Experimental investigation on Biodiesel Production through Microwave Irradiation

A Thesis submitted to the Delhi Technological University, Delhi in fulfilment of the requirements for the award of the degree of

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Mechanical Engineering

by

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DECLARATION

I hereby declare that the thesis entitled **"Experimental investigation on Biodiesel Production through Microwave Irradiation"** is an original work carried out by me under the supervision of **Dr. Amit Pal**, Professor, Mechanical Engineering Department, Delhi Technological University, Delhi. This thesis has been prepared in conformity with the rules and regulations of the Delhi Technological University, Delhi. The research work reported and results presented in the thesis has not been submitted either in part or full to any other university or institute for the award of any other degree or diploma.

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CERTIFICATE

This is to certify that the work embodied in the thesis entitled **"Experimental investigation on Biodiesel Production through Microwave Irradiation"** by **Naveen Kumar Garg, Roll No. 2K16/Ph.D.ME/15** as a Ph.D. scholar in Mechanical Engineering Department, Delhi Technological University, Delhi is satisfactory and meets the Ph.D requirements. He has completed his thesis work under my guidance and supervision. The matter embodied in this research work has not been submitted earlier for the award of any degree or diploma etc. to the best of my knowledge and belief.

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Abstract

As the levels of pollution rise, so does interest in investigating sustainable energy sources. Governing authorities throughout the world are embracing more dependable and sustainable techniques of dealing with energy than just using non-renewable energy sources, with biofuel driving the investigation farther. The biodiesel derived from cottonseed and other biodiesel sources may eventually replace depleting diesel reserves. On the other hand, there is not enough of awareness regarding the process for producing biodiesel using microwave irradiation methodology as well as its combustion impact and performance analysis in compression ignition (CI) engines.

To begin with, a domestic microwave oven was modified to keep the entire process economically viable. Three different feedstocks which are available in sufficient quantity in the surroundings were chosen, and its process parameters were defined and improved using the statistical software Minitab 17. It was found during the literature review that usage of catalysts also affects the biodiesel yield and also the ecology of the environment therefore a catalyst prepared from aluminium foil was used to process the waste cooking oil for obtaining the biodiesel from it.

Design of experiment (DoE) has provided different combinations to reduce the number of trials and maximise the yield. Different levels of input power (200W, 300W, 400W and 500W), methanol: oil molar ratio (3:1 to 15:1) concentration of catalyst, duration of reaction, speed of the turntable (10 to 40 rpm) as well as speed of the cooling fan (800.1000,1200,1500 rpm) of microwave reactor were selected as useful variable process parameters.

The pilot study of biodiesel manufacturing revealed that a high-quality yield of 99.5 % was obtained in 11 minutes with an input power of 500W, methanol: oil molar ratio of 4.5:1, catalyst-1%, turntable and the cooling fan speed of 40 rpm and 1500 rpm respectively. During the conversion of cottonseed biodiesel CBD with microwave (MW) methodology from cottonseed oil, there was a saving of around 56% in the duration of the reaction or the time taken including a yield nearly 63% higher than the conventional magnetic stirrer (MS) process.

For kalonji oil, parameters included system wattage, reaction time, stirrer speed, methanol to oil molar ratio and catalyst loading. The optimum values for different parameters were found to be 500W, 20 minutes, 30 rpm, 9:1 and 1.5(wt%) respectively, for this study. The kalonji biodiesel maximum yield was recorded as 95.47 percent.

The prepared catalyst $NaOH/\gamma\text{-}Al_2O_3$ (3% by wt. of oil) has been used with waste cooking oil (WCO) and methyl alcohol having a molar ratio of 15:1 for a reaction time of 1 hour in microwave based processor. The biodiesel yield has been recorded as 98%.

Finally, different blends (B10, B20 and B30) of cottonseed biodiesel and diesel were prepared and tested to run a multi cylinder CI engine. The examination of blends of cottonseed biodiesel in CI engine revealed that this biodiesel prepared through microwave assistance can be used suitably without any modification in engine.

Test run indicated that the brake specific fuel consumption (BSFC) of blended fuel increased by about 15% in comparison to fossil –diesel at lower loads. As the load percentage is raised the BSFC fell down consistently however, found to be higher than fossil –diesel owing to higher viscosity, higher density and lower heating value of the blended fuel as compared with diesel.

The brake thermal efficiency (BTE) of biodiesel blends observed to be slightly lesser as compared to diesel and blended biodiesel fuel. However, the proportion between the drop in BTE and the rise in biodiesel content is not equal. A 20-25% reduction in concentration of carbon monoxide and hydrocarbon (THC) emissions has been observed when operating with the blends of biodiesel. The extra oxygen present in the biodiesel fuel, boosting combustion in the cylinder, may be the cause of the decrease in CO and THC emissions from biodiesel and its mixes.

It is found to be one of the promising fuel for CI engines because utilizing this fuel all the emissions were reduced to a great extent except the nitrogen oxide (NO) emission. Which could have been controlled by some other methods such as intake air temperature management, controlling with injection timing and exhaust gas recirculation etc.

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List of Abbreviations and Nomenclature

AKOME	Acorn Kernel Oil Methyl Ester
ANOVA	Analysis of Variation
Approx.	approximately
BMEP	Brake Mean Effective Pressure
BP	Brake Power
BSEC	Brake Specific Fuel Consumption
BSFC	Brake Specific Energy Consumption
BTE	Brake Thermal Efficiency
CBD	Cottonseed Biodiesel
CCD	Central Composite Design
CFPP	Cold Filter Plug Point
CH_3OH	Methyl alcohol
CI	Compression Ignition
CN	Cetane Number
CP	Cloud Point
CRDI	Common Rail Direct Injection
CSO	Cottonseed oil
CV	Calorific Value
DOE	Design of Experiment
ECU	Electronic Control Unit
EGR	Exhaust Gas Recirculation
FAMEs	Fatty Acid Methyl Esters
FFA	Free Fatty Acid

FID	Flame Ionisation Detector
g/cm^3	gram/ centimeter^3
g/ml	gram/ millilitre
GC	Gas chromatography
GHG	Greenhouse Gas Emissions
IMEP	Indicated Mean Effective Pressure
INR	Indian National Rupee
KOH	Potassium Hydroxide
KOMe	Potassium methoxide
kWh	kilo watt hour
LDL	low-density lipoprotein
mm^2/s	Millimetre^2/ second
MMT	Metric Million Tonne
MS	Magnetic Stirrer
MW	Microwave
NaOH	Sodium Hydroxide
NaOMe	Sodium Methoxide
NDIR	Nondispersive Infrared
psi	Pound/ inch^2
RPM	Revolutions Per Minute
RSM	Response Surface Methodology
S/N	Signal to Noise
SCMH	Synchronously Chilling and Heating by Microwave
SI	Spark Ignition

TAG	Triacylglyceride
TDC	Top Dead Centre
THC	Total Hydrocarbon
USD	US Dollar
v/v	volume/ volume
WCO	Waste Cooking Oil
WHO	World Health Organisation
wt	weight
XRD	X- Ray Diffraction
ρ	Density

CHAPTER 1

INTRODUCTION

1.1. Contextual

In present scenario, diesel engine plays an important role in transportation, generation of power, various industries, agriculture and marine applications. Thus, fossil -diesel fuel has been used to a great extent in every single place as first choice to be used as source of fuel to produce the required power. It is mostly owing to the lower cost of fossil -diesel and its higher thermal efficiency. Although, the excessive burning of diesel also resulted in harmful impacts on environment; there happen to be dire necessity for unconventional alternative fuels that happen to be equally suitable for compression ignition (CI) engines. These proposed unconventional alternative fuels must have comparatively lesser harmful impacts on environment due to their burning, and, they must be procured from inexhaustible sources such as produced from various crops. Such unconventional alternative fuels may enjoy the status of becoming more favourite now a days. Culinary oils are obtained from renewable energy source with energy content reasonably equivalent to fossil -diesel in them. In 1885, the inventor of diesel engine Rudolf Diesel used peanut oil as fuel for CI engine[1]. During emergency or situation of crisis in the decade of 1930s and 1940s, vegetable oils replaced fossil –diesel as main source of energy. Now, the focus has been transferred on the use of vegetable oil derived biodiesel fuel[2].

Numerous types of vegetable oil as well as biodiesel have actually put to the test or experimented in CI engines powered by the fossil –diesel, on certain occasions. The main advantage behind promoting the use of vegetable oils and biodiesel includes reduction in greenhouse gases, plummeting a country's dependency on imports of fossil fuel as well as the different agriculture crops of any country will be getting additional new buyers, furthermore higher lubricity of these fuels lessens the requirement of friction reducing agents. The acceptance of blend of biodiesel as source of fuel in locomotive industry is another reason behind their bright future projections [3].

Approximately, there are more than 350 plant species [4][5] whose seeds can be used to harvest unfinished vegetable oils, out of which only jatropha, sunflower, soya bean, cottonseed, rapeseed[6] and thumba[7] oils ought to be potential choice for obtaining biodiesel fuels for diesel engines.

1.2. Motivation

The soaring prices of petrol/Diesel in recent years cause disturbance in budget and make things difficult to work out on individual basis. Though Government of India put their best efforts to tackle the situation of inflation through various measures by working on its international policies and avoid reaching critical situations, but still the heat of petrol/diesel is felt by my fellow Indians in terms of hyperinflation. People are certainly determined to improve the current scenario. It is well known fact that India has poor reserves of oil, but vast agricultural land and produce. To alleviate the problem related to oil imports, production of indigenous oil is a possible solution. The indigenous production of oil may reduce the existing need of oil import to some extent. For this reason, it was decided to shift focus on biodiesel as a strong contender. Although, since its inception, the main lacuna in absorbing biodiesel is its production cost, which is quite high due to low production yield. Therefore, this work revolves around the development of process which may enhance the production yield and bring down the overall production cost.

1.3. Need for alternative fuels

Due to rapid urbanization and industrialization there is sharp decrement in fossil fuel deposits[4].The key motivations for development and generation of the different fuels are given as under:

1.3.1. Depleting oil reserves

In 2011-21, India has recorded an increase of 2.9% in primary energy consumption per capita whereas, for the whole world it appeared to be 0.2%. Total liquid fuels (oil) consumption for India remained 4954 thousands of barrels per day and there was an increase of 3.5% and that contributed to 5.1% of the total liquid fuel consumption of the world in the same decade [8]. The gasoline and diesel are obtained from crude oil, which is driven out from the earth’s crust. However, the quantity of petroleum inside the earth is not unlimited and these storages of the oil are diminishing at a very high pace. In very near future, a day will come when these pools would be absolutely finished, when that happened there shall be neither gasoline nor diesel remain left to operate the industries, automobiles etc. Hence, necessity of an alternative fuel may decrease the dependence on fossil fuels. Considering a nation such as India, around 80% of the total requirement of the fuel is being imported from different countries, it is probably among the fundamental contributors of having a large trade asymmetry. It is expected that the energy imports may up by 43.6% in current financial year for India. This rise includes the amount to be spent on procurement of fossil fuels and will be around 36% of the country's total expenditure on imports. With the same rate of procurement (if continues) India will be spending approximately USD 1 trillion by 2026 on it. In the current world scenario, one can't be assured of the consistent supply of crude oil. Also, the unstable rate of the crude oil possibly will worsen the things. Hence, as a consequence of this, it's is of the utmost importance for nations such as India to make the shift towards non-traditional or alternative fuels so they can contribute to the resolution of the aforementioned problems. According to Hubbert peak theory[9], generation starts off slowly, rises faster and faster, next slows down and flattens until it reaches a peak, after which generation declines. In the late stage, generation often enters a period of exponential decline in which the decline become less and less steep. The extraction of petroleum might never reach empty, yet it is going to be exceptionally low.

1.3.2. High Emissions

The atmospheric air consists of complex composition of gases. The aforementioned gases must be available for species to exist on Earth. The stratosphere of the atmosphere gets depleted because of the pollution. Eco-systems of the earth and human life are in a great problem due to the air pollution. If it is to ascertain the two most severe problems related to the air pollution, it can be coined as problems because of the poor atmospheric quality of urban areas and the problems arising due in-house atmospheric air pollution. According to the 2014 WHO report, around 7 million people died worldwide due to atmospheric air pollution in 2012[10].

The term "air pollutants" refers to dangerous substances that exist in the atmosphere and have the potential to harm both people and the environment. These contaminants might be in all the three states of the matter. A pollutant may be created by human or may be due to natural causes. Air pollutants which are created due to natural phenomenon such as ash coming out of the volcanic eruptions are termed as main or primary pollutants. There can be some other examples of the reasons behind the primary pollutant creation like the exhaust coming out because of the burning of the fossil fuels in the internal combustion engines and the gases such as Sulphur dioxide being emitted from the chimneys of the factories. The other classification of the air pollutants, termed as secondary pollutants are defined as the pollutants, that are not directly secreted nevertheless they react with primary pollutants already present in the atmosphere and form new compound. Ozone in ground level is the best example of secondary pollutant. There are some other pollutants which can be both primary as well as secondary, because they are emitted directly in air and sometimes also formed by reaction with primary pollutants. During combustion of the fossil fuels in automobiles a large amount of carbon monoxide (CO), carbon dioxide (CO_2), unburnt hydrocarbons, oxide of nitrogen (NOx) are emitted which has harmful effect on health. To reduce the emission of these harmful gases much cleaner fuels, advances in engine and combustion technology etc. are very much required. The cleaner or alternative fuels must possess good performance as well as low emission characteristics.

