air ratio) is low at high loads and vice-versa. Further, heterogeneity in fuel to air distribution in the combustion chamber leads to the formation of local rich mixtures in core

regions and local lean mixtures in outer regions of fuel spray. Therefore, care must be taken for fine dispersion of fuel to promote thorough mixing and to ensure near complete combustion.

1.8. Combustion in Diesel Engines:

The nature of combustion in diesel engines is 3-D, diffusive, heterogeneous and unsteady. Engine noise, power developed, pollutant formation and amount of emissions released into the atmosphere largely depend on the combustion pattern and its completeness. The finely atomized fuel that is injected into the hot compressed air evaporates and forms combustible fuel-air mixture. Combustion efficiency primarily depends on amount of air induced, fuel characteristics, operating parameters and engine geometry. Combustion in diesel engines starts after a short delay from the commencement of fuel injection, called ignition delay, and completes in two phases viz., premixed and diffusion combustion phases.

A portion of total fuel injected that is initially mixed with air and within flammability limits gets combusted rapidly in the first phase, called premixed combustion phase. This phase lasts for a small period during which combustion takes place very rapidly resulting in high heat release peaks and pressure release peaks.

Combustion rate is relatively slow in diffusion combustion phase owing to controlled mixing of remaining fuel-air mixture with burnt gases. This combustion phase is further divided into controlled combustion and after burning. During controlled combustion, fuel oxidation takes place under reduced oxygen availability in the presence of high in-cylinder temperatures, promoting soot formation. Final oxidation of remaining fuel and partially oxidized fuel takes place during after burning. Chemical reactions rates reduce significantly during this combustion phase owing to the reduction in oxygen supply and presence of low in-cylinder temperatures (as a result of start of expansion stroke).

1.9. Use of Biodiesel Blends in Diesel Engines:

Blends of biodiesel can be used in diesel engines. Generally, B-factor is used to indicate the amount of biodiesel present in the blend. B20 represents 20% biodiesel blended in 80% of diesel. Blends of 20% diesel or below can be directly used in diesel engine without any modifications to the engine. B20 is a common blend because it represents a good balance

of cost, emissions, cold-weather performance, materials compatibility and ability to act as a solvent. Most biodiesel users purchase B20 or lower blends from their normal fuel distributors or from biodiesel marketers.

The Environmental Protection Agency (EPA) in the United States used statistical regression analysis to correlate the biodiesel ratio with changes in pollutants as well as the average impact of biodiesel on heavy-duty diesel engines. While HC, CO, and PM emissions were significantly reduced, NOx emissions increased with the percentage of biodiesel and increased by 10% at B100. The oxygen content of biodiesel is responsible for the considerable decrease in HC, CO, and PM emissions [14,15]. It has been observed that using biodiesel in diesel engines causes NO_x levels to rise. Several attempts have been made to comprehend the mechanism of formation and eliminate this rise in NO_x levels. There are several primary causes that have been put forth.

- Advanced injection timing
- Oxygen content in biodiesel
- double bond
- Radiative heat transfer
- Higher adiabatic flame temperature

There are several mechanisms that contribute to the NO_x increase in biodiesel-fuelled engines and the relative importance of each mechanism may change depending on the operating conditions. It has been suggested that the air/fuel mixture close to stoichiometric at ignition and in the standing premixed auto-ignition zone close to flame lift-off length may be the key factors in explaining the NO_x increase could have an impact on higher local and average in-cylinder temperature and lower radiant heat flux [16].

Chapter-2

LITERATURE SURVEY

This chapter deals with some of the prominent studies on biodiesel preparation, feedstock selection and optimization of the process parameters of biodiesel preparation. Key findings with the use of various cooking oil based biodiesel blends on performance and emission characteristics are also provided.

Jaichandar and Annamalai [17] observed that substituting vegetable oil for diesel fuel poses problems due to high viscosity and low volatility but transesterification can reduce viscosity and produce biodiesel. Base catalysts were found to perform better than acid catalysts and enzymes in biodiesel production. Biodiesel has similar fuel and combustion characteristics to diesel with lesser exhaust emissions and potential for carbon neutrality. While biodiesel engines emit more NO_x, strategies such as cetane improvers, injection timing retardation, and exhaust gas recirculation can control emissions. They recommended further investigations and improved engine design to fully explore the potential of biodiesel engines.

Carlos *et. al.* [18] felt that biodiesel can be used as an alternative fuel to petroleum-based diesel, but the cost and availability of feedstock are major challenges. Authors opined that used cooking oil collection can reduce the cost of biodiesel production and minimize the negative impact of waste oil disposal. However, impurities in used cooking oil require an additional pre-treatment step. They declared that NaOH and KOH are the most used catalysts, and methanol is the most common esterification agent in the conventional transesterification reaction.

Suhel Aktas *et. al.* [19] declared that biodiesel obtained from vegetable and animal oils can be used as a renewable alternative fuel source. The transesterification process which reduces viscosity is commonly used to produce biodiesel. However, the treatment of wastewater generated during the production process is an area that requires further research. Advanced oxidation processes, such as Fenton, have been shown to be effective, but more effective methods are needed.

Canakci and Van Gerpen [20] studied the effects of molar ratio, reaction temperature, catalyst amount, and reaction time on ester formation in acid-catalyzed biodiesel production process. They found that acid-catalyzed transesterification is slower than alkali-catalyzed, and a higher molar ratio is required for acid-catalyzed esterification. The completeness of ester formation increases with increasing acid catalyst amount, while the presence of water in the oil can inhibit ester conversion. Free fatty acid levels above 5% can also lower the ester conversion rate below 90%.

Ehsana *et. al*. [21] examines the feasibility and cost-effectiveness of producing biodiesel for a fast food restaurant in Dhaka using alkaline-based catalyst from waste cooking oil. The study showed that the additional monthly saving could be around 4% of fuel costs if there is no cost associated with dumping waste cooking oil. The cost of chemicals and their recovery units are crucial factors affecting the feasibility. The study suggests that the processing cost for biodiesel may not be very feasible unless the cost of properly dumping waste cooking oil is high enough.

Sunil Kumar and Vikas Deswal [22] have optimized the biodiesel production from soybean oil using the trans-low temperature esterification process technique. The catalyst concentration, reaction time, and molar ratio for transesterification were optimized using the Box-Behnken Design. A biodiesel yield of 80.86% was achieved with a molar ratio of 8:1 and NaOH catalyst (1.8% w/w) in 34 minutes. The concentration of the catalyst, reaction time, and volume ratio of methanol/oil significantly affected the soybean oil yield. Response surface methodology was used to optimize the operating conditions to be employed for maximum production of methyl esters.

Rodríguez *et. al.* [23] produced the biodiesel from blends of castor oil and waste cooking oil using transesterification. The mixture of 85% castor oil and 15% waste cooking oil obtained the highest yield and calorific value (36645 J/g) compared to other blends evaluated. The cost of biodiesel production was reduced by 30% using blends of castor oil and waste cooking oil compared to using only castor oil. This work showed the possibility of integrating waste cooking oil into productive processes to reduce costs and environmental impact.

Gnanaprakasam *et. al*. [24] felt that the use of waste cooking oil as feedstock for biodiesel production can reduce costs, but pre-treatment with acid catalyst may be necessary to reduce high fatty acid content. Methanol is the most suitable alcohol for the transesterification reaction, with a methanol to oil ratio dependent on the amount of free fatty acid. The concentration of catalyst used depends on its nature, and optimum stirrer speed is typically maintained at 200-250 rpm. They employed stepwise reaction mechanisms to eliminate inhibition of acid catalysts by water produced during the esterification process.

Anwar [25] analyzed the suitability of sixteen biodiesel feedstocks based on economic and environmental criteria, as well as physicochemical properties. Four Multi-Criteria Decision Analysis (MCDA) systems were used, with different weighing methods. Coconut was found to be the most suitable edible biodiesel source, while Moringa was the ideal non-edible feedstock. Tallow and Beauty leaf were also considered alternative sources. This study provides useful information for decision-makers in choosing sustainable biodiesel feedstocks. Further research using other MCDA processes was recommended.

Barnwal *et. al.* [9] declared that high viscosity, low volatility, and poor cold flow properties of triglycerides have hindered their direct use in diesel engines. Catalytic transesterification and supercritical method of producing biodiesel could improve fuel properties of triglycerides. Biodiesel obtained from non-edible oils is cheaper than that from edible oils and it provides similar engine performance with low emission levels.

Agarwal and Das [26] proved that biodiesel can be used as an alternative fuel for conventional diesel engines without significant modifications. Esterification can reduce the viscosity and unsaturation of vegetable oils making them suitable for use in diesel engines and also prevents long-term engine problems associated with using vegetable oils as fuel. Biodiesel blends of up to 20% were found to be optimal which improved engine performance and emissions.

Aditya *et. al*. [27] examined the production of biodiesel from waste cooking oil using Response Surface Methodology (RSM) optimization and Artificial Neural Network (ANN)modelling. The study found that NaOH catalyst was the most significant parameter for achieving maximum biodiesel yield. RSM optimization resulted in a maximum yield of

91.30%, while ANN modelling predicted a yield of 92.88%. The fuel properties of the biodiesel met international standards. The study concluded that waste cooking oil is a suitable feedstock for biodiesel production and optimization and modeling tools can help achieve better yields.

Vinod Babu *et. al.*[28] studied on Sterculia Foetida based biodiesel production and concluded that biodiesel can be produced through a single-step trans-esterification process due to its low FFA content. Optimum conditions for the process were determined as methanol-to-oil ratio of 26%, reaction time of 135 minutes, catalyst concentration of 1.09% and reaction temperature of 63°C. The yield and properties of the resulting biodiesel met the requirements of ASTM D6751 and EN 14214 standards and it is suitable for use in CI engines.

Vijayan *et. al.* [29] felt that the transesterification process is the easiest and most economical way to produce biodiesel from edible and non-edible oils, with parameters such as reaction time, molar ratio, type and amount of catalyst, stirring time, and operating temperature affecting the yield. NaOH is commonly used as a catalyst for non-edible oil, with a molar ratio of 6:1, operating temperature of 60-70°C, and reaction time of 1-3 hours resulting in a yield of at least 80%. 70% of biodiesel is produced from edible oil and 30% from non-edible oil, with non-edible oil producing over 90% yield using transesterification.

Ribeiro *et. al.* [30] discussed various aspects of biodiesel production and highlighted the need for continuous improvement in the technology. The study showed that the quality and source of feedstock oil affects the operating and processing parameters of transesterification. The alkali-catalyzed process is suitable for low FFA content oil, while a two-step process is required for higher FFA content oil. The study concludes that the produced biodiesel from tested feedstocks met EN14214 standards and can be used in diesel engines without a decrease in engine efficiency.

Shashikant and Raheman [31] investigated the effect of pre-treatment of crude mahua oil to reduce its high FFA content to less than 1% using methanol and H_2SO_4 as a catalyst. A second-order model was developed to predict acid value as a function of methanol-to-oil ratio, catalyst concentration, and reaction time. After pre-treatment, settling time was

required to remove methanol-water mixture before proceeding to the final alkali-catalyzed transesterification reaction with methanol to produce biodiesel.This process yielded a 98% mahua biodiesel product that satisfied both American and European standards for biodiesel.