1.4. Prospective alternative fuels

A major substitute for fossil fuels used in an internal combustion engine can be inexhaustible type of fuels. Some of the prominent alternative fuels can be described as follows:

1.4.1. Biodiesel

Now a days, biodiesel can be classified as a promising substitute fuel to the fossil diesel. The most environmentally friendly alternative to fossil -

diesel fuel, biodiesel is a less polluted, renewable fuel that may be utilized in compression ignition (CI) engines without requiring any major changes. Oils extracted from numerous vegetable seed appeared to be attractive source to be explored as raw material for production of biodiesel due to their renewability in nature. These oils possess the capability for producing biodiesel at a more widespread basis while also being ecologically beneficial. The benefits of biodiesel in comparison to fossil -diesel fuel are enlisted here as under:

i. Biodiesel serves as a superior lubricating agent, outperforming petroleum diesel by approx. 60%[11].

ii. Since it is free of sulphur and aromatics, biodiesel emits less smoke and particulate matter[12][13].

iii. Biodiesel has a higher cetane number and enhanced anti-knocking characteristic. Thereby, it emits lesser carbon monoxide and hydrocarbons[14].

iv. Biodiesel is renewable, eco-friendly, and non-toxic[12].

The following salient traits distinguish biodiesel from fossil diesel fuel:

i. Energy content (lower heating value (by about 10-12%))[15]

ii. Higher cetane value (typically 50-60) [14][16]

iii. Presence of oxygen content around 11% [17] (oxygen missing in fossil -diesel)

iv. Aromatics content is almost negligible[12][13][14]

v. No sulphur or extremely low sulphur content[13]

vi. Better lubricity[13], [15], [18]

vii. Higher viscosity[12], [15], [18]

viii. Higher freezing temperature (higher cloud point and pour point)[1], [18]–[20]

Biodiesel is produced by the process of trans- esterification of animal fat, vegetable oil, waste cooking oil and many inexhaustible resources, with short chain alcohol such as ethanol or methanol. In the Biodiesel, percentage of oxygen content is more than petroleum- diesel. Consequently, on its use in engines powered by the fossil -diesel, the emissions must be considerably drop down. Because oily substances extracted from vegetable seeds are considered unconventional energy sources due to the fact that they have been generated from agricultural by-products or unprocessed substances generated through the mechanism of photosynthesis fixing of carbon, they have no impact towards overall the surrounding atmosphere greenhouse gas emissions (GHG) levels when burned. Biodiesel has been categorized into a couple of categories based on where it comes from are given as under:

i. Plant based: Jatropha for example is used to produce biodiesel. However, with very high population density and limited food supply, it is imperative to first address the food security problem rather than utilizing agricultural land for planting the plants for oil production.

ii. Culinary oils based: The other important source for producing biodiesel is culinary or eatable oils. But, it should be kept in mind that India is one of the larger importer of these oils. Nobody could hardly contemplate the process of transitioning.

1.4.2. Hydrogen

Hydrogen is the first element of the periodic table and lightest in weight. It rises up in the atmosphere and mixes with other elements easily, which makes procuring hydrogen in its purest form a very cumbersome task. During the phenomenon of combustion of hydrogen gas in the presence of atmospheric air, the hydrogen reacts with oxygen present in atmosphere, it releases energy and gives out water as the exhaust. The main reason behind consideration of hydrogen as a prospective alternative fuel in spark ignition (SI) engines is the high amount of energy it releases on burning. It has the higher calorific value and comparatively cleaner fuel with some drawbacks such as higher NO_x amount and smaller power output[21]. Electric devices and automobiles can be powered with the help of fuel cells. These fuel cells work on the principle of electrochemical reactions in which the chemical energy present in the hydrogen fuel gets converted into electricity without involving any external media. It will take some more time to popularize the use of hydrogen in internal combustion engines. Hydrogen gas is being promoted as a promising fuel for the aviation industry. Hydrogen can be produced in abundance through different electrolysis and biological processes. While using hydrogen in electrochemical devices such as fuel cells they are said to be highly efficient and less polluting.

Since hydrogen gas is not easily available, its use is restricted as an energy carrier. There are very limited places where hydrogen gas is preferred as an independent source of energy. The amount of energy required to generate hydrogen from different sources is said to be higher than the energy received from it by using it as fuel. Moreover, the process of generation itself requires a high skill. In future, hydrogen may get acceptability to be commercially used for passenger vehicles.

1.4.3. Alcohols

Alcohol has been used as a substitute to the conventional fossil fuel since ages. The procurement or synthetization of alcohol is a comparatively easy as it can be achieved chemically as well as biologically, from waste biomass like an agricultural waste. The first four members of the alcohol family i.e. methanol, ethanol, butanol and propanol have had domination over the rest and are chosen to be used as fuels in internal combustion engines owing to their characteristics such as high octane number, easy bendability with gasoline, etc.

Commercially, about two third of the methanol is produced from natural gas, slightly less than one third using coal and remaining from other sources[22]. Biomass can be used as feedstock to harness methanol with the help of chemical processes. Ethanol can be derived from various biological substances using the fermentation technique. It can also be procured from the petroleum products by transformation of ethylene. However, the ethanol acquired from the latter technique contains approximately 5% methanol. Bio-butanol can be procured from the same biomass as for ethanol. However, the process of procurement of bio-butanol is comparatively difficult. The factor that makes bio-butanol a better fuel than ethanol is its higher energy density as compared to ethanol. Furthermore the energy density of bio-butanol is at par with that of gasoline.

As discussed earlier, since the bio-alcohols or alcohols are extracted from biomass or agriculture residue, they contribute to waste management. Another advantage associated with the utilisation of alcohols is its cost-effectiveness. As an independent alternative fuel or a blend with conventional fossil fuel, it does not require any major modification to the engine system. Furthermore, the logistics and handling of alcohols has proven to be simpler than that of fossil fuels.

1.5. Biodiesel synthesizing process

There are several techniques to synthesis biodiesel, which are listed below:

1.5.1. Direct use and blending

It is not suggested to utilise vegetable oils directly in internal combustion engines. There are some regarding working conditions. Vegetable oils have qualities comparable to biodiesel fuel. Despite this, some chemical transition is necessary. Before it can be utilised as a fuel in an engine, first it must be treated like remove the impurities and modifications in desirable properties. There has been extensive research during the last few decades.

1.5.2. Emulsification procedure

The higher viscidity possessed by the oils extracted from vegetable seeds is a challenge that micro emulsions can address. It utilizes ethanol, methanol, and 1-butanol as solvents. A colloidal equilibrium dispersion of optically isotropic fluid microstructures with sizes ranging from 1-150 nm, produced spontaneously from two normally not comparable fluids including one or more ionic or non-ionic amphiphiles, is how this process is described.

Micro-emulsion causes an increment in cetane number simultaneously a decrease in viscidity. That gives the biodiesel a nice spray flavour.

However, ongoing usage of such processed (micro emulsified) fuel in CI engines has major drawback. Its injector plunger may get stuck, there may be building up of carbon deposition, and inefficient burning of fuel particles.

1.5.3. Thermal cracking

Pyrolysis or thermal cracking is a process in which heat acts as a catalyst to transform one material into another in the absence of oxygen. The breaking of bonds takes place and different kind of tiny molecules are being created. Various studies has been done on production of biodiesel through this process[2], [16], [23]–[28]. It is used in various amounts to generate alkenes, aromatics, alkanes, alkadienes, and carboxylic acids. The equipment needed in the thermal cracking and pyrolysis processes is expensive. It is an expensive procedure to produce biodiesel. The primary drawback of pyrolysis is that it requires isolated distillation apparatus for separation.

1.5.4. Trans-esterification

Transesterification is analogous to the chemical reaction processes that use a catalyst. When the oils and spirits (any alcohol) react, formation of biodiesel and glycerol takes place.

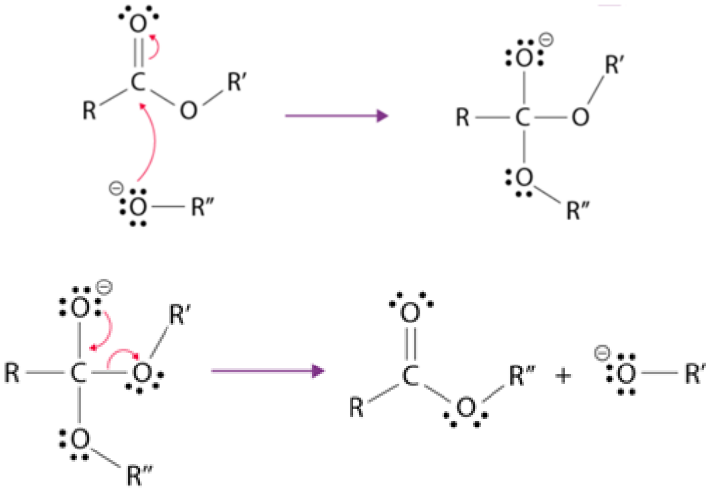


Figure1.1: Transesterification process

Catalysts of basic nature, such as NaOH, KOH, or sodium methylate, is utilized in this procedure. The employment of a catalyst escalates the reaction rate and the final output. Because of the reversible response, an excessive amount of alcohol is consumed. The additional alcohol is used to tilt the scales in the appropriate direction. Because it is reasonably priced, methanol is used as an alcohol in chemical processes. Vegetable oil reacts with methanol fast, which is advantageous. NaOH may pass through it easily.

A molar ratio of 3:1 is necessary for the completion of the trans-esterification process, i.e. number of alcohol moles to be three times of the moles of triglycerides. Commonly, the molar ratio can be increased in such a manner that the chemical reaction moves towards the maximization of the final output of the biodiesel. The chemical process of triglyceride with an appropriate alcoholic substance under a predetermined temperature and duration in the presence of a substance called a catalyst can be defined as trans-esterification. The end products include esters of the alkyl and glycerol. Equation under basic circumstances, the transesterification process is shown in the figure 1.1.

1.6. India's biodiesel production

India deals with the shortage of the culinary oils, to overcome it, 40% of the needs are fulfilled by the imports of such oils. The price of the imported oil therefore depends upon the international market and are mostly soared compared to the fossil –diesel. In such a scenario, it is not feasible to use the culinary oil ready to be consumed. However, it can still be treated as a strong contender, since the culinary oils doesn’t get rancid. In comparison, non-culinary oils get rancid easily and hence, it necessitates their pretreatment. Together with the Government Policy (National Policy on Biofuels-2018) Amendment, 2022[29] in picture, which says non eatable oilseeds, utilised kitchen oil , animal tallow, acid oil, short gestation non-edible oil rich crops, algal, and other materials be investigated for biodiesel production, with a target of 5% biodiesel mixing in diesel/direct sale of biodiesel by 2030. Plantation for obtaining non eatable oilseeds etc. can be done on degraded, waste, and marginal lands. Whereas, plantation in fertile, irrigated lands would not be encouraged. The development of innovative technology for biofuel conversion must also be promoted.

1.7. Factors affecting biodiesel generation by trans-esterification

1.7.1. Temperature of the reaction

Among the various factors which influence the yield of biodiesel, temperature of the reactants or reaction temperature is said to be a crucial one. Speed of reaction is directly proportional to the reaction temperature. Whereas, the speed of the reaction is inversely proportional to the reaction time. With an escalation in the reaction temperature, the viscidness of the reactants drops down rapidly. However, on increasing the temperature past boiling temperature of alcohol, it causes the alcohol to vanish, due to which yield decreases. Saponification of triglycerides also occurs at high temperature. Due to this reason the temperature of reaction should be kept below the boiling point of alcohol, so the evaporation of alcohol can be prevented or minimised. Hence, the range of optimal temperature of trans-esterification varies between 50ºC- 60ºC according to type of fats or oils. That is why; the temperature of reaction should be kept around the boiling point of the methanol, which is approved by various works of literature. At room temperature, there is up to 78% conversion after 60 minutes.

1.7.2. Duration of the Reaction

With an escalation in the duration of the chemical reaction i.e. transesterification, the transition of esters of fatty acid take place at a faster rate. The rate of reaction is comparatively slower in the beginning because the integration and dispersion between alcoholic substance and the oil consumes a bit longer. As soon as the progression of integration completes the pace of the chemical reaction process escalates by leaps and bounds.

The most significant production of the biodiesel on the other hand, takes place in a period of no more than ninety minutes. When the duration of the chemical reaction further increases there is no significant improvement in the biodiesel yield is observed rather the output of the biodiesel production drops down and there happen to be fair enough chances of formation of soap from the chemical reaction.

1.7.3. Methanol to oil molar ratio

The quantity of alcohol used during the transesterification process plays an important role in it. Methanol reacts with triglycerides present in the vegetable oil to form glycerine. The molar ratio of methanol to oil is a crucial factor impacting the biodiesel production process. In a chemically correct mixture 3 moles of methanol and 1 mole of triglyceride are required for trans-esterification. As a result, 3 moles of fatty acid methyl esters and 1 mole of glycerol will be produced. It fluctuates between 3:1 and 7.5:1. The equilibrium of the reaction shifts towards the right when the amount of methanol is increased, boosting the creation of biodiesel.

1.7.4. Type of Catalyst

The biodiesel yield is a function of amount of catalyst used during the transesterification process. Numerous type of catalysts are used in transesterification of vegetable oils to obtain biodiesel from them. Among the variety of catalysts potassium hydroxide (KOH) and sodium hydroxide (NaOH) are the most preferred ones. The quality of the feedstock determines the type of catalyst to use and how much there should be.

The yield of fatty acid alkyl esters increases when catalyst concentration is increased. The implementation of homogeneous catalysts carries multiple significant limitations, including the fact that following transesterification, the catalyst melts completely inside the glycerol successively as well as partially in the biodiesel, making differentiation of the products not easy. Therefore, biodiesel necessitates a time-consuming, labor-intensive, and ecologically unpleasant water-based cleaning procedure to eliminate surplus catalyst. Glycerine with catalysts component in it encounters less economic worth now, and disposing of it is a further obstacle. The previously mentioned issues connected with homogeneous catalysts might be overcome through the application of heterogeneous catalysts. Which are comparatively easy to be removed easily from the mixture of reactions and may be reclaimed and reused, making the method less harmful to the environment. Furthermore, they are more resistant to corrosion than homogeneous catalysts. To overcome these challenges, a heterogeneous catalyst has also been prepared during this work and selected for biodiesel synthesis from waste cooking oil. Also the conventional basic homogeneous catalysts were utilized with cottonseed oil.

1.7.5. Free Fatty Acid (FFA)

The following criterion is commonly used to define the steps involved in transesterification process for an oil:

Criterion	Process
FFA of oil to be processed <2%	Transesterification in single step
FFA of oil to be processed ≥ 2%	Transesterification in two step

1.8. Available technologies for biodiesel production

1.8.1. Mechanical/ Magnetic stirring

The transesterification is largely carried out in a batch type apparatuses that utilizes electrical energy to heat the reactants heating and magnetic/ mechanical stirring to agitate them. It is the most common and widely used process for biodiesel production. Different parts of conventional mechanical/ magnetic stirring method are shown in figure 2.

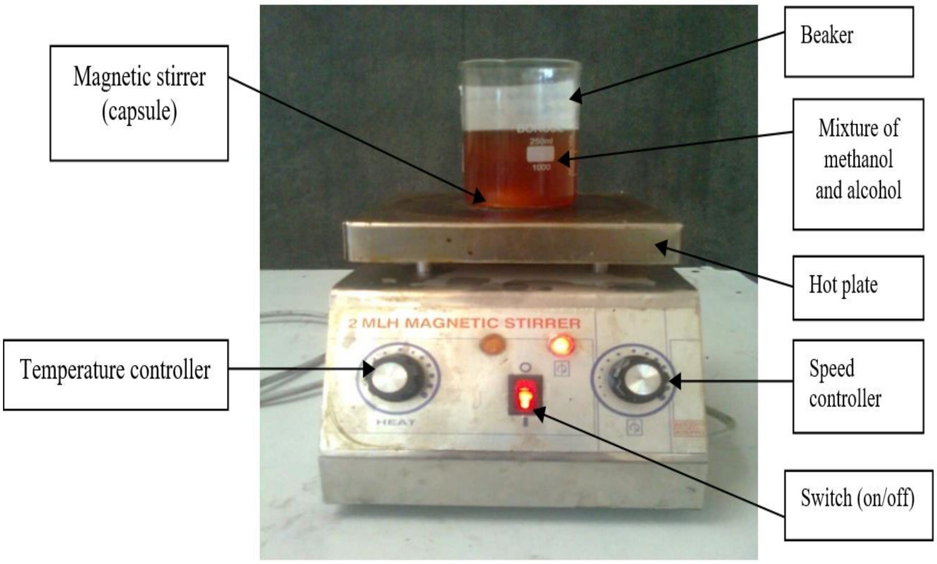


Figure1.2: Conventional mechanical/ magnetic stirring method

This process includes the production of biodiesel using methanol. The very existence of methanol causes the vegetable/seed oil to undergo transformation and be transformed into glycerol and ester. This process often uses electricity for heating and stirring for quite longer duration. In order to produce biodiesel, a few different energy efficient techniques were also experimented. Additionally, mechanical/ magnetic stirring method has a fundamental flaw called mass transfer resistance, which results in a slower response rate and a greater cost of production. Though, the biodiesel generated through this technology meets various biodiesel specifications and attributes. In an attempt to escalate the affordability of biodiesel production paralleled to fossil -diesel fuel, process magnification has been essential.

1.8.2. Hydrodynamic Cavitation

In the hydro dynamic cavitation technique, the system's geometry is used to induce velocity shifts, which in turn produces cavitation situations due to a difference in pressure, which in turn facilitates the mixing of two phases of reactants. It would be plausible to say that cavitation is caused by regulated liquid flow via basic geometries like venturi tubes and/or orifice plates. Pressure and temperature vibrations are created when the throat pressure drops below the liquid's vapour pressure, causing the liquid to flash and create a large number of cavities that then oscillate and produce increased and energy-stimulating transesterification by more evenly mixing liquids that are immiscible[30]. Schematic diagram of hydrodynamic assisted cavitation is given in figure 1.3.

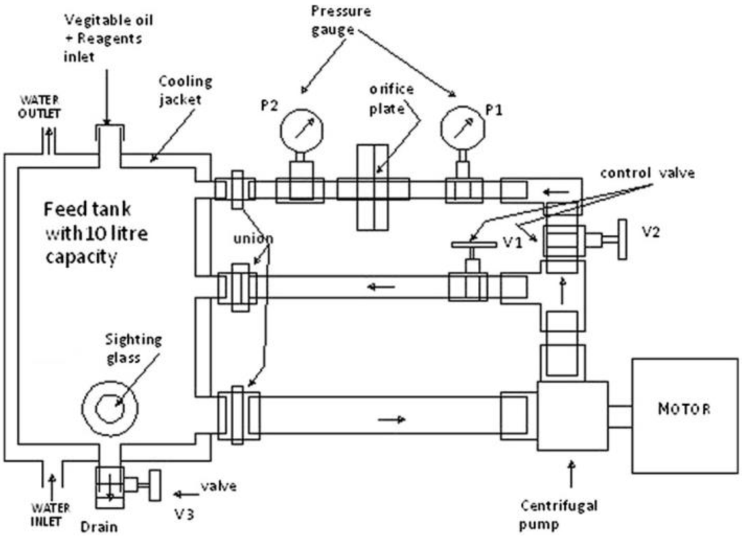


Figure 1.3: Schematic diagram of hydrodynamic assisted cavitation[30]

1.8.3. Ultrasonic Cavitation

The fundamental working principle of ultrasonic cavitation assisted trans-esterification technique is creation of cavities in any fluid due to the irradiation of power ultrasonic with sufficient energy. These cavities can be created in immiscible liquids also. As a consequence of this, microfine soap suds formation takes place and these soap suds collapse at different places in the reaction chamber, disrupting the phase restrictions between liquids that are immiscible therefore resulting in emulsifying of the combination with one another. A maximum energy discharge ranging from 1 to 1018 kW/m^3 all through the ultrasonic cavitation process elevates the total internal temperature of the reactant mixture to the temperature that's required to kick start the trans-esterification process, eliminating the need for an external heating source.

Different oily feedstocks were investigated by the previous researchers such as *Cedrus deodara*[31], soybean oil[32], palm oil[27], Neem oil[33] and waste cooking oil[34]. Different catalysts including solid base catalyst[27] and different geometries of the ultrasonic horn[31] were also investigated in combination with ultrasonic energy. Figure 4 given below shows the schematic diagram of ultrasonic cavitation.

1.8.4. Microwave

The microwave (MW) oven was one of the most significant developments of the twentieth century. Microwaves have been used to heat food items from the 1940s. Microwaves have been employed in a variety of industries, including the food, chemical, medical, and telecommunications industries.

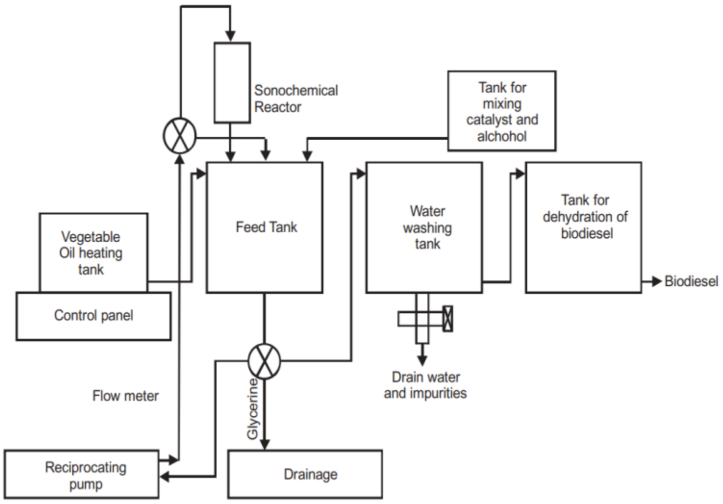


Figure 1.4: Schematic diagram of ultrasonic assisted cavitation[31]

Microwave energy is still the most widely used in the food business, where it is used for many purposes. Microwave heating has the advantages of being fast and volumetric. However, non-uniform hotness is a drawback of MW heating. The complicated interaction of microwaves with food results in non-uniform heating.

The primary benefit of heating with microwaves is the fact that it is not physical heating, hence it can be described simply energy transfer as opposed to heat transfer, which aids towards the lowering of the temperature gradients set up in the mixture and produces rapid heating.

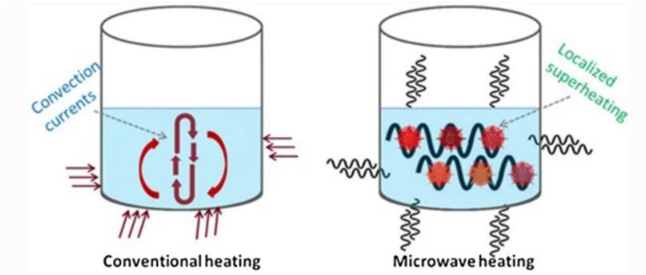


Figure 1.5 Comparison between conventional and microwave heating[35]

The response starts extremely quickly and is quite simple to stop. Microwave heating is referred to as the reverse thermal effect since it heats a material's inside first. The difference between conventional and microwave heating method is shown in figure 1.5. For production of biodiesel it can be stated that it is more effective, faster effect of catalysts, compact equipment, easier to control heating, faster start-up, increased generation, and process steps can be reduced[28], [36], [37]. The new classification of biodiesel feedstock based on sustainability issue is shown in figure 1.6.

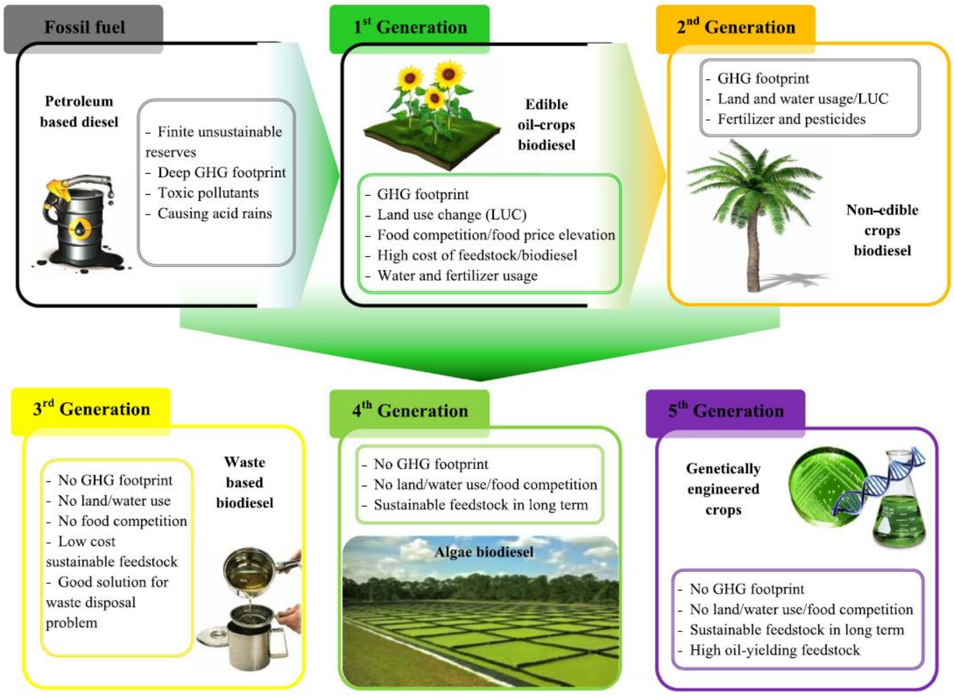


Figure 1.6: New classification of biodiesel feedstocks based on the sustainability issue[38]

1.9. Organization of Thesis

The present dissertation has been organized into seven chapters, which are as follows:

The **chapter 1** discusses the present-day energy sector in India alongside others around the world, as well as worries about energy security and environmental damage caused by the excessive mining of petroleum and coal. The possible applications of biodiesel as an environmentally friendly substitute to fossil -diesel is investigated further, as well as the benefits in addition to limitations. Both the chemical and physical features of the biodiesel have been addressed. Other methodologies for utilising oils derived from vegetable seeds inside the compression ignition engines, as well as other transesterification procedures, are also being researched.

Chapter 2 is a review of the literature, which summarises research publications on biodiesel production. This section covers studies undertaken by multiple researchers that provided the foundation for the current research activity. The fundamental components to obtain biodiesel synthesis, reaction parameter optimization, traditional and non-traditional production methods for biodiesel technologies, the financial and environmental implications regarding biodiesel, as well as biodiesel sustainability are all covered in the literature review.