Nedambale *et. al.* [32] conducted simple and cost-effective tests to evaluate the quality of biodiesel, requiring minimal equipment. These tests offered a good indication of the quality of the biodiesel which were cheaper than sending samples for official testing. Although they fail to provide a full testing package, tests provided by them were helpful in evaluating the production process. The total cost of these tests was found to be Rs. 898.60 against the cost of sending samples for official testing, which was Rs. 8,000.

Sahar *et. al.* [33] studied conversion of WCO to biodiesel using mineral acid pre-treatment and base catalyzed transesterification. H_2SO_4 was found to be the most efficient mineral acid for esterifying the free fatty acids (FFA) in WCO. The biodiesel produced had a 94% FAME yield and met ASTM D6751 standards.

Zhang *et. al.*[34]designed four continuous flowsheets and simulated for producing biodiesel using virgin vegetable oil or waste cooking oil as raw materials. All processes proved feasible for producing high-quality biodiesel and glycerine by-products with some limitations. The alkali-catalyzed process using virgin oil was the simplest, while the acid-catalyzed process using waste cooking oil was the most complex. If raw material cost is a concern, the acid-catalyzed process using waste cooking oil is a competitive alternative to other processes.

Meng *et. al*. [35] declared that compared to other oils, WCO suffers from lower conversion rates while undergoing transesterification for biodiesel production. Quality upgrading treatment can improve the quality of biodiesel from WCO for use as a fuel in diesel engines. They identified optimized reaction conditions for producing high-quality biodiesel from WCO. Engine testing with B20 blends significantly reduced particles, HC, and CO emissions. They recommended the use of waste oil resources for biodiesel production to ensure national diesel security for China.

Sudhir *et.al*. [36] felt that biodiesel made from WCO is an environmentally friendly liquid fuel, as WCO is a post-consumer waste product. The paper recommends converting WCO into WCO-biodiesel chemically, as using recycled WCO for human consumption is not advisable. WCO-biodiesel showed impressive performance and emission characteristics in CI engine tests with a slight decrease in performance at high loads and lower hydrocarbon emissions compared to baseline diesel. They reported that NO_x, CO, and CO_2 emissions were similar to baseline diesel.

Enweremadu and Rutto [37] concluded that UCO based biodiesel has similar engine performance and combustion characteristics as fresh vegetable oil biodiesel in their literature review and the differences are mainly due to the higher viscosity and lower calorific value of UCO. Decrease in ignition delay and increase in peak pressure were observed with UCO biodiesel compared to diesel fuel, while emissions characteristics vary depending on engine and operating conditions. they felt that UCO biodiesel is a cheap and environmentally friendly liquid fuel option due to its use of post-consumer waste products.

2.1. Problem Statement:

Numerous researchers have studied into the production of biodiesel using different feedstocks but majority of the work was limited to the different edible and non-edible feedstocks. Recently, more researchers are focusing on the use of waste cooking oil for the production of biodiesel but the origin of this waste cooking oil varies from user to user depending on the type of usage. There is enormous scope for the production of biodiesel from different origins of this WCO and utilization of this feedstock for the biodiesel production has not reached even 20% of its potential. further, depending on the usage of WCO, its properties tend to change and hence there is a need for optimizing the process parameters involved in each type of feedstock used for biodiesel production. Hence, this study is aimed at optimization of biodiesel production process parameters and the application of the biodiesel thus obtained in CI engines to investigate its effect engine performance and emission parameters.

Chapter 3

METHODOLOGY

This chapter describes the details of materials, process parameters involved in transesterification, various steps involved in biodiesel synthesis and the engine details used for experimentation.

3.1. Materials:

Raw oils	:	Used Cooking Oil (UCO).
Chemicals	:	Sodium hydroxide, methanol.
Deionized water	:	Double-distilled
Glassware	:	Conical flasks (250 ml, 500 ml& 1000 ml), volumetric flask(1000 ml), separating funnels (500 ml), beakers (1 litre& 2 litres), measuring jars (50 ml, 250 ml& 500 ml).
Others	:	Digital weighing balance, Thermometers, magnetic beads, hot plate magnetic stirrer, heating mantel

Used cooking oil was collected from Manikanta Food Court (MFC) which is close to our campus. Methanol (CH_3OH) and Sodium Hydroxide (NaOH) are procured from Organic Chemistry Lab of ANITS (Anil Neerukonda Institute of Technology & Sciences, Visakhapatnam). Specifications of ingredients used for transesterification are provided in Table 3.1 and Table 3.2.

Equipment required for measurement of ingredients (Digital weighing balance, measuring jars), methoxide preparation and transesterification setup (Magnetic stirrer with heating coil, volumetric flask), decanting and water washing setup (Conical flasks, separating funnel, heating mantel) are procured from Thermal Engineering and Organic Chemistry Laboratories of ANITS.

Table 3.1: Specifications of Sodium Hydroxide (NaOH)

Minimum assay	97.0%
Identification (as NaOH)	Passes test
Solubility	Passes test
Maximum limits of impurities	
Carbonate (Na_2CO_3)	2%
Chloride (Cl)	0.01%
Sulphate (SO_4)	0.05%
Potassium(K)	0.1%
Silicate (SiO_2)	0.05%
Zinc (Zn)	0.02%
Heavy metal (as Pb)	0.002%
Iron (Fe)	0.002%

3.2. Process Parameters Involved in Biodiesel Preparation:

Catalyst concentration, free fatty acid (FFA) content, methanol ratio, reaction temperature and reaction time are important process parameters affecting the biodiesel yield and quality.

3.2.1. Catalyst concentration:

Alkali metal alkoxides are found to be more effective transesterification catalysts compared to acidic catalysts. Sodium alkoxides are the most efficient catalysts. A concentration in the range of 0.5–1% (w/w) has been found to yield 94–99% conversion to vegetable oil esters, and further increase in catalyst concentration does not affect the conversion but adds to extra cost, as the catalyst needs to be removed from the reaction mixture after completion of the reaction. Since UCO has been employed in this study, extra amount of catalyst was considered to compensate the catalyst lost while saponification reaction during transesterification.

Table 3.2: Specifications of the Methanol

Minimum assay (by GC)			99.9%
Maximum limits of impurities			
Water			0.05%
Residue on evaporation			0.0005%
Acidity (As HCl)			0.001%
Alkalinity (as NaOH)			0.0003%
Suitability for gradient analysis :			
Maximum absorbance of largest eluted peak			
At 235nm			0.002 A.U.
At 254 nm			0.001 A.U.
(linear from 95% water (HPLC grade) to 100% methanol			
Over 20 min, followed by 100% methanol for further 10min.)			
Maximum absorbance (1.0 cm cell-vs H_2O)			
205 nm	1.000	225nm	0.160
250 nm	0.020	300nm	0.005
400 nm	0.005		

3.2.2. Free fatty acid content:

FFA content in the feedstock plays a significant part in reaction during biodiesel preparation. Higher FFA content in the reactant oils leads to the formation of soaps and emulsions. This prevents the biodiesel separation from glycerol which in turn lowers biodiesel yield. Therefore, it is necessary either to select feedstocks with lower FFA or to reduce FFA level in the oils before transesterification. The maximum amount of free fatty acids acceptable in an alkali- catalyzed system is below 3 wt. % of FFA. If the oil or fat feedstock has a FFA content over 3 wt. %, a pre-treatment step is necessary before the transesterification process.

3.2.3. Methanol ratio:

Although a 3:1 stoichiometric ratio is necessary, the transesterification process is typically carried out with additional alcohol in order to move the equilibrium to the methyl ester side, where the desired product lies. The catalyst was deactivated by too much methanol, which reduced its effectiveness and resulted in a drop in yield at molar ratios of 9:1 and

12:1. Glycerine created during transesterification settled at the bottom layer, with biodiesel at the top. Therefore, the excess methanol also tended to blur the separation border between glycerin and biodiesel, making it difficult to extract the biodiesel.

3.2.4. Reaction temperature:

Reaction temperature plays an important role in preparation of biodiesel. It was found that reaction aggravates with the increase in reaction temperature up to the boiling point of alcohol used. However, very small improvement has been observed with increase in temperatures above 55°C when methanol was used as alcohol.

3.2.5. Reaction time:

Another crucial parameter in biodiesel preparation is the reaction time. It has been found that rate of reaction starts immediately after the addition of methoxide to base oil at desired temperatures (around 50°C) and majority of reaction completes within first ten minutes and reaction completes with further increase in time which depends on the feedstock oil used.

3.3. Biodiesel Synthesis:

Preparation of biodiesel involves various steps from pre-treatment of raw oil to heating of the final product called Fatty Acid Methyl Esters (FAME). Detailed procedure is explained here under.

3.3.1. Pre-treatment of used cooking oil:

The obtained used cooking oil has been filtered using coarse filter papers for the removal of solid impurities present. To ensure that all water present in the filtered UCO is evaporated, it is heated up to 120^{o}C and then cooled back to room temperature. A sample of 250ml of UCO is then transferred to the volumetric flask and then the sample is heated using a magnetic stirrer with a stirring speed of 400 rpm. Pre-treated UCO is shown in Fig. 3.1 (a).

3.3.2. Preparation of methoxide:

Sodium methoxide was prepared with desired levels of NaOH and methanol through careful measurements of the ingredients. Methanol needed for the run is measured and added to a 250ml conical flask along with the measured sodium hydroxide. Quick transfer of the measured NaOH to the conical flask prevents interaction with the water vapour. The solution containing methanol and sodium hydroxide is agitated using magnetic stirrer to maintain the homogeneity. Enough care was taken to prepare homogeneous solution of methoxide. Methoxide solution is shown in Fig. 3.1 (b).

3.3.3. Transesterification process:

Methoxide has been added to the previously heated and filtered UCO at a temperature of 55^{o}C keeping a constant stirring speed of 400 rpm. The temperature is continuously monitored during the reaction and a constant reaction temperature of 60^{o}C is maintained throughout the reaction. Conical flask is covered with a lid to prevent the escape of methanol. Setup used for transesterification is shown in Fig. 3.1 (c).

3.3.4. Separation of crude biodiesel and glycerol:

Reaction is continued until the run time and is then poured into a separating funnel. Separation of glycerol was observed within few minutes due to difference in the densities of crude biodiesel and glycerol.. After settling down for 8hours, clear separation of methyl esters from glycerol was observed as shown in Fig. 3.1 (d). Glycerol was then removed from separating funnel carefully and the raw FAME was taken into a conical flask for water washing.

3.3.5. Water washing:

The raw FAME needs to be thoroughly water washed to remove the excess catalyst, methanol that is unreacted during the process and the dissolved glycerine. Distilled water is added to the conical flask containing raw FAME and the mixture was then heated using a heating mantel until the bubbles appear.

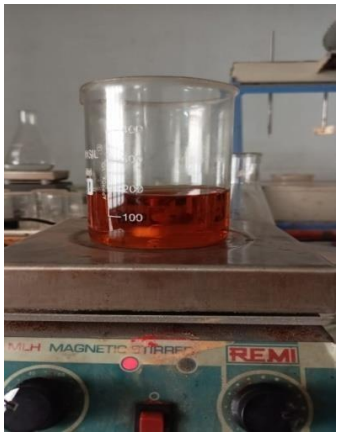
(a) Filtered UCO

(b) Methoxide solution

(c)Transesterification reaction

(d)Separation of glycerol

(e) Final stage of water washing



(f) Post-heating of FAME

Fig. 3.1. Stages involved in biodiesel synthesis

Then it is taken out from the heating mantel and poured into the separating funnel after agitating the mixture to ensure proper mixing of water with the raw FAME. After observing a separation of both layers, water was removed from the separating funnel and the procedure was repeated several times until a clear separation of both the layers as shown in the Fig. 3.1 (e).