Chapter 3 elucidates organization of the research. In the beginning a thorough study of the literature had been carried out. The shortages in the published review revealed that investigations were carried out in the past were majority of them with industrial microwave reactors and that too with few popularly known operating variables. With a particular focus on microwave technology, a biodiesel reactor for small scale production process has been developed. Some new operating factors were also decided and optimised which could have affect the biodiesel output. Both popular and non-popular oils were investigated for biodiesel yield. Simultaneously conventional and non-conventional catalysts were also selected for ensuring their usefulness in biodiesel industry.

Chapter 4 discusses production of biodiesel using modified microwave reactor. Biodiesel is produced on a small scale in a modified household microwave reactor. Production of biodiesel was successfully done from selected different feedstocks using different catalysts like single use homogeneous base catalysts, single use homogeneous acid catalysts and reusable heterogeneous catalyst prepared from waste aluminium foil. Optimization of various process parameters of biodiesel production was established using Taguchi methodology.

Chapter 5 deals with the test run of the CI engine fuelled with produced biodiesel blended with fossil –diesel. Three blends were prepared and successfully driven the test engine. Different performance and emission parameters were also investigated and results were analysed accordingly.

Chapter 6 addresses the financial repercussions associated with a biodiesel producing unit/industry. This provides the tentative cost and/ or the processing cost of the biodiesel produced which may help in industrial planning of biodiesel production.

Chapter 7 scrutinises the research work's thesis conclusions and provide an insight to probable future study directions of increase in biodiesel production.

1.10. Summary

Modernization is causing massive pollution and the exhaustion of reserves of petroleum to grow exponentially. Thus, if the rate of exploitation doesn't decrease down from the current rate, it won't be long until the fossil fuel reserves are no longer accessible. The worst affected countries will be those that are developing or underdeveloped. Everyone needs to explore for alternate energy sources that won't significantly increase the expense of infrastructure construction. Such energy sources ought to work well with the ones that are already in use. Biodiesel seems to be the option that best meets the conditions at hand. There are many potential raw material sources to investigate, and enough advancement has already been made in their processing. The process of development is still ongoing, and effective ways to continue this journey are being found.

CHAPTER 2

LITERATURE REVIEW

2.1. Related to feedstocks

In their research, Demirbas et al. [6] mentioned that biodiesel a substitute for fossils fuels can be obtained from various feedstocks like cooking oil, animal fats, and vegetable oils. It was also found that the soyabean, rapeseed, sunflower, and palm could be used primarily in the production of biodiesel. The pricing of biodiesel being a crucial factor behind the promotion of biodiesel as an alternative fuel for automobiles, power generation units powered by fossil -diesel. Their work identified variety of non-edible oil possibilities as important feedstocks, the biodiesel production process.

Pal et al.[39]**,** reported cotton seed has a high potential for biodiesel generation, according to their research. Cotton seed's most important feature lies in the fact it develops like a climber in sandy soil in a bi- annual harvest period. It is well known fact that an abundance of unprocessed materials affects the financial feasibility of an item. As a consequence, in order to investigate the advantages associated with the cotton seed oil plant, there must be efficient procedures for the plantation of ignored plants and their use. Bio-diesel has been proved to be a more ecologically friendly alternative to fossil -diesel. The overall standard associated with biodiesel fuel's composition has consistently proven to be important to support its application in CI engines as well as potential substitution of non-recyclable petroleum-based fossil -diesel. The composition belonging to the biodiesel has been realized to be the key behind its performance in CI engines along with potential replacement of fossilized petroleum-based fuels. Cottonseed biodiesel produced results compatible to petroleum diesel. The yield achieved at 1% KOH found to be greater. Cotton seed oil yields up to 97.90% when using the mechanical stirrer process. According to the findings of an experimental investigation of cotton seed biodiesel, it has qualities similar to diesel. As a result, blends have the ability to lessen the overburdening of diesel fuel imports.

Malhotra et al.[12], reviewed different feedstocks that may be employed in the production of biodiesel which can be used to power engines powered by diesel, as well as feedstocks for producing fuels for petrol engines. It was also suggested that the blend of biodiesel and diesel will be helpful in reduction of emission to an extent.

2.2. Microwave Assisted Transesterification

Previous studies[40][41], [42] done for comparison of conventional and non-conventional methods, has shown that microwave and ultrasonic irradiated processes significantly reduced the reaction time and energy requirements.

Anvita Sharma et al.[40], compared the traditional stirring, microwave and ultrasonic methods for harnessing the biodiesel from rejected culinary oil (WCO). The reported 98% yield of biodiesel was acquired with ultrasonic method, 96.45% via microwave and 88.03% using traditional stirring. It was also reported that the consumption of activation energy in kJ/mol was maximum in traditional method followed by the microwave method and ultrasonic method respectively. It was also concluded that the ultrasonic and microwave methods were more energy efficient.

Chellappan et al.[41], harnessed biodiesel using microwave assistance for agitating millettia pinnata seed oil using an eco-friendly catalyst. The optimum variable parameters such as temperature of reaction around 65 degree Celsius, oil to alcohol proportion of 11:1, the amount of catalyst equal to 3% of the oil, and the duration of the trans-esterification process of 45 minutes yielded a final yield of biodiesel of 98.7%. Biodiesel may be harnessed using the new ecologically friendly catalyst that was economically produced from biomass.

Gude et al.[42], investigated that hybridisation of microwave and ultrasonic assisted production methodologies for biodiesel production will help in reducing the time and energy requirement. Reduction in these two parameters will affect the overall biodiesel production cost thereby increase its probability of using in diesel engines.

Kapilan et al.[24], reviewed that a higher volume of methanol and catalysts (in the case of MS and ultrasonic processes) guaranteed higher yield (to some percent) but at the same time increased wastage of energy and hence the cost of production. In addition, it also served the role of another catalytic force applying heating in conjunction with mixing, thereby increasing the yield in a shorter period. Therefore it can be acknowledged that the microwave-assisted transesterification process was simpler and provided various advantages over other methods.

Datta et al.[43], narrated that the key to automated heating is the combination of different heating modes. The microwave energy-based transesterification has been favoured for overcoming the drawbacks of different modes. The electromagnetic waves were found to be responsible for oscillations of reactant molecules in a microwave oven; this in turn elevated the reaction temperature and thus helped to better amalgamate the various reactants.

Several researchers have already employed industrial microwave irradiation to treat different feedstocks. Azcan et al.[44], communicated that the microwave aided transesterification was successful in producing cottonseed methyl ester. The reaction time was significantly shortened from 30 minutes (for a typical heating system) to 7 minutes. Microwave irradiation offers a quick substitute for the standard procedure together with great product yields. Both time and money are significantly saved as a result.

Azcan et al.[45], utilized a microwave to transesterify used culinary oil in the existence of sodium methoxide. At 60°C and 5 minutes of reaction time 98.87% yield was reported. A viable method for producing bio-diesel quickly and with a high product yield is microwave heating.

Hindarso et al.[46], generated the bio-diesel from microalgae using microwaves. It was also studied that increasing the operating temperature from 50°C to 60°C increased the biodiesel amount that could be produced; however, when the temperature rose from 60 to 65 degrees Celsius, the yield dropped. The effect of two different catalysts was examined. The biodiesel output was also impacted by the duration of the reaction. Reported biodiesel yield rose when the duration of the reaction was ranging from 1- 3 minutes; it declined between 3 and 5 minutes. The best conditions were attained by utilizing 3%

MgO catalyst during a 3 minute reaction at 60°C, which resulted in a 99.35% biodiesel yield.

Fernandes et al.[47], used laboratory microwave oven to help in the transformation of trap grease (brown grease). It was communicated that a biodiesel yield of 96% was obtained from the chosen feedstock in 10 minutes using acidic catalyst. Programmable laboratory microwave apparatus of input power of 300W was used to perform the experimentations. It was suggested that the microwave assistance could be used for industrial application.

Ozturk et al.[48] and Duz et al. [49], used microwave energy to transesterify maize oil and safflower (*Carthamus tinctorius L.*) oil into bio-diesel respectively. The researchers exploited the experimental conditions with homogeneous alkali based catalysts and laboratory microwave with input power of 400W. Reported yield of 98% was achieved in short duration of 5 minutes for localized feedstock of maize.

Gülyurt et al. [50], experimented with microwave-assisted production of bio-diesel from Chlorella protothecoides. As a result of their experimentations, which was optimised using the factorial design method and it was possible to harness the maximum from the feedstock using alkali based catalyst, 9:1 methanol to oil molar ratio yield in 10 minutes.

Perin et al.[51], produced biodiesel from castor oil using microwave assistance. To obtain the highest yield, they preferred molar ratio of 6:1 and concentration of catalyst which kept as 10% of the mass of the oil and alkaline alumina was used as catalytic agent in a duration of 5 minutes.

Andrew et al. [52], used microwave reactor to transesterify palm oil. It was reported that a biodiesel output of slightly higher than 97% was obtained in a duration of 10 minutes. The amount of alcohol was kept as 6 times the amount of oil and alkali based catalyst agent was preferred to complete the reaction. Process parameter of duration of reaction was selected to be 2, 4, 6, 8 and10 minutes.

Khatun, M.S.[53] and Abdurakhman et al.[54], preferred microwave reactor over other methodologies to agitate waste cooking vegetable oil and procure biodiesel. In their experiment, they harnessed a yield as high as 99.5%.

Pereira et al.[55], experimented with macauba oil (Acrocomia Aculeata) with the help of a microwave reactor and communicated the effectivity of the process. They processed the vegetable oil extracted from local Brazilian palm tree known as macauba oil. It is communicated that in a duration of 60 seconds using ethanol as alcohol and potassium hydroxide as catalyst in moderate quantities helped to attain a yield of about 97% having microwaves assisted reactor.

Nayak M.G. et al.[56], found it handy to use microwave over other reactors to work with papaya oil to fulfil the requirement of biodiesel. They harnessed a yield of 99.3% from the oil in 3.30 minutes using microwave reactor supplied with a constant input power of 700W and the quantities of reactants including methanol to oil ratio of 9.5:1 along with catalyst loading of 0.95%. Response surface methodology was used to optimize the process.

Silitonga et al.[57], used an industrial microwave to process ceiba pentandra oil and obtained biodiesel from it. It was communicated through their work that the microwaves' assistance in biodiesel production found to highly time saving methodology. They obtained a yield of biodiesel as high as 95.42% in 388 seconds under controlled temperature and moderate reactants quantities. They used laboratory microwave for their studies and BB design of optimization was applied for process optimization.

Anvita Sharma et al.[28], experimented with microwave for production of biodiesel from waste cooking oil. In their work they reported that the two preferred catalysts i.e. calcium oxide and potassium hydroxide over others. The outcomes of the experiment demonstrated that biodiesel conversion percentage of 90.4% was obtained in a response period of 10 minutes, molar ratio of 15:1 and 0.5 wt% of calcium oxide catalytic agent. A yield of 93.8% was reported in a reaction duration of 9 minutes and methanol to oil ratio of 10:1 utilising potassium hydroxide as catalytic agent.

In their work, Devasan et al. [58] used microwave for production of biodiesel using a novel heterogeneous catalyst synthesized from sulfonic acid functionalized banana peel. Biodiesel yield of 97.09 plus minus 0.7% was obtained.

Saifuddin et al.[59], reviewed that microwave heating has exceptionally well heating qualities and gives tremendous thermal and non-thermal effects. Microwave method gives higher yield in shorter reaction time.

Saifuddin et al. [60] transesterified used frying oil ganged up with sodium hydroxide (NaOH) as catalyst with ethanol and microwave. Microwave oven model RM 800 of Wroclaw, Poland with output Power of 750 was shortlisted as the reactor for their batch production study. Potassium hydroxide and sodium hydroxide were selected as the reactants in different proportions. A conversion yield of 87% was obtained in a short time span of 4 minutes. It was concluded that microwave assisted reaction agreements a profligate and nonviolent substitute to the common process.

Lertsathapornsuk et al.[61], used waste frying palm oil in a modified domestic microwave of 800 Watts in continuous mode for their study. They used sodium hydroxide with ethanol as reactants for their study. They obtained 97% biodiesel yield in a reaction time of 30 seconds.

Yaakob et al.[62], also selected waste frying palm oil for their studies and used Deb microwave MW650 with a power range of 1250 watts. During their study, a methanol-to-oil ratio of 12:1 has been employed with a catalyst concentration ranging from 0.5 to 1.5%. The reaction temperature was maintained between 60º Celsius and 70º Celsius. A yield of 88.63% was reported at a reaction temperature of 65º Celsius and 7 minutes. Rahmanlar et al.[63], picked waste frying oil as feedstock with milestone microsynth microwave as reactor for their exertion. The reported yield was 97.2% in 3 minutes with a molar ratio of 9:1 and sodium hydroxide being the triggering agent.

Patil et al.[64], also considered waste cooking oil as feedstock with modified domestic microwave of 800 watts and obtained 92% of yield with 10% lesser energy consumption. Methanol to oil ratio of 9:1 was confirmed to be optimal for this work.

Chen et al.[65], synthesized biodiesel from waste cooking oil using microwave synthesis reactor and NS235 of Panasonic having a power range of 200 Watt to 750 Watt. A 6:1 methanol-to-oil ratio with sodium methoxide as stimulus were selected with other process parameters to complete the successful reaction.