3.3.6. Post-heating of raw FAME:

The product obtained after water washing is collected into a beaker and is then heated to around 120^{o}C to ensure the removal of methanol and water left with the raw FAME to obtain the biodiesel (FAME). Post-heating process is shown Fig. 3.1 (f). The final product is then cooled to room temperature and collected into a glass container with a tight lid.

3.4. Preparation of Biodiesel/Diesel Blends:

Fuel blends were prepared in the volume proportion using petro-diesel and UCO based biodiesel by measuring the proportions carefully using a calibrated measuring jar. The mixture was taken into a conical flask with lid and is stirred for 30 minutes using magnetic stirrer at 1200 rpm to ensure proper mixing. Blends were prepared in different proportions by varying biodiesel content from 5% to 25% by volume. All the blends were prepared 30 minutes before their utilization in the engine to ensure homogeneity of the blends and hence no separation of denser and lighter fractions of constituents. Details are listed in Table 3.3.

Table 3.3: Biodiesel blends prepared

Blend	Amount of diesel (ml)	Amount of biodiesel (ml)
B5	950	50
B10	900	100
B15	850	150
B20	800	200
B25	750	250

3.5. Fuel Characterization:

Due to slight differences in composition and physicochemical features between biodiesel and conventional fossil diesel, different inferences can be drawn about engine performance, combustion, and emissions characteristics. Whereas biodiesel is primarily formed of complex esters, fossil diesel is primarily composed of straight-chain hydrocarbons with carbon numbers between 12 and 24. Although different biodiesels have varying esters by composition, it may be said that they all have similar fuel qualities to diesel. Varying fatty ester qualities in biodiesel will alter the physicochemical properties of the fuel by determining its characteristics. Comparison of some important properties of biodiesel with International Standards are provided in Table 3.4.

Table 3.4:Comparison between the properties of biodiesel and International Standards

Properties	ASTM-D6751	EN-14214	Biodiesel
Density at 15^{o}C (kg/m^{3})	--	860-900	869
Flash point (^{o}C)	Min 130	Min 101	168
Fire point (^{o}C)	--	--	174
Kinematic viscosity at 40^{o}C (mm^{2}/s)	1.9-6.0	3.5-5.0	4.08

Important physico-chemical properties of test fuels were determined using standard test procedures. Properties of test fuels are listed in Table 3.5. Apparatus used for the measurement of calorific value, viscosity, density, flash point and fire points of the fuels employed in this study are shown in Fig. 3.2.

Table 3.5: Properties of test fuels

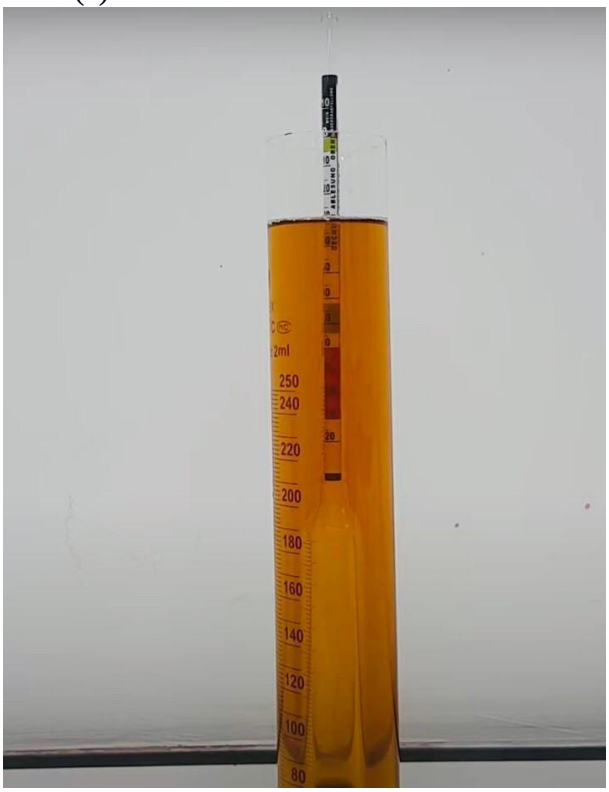
Fuel blend	LCV (kJ/kg)	Kinematic viscosity (cSt)	Density (kg/m^3)	Flash point (^oC)	Fire point (^oC)
D100	42000	3.2	831	>52	--
B100	39640	4.08	869	168	174
B5	41880	--	833	--	--
B10	41760	--	835	--	--
B15	41640	--	838	--	--
B20	41520	--	841	--	--
B25	41400	--	845	--	--

3.6. Engine Description:

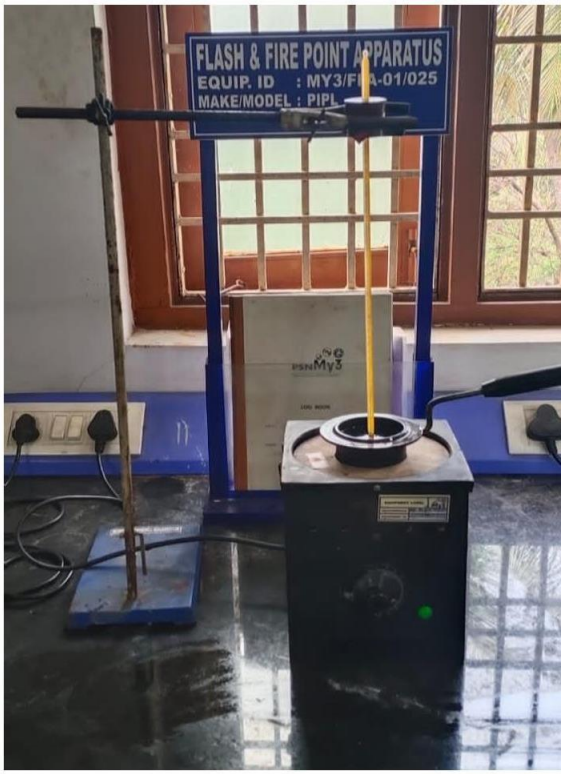
Experimentation was carried out using the facilities available in Thermal Engineering Laboratory, ANITS. The schematic of the test rig used for experimentation is shown in Fig. 3.3. The test rig used for the experimentation is a four stroke, single cylinder, constant speed, water-cooled diesel engine. This engine is provided with a crank handle for starting and is mounted with a rope brake dynamometer. The engine set up is also provided with burette, graduations duly marked and a 3way valve is used to measure the fuel flow rate. Specifications of the engine used are provided in Table 3.6.

(a) Measurement of calorific value

(b) Measurement of viscosity



(c) Measurement of density



(d) Measurement of flash and fire points

Fig. 3.2. Apparatus used for the measurement of fuel properties

Fig. 3.3. Test rig used for experimentation

Table 3.6: Specifications of the engine

Name	Single-cylinder, 4-stroke diesel engine
Engine Make	M/S Kirloskar
Cylinder Position	Vertical
Brake Power	5Hp
Speed	1500RPM
Bore	80mm
Stroke	110mm
Compression ratio	17.5:1
Air Box Orifice Diameter	20mm
Cooling	Water cooled
Starting	Hand Cranking
Dynamometer	Rope brake

3.7. Evaluation of Performance and Emission Parameters:

Load test on a single-cylinder, four-stroke diesel engine has been carried out using diesel fuel and different blends *viz*. B5, B10, B15, B20, B25 to analyze the performance and emission characteristics of the engine. Following procedure was adopted.

- Fuel and lubricating oil levels, water supply to the engine and brake drum were checked before starting the engine.
- Engine was allowed to start at no load condition keeping the 3-way cock in open position so that fuel flows from the tank to the engine filling burette.
- Engine was allowed to pick up rated speed and then coolant flow rate was adjusted.
- Engine was allowed to reach steady state condition after which required readings (time taken for fuel consumption keeping the 3-way cock in closed position, tail pipe emissions) were noted down at no load condition.
- Load was varied from no load condition to full load condition in equal intervals and coolant flow rate was adjusted for all the loads.
- Engine was allowed to reach steady state condition after applying the load and required readings were noted down at all load conditions.

- It was ensured that no fuel was present in the fuel tank before filling the tank with a new fuel blend and the same procedure was repeated for all the fuel blends.
- While taking emissions readings, enough care was taken to compensate the fluctuations and readings were noted once readings were stabilized.
- All the readings were taken thrice and the average of readings was employed for calculations and plotting graphs to minimize the experimental errors.

Chapter 4

PARAMETRIC OPTIMIZATION OF BIODIESEL PROCESS PARAMETERS

This chapter deals with the optimization of process parameters involved in the biodiesel preparation using UCO as feedstock. RSM was employed for the design of experiments, which helped in conducting the experiments with fewer test runs.

4.1. Introduction to Design of Experiments:

Science, engineering, and business study all heavily rely on experimentation and are largely empirical. A scientific method of planning the trial must be used to carry out the experiments as effectively as possible. Design of experiments (DOE) is the process of organizing an experiment to gather relevant data for inferences that have significance. Design of experiments include:

- selection of response variables,
- choice of factors, levels and ranges,
- choice of experimental design, and
- selection of an empirical model.

It is crucial to keep the experimental goals in mind when choosing the style. By entering information about the number of factors, levels, and ranges, interactive statistical software programs can be used to design experiments. For use in carrying out the experiment, these programs typically also offer a worksheet with the randomization of the sequence of the runs. When conducting experiments, careful process tracking is crucial to ensuring the validity of the results.

Experiments with statistically sound design provide a solid foundation for creating an empirical model of the system under study. Model is the mathematical equation that represents the relationship between the key design parameters and the reaction.

Model sufficiency testing and residual analysis are both parts of statistical analysis. To analyse the data and arrive at findings and conclusions, graphical methods and empirical

models are both helpful. To verify the validity of the experiment's findings, additional trials and confirmation testing must be carried out after data analysis

4.2. Introduction to Design Expert:

Stat-Ease Inc. offers a statistical software program called Design-Expert that is devoted to carrying out design of experiments. (DOE). Comparative tests, screening, characterization, optimization, resilient parameter design, mixture designs, and combined designs are all provided by Design-Expert.

Analysis of variance (ANOVA) is used to determine the statistical importance of these variables. The effects of each factor on the intended outcomes are shown graphically, and anomalies in the data are shown graphically. Up to 50 variables can be screened using test matrices provided by Design-Expert.

Calculating the required number of test runs is assisted by a power calculator. To establish statistical significance, ANOVA is offered. A numerical optimizer assists the user in choosing the optimal values for each of the experiment's parameters based on the proven predictive models. To analyse the residuals, Design-Expert includes 11 graphics in addition to text output.

By changing the values of all components simultaneously, the software can determine both the primary effects of each factor and their interactions. A robust statistical software programme with a focus on design of experiments (DOE), multivariate analysis, and optimisation is called Design Expert.

4.3. Features of Design Expert:

Design Expert software is an effective tool for data analysis and experimental design due to its extensive statistical features and interface when compared to other statistical softwares. They are briefly explained below.

4.3.1. Design Expert is specifically designed for DOE:

The only piece of software devoted exclusively to DOE is Design Expert. It enables users to quickly build and analyse DOE experiments using a variety of designs including factorial, response surface, mixed, and Taguchi designs.

4.3.2. Advanced visualization capabilities:

Powerful visualisation tools provided by Design Expert make it simple to comprehend complex data sets and improve procedures. It provides contour plots, response surface plots, and optimisation plots, for instance, which can be used to see how different variables relate to one another and find the best conditions.