Azcan et al.[45], selected Start S model microwave for trans- esterifying waste frying oil and the reported yield was 90.04% to 98.85% within a time range of 3 minutes to 29 minutes. The reaction temperature was maintained between 55º Celsius and 65º Celsius.

Mazubert et al.[66], preferred CEM discover SP microwave reactor of 300 Watt power for harnessing biodiesel from waste cooking oil. The procedure's settings were preserved as follows: methanol to oil ratios that varied from 4:1 – 6:1, stirring at approximately 300 RPM for a batch size of 80ml. The reaction temperature was kept between 30 degrees Celsius and 60 degrees Celsius and reaction time was taken 20- 45 seconds. The ester production was reported as 91.3% with an activation energy consumption of 37.1 KJ per mole.

Priambodo et al.[67], used Panasonic NSF 550 microwave reactor having power of 1000 Watt for transesterifying waste cooking oil. 93% yield was reported in their work with methanol: oil molar ratio of 6:1 in 3 minutes.

In Kwon Hong et al.[68], preferred microwave assisted transesterification using Mars 2 microwave for transesterifying waste cooking oil. 96.5% yield was obtained in their work with input power of 500 Watt, methanol to oil ratio of 8:1, reaction time- 6 minute and sodium hydroxide was taken as 1.2 wt% of the reactants.

Tangy et al.[69], used modified domestic microwave as reactor for their reaction with waste cooking oil. For experimentation work a batch size of 15 g along with silicon oxide was preferred. The reaction time was taken as 10 second and the reaction temperature was maintained at 333 K. It was reported that yield of 99.4% was obtained with this configuration.

Tangy et al.[70], picked flow SYNTH microwave system having 1000 W power for their study. The experiment was a single synthesis for 190 ml batch with an O: M ratio ranging from 1:6 to 1:12, SrO the catalyst as 5 to 8.75 gm, feed rate of 0.06 to 340 m/min, with pressure of 30 bar. In their study, they reported a 99.2% yield in minutes.

Panadare et al.[71], preferred ANTON-PAAR Multiwave PRO model of microwave having input power- 70 W for their studies. Waste cooking oil happens to be the promising feed stock for ester production via batch size pf 20 ml. Dimethyl carbonate: waste cooking oil (WCO) ratio was taken between 2–12:1, enzyme ranging from 5–15 wt % along with reaction temperature 50-70º C. 94% yield within a reaction time of 240 seconds alongwith first order kinetics activation energy of 18.96 kJ/mol was reported in their findings.

Thirugnanasambandham et al.[72], selected modified domestic microwave reactor for their experimental studies. The process parameters were optimized with Box Behnken (BB) model of optimization. The study was conducted for a batch size of 30 gram of waste cooking oil. It was observed that for an optimum power of 325Watt and reaction time of 200 seconds 97% biodiesel yield was reported. The reaction temperature reported to be 70º Celcius with potassium hydroxide (KOH) wt%-1 g/g.

Xiang et al.[73], harnessed biodiesel yield of 94.91% from waste cooking oil batch considered in their experimentation. They exploited modified domestic microwave for batch production of batch size of 10 ml, methanol: oil ratio of 3:1 to 11:1 with fly-ash g/g% ranging from 2 to 10% and reaction temperature within the range of 50–90ºC. Central Composite Design (CCD) technique was successfully applied to harness this biodiesel yield of 94.91% with methanol: oil ratio of 9.67:1, Fly-ash g/g %- 3.99%. The average reaction temperature recorded to be 66.20ºC in 6 minutes.

Gupta et al.[74], explored waste cooking oil for biodiesel production using multiwave PRO model of microwave reactor. Batch size of 100 ml was taken for methanol to oil ratio of 6:1 to 9:1. BB design of optimization was applied for process optimization. Optimal methanol: oil ratio reported to be 7.46:1 for maximum biodiesel yield.

As per the national biofuel policy of Government of India 2018 amended in 2022 [29] it is proposed to achieve a target of 5% blending of biodiesel in diesel by 2030. To achieve this target, it is proposed that the new feedstocks such as used cooking oil (UCO) for biofuels must be identified and development of new technologies for conversion of biofuels maybe set up.

Marchetti et al. [75] suggested many techniques like pyrolysis, micro-emulsification, traditional magnetic stirrer (MS) and ultrasonic whereas

Gude et al.[36], explored microwave-assisted transesterification that assisted in the production of bio-diesel from the different feedstock. Generally, biodiesel, irrespective of feedstock, is associated with lower air pollutants, with the exception of NO_X, than petro diesel[76].

Kim et al.[77], revealed in their work that in America and European Union, fats from animals and leftover frying oil are getting promotion as base material for production of biodiesel from them. It was also reported that the performance and emission characteristics of any biodiesel fuel are independent of type of feedstock.

Bergthorson et al.[16], in their investigation, they discussed the impact of biodiesel blended fuel on both performance and emissions characteristics of engines igniting the fuel by compression (CI). Alongside, it was also reported that, the cetane number (CN) of fatty acid methyl esters biodiesels ranges from 48 and 65. Because of the presence of more oxygen content in blended fuel, comparatively lean fuel: air mixture was supplied during ignition and lesser engine deposits and coking were observed. Whereas, an increment in the pollutants in the form of oxide of nitrogen have been observed.

Aydin and Bayndir [78], explored both the emission and performance characteristics of a single cylinder diesel engine powered with the help of blend of fossil –diesel and cottonseed oil methyl ester in it.

S. Awad et al.[79], investigated the alkali catalysed three kinds of wasted cooking oil, or WCO, along with methanol as well as ethanol. Sodium hydroxide had been utilised as the catalyst in this investigation. The temperature, catalysts amount, ethanol to oil ratio, and reaction time all had an impact on yield. The key variables impacting biodiesel yield comprised the operating temperature and the concentration of catalyst. The method had been influenced by the amount of FFA in the waste cooking oil and the verities of spirits used. The amount production of biodiesel varied from 97% at the lowest acidity (0.4% FFA WCO) to 76% at the highest acidity (3.25% FFA WCO). The ethyl esters yields were lower, and the distinction grew as the quantity of

FFA in the oil rose; their greatest outputs reached 95% and 73% with the lowest and medium acidities, respectively, and there was no reaction with the highest. The chromatography evaluation of the biodiesel produced revealed significant levels of fatty acid methyl esters ranging from 96.5% to 98%.

T Sathya et al.[80], reported that vegetable oils (primarily non eatable) containing large levels of free fatty acids (FFA) (more than 2%) cannot be transesterified using an alkaline catalyst because the alkaline catalyst reacts with the FFA to generate soap. As a result, a two-step procedure was established in this work to convert FFA to its methylester. The initial phase was an acid usage, which decreased the FFA content of the oil to less than 2% using sulphuric acid (H_2SO_4) 0.5% v/v reacting with methanol (0.45 v/v) at 50ºC and 45 minutes. Esterification observed to be the first stage. Following acid treatment, an alkaline trans-esterification process was performed at 1% potassium hydroxide (KOH), 30% methanol, 55ºC, and 60 minutes. Trans-esterification called as the second stage. The yield reported lied between 88 to 92 percent. In each stage process, the contribution of molar ratio, catalyst used, temperature of reaction, and response duration was investigated. Excessive sulphuric acid addition stained the end product, resulting in higher generating costs.

Umer Rashid et al.[81] generated trans-esterified biodiesel from cottonseed oil utilizing the following elements: NaOH, KOH, sodium methoxide, and potassium methoxide as catalysts with methanol to accomplish this. A series of studies were conducted to evaluate the impact from parameters related to the reaction including methanol/oil molar ratio ranging from 3:1 to15:1, the amount of catalyst from 0.25% to 1.50%, its temperature from 25 to 65°C, and agitation speed ranging from 180 to 600 rpm on yield and quality. The highest achievable synthesized methylester production level of 96.9% was obtained by optimizing the parameters such as 6:1 (methanol: oil proportion), sodium methoxide (0.75 wt.%), reaction optimum temperature of 65 degrees Celsius, movement speed of 600 rpm, and response duration of 90 minutes.

Leung et al.[82], Leung et al. determined that four important variables govern biodiesel yield: alcohol amount, duration of reaction, temperature of reaction, and catalyst percentage. For certain an efficient transesterification reaction, raise the molar ratio of alcohol to oil to 6:1 using an alkali catalyst. A larger molar ratio has to be used for acid assisted transesterification of discarded culinary oils or oils having an elevated fraction of FFA. As the transformation rate of fatty acid esters rises with the duration of the reaction, the final amount of the biodiesel becomes highest at the ideal duration of reaction. Greater temperatures of reaction may decrease oil viscosity, increasing reaction speed. Depending on the oil used, the optimum temperature fluctuated between 50oC and 60oC. The ideal catalyst concentration for sodium hydroxide, the most often used catalyst, has been around 1.5 wt.%.

According to Tesfaye et al. [83], the microwave supported transesterification process of soy-bean oil resulted in highest biodiesel transformation of 99% in a reaction duration of 5 minutes. Because the reaction is reversible, the conversion decreases when the reaction time exceeds 5 minutes. A comparable conclusion may have been demonstrated regarding both cloud point and pour point, whereby a rise in transformation showed a beneficial impact upon cloud point however had little impact regarding pour point.

Sinha et al.[84]**,** investigated waste cotton seed oil. The highest possible biodiesel output was 92% when the reaction's temperature, as well as reaction duration, methanol: oil ratio, and catalytic rate of addition had been set at 60°C, 50 minutes, 12:1, and 3% correspondingly. The usage of blended biodiesel lowered the amount of carbon monoxide along with hydrocarbon releases while slightly enhancing nitrogen oxides (NO_x) emissions.

According to Gude et al.[35], Microwave-enhanced organic/inorganic synthesis is a popular approach for a variety of reasons, including declined consumption of energy, significant decreases in processing time frames and solvents demands, higher precision, and enhanced transformations with fewer by-product production. Numerous tasks that are not possible with standard heating approaches may be performed with high outputs utilizing microwave irradiation. Because of their capacity to interact with a wide range of reagents, microwaves possess the potential for large-scale applications, particularly in biodiesel synthesis.

According to Panadare et al. [71], In the vicinity of temperatures that are lower, enzymatic activities outperform chemical catalytic processes procedures. The lipase enzyme was shown to be most active at temperatures ranging from 323 K to 353 K. As the temperature rises, the structural conformation of the enzyme deteriorates, resulting in a decrease of enzyme activity. The greatest transformation of 85.1% had reached at reaction temperature of 343 K, which happen to be slightly greater than the 83.3% conversion produced at 333 K. Because of the similarity in findings, Energy utilization and activity of the enzymes have been used as criterion for determining the optimal reaction temperature. It goes without saying that rising temperatures increase energy consumption. The microwave power imparted to maintain the requisite temperature was increased by 3W from 48W as the temperature of the reaction rose by 10K from 333 K. It is possible that an extremely small amount of catalyst loading will occur in a sluggish reaction rate, whereas large catalyst loading raises the reaction's operating cost. As a result, enzymatic concentration between the 5-15% (weighted average) ranges was investigated for biodiesel generation. Excess DMC neutralizes the oily content in addition to bring down overall collision rate of the reacting substances and trigger at higher mole fraction ratio. As a result, 4.5:1 has been found to be the best ratio to use for the specified procedure in order to achieve an optimal yield of 84.1%. The optimal ratio for the response of the sample investigated without the use of a microwave was 6:1 that happened to be greater as compared to the ideal value for the microwave aided process. It has been demonstrated that a traditional reaction took around 1380 minutes to achieve a state of equilibrium but a microwave supported procedure establishes just 240 minutes. As a result, this process having microwave irradiation accomplished in a relatively brief duration of time involving homogeneous heating up, as opposed with longer reactions with considerable losses of energy using the traditional approach.

It was discovered by Teo et al.[85]**,** that synchronously chilling and heating by microwave (SCMH) trans-esterification used 40.26% of the total energy consumption as compared to the conventional approach of biodiesel production. When compared to traditional trans-esterification and microwave aided trans-esterification, the biodiesel productivity efficiency attained in SCMH trans-esterification was the greatest. Furthermore, the optimisation of a parametric research on SCMH accomplished effectively in order to boost the productiveness of biodiesel conversion. In the end, it was discovered that the grade of biodiesel produced by SCMH transesterification found to much better in terms of creating fatty acid methyl esters (FAMEs) with low carbon content(less than 19), displayed fair cetane number and iodine value, and so has both good ignition and friction reducing qualities.

Onukwuli et al.[86]**,** focused on synthesis of bio- diesel using batch transesterification with refined oil from the oil that had been refined and extracted from cottonseeds utilising the two reagents methanol as alcohol and potassium hydroxide as an activator for the process. The characteristics of the oil extracted from cottonseeds as well as of the biodiesel processed from it reported to be a unique fuel for engines powered with fossil –diesel were established using ASTM fuel test guidelines. The chemical structures of biodiesel have been investigated employing Fourier transform infrared spectroscopy. The batch method had been implemented to test the effect of various operational factors. These operational factors were optimized using response surface methodology (RSM) and analysis of variance (ANOVA). A full factorial central composite design was used to determine the relevance of these various process factors and their combined impact on trans-esterification efficiency. The findings are in line with previously released information regarding biodiesels that transformed from different vegetable oil in addition to numerous worldwide biodiesel fuel requirements. A 96% yield was obtained using an ideal methanol: oil value of 6:1, a preferred reaction temperature of 550°C for 60 minutes, and an appropriate catalyst concentration of 0.6%. Oil extracted from cottonseeds of Nigeria, according to this study, might be used to make biodiesel.

Lee et al.[87], developed an exceptionally well lesser polluting power generation system utilising a 2.0 litre capacity CRDI engine powered with fossil –diesel. In addition to aforesaid test equipment an after-treatment equipment termed as re-combustor was existed in the test set up. This a combination of technologies concept generated the electricity while also recovering unwanted heat that coming from emissions from the engine. The re-combustor has been placed near the system's exhaust gases exit that allows an additional combustion of the gases that leave the system, boosting the system's thermal efficiency and minimizing exhaust emissions of gases. The above re-combustor's main components consist of coiling of pyromax wires mounted inside the ceramic cover, a diesel oxidation catalyst, and a diesel particulate filtration system. A total of four distinct rates of flow of water and four different electrical energy outputs have been utilized in the research. In broad terms, determining the perfect experimental conditions for the highest possible effectiveness of a co-generation equipment involves a significant amount of time and money. Most appropriate conditions for experiments for the existing system, on the other hand, are identified using the method described by Taguchi and analysis of variance (ANOVA), enabling for significant time and cost savings. The present co-generation system has an overall efficiency of 85.7% and decreases carbon monoxide, nitrogen oxides as well as Particulate by 73.3%, 34.3%, and 94%, respectively, according to the data.

Buasri et al. [88] The influence of experimental parameters such as the duration of the reaction, temperature at which the reaction occurs, methanol/oil molar ratio, and the quantity of catalyst on biodiesel trans-esterification been thoroughly explored. A Taguchi L9 (34) orthogonal array has been employed to analyze the characteristics influencing palm oil conversion to biodiesel. This scallop waste shell was calcined at 1,000oC for a period of four hours, and the catalyst was further investigated using different advanced techniques. At these optimal reaction conditions of 10% catalyst concentration, 9:1 ratio of methanol to oil, and 65ºC temperature, biodiesel conversion was actually 95.44% in three hours of operation. The scallop waste shell catalyst had been found to have outstanding catalytic activity and ecologically sound properties, with the potential to be used as a heterogeneous base catalyst in the biodiesel synthesis process. A list of specifications was established in order to provide a high-quality outcome that would be a less expensive alternative. The catalyst in question reacted in the same way as laboratory grade CaO. As a result, scallop waste shell is an appealing raw material for the manufacture of an inexpensive transesterification catalyst. The waste shell catalyst's surface area was enhanced by calcination, which boosted its catalytic activity. The end product is a light brown substance that satisfies Thai biodiesel standards.

Wu et al.[89], studied the Taguchi approach for establishing optimal concentration combinations for a CI engine running on fossil-diesel and/or mixtures of fossil-diesel and biodiesel fuels with Hydrogen along with cooled exhaust gas recirculation (EGR) at the intake manifold. At various engine loads and at 1500 rpm, these authors found the best operating variables for attaining good combustion performance, minimal oxides of nitrogen and smoke. In addition, the optimum combination of variables' combustion performance and emissions, alongside those of the conventional basic CI engine, have been examined. With an acceptance rate of 95%, Taguchi's estimations were found to be in reasonable conformity with the verified results, and the strategy conserved 67% of the time that was necessary to complete the experiment. An amalgamation of B20, 30% hydrogen, and 40% blending of exhaust gases to the fresh charge provides the best brake thermal efficiency (BTE), brake specific fuel consumption (BSFC), nitrogen oxides (NO) as well as smoke at every load. Furthermore, with an adjustable specific heat ratio, the experimental cylinder pressure has been utilized to determine the heat release rate. The arrangement of components appears better matched than the standard CI engine for obtaining several characteristics that affect efficiency of the combustion like brake thermal efficiency, pressure of engine cylinders and heat release rate, for different loads. Additionally, the right mix reduces BSFC while simultaneously reducing nitrogen oxides and smoke emissions as well. At 0.6 times the rated load, the BSFC drop percentage is slightly more than 25%, the nitrogen oxide reduction rate is 74%, and the smoke decline rate is slightly lesser than 30%. The Taguchi method was successful in determining these optimum combinations. With an acceptable level of confidence of 95%, the predictions created using Taguchi's method are consistent with the confirmation results, and the strategy saved approximately 67% of the total amount of time. The factor of blend percentage of biodiesel and fossil - diesel influences brake thermal efficiency with a nearly 34% contribution at a third load, oxides of nitrogen with a approx. 23% contribution at one third load, and smoke by a 67% contribution at approximately sixty percent load, based to the ANOVA approach. At various loads, the hydrogen ratio component contributed in excess of 87%, and the amount of smoke released approximately twenty percent at 45% load. The effect of the exhaust gas recirculation ratio on nitrogen oxides has been found to be more significant than all other intended goals.

Cheng-Han Hung et al.[90], studied characteristics of the combustion and determined best operation variables for a CI engine employing blends of biodiesel with fossil –diesel along with liquefied petroleum gas and low temperature exhaust gases introduced in the inlet manifold using the Taguchi technique. For 1500 revolutions per minute with a variety of loads, the optimum operating conditions for getting the maximum utilization of fuel duration, the minimum amount of smoke and nitrogen oxides have been identified. Likewise, the optimal and baseline CI engines' combustion traits such as heat release rate and ignition delay alongside emissions have been examined. The results state that the Taguchi approach predicts amount of atmospheric air required, which is compatible and at par with the confirmation results, and which means that this strategy minimizes the total number of experimentation trials in the research. Combination of B10, 40% LPG, and 20% EGR yields the best fuel consumption time, smoke, and NO_x levels at each load. Furthermore, for engine settings, the heat release rate is computed by dividing the experimental in-cylinder pressure by a variable specific heat ratio. Additionally, the optimal combination reduces both smoke and NO_x emissions. At 60% engine load, this drop rate is 52% for smoke and 31% for NO_x.

The L9 orthogonal array of the Taguchi technique was explored by Zhan-Yi Wu et al. [91] with the objective to figure out the most suitable hydrogen injection timing, ratio of hydrogen in the total energy supplied, and amount of blend of exhaust gases in a single cylinder direct injection compression ignition engine. An electronic control unit (ECU) controls the injection time, while a hydrogen flow controller controls the amount of hydrogen. The researchers’ appraised the ideal functional variables for lowering down the brake specific fuel consumption, oxides of nitrogen as well as smoke for different engine loads. In addition, using different specific heat ratios, the experiment's in-cylinder pressure has been used to compute the net heat release rate. On a 95% confidence interval, such forecasting developed with the help of Taguchi's approach and the results found to be at par with them. At 45% and 60% loads, the optimal factor combination decreases about 14% in case of BSFC, 60% for oxide of nitrogen, and 42% smoke while improving engine performance with different attributes like an increment in pressure inside the combustion cylinder along with net heat release rate when compared to the conventional CI engine. Due to the weaker COV of IMEP, adding hydrogen and EGR would not result in unstable combustion.

Ganapathy et al.[92], put forth an approach for studying the thermodynamic modeling of an engine fueled with Jatropha biodiesel join up with Taguchi's optimisation strategy in order to determine the optimal engine's design and operating parameters. To predict the performance of the Jatropha biodiesel engine, a thermodynamic model based on two-zone Weibe's heat release function was used. Ten crucial engine design and operating parameters were chosen from among the significant engine design and operating parameters, assuming no interactions between the parameters. An L16 orthogonal array was used to determine the engine test trial arrangement using linear graph theory and the Taguchi technique. The signal to noise ratio linked to higher the better quality attributes was employed to optimise the performance of the Jatropha biodiesel engine. When compared to other characteristics, the current technique properly forecast the different variables affecting engine performance.

Hülya Karabas [93]**,** investigated the use of oil extracted from acorn kernel with a higher free fatty acid content as a feedstock for biodiesel production. After getting the acorn kernel oil, biodiesel is produced in two phases. In the first phase, the 3.38% free fatty acid content is reduced to 0.14%, and the acid ester biodiesel is created in the second stage by an alkaline trans-esterification process. The alcohol: oil molar ratio, catalyst concentration, reaction temperature, and reaction time are the biodiesel production process parameters. Taguchi experimental design is used to optimise process parameters for acorn kernel oil methyl ester production. A catalyst amounting to 0.7 wt%, alcohol to oil molecular ratio of 8:1, an average reaction temperature of 50oC, and a response duration of 40 minutes utilizing pottasium hydroxide catalyst are suitable for the procedure. The Taguchi method determines the most efficient process parameter in the synthesis of acorn kernel oil methyl ester. Finally, the acorn kernel oil methyl ester yield is 90% when the Taguchi method's recommended parameters for processing are implemented. Reaction length at first level i.e. 40 minutes, alcohol to oil molar ratio at the second level@ 8:1, the temperature of the reaction at level 1 @ 500ºC, and catalyst concentration at level 2- i.e. 0.7% are the most suitable parameters based on the S/N ratio. Considering such circumstances, the AKOME yield for the above confirmation investigation has emerged as 90%.

Je-Lueng Shie et al. [94], investigated the pecuniary expenses pertaining to three manufacturing units of biodiesel having annual production capacity of 8k, 30k, and 100k tonnes of biodiesel. These units utilised continuous processes using an alkali catalyst and soybean oil as a raw source. Six significant economic cost components were calculated and investigated. The plant with an annual output of 100,000 tonnes reported to be economically viable. Among the operational components of the manufacturing facilities evaluated, the capacity of the plant, feedstock, glycerol and yields etc. happen to be the most critical operating characteristics impacting the monetary practicability of biodiesel production.

Srinivasa rao et al.[95], presented investigation on biodiesel harnessing from left-over chicken fat along with price calculation. The process involves the expenses associated with the production of biofuel from chicken fat in addition to the various expenses for raw materials, energy, and labour. The trans-esterification method was used to make chicken fat biodiesel, and the cost of production was evaluated using current data and appropriate assumptions. The cost of producing chicken fat biodiesel is projected to be roughly 25 USD per litre. As a result, discarded remains of the chicken could be accepted for manufacturing of the biodiesel, harmless waste disposal, and economically feasible.

2.3. Gaps in Literature Review

As per the exhaustive literature review conducted following research gaps are identified:

i. As discussed, different feedstocks has numerous chemical as well as physical characteristics. This literature review noticed that two-step transesterification using microwave’s assistance is one area where there is more research is needed.

ii. Different operating factors such as applied power, molar ratio (methanol: oil proportion), amount of the catalytic agent, duration of the reaction etc. have been discussed by most of the researches reviewed. However, the consequence of rotational speed of microwave’s turn table on biodiesel yield hasn’t discussed in the literature reviewed and this may need further investigations.

iii. As per the literature reviewed, the effect of cooling fan speed on biodiesel yield is another parameter which may be given more attention towards it. It is presumed that the heating of components of microwave may affect the efficiency of the equipment as well as the biodiesel synthesis.

iv. Only a few discussions were found on economic analysis of microwave aided biodiesel production but they aren’t found conclusive.

2.4. Objective of the present work

i. To upgrade an existing microwave oven in a suitable and cost-effective manner for biodiesel synthesis.

ii. To optimize the various operational variables for high yields by microwave irradiation method.

iii. To investigate the impact of various operating parameters on the biodiesel yield using microwave irradiation method.

iv. To compare the characteristics of the biodiesel produced by microwave method to conventional method using standard tests.

v. To run a compression ignition (CI) engine fueled with various blends of produced biodiesel and petro diesel.

vi. To prepare a comprehensive economic analysis of microwave assisted biodiesel production.

2.5. Summary

From the literature review, it has been summarized that microwave heating is fundamentally non-uniform and volumetric. Microwaves generate echo like sequences within the steel sidewalls of the oven. These patterns are areas of high- and low-intensity electric fields. This is one of the causes of hot and cold areas in the heated meal. Less energy is left for the waves to penetrate farther when they pass through a substance that absorbs microwave radiation. As a result, energy absorption is uneven. Against all odds the microwave reactors have successfully employed in treating different feedstocks to synthesize biodiesel from them. Microwave assisted reactors found to be compatible with different catalysts and other reactants. Microwave assistance is effectively utilized to produce biodiesel economically and the final produce observed to be compatible with the produce from other techniques. Moreover, it was found at par with different international standards. It has also been established that different feedstocks like waste cooking oil, cottonseed oil and other feedstocks which are found in abundance in countries like India can be investigated using microwave assisted transesterification. Different operating parameters can be optimized with the help of optimization techniques such as Taguchi and ANOVA. Number of research gaps were also observed while reviewing the literature. These research gaps has been enlisted as above in section 2.3 of the chapter.

CHAPTER 3

RESEARCH METHODOLOGY

3.1. Introduction

Implementing an effective research methodology is essential for producing precise findings in study that may be generalized. It may be described from a variety of angles, such as technique, approaches, tactics, and systems that are linked to combine and compile all pertinent data for the investigation.

This chapter discusses how to define and formulate research questions based on a thorough literature review. It also demonstrates how to obtain raw materials, characterize raw cottonseed oil, kalonji oil, and waste cooking oil, design experiments, optimize reaction parameters, test properties, and analyze the economics of producing biodiesel. This chapter also includes a list of the equipment, along with information on calibration and error estimation. A flow chart that offers a summary of the study is shown in Figure 3.1.