4.3.3. User-friendly interface:

Even for consumers with limited statistical background, Design Expert features a simple to use graphical user interface. Without requiring substantial training, it enables users to generate designs, analyse data, and improve processes quickly.

4.3.4. Comprehensive statistical tools:

A variety of statistical techniques are available from Design Expert, including Taguchi methods, mixture designs, and response surface approach. ANOVA, regression analysis, and data manipulation are just a few of the built-in features.

4.3.5. Integration with other software:

To expand its functionality and let users deal with bigger data sets, Design Expert can be connected with other programmes like JMP and Minitab. In conclusion, Design Expert is a particular statistical software programme with a user-friendly interface that focuses on DOE and optimisation. It also has powerful visualisation features.

4.4. Introduction to Response Surface Methodology:

Response surface methodology (RSM) is a statistical technique that finds the ideal set of input variables to maximise or minimise an interest response. It is used to optimise complicated processes or systems. The foundation of RSM is the concept of fitting a mathematical model to experimental data in order to forecast how the system will react as a function of the input variables. The mathematical model may be expressed as a linear, quadratic, or higher-order polynomial equation, among other shapes.

A series of trial runs are created to apply RSM based on the input variables and the desired response. These tests are carried out, and there is curiosity in the results. The outcomes of

these tests are utilised to create a mathematical model that explains the relationship between the input factors and the response.

Depending on the optimisation goal, after the model has been constructed, optimisation techniques are employed to identify the combination of input variables that yields the highest or least response. These methods include global optimisation algorithms like simulated annealing or genetic algorithms as well as gradient-based methods like the steepest descent or Newton's method.

RSM is frequently used in business, engineering, and academia to optimise a variety of intricate systems and procedures, including chemical reactions, production procedures, and product compositions. By determining the ideal input parameters for the system or process being optimised, it is a potent tool for lowering costs, increasing efficiency, and enhancing quality.

4.5. Central Composition Design:

A type of response surface methodology (RSM) used in experimental design is called central composite design (CCD). It is a commonly used design technique in the robust software programme Design Expert, which can be used to design tests, analyse data, and optimise procedures.

A response surface design is employed in the main composite design to fit second-order models. It is made up of a number of experimental runs that combine various parameters at various degrees. The design has a number of axial points that are situated outside the experimental domain's borders and centre points that are located in the middle of the domain. The response surface is typically optimised using the design, and the best values for the variables that maximise or minimise the response are found.

A user-friendly interface is offered by Design Expert for the creation and analysis of central composite designs. To analyse the data and identify the importance of the components, it provides a wide range of statistical methods. The software offers graphic capabilities as well for visualising the response surfaces and determining the ideal circumstances for the process. Central composite design, as a whole, is a potent

experimental design technique utilised in Design Expert to streamline procedures and raise product quality.

4.6. Experimental Design and Statistical Analysis:

The experimental matrix was constructed using Design Expert, a graphical and statistical software version 13, using the central composite full design of RSM. The design consists of three variables and 20 base runs. The design and outcomes analysis employed the approach described below and included it as one of the five tiers of input factors. A steady stir speed of 400 rpm and a constant reaction temperature of 60°C were maintained throughout the reaction duration. The experiments were completed according to the planned run order, and the outcome, the biodiesel yield, was documented. The details of experimental design with levels of input factors are provided in Table 4.1.

Table 4.1: Numeric Factors and Levels

Input Factor (Units)	Low	Medium	High	- alpha	+ alpha
A: Methanol Ratio (% vol)	18	23	28	14.591	31.409
B: Catalyst Concentration (% wt.)	0.9	1.1	1.3	0.763641	1.43636
C: Reaction Time (minutes)	60	90	120	39.5462	140.454

Response surface regression was used to examine the experimental data acquired by the aforementioned procedure using the polynomial Eq. (1).

$$yiel(Y) = C_0 + \sum_{i=1}^{k} CC_i X_i + \sum_{i=1}^{k} C_{ii} X_i^2 + \sum_{i,i>j}^{k} \sum_{i=1}^{k} C_{ij} X_i X_j + \text{e} \quad \text{..... Eq. (1)}$$

Where Y is yield, i and j are linear & quadratic coefficients respectively. X_i and X_j are independent non-coded variables, C_0 is constant, Ci is regression coefficient, k is quantity of factors optimized and studied within the experiment. C_{ij} is the regression coefficient of the product term, C_{ii} is the regression coefficient of the product term *i*th predictor variable, e is the value that is attributed to uncertainty of Y.

Analysis of variance (ANOVA) and multiple regression analysis were used to check the p-values, model summaries, regression equations, and the percentage contribution of each component. The ideal values of the input factors were discovered using the desirability approach.

Statistical analysis with 95% confidence level was performed using Analysis of variance (ANOVA) and multiple regression analysis to examine the statistical significance of the polynomial model. Based on regression equations, response surface plots were developed to exemplify interactive and main effects of input factors on responses. Finally, desirability approach was employed to evaluate optimal operating conditions that result in higher biodiesel yield.

4.7. Biodiesel Yield:

Based on the experimental design using central composite design of RSM, biodiesel synthesis has been carried out as per the run order and the values of response parameter i.e. biodiesel yield were noted. Table 4.2 presents the details of input factors and response parameter used in the present study. Desirability approach was then adapted to maximize the biodiesel yield through optimization of input factors following the criteria of response surface optimization.

Table 4.2: Experimental Design Matrix

Run Order	A: Methanal Ratio (% v/v.)	B: Catalyst Concentration (% w/w)	C: Reaction Time (Min.)	Biodiesel Yield (ml.)
1	23	1.1	90	230
2	18	0.9	120	218
3	28	0.9	60	225
4	23	1.43636	90	200
5	18	1.3	60	219
6	23	1.1	90	230
7	18	0.9	60	221
8	18	1.3	120	208
9	23	1.1	90	228
10	23	1.1	39.5462	219
11	23	1.1	90	231
12	14.591	1.1	90	221
13	23	0.763641	90	222
14	23	1.1	140.454	218
15	23	1.1	90	229
16	28	0.9	120	229
17	23	1.1	90	232
18	31.409	1.1	90	223
19	28	1.3	120	203
20	28	1.3	60	205

4.8. Analysis and Assessment of the Model:

Regression analysis results show that polynomial models are suitable to analyze experimental data. Quadratic regression models in terms of coded factors for response parameter (Biodiesel yield) was developed and given by the following equation.

Biodiesel Yield (vol. %) = -141.904 + 8.82072*A+ 463.188*B + 0.810329*C - 4.25*AB + 0.0133333*AC - 0.291667*BC + 0.116414*A^2 - 169.986*B^2 -0.00460865*C^2

Here, the terms with positive sign indicate synergistic effect, whereas terms with negative sign indicate antagonistic effect on the responses. This equation can be used to make predictions about the response for given levels of each factor. The equations developed in this study are valid for the following input ranges: Methanol ratio (A): 18 to 28% by volume; Catalyst concentration (B): 0.9 to 1.3% by weight; Reaction time (C): 60 to 120minutes.

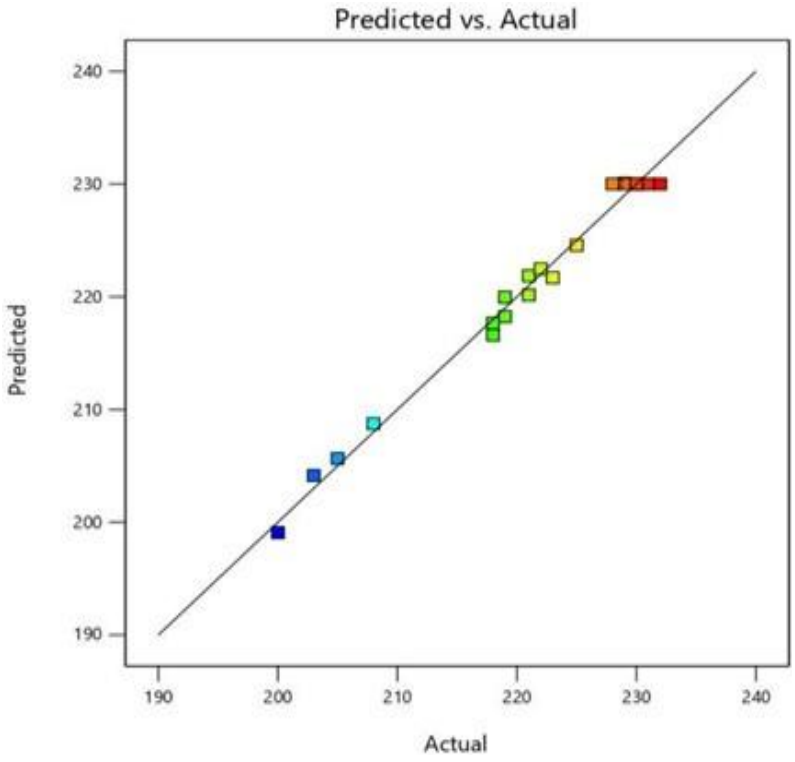


Fig 4.1. Predicted versus actual Bio diesel (%) yield values.

The adequacy and fitness of the models were verified using ANOVA. The plot of predicted and actual values of biodiesel yield as shown in Fig. 4.1 signifies that the model is a good estimate for predicting the actual values of the yield, since the predicted and actual values are very close. Normal probability plot and the analysis of the residuals are shown in Fig. 4.2 and Fig. 4.3 respectively. These plots show no significant deviations from normality. Since there were no discernible patterns in the plot, it could be assumed that the residuals

have a constant variance. The different colours in the plots represent the values of the yield of biodiesel ranging from 200 to 234, where blue is the lowest yield and red is the highest.

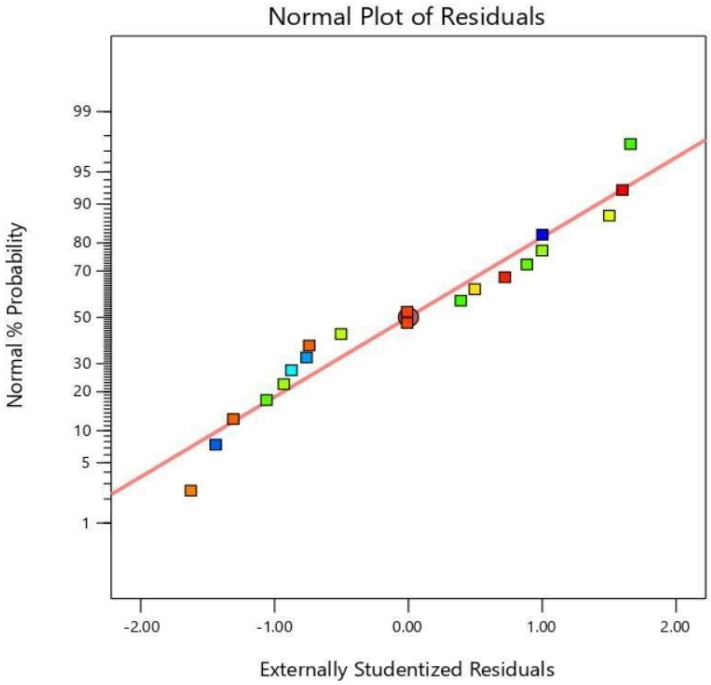


Fig. 4.2. Normal Probability Plot

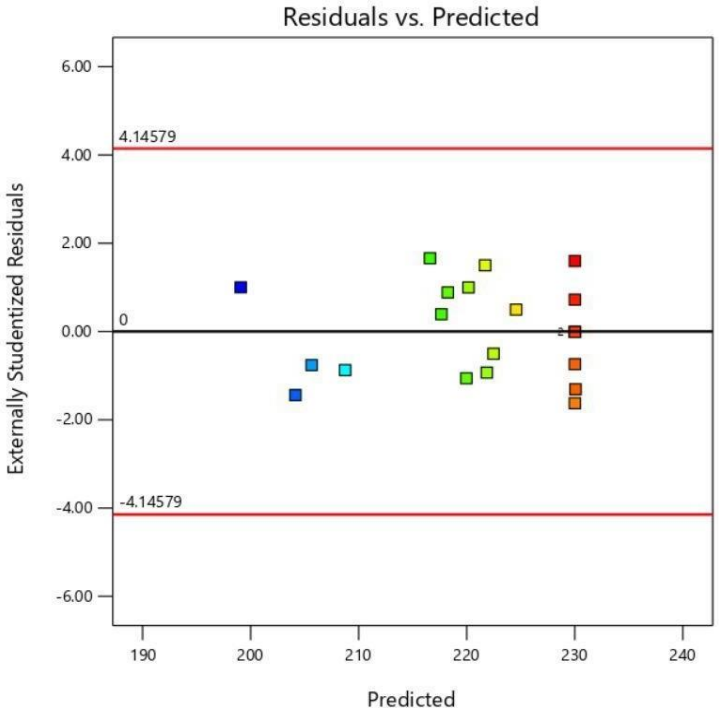


Fig. 4.3. Residuals Plot

The reliability of the created model to establish a correlation between the process variables and the biodiesel yield is validated through ANOVA using response surface methodology. Higher F-value (91.927) and lower p-value (<0.0001) of the model suggest that the model is significant. Further, parameters having p-values less than 0.05 are significant and the parameters having higher p-values are insignificant and hence can be omitted from the model.

Table 4.3 presents the summary of ANOVA results of the model developed in this study. Higher values of R-Squared, adjusted R-Squared, and predicted R-Squared correlation coefficients suggest that the regression model was very significant at the 95% confidence level. This shows that there is just a small variation between the actual and predicted numbers. The chosen model may be able to accurately represent the actual value without the need for a more complex one because the correlation coefficients R-squared and expected R- Squared have strong agreement in values.

Table 4.3: ANOVA results of the model

Source	Sum of Squares	DOF	Mean Square	F-value	p-value
Model	1771.537	9	196.837	91.927	<0.0001
A	0.029657	1	0.0296	0.0138	0.90864
B	660.8325	1	660.832	308.623	<0.0001
C	13.706	1	13.706	6.4013	0.02986
AB	144.5	1	144.5	67.484	<0.0001
AC	32	1	32	14.944	0.00313
BC	24.5	1	24.5	11.442	0.00697
A^2	0.652	1	122.065	57.007	<0.0001
B^2	666.26	1	666.266	311.161	<0.0001
C^2	247.933	1	247.933	115.790	<0.0001
Residual	21.41	10	2.141		
Lack of Fit	11.412	5	2.282	1.1412	0.44413
Pure Error	10	5			
Total Correlation	1792.95	19			

4.9. Individual Effects of Process Variables on Biodiesel Yield:

Perturbation plots show the effects of variation of individual operating parameters on the response parameters. Fig. 4.4 shows the effect of methanol ratio (A), catalyst concentration (B) and reaction time (C) on biodiesel yield. One of the important factors in biodiesel production is the molar ratio. Methanol plays a fundamental role in biodiesel conversion. The yield of biodiesel increases with increase in the methanol ratio and once the maximum point is reached, it begins to decrease due to excess methanol. This could be as a result of the increased methanol addition causing triglycerides to react more quickly increasing the production of biodiesel. If the methanol Concentration to oil is not selected properly, the reaction between the free fatty acids and the catalyst leads to soap formation. Initial rise in biodiesel yield with increase in reaction time can be attributed to the increase in conversion rate of fatty acids over time.

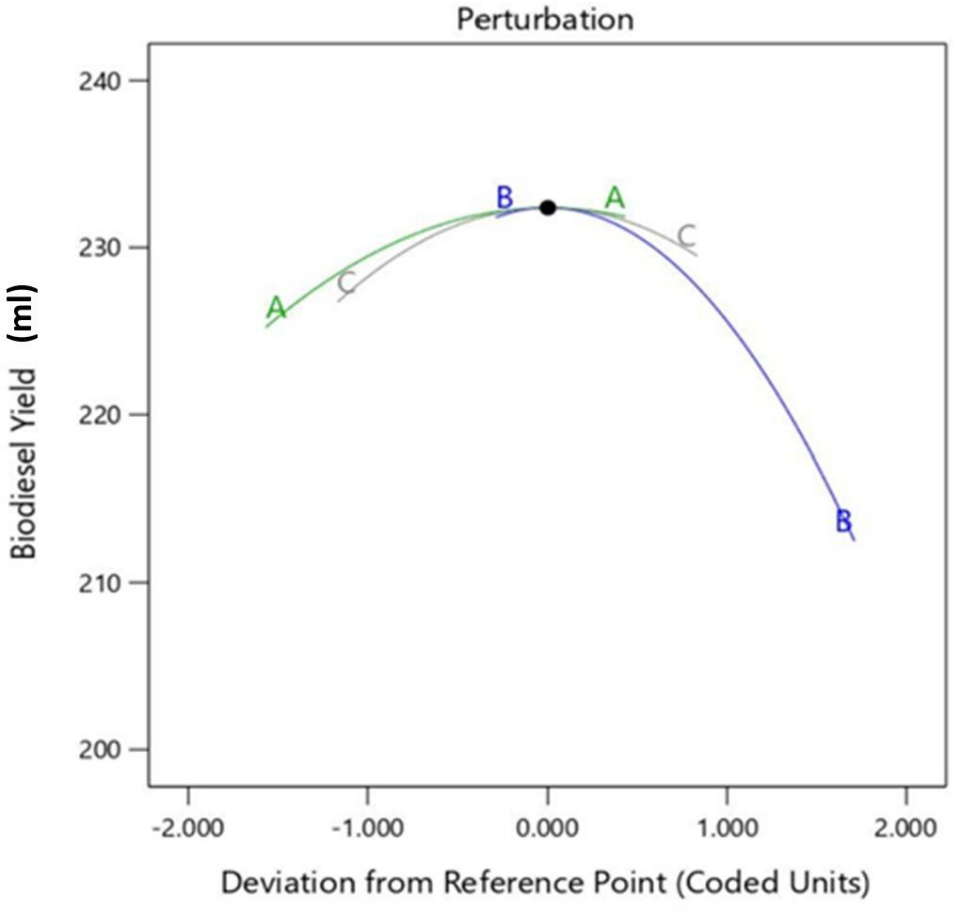


Fig. 4.4. Perturbation Plots Showing the Effects of Process Parameters on Biodiesel Yield

Since the transesterification reaction is reversible by nature, it is discovered that the yield of biodiesel falls as the amount of catalyst is increased. Thus, additional alcohol is supplied to ensure the complete conversion of triglycerides. As the catalyst concentration rises, the biodiesel yield falls. In general, more catalyst would result in higher biodiesel conversion, but too much catalyst would speed up the transesterification of free fatty acids, produce more water in a shorter amount of time, and deactivate the acidic hydroxyl groups.

4.10. Effects of Interaction Parameters on Biodiesel Yield:

The interaction effect of the methanol ratio (% vol) and catalyst concentration (% wt.) on biodiesel yield is shown in Fig. 4.5keeping the reaction time fixed at optimized value. It can be seen from the figure that the value of the methanol-to-oil ratio leading to the highest

yield is in the range of 22 to 26 % by volume and that of catalyst concentration is between 0.95 to 1.15 % by weight for the optimum reaction time.

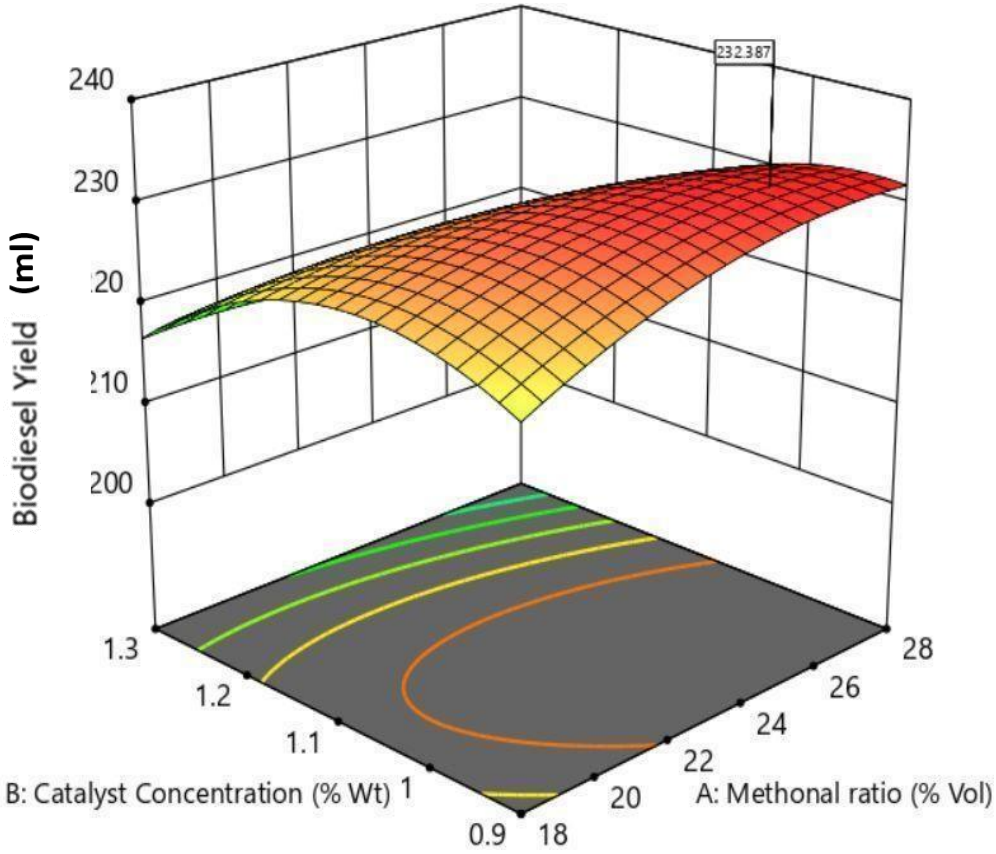


Fig. 4.5. Effect of Interaction of Methanol ratio (% vol) and Catalyst Concentration (% wt.) on Biodiesel Yield.

Interaction effect of methanol ratio and reaction time is shown in Fig. 4.6.It can be observed that the biodiesel yield rises with increase in methanol ratio up to 25% by volume and reaction time in the range of 80-100 minutes for the optimum catalyst concentration. Any further increase in either of the parameter does not contribute to rise in the biodiesel yield.