3.2. Formulation of research problem

Many studies have been conducted on the generation of biodiesel from various feedstocks, alcohols, and catalysts using both standard and unorthodox approaches. The unorthodox procedures include transesterification by microwave irradiation, supercritical alcohol process, hydrodynamic cavitation, and ultrasonic cavitation. The promptness and a greater biodiesel output are advantages of unorthodox methods. When compared to the production of biodiesel using cavitation methods, it was discovered that microwave irradiation and non-catalytic supercritical methanol transesterification had a quicker reaction rate and better yield. However, the high energy requirements of both of these methods restrict their usage in the commercial synthesis of biodiesel.

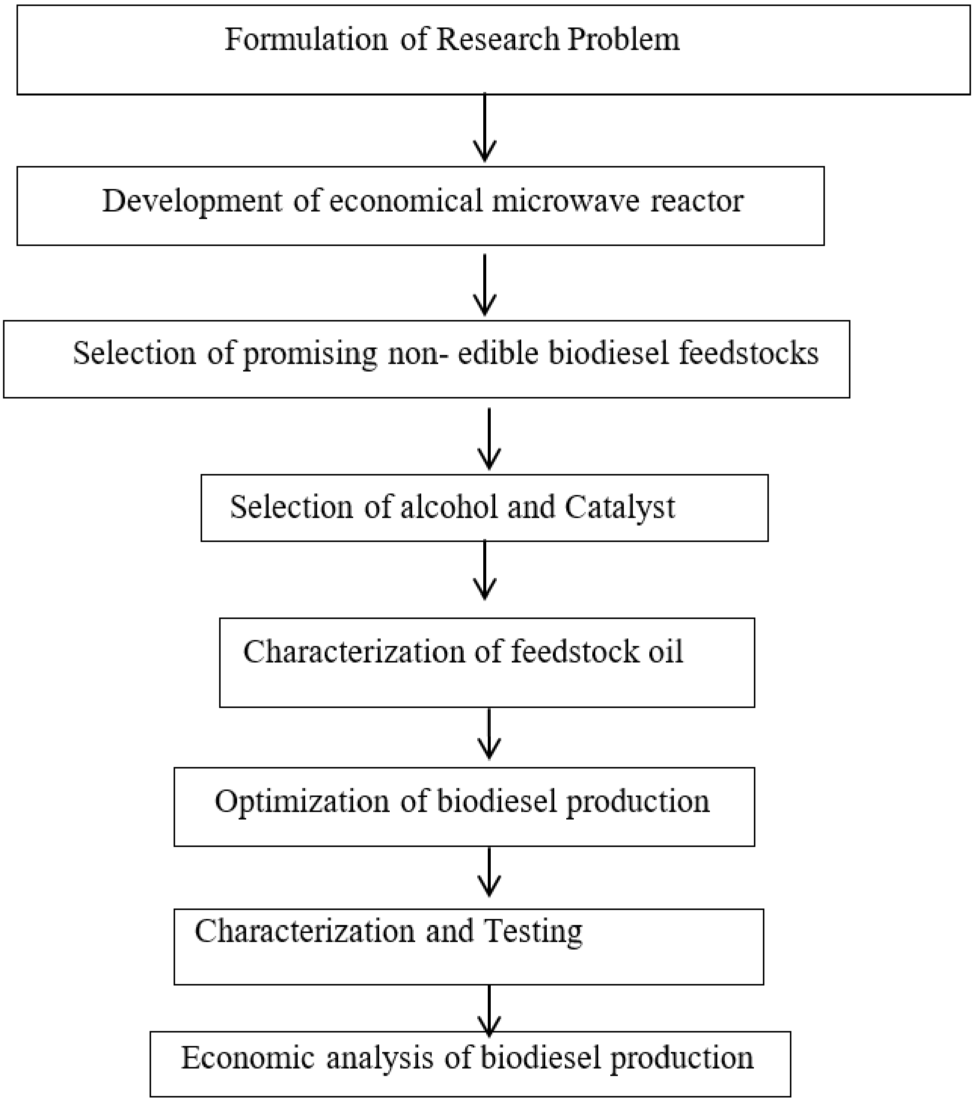
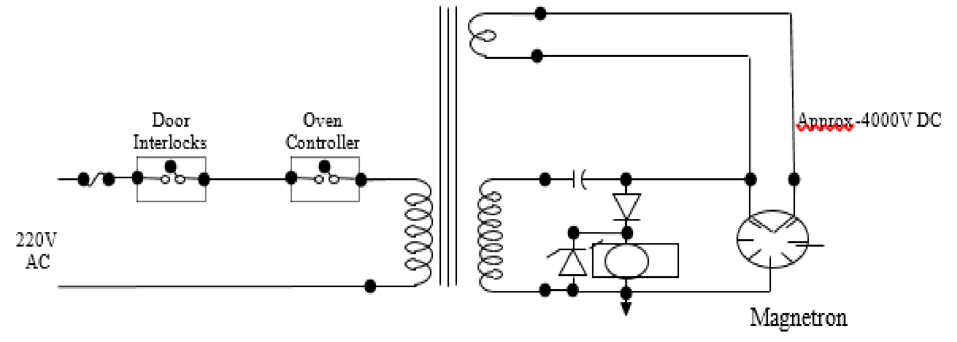


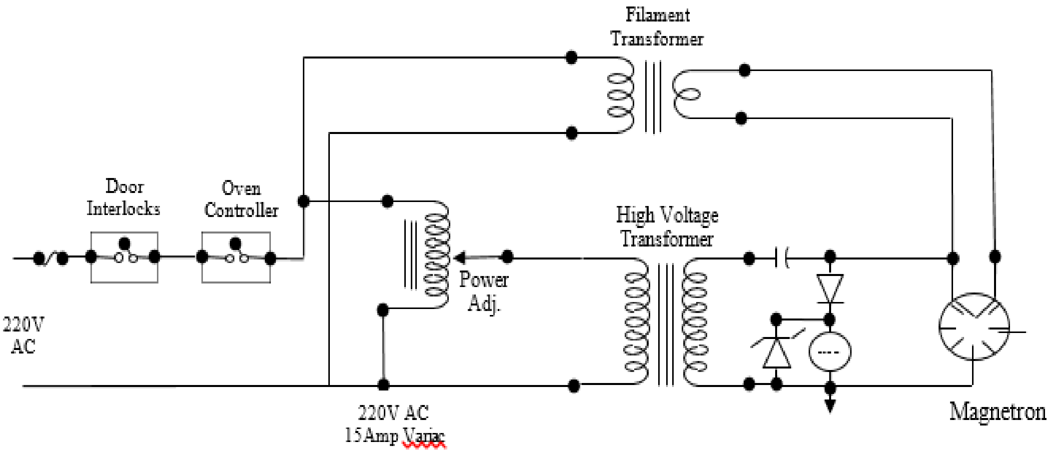
Figure 3.1: Flow diagram of the research methodology

3.3. Reactor Set up

A 23.0-litre stainless steel domestic microwave (Make-SANYO, Model- EM-G3686WY, Power/source 230V, 50Hz, Output Microwave 800W, 2450MHz, and Power consumption- 1250W) was used in this case as a reactor with few modifications in it to carry out the transesterification reactions. The specifications of the different components used to modified microwave reactor are given in table 3.1. Figure 3.2 (a) below shows the circuit diagram without any modification. Whereas, figure 3.2 (b) shows the circuit diagram for a modified microwave oven used in this study.



(a) circuit diagram for non- modified oven



(b) circuit diagram for modified oven

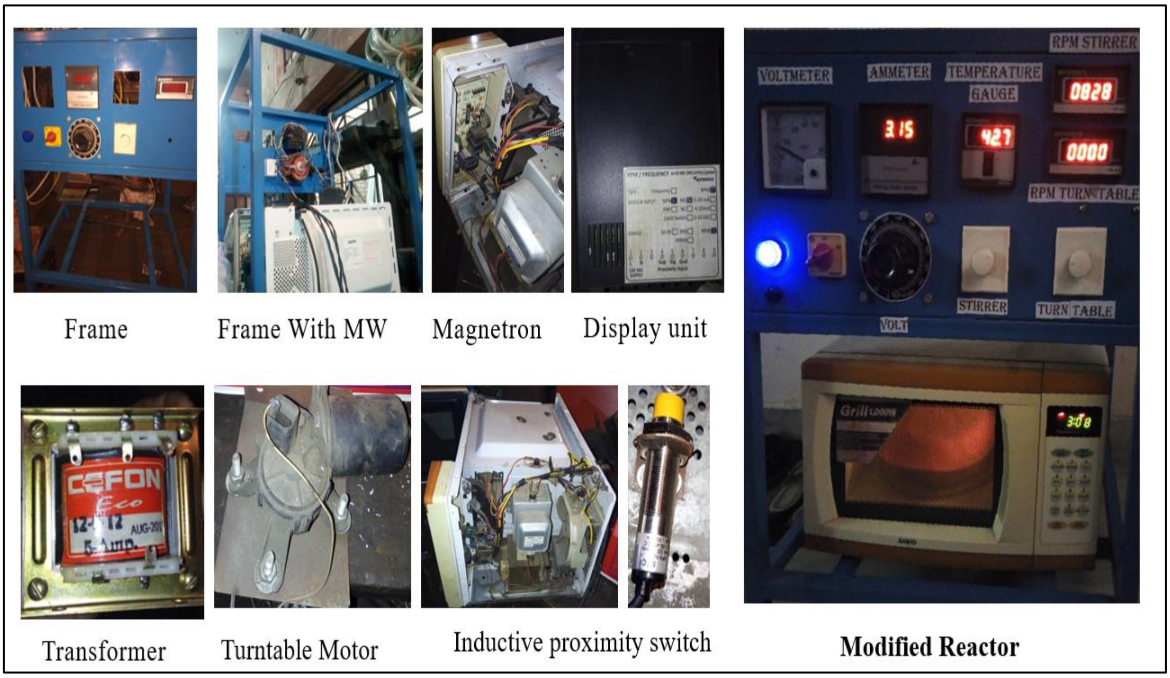
Figure 3.2: Comparison of circuit diagram for domestic microwave oven

The figure 3.3 given below show the different components used for modifications and the actual modified microwave used in the present study.

Table 3.1: Specification of the different components used to modified microwave reactor

Component	Rated Values
Transformer	Continuously variable autotransformer giving different output voltage range of (0-260)V at an input voltage of 230V
Voltmeter	Range- (0-500)V, Analog(confirming to Indian standards IS.1248-68)
Ammeter	Range- (0-20)A, Digital

Temperature indicator	Model DTI- 200M, Type-K, Range- (0-1200)°C coupled to a thermocouple (for measuring the temperature of the cavity)
Inductive proximity switch- 1	Type- NPN, NO, Sensing range- (0-8)mm, Outside diameter- 18mm, Operating Voltage- (5-40)V, DC, Maximum load- (0-200)mA



Frame Frame With MW Magnetron Display unit

Transformer Turntable Motor Inductive proximity switch **Modified Reactor**

Figure 3.3: Different components used to modify microwave reactor

3.4. Identification and characterization of feedstocks

The world's two most significant cotton growers, China as well as India, account for around half of global production, while the most prominent four cotton producers account for two third of global production. India has risen above China to become the world's number-one cotton grower. Cotton is one of the most significant commercial crops grown in India, accounting for around 25% of total world cotton output. India is the globe's highest cotton grower and third major exporter. It is also the world's biggest consumer of cotton. Gujarat and Maharashtra are the top cotton growing states in India. Production volume of cotton oilseed across India is shown below in figure 3.4 since 2015-2022.

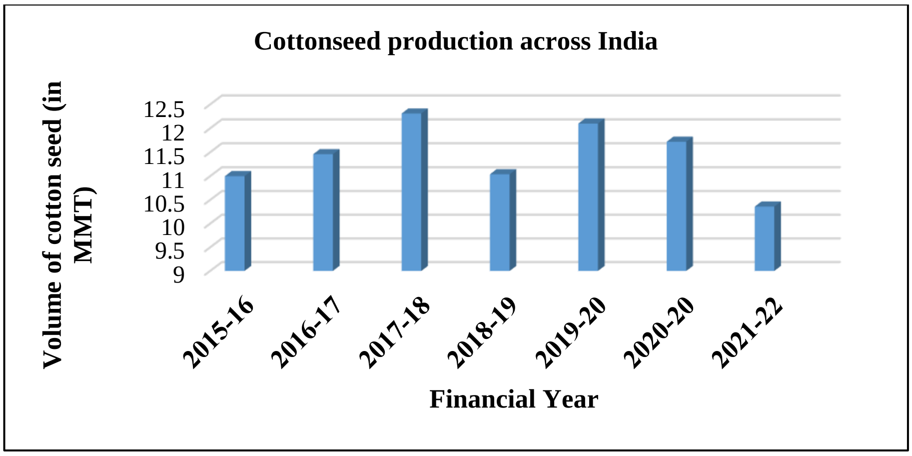


Figure 3.4: Production volume of cotton oilseed across India

Kalonji (Nigella sativa L.) belongs to the Ranunculaceae group annually flowering seed herb widespread to Southern and Southwest Asia. Black cumin has been another title for kalonji in English, kalonji in both Hindi and Urdu, krishnajirika in the ancient Indian language of sanskrit, kalajira in Bangali, in addition to shonaiz in Persian. [96]. It grows in the Mediterranean region and world's large producers are shown in figure 3.5 [97]. It is grown commercially in West Bengal, Punjab, Jharkhand, Himachal Pradesh, Bihar, and Assam in India. Small-scale farming is also practised in the states of Uttar Pradesh, Rajasthan, Madhya Pradesh, and Tamil Nadu[98]. The crop was found to be fairly salt water accommodating, suggesting that it represents a glycophyte. Kalonji develops to an average height of 200-300 mm, with upright, perfectly divided leaves. The vegetation develops in around 135-150 days after sowing.