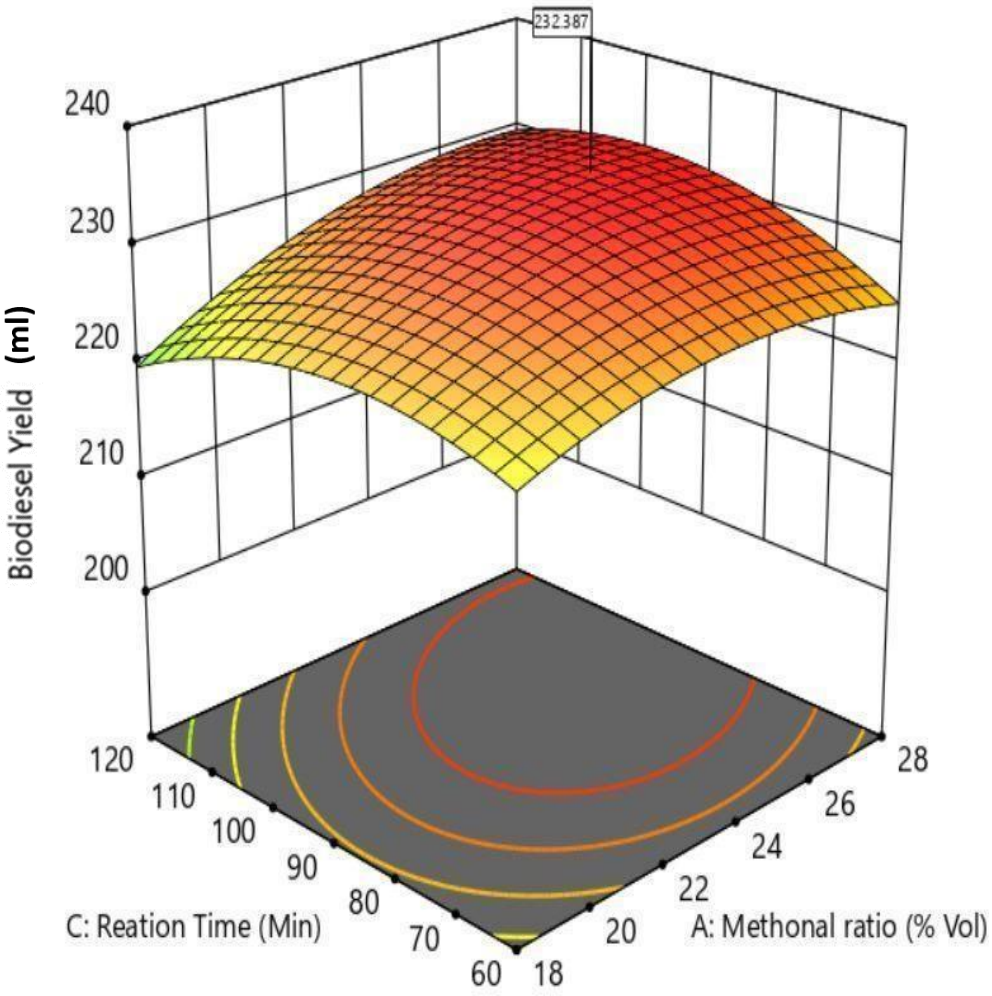


Fig. 4.6. Effect of Interaction of Methanol ratio (% vol) and Reaction Time on Biodiesel Yield

The interaction effects of catalyst concentration and reaction time are shown in Fig. 4.7.It is evident from the figure that the biodiesel yield increases slightly with increase in catalyst concentration up to 1.05% by weight and reduces significantly thereafter. Further, an optimum yield of biodiesel could be observed with the reaction time in the range of 80-100 minutes and reduction in yield with too less and very high reaction times.

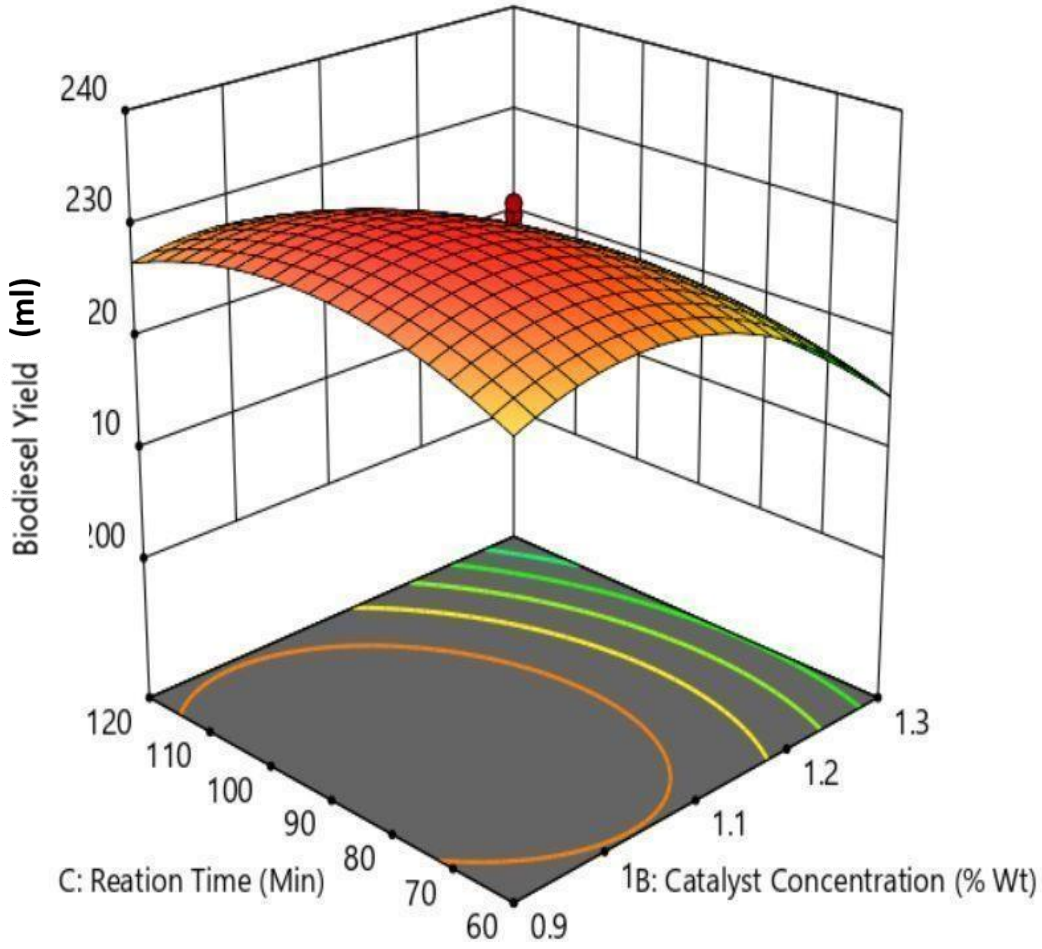


Fig. 4.7.Effect of Interaction of Catalyst Concentration (% wt.) and Reaction Time (Min) on Biodiesel Yield.

4.11. Desirability Approach:

One of the key features of Design-Expert is the desirability function approach, which allows users to set target values for multiple response variables and optimize the factor settings to achieve these targets. In Design-Expert, the desirability approach is implemented through the use of a desirability function, which is a mathematical expression that combines multiple response variables into a single value that represents the overall desirability of the experimental outcome. The desirability function can be customized by the user to reflect their preferences for each response variable, and different weighting schemes can be used to adjust the importance of each variable in the overall calculation.

Once the desirability function is defined, Design-Expert can be used to optimize the factor settings by searching for the combination of factors that maximizes the desirability function. This can be done using a range of optimization algorithms, including response surface methodology (RSM) and genetic algorithms.

4.12. Optimization and Confirmatory Testing:

To obtain the optimal parameters that result in higher biodiesel yield, desirability approach was adapted with a goal of maximizing the biodiesel yield. A solution with highest desirability predicted by the optimization model was selected. Optimization results showed that biodiesel yield of 232.387 ml. (92.95%) could be obtained by employing catalyst concentration of 0.958 (Wt. %), methanol ratio of 25.83 (Vol. %), and a reaction time of 94.96 minutes with desirability value of 0.925 i.e. 92.5 % chances of obtaining the result. Optimization results are provided in Table 4.4 and the details of optimization are shown in Fig. 4.8 and Fig. 4.9.

Table 4.4: Optimized values of process parameters

Solution Number	Methanol Ratio (v/v)	Catalyst Concentration (w/w)	Reaction Time (min.)	Biodiesel Yield (ml)	Desirability
1	25.84694	0.9577611	94.9950	232.3865	0.925330

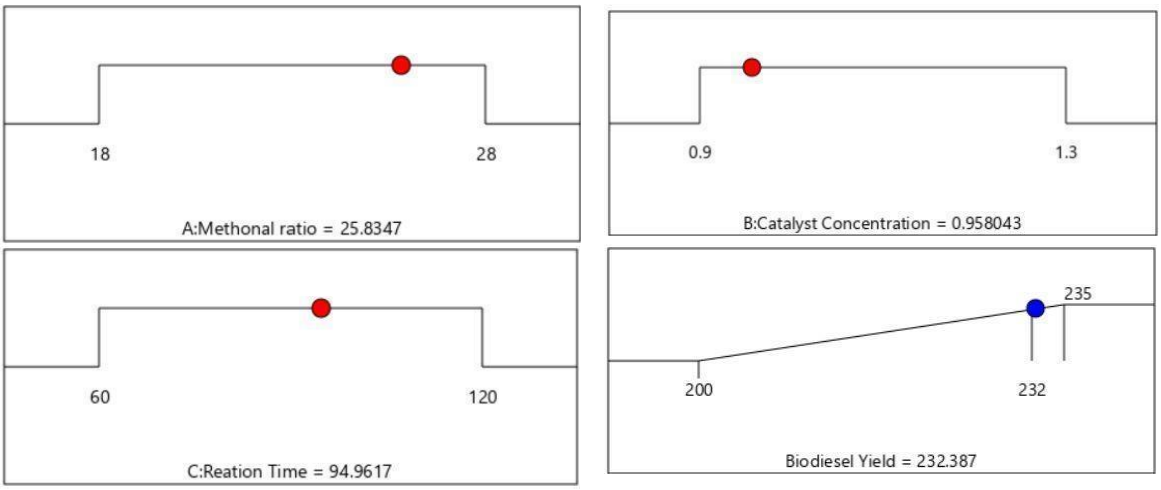


Fig. 4.8. Optimized values of Process and Response Parameters

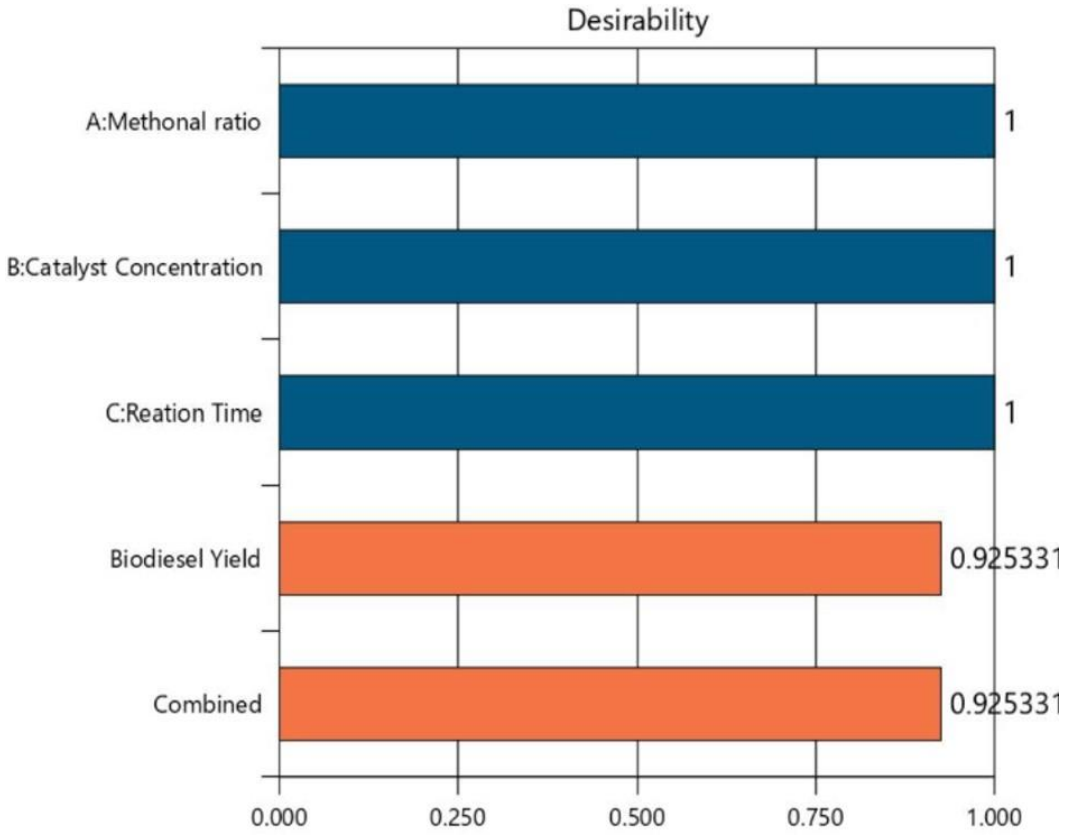


Fig. 4.9. Desirability Values of Parameters

Confirmatory tests were conducted using the optimal values of process parameters that were obtained and the results are provided in Table 4.5.With the use of rounded-off values of process parameters close to the optimized values (methanol ratio of 26 Vol. %, catalyst concentration of 0.96 Wt.%, reaction time of 95 minutes), a biodiesel yield of 231.33 ml.(average of 3 tests) was obtained which is in very close agreement with the predicted yield value by the model developed.

Table 4.5: Confirmatory test results showing the validation of the model

Analyzed Parameter	Predicted Mean	Std. Dev.	95% PI low	95% PI high	Confirmatory Test Result (Average of three tests)
Biodiesel Yield	232.381	1.463	230.035	234.727	231.33

4.13. Summary:

Optimization study was carried out to optimize used cooking oil based biodiesel production process parameters using RSM. Empirical model was developed to predict the biodiesel yield and optimization analysis was performed to optimize response parameter. Confirmatory tests were conducted at design conditions (nearest possible) to validate the results predicted by the model. The following are the main conclusions drawn based on the present investigation.

(i) The values of regression statistics goodness of fit (R^2), the goodness of adjusted and predicted (R^2 adj. and R^2 pred.) values indicate that the data fitted very well in the regression model and the variation in the response parameters can be estimated convincingly by the model in the range studied.

(ii) The errors between predicted and experimental outcomes were found to be well within the acceptable limit (<5 percent) indicating the adequacy of models developed.

(iii) The following ranges of operating parameters are recommended for obtaining higher biodiesel yield: reaction time (90 to 100 min.), methanol-to-oil ratio (24 to 26% v/v), and catalyst concentration (0.95 to 1.05% w/w).

Chapter 5

PERFORMANCE AND EMISSION PARAMETERS

Experimentation on a single-cylinder, water cooled, four-stroke diesel engine has been carried out using petro-diesel and biodiesel/diesel blends following the standard test procedure to evaluate various performance and emission parameters and the results are discussed below:

5.1. Performance Characteristics:

5.1.1. Variation of brake specific fuel consumption with load:

The variation in brake specific fuel consumption with engine load for diesel fuel and waste cooking oil biodiesel is shown in Fig. 5.1. Brake Specific fuel consumption for biodiesel blends is higher than diesel and is increased with the proportion of biodiesel in the fuel. When using biodiesel blends, diesel engine uses more fuel to operate at same power as diesel fuel. This rise in brake specific fuel consumption has been attributed to the lower calorific value of biodiesel, and perhaps higher density when compared to diesel fuel.

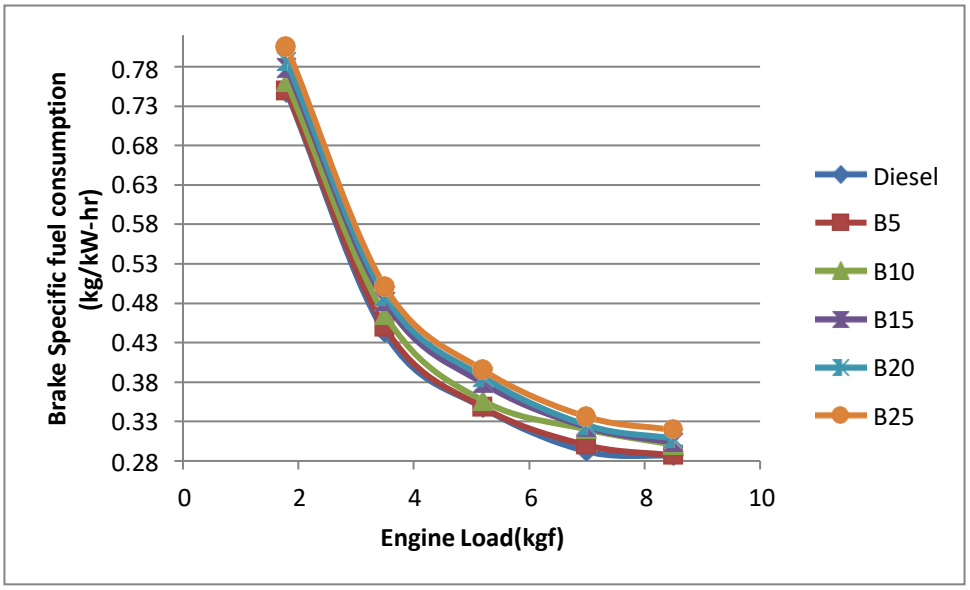


Fig. 5.1. Variation of Brake Specific Fuel Consumption with Brake Power

5.1.2. Variation of brake thermal efficiency with load:

Brake thermal efficiency tells how efficiently the heat is converted to mechanical work. The thermal efficiency of diesel engine fuelled with diesel and waste cooking oil blends is shown in Fig 5.2. Lower brake thermal efficiency of engine has been observed for waste cooking oil biodiesel than diesel fuel. Biodiesel has lower calorific value due to the presence of oxygen but has high specific gravity compared to conventional diesel fuel. Various studies have been published comparing the brake thermal efficiency of used cooking oil biodiesel and it blends to diesel fuel.

Some reports showed lower thermal efficiency for used cooking oil biodiesel and its blends compared to diesel fuel, other authors find similar thermal efficiency to diesel fuel when using biodiesel and/or its blends. The waste cooking oil biodiesel's lower thermal performance as compared to diesel may be attributed by its higher viscosity.

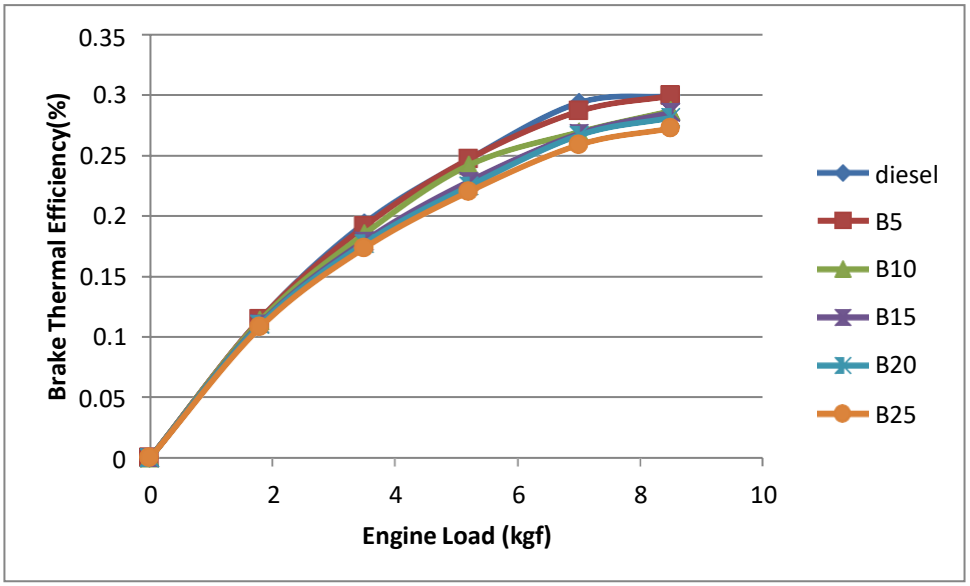


Fig. 5.2. Variation of Brake Thermal Efficiency with Engine Load

5.2. Emission Characteristics:

5.2.1. Variation of CO Emissions with load:

The use of waste cooking oil and/or its blends generally reduces CO emissions according to most of the literature reviewed. Fig. 5.3 shows the variation in CO emissions with increasing load on engine for different fuels/blends. CO emissions decreased with

increasing engine brake power at lower loads and then increased at higher loads. More oxygen molecules and less carbon content present in biodiesel blends led to better combustion than diesel fuel resulting in less CO emissions.

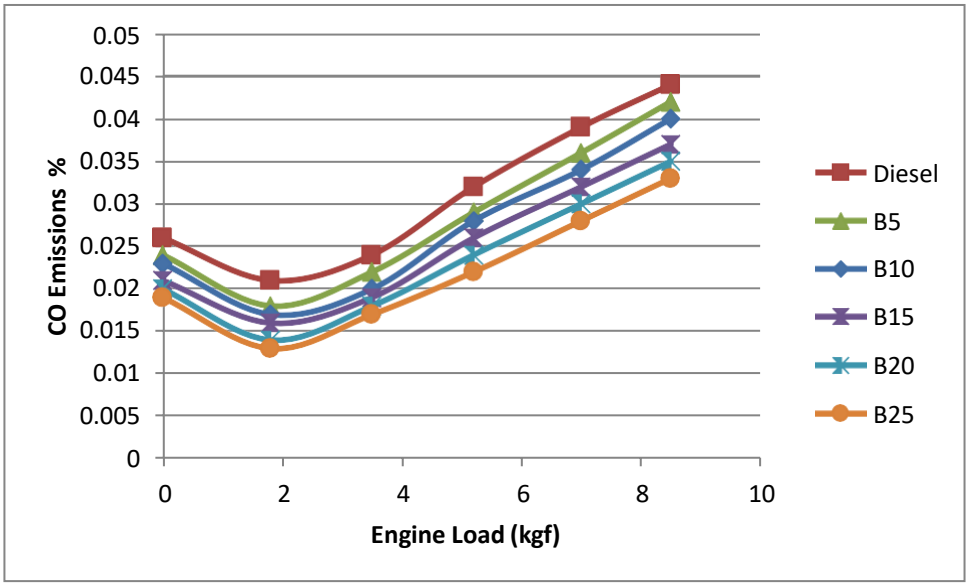


Fig. 5.3. Variation in CO emissions with Engine Load

5.2.2. Variation of HC Emissions with load:

The variation in HC emissions with increasing engine load is depicted in Fig. 5.4 for different fuels. HC emission increases with increase of engine load. Biodiesel blends with diesel fuel produced lower HC emissions at all engine loads compared to diesel fuel. Increase in HC emission for diesel is mainly due to incomplete combustion. When the percentage of biodiesel in the fuel blend increased, HC emissions dropped as a result of better combustion caused by the higher oxygen content of biodiesel. Some researchers have obtained up to 50% unburnt hydrocarbon emissions reduction when using pure biodiesel, independent of where it comes from, and it has been determined that the source has no effect on HC emissions.

5.2.3. Variation of CO_2 Emissions with load:

The variation in CO_2 emissions with increasing engine load is portrayed in Fig. 5.5 for different fuels. CO_2 emission is more for biodiesel and its blends than that for diesel fuel. The rising trend of CO_2 emission with engine load was due to the higher fuel consumption

rate as the load increased. CO_2 emissions are increased with the increase in blend proportion and were due to higher oxygen content in biodiesel blends. B25 have lower CO emission than diesel because of the higher oxygen content leading to higher temperature in the combustion chamber and resulting in more conversion of CO to CO_2.

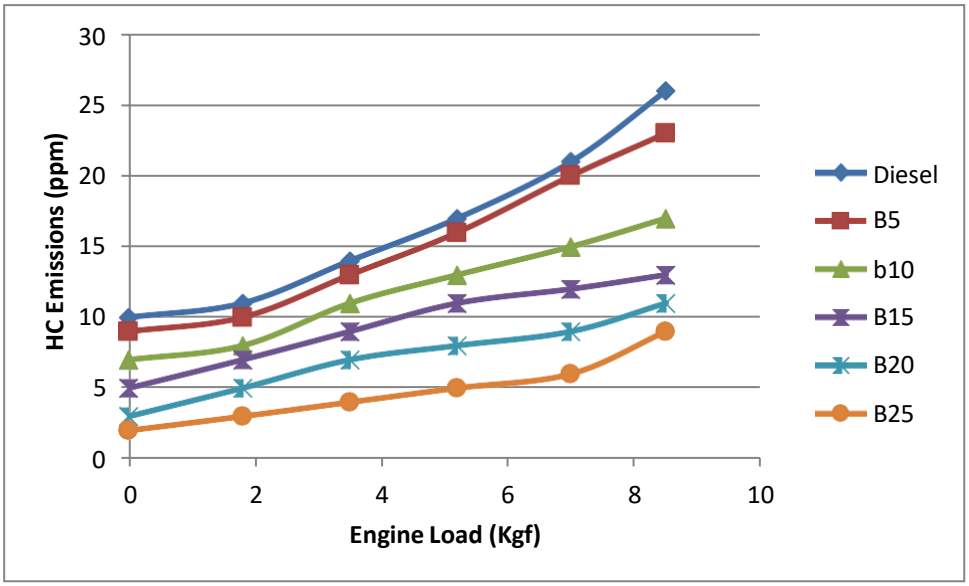


Fig. 5.4. Variation in HC emissions with Engine Load

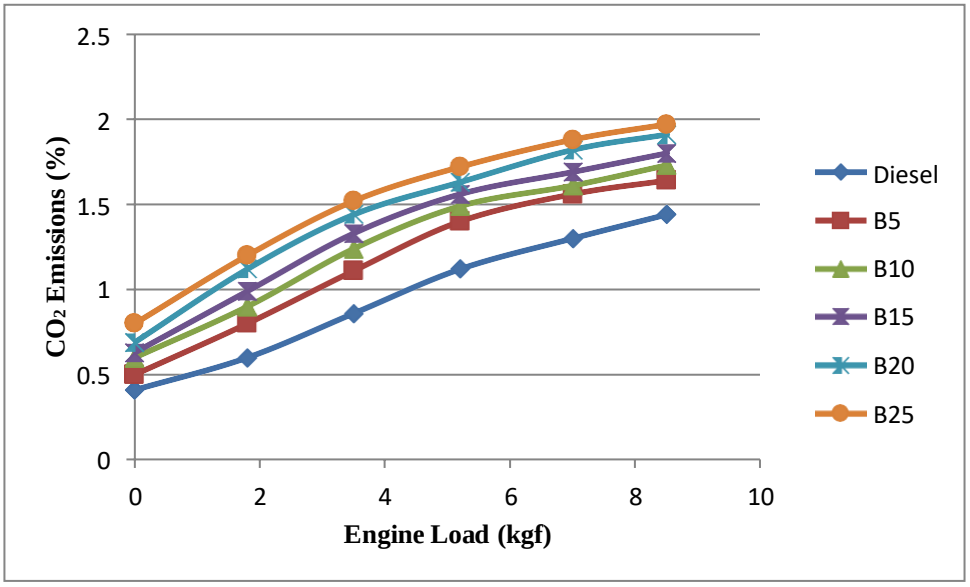


Fig. 5.5. Variation in CO_2 emissions with Engine Load

Chapter 6

CONCLUSIONS AND FUTURE SCOPE

6.1. Conclusions:

The present study dealt response surface optimization of biodiesel preparation process parameters. Optimized biodiesel was tested for its properties. Single cylinder four stroke diesel engine was run on biodiesel and its blends. Performance and exhaust emissions were measured at different engine loads at a constant engine speed of 1500 rpm. The results showed that used cooking oil may be transesterified into biodiesel, resolving the oil disposal difficulties in the restaurant business and converting waste into profit. When the catalyst is too high, soap and gel may form, preventing the separation of the ester layer. With a methanol concentration ratio (26 vol. %) and NaOH as catalyst (0.95Wt. %), biodiesel yield of 93% was reached in 95 minutes at constant stir speed of400 rpm and 60^{o}C temperature. The optimized biodiesel was test for its properties and were compared to ASTM-D6751 and EN-14214 and was found out to be within the limits mentioned. So, no engine modification should be required if diesel–biodiesel blend (with lower per cent of biodiesel) is used. Decrease in CO and HC emissions was observed at all engine loads compared to diesel fuel. It was found that CO_2 emissions were higher for biodiesel and its blends than for diesel fuel. It is also observed that with the increase in the percentage of biodiesel in the blends brake specific fuel consumption increases and brake thermal efficiency of engine decreases slightly.

6.2. Future Scope:

In the present study optimization of biodiesel preparation parameters was carried out and performance and emissions testing was carried out to find the variation in CO, HC and CO_2 emissions with the application of biodiesel/diesel blends. Further investigations can be carried out in the following aspects:

- Use of pure biodiesel (B100) and higher biodiesel fraction in blends.
- Smoke analysis and NO_x analysis for all the fuels.

- Effects of injection timing and injection pressures for different fuel blends.
- Application of Exhaust Gas Recirculation.
- Use of ternary blends and port fuel techniques.

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APPENDIX

Table A1: Performance and emission parameters of CI engine fuelled with Diesel (D100)

DIESEL								
S.no	Load on the brake drum (W-s) (kgf)	Time for 10 cc fuel consumption (sec)	FC (kg/hr)	B.P (kW)	B.S.F.C (kg/kW-hr)	CO (%)	CO_2 (%)	HC (ppm)
1	0	79.5	0.3763	0	-	0.026	0.41	10
2	1.8	67.4	0.44386	0.594	0.74723	0.021	0.6	11
3	3.5	58.63	0.51025	1.155	0.44178	0.024	0.86	14
4	5.2	50.22	0.5957	1.716	0.34714	0.032	1.12	17
5	7	44.36	0.67439	2.31	0.29194	0.039	1.3	21
6	8.5	37.22	0.80376	2.805	0.28655	0.044	1.44	26

Table A2: Performance and emission parameters of CI engine fuelled with 5% biodiesel (B5)

B5								
S.no	Load on the brake drum (W-s) (kgf)	Time for 10 cc fuel consumption (sec)	FC (kg/hr)	B.P (kW)	B.S.F.C (kg/kW-hr)	CO (%)	CO_2 (%)	HC (ppm)
1	0	78.81	0.38051	0	-	0.024	0.5	9
2	1.8	67.4	0.44493	0.594	0.74903	0.018	0.8	10
3	3.5	57.8	0.51882	1.155	0.4492	0.022	1.11	13
4	5.2	50.22	0.59713	1.716	0.34798	0.029	1.4	16
5	7	43.28	0.69288	2.31	0.29995	0.036	1.56	20
6	8.5	37.22	0.8057	2.805	0.28724	0.042	1.64	23

Table A3: Performance and emission parameters of CI engine fuelled with 10% biodiesel (B10)

B10								
S.no	Load on the brake drum (W-s) (kgf)	Time for 10 cc fuel consumption (sec)	FC (kg/hr)	B.P (kW)	B.S.F.C (kg/kW-hr)	CO (%)	CO_2 (%)	HC (ppm)
1	0	76.86	0.3911	0	-	0.023	0.6	7
2	1.8	66.6	0.45135	0.594	0.75985	0.017	0.9	8
3	3.5	55.91	0.53765	1.155	0.4655	0.02	1.24	11
4	5.2	49.16	0.61147	1.716	0.35634	0.028	1.49	13
5	7	40.66	0.7393	2.31	0.32004	0.034	1.61	15
6	8.5	35.66	0.84296	2.805	0.30052	0.04	1.73	17

Table A4: Performance and emission parameters of CI engine fuelled with 15% biodiesel (B15)

B15								
S.no	Load on the brake drum (W-s) (kgf)	Time for 10 cc fuel consumption (sec)	FC (kg/hr)	B.P (kW)	B.S.F.C (kg/kW-hr)	CO (%)	CO_2 (%)	HC (ppm)
1	0	76.56	0.39404	0	-	0.021	0.63	5
2	1.8	65.25	0.46234	0.594	0.77836	0.016	0.99	7
3	3.5	54.18	0.55681	1.155	0.48209	0.019	1.33	9
4	5.2	46.47	0.64919	1.716	0.37832	0.026	1.56	11
5	7	40.47	0.74544	2.31	0.3227	0.032	1.69	12
6	8.5	35.5	0.8498	2.805	0.30296	0.037	1.8	13

Table A5: Performance and emission parameters of CI engine fuelled with 20% biodiesel (B20)

B20								
S.no	Load on the brake drum (W-s) (kgf)	Time for 10 cc fuel consumption (sec)	FC (kg/hr)	B.P (kW)	B.S.F.C (kg/kW-hr)	CO (%)	CO_2 (%)	HC (ppm)
1	0	75.99	0.39842	0	-	0.02	0.69	3
2	1.8	64.85	0.46686	0.594	0.78596	0.014	1.12	5
3	3.5	53.6	0.56485	1.155	0.48905	0.018	1.44	7
4	5.2	45.68	0.66278	1.716	0.38624	0.024	1.63	8
5	7	40.31	0.75108	2.31	0.32514	0.03	1.82	9
6	8.5	35	0.86503	2.805	0.30839	0.035	1.91	11

Table A6: Performance and emission parameters of CI engine fuelled with 25% biodiesel (B25)

B25								
S.no	Load on the brake drum (W-s) (kgf)	Time for 10 cc fuel consumption (sec)	FC (kg/hr)	B.P (kW)	B.S.F.C (kg/kW-hr)	CO (%)	CO_2 (%)	HC (ppm)
1	0	75.69	0.4019	0	-	0.019	0.8	2
2	1.8	63.63	0.47808	0.594	0.80484	0.013	1.2	3
3	3.5	52.59	0.57844	1.155	0.50081	0.017	1.52	4
4	5.2	44.86	0.67811	1.716	0.39517	0.022	1.72	5
5	7	39.2	0.77602	2.31	0.33594	0.028	1.88	6
6	8.5	33.97	0.8955	2.805	0.31925	0.033	1.97	9