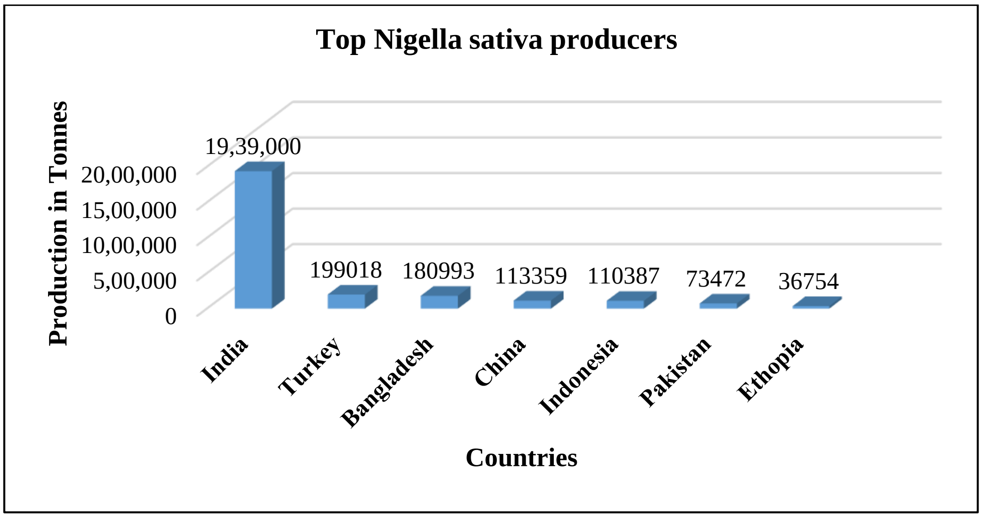


Figure 3.5: Worldwide leading Kalonji oilseed producers in 2019

It should be picked once the seed has reached full maturity in the capsule and has turned a deep brown/ black hue. Seeds may break as a result of the harvesting delay. One hectare of land may provide an average seed output of 5-10 quintals. Plants are rubbed or beaten with a stick to separate the seeds. The seeds are then winnowed and dried[99]. Figure 3.6 [100] depicts the plant's blossoms, which are vivid, light blue, and white. The fruit of the kalonji shrub resembled a big and bloated shell containing several seeds that served as spice. Different homogeneous alkaline, acidic, heterogeneous, and enzymatic catalysts have been employed to economically revolutionize plant oil transformation to biodiesel [101].

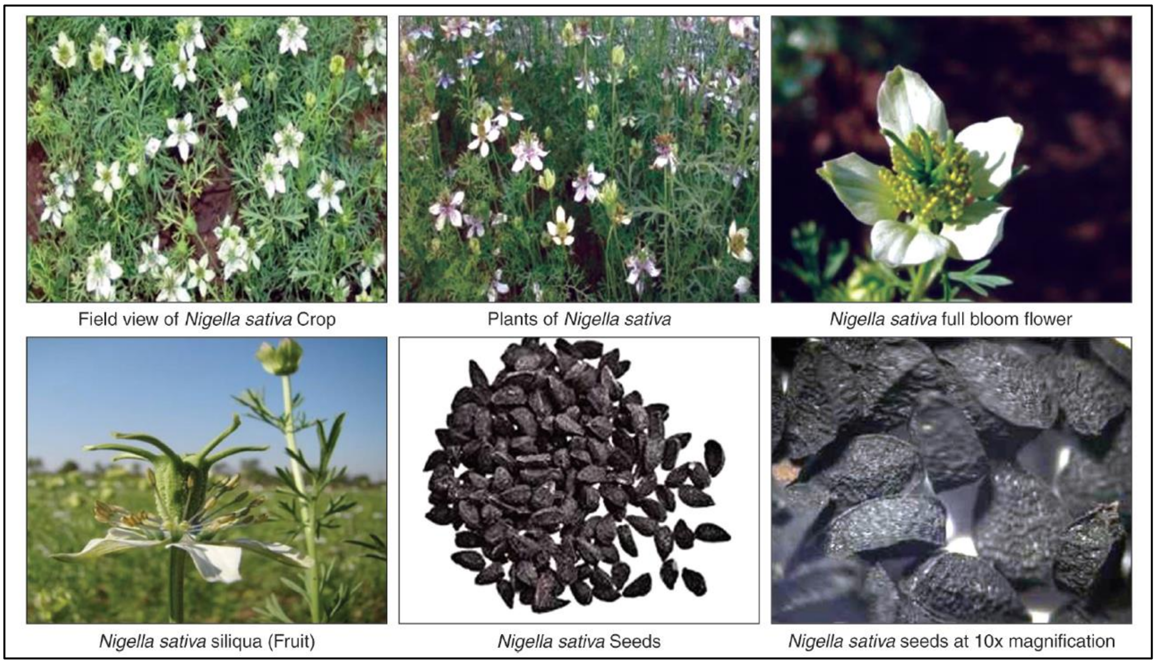


Figure 3.6: Plant, flower, fruit and seeds of kalonji (Nigella sativa L.)

Alkali-catalyzed transesterification has been one of the preferred pathways because of following advantages:

i. the reaction conditions involve moderate temperature and atmospheric conditions,
ii. fewer ingredients involved,
iii. lower reaction time
iv. relatively lower cost,

especially when compared to some of the other counter-processes involved[5], [24], [102], [103]. However, the aforesaid method found to be suitable for the feedstock oils containing less than 2% FFA components. More FFA disrupts the reaction mechanism, causing unwanted soap formation and, as an outcome, decreased efficiency[101].

3.5. Gas chromatography (GC) analysis of feedstock

For gas chromatography (GC) of feedstock, a centurion scientific gas chromatographer (Model- 5770 GC) which included a flame ionisation detector (FID), column BPX-70 having column length of 30 m with an inner diameter of 0.25 mm, and film thickness equivalent to 0.25micron was utilized. Nitrogen gas has been supplied at 1ml per minute for the carrier gas function.

Figure 3.7: Gas chromatographer (Model- 5770 GC)

The oven temperature was set to 40 degrees Celsius/ minute, with a holding duration of 20 minutes.

3.5.1. GC analysis of cottonseed oil

Biodiesel feedstock is obtained from several biomass sources. These feedstocks have various functional groups, which impact their physicochemical characteristics. Table 3.2 compares the values of important physicochemical parameters of cottonseed oil utilised in the current investigation with some of previously published studies.

Table 3.2: Comparison of physicochemical properties of cottonseed oil used in the present study amid those accessible in the literature

Physicochemical Properties	**Test Method**	**Present study**	**Azcan.et al.[44]**	**Literature values*[104]**
Color	-	Light Golden	-	-
Acid value (mg KOH/g oil)	D 664	0.08	0.22	0.20–0.24
FFA content (%)	D 664	0.07	-	-
Iodine value	EN 14214	107	99.3	99.0–119.0
Saponification value	-	191	-	-
Kinematic viscosity at 40°C (mm^2/s)	D 445	34.6	35.7	35.7
Relative Density at 25 °C (g/ml)	D 1298	0.917	0.924	0.918–0.926
Cloud point (°C)	D 6371	-1		
Flash point (°C)	D 93	329	262	>250

(*Danisman, A.; X. Fan, Xi Wang, and F. Chen)

Gas chromatography was used to assess the composition of fatty acids present in the cottonseed oil taken for this experimental study. The obtained values are tabulated in table 3.3 and compared with the values available in the literature. Besides the non-existence of very small amount of myristic and cyclopropenoid acids, the values revealed are in line with previous studies.

Table 3.3: Comparison between FFA compositions (%) of cottonseed oil used in the current study and literature values

Parameter	Present Study	X. Fan et al. [104]	Azcan et al. [44]	Salunkhe et al. 1992 [120]
C14:0	-	-	0.71	0.5-2
C16:0	24.7982	23.67	25.48	20-25
C16:1	0.8358	-	0.49	0.5-2
C18:0	2.9231	-	2.38	2-7
C18:1	19.2791	17.09	15.71	18-30
C18:2	52.1637	50.33	55.23	40-55
Cyclopropenoid acids	-	-	-	0.5-2

Figure 3.8 depicts the gas chromatogram of cottonseed oil used in the present study. The values of this chromatogram have already been tabulated in table 3.3.

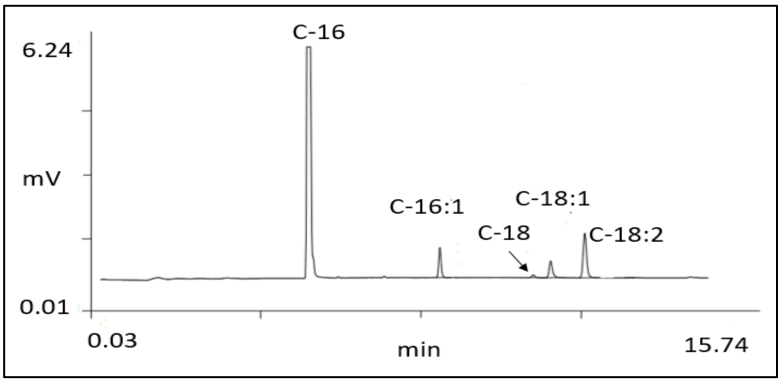


Figure 3.8: Gas chromatogram of cottonseed oil used in the present study

3.5.2. GC analysis of kalonji oil

The FFA concentration of the oil has been one of the most important elements in assessing its eligibility for transesterification processing. The amounts of free fatty acids were evaluated using the titrimetric technique in this study. The current study's average reported value was 4.7%. The quantity of FFA results in a higher level of oil hydrolysis. Chemicals that happen to be typically dissolve in oils and fats yet do not become saponifiable with caustic alkali nevertheless disintegrate in common fat solvents such as hydrocarbons are examples of unsaponifiable content. Peak evaluation of fatty acids in the analysed samples was accomplished by comparing mass spectra. Table 3.4 shows the average proportion of saturated and unsaturated fatty acids found in the specimen kalonji oil samples.

Table 3.4: The percentage of saturated and unsaturated fatty acids in kalonji oil specimen

Parameter	**Present Study**	**Aftab, A. K et al. [105]**	**Mohammed Yahya Hadi et al. [106]**	**Gharby et al.[107]**
C14:0	3.3	0.12±0.004	0.312	0.2±0.1
C15:0	0.55	-	-	
C16:0	20.59	12.68±0.5	13.775	11.9±0.1
C16:1	0.52	0.12±0.005	1.651	0.2±0.1
C17:0	0.13	-	-	-
C18:0	6.23	3.99±0.15	1.817	3.2±0.1
C18:1	28.94	28.55±0.75	18.643	24.9±0.5
C18:2	37.01	51.80±1.45	18.061	56.5±0.7
C20:0	0.35	0.21±0.003	20.43	0.2±0.1
C20:1	0.53	0.48±0.01	0.69	-
C20:2	1.84	2.05±0.05	3.085	-
C22:0	-	-	21.536	-
X1	1.85	-	-	-

The presence of Oleic acid has been linked to lower low-density lipoprotein (LDL) cholesterol levels. PUFA, on the other hand, has beneficial effects on both good health and terminal conditions, such as lipid regulation[105]. Table 3.5 depicts the comparison of the physicochemical properties of kalonji oil/sample used in this study.

Table 3.5: A comparison of the physicochemical properties of kalonji oil/sample

Property	**Test Method**	**Present study**	**Aftab, A. K et al.[105]**	**Gharby et al.**[106]	**Wahidu Zzaman, et al.**[107]
Color	-	Deep	-	-	Green-
Acid value (mg KOH/g oil)	D 664	0.35±0.01	-	-	0.41±0.01 (as oleic %)

FFA content (%)	D 664	4.75±0.02	4.69±0.15	2.3±0.5	0.20±0.01 (as
Iodine value (Wij’s)	EN 14214	127±2	123.4±1.52	126±4	-
Saponification	ISO	195±3.2	205.21±2.4	-	132.75±1
Kinematic	D 445	64.6	-	-	-
Relative Density at 25 °C (g/cm^3)	D 1298	0.9167±0.07	0.9158±0.02	-	0.93±0.04
Refractive Index	-	1.425±0.0	1.465±0.05	1.473±0.00	-
Unsaponifiable Matter (%)	-	-	0.33±0.01	-	-

3.6. Waste cooking oil

The vegetable oil after repeated frying becomes non-edible, is referred to here as waste cooking oil (WCO). A huge quantity of WCO is produced by caterers all over the world. WCO was collected from reputed caterers in kamla nagar, Delhi. WCO contains moisture and food particles. Food particles were removed by filtering through sieves and moisture was removed by heating the oil to 120°C. The WCO was then cooled and kept in an airtight glass container after filtration. FFA, iodine value, saponification, value, etc. of WCO were determined and recorded in table 3.6 by using standard methods.

Table 3.6: Properties of WCO

Property	**Measured Value**	**Test Method**
Kinematic viscosity @ 40°C (mm^2/s)	51.5	D 445
Acid Value (KOH/g)	5.4 mg	D 664
Iodine number (g I_2/100g)	58.7	EN 14214
Saponification Value (mg KOH/g)	203.7	ISO 1385-1:1977
FFA (%)	1.04	D664

3.7. Catalysts selection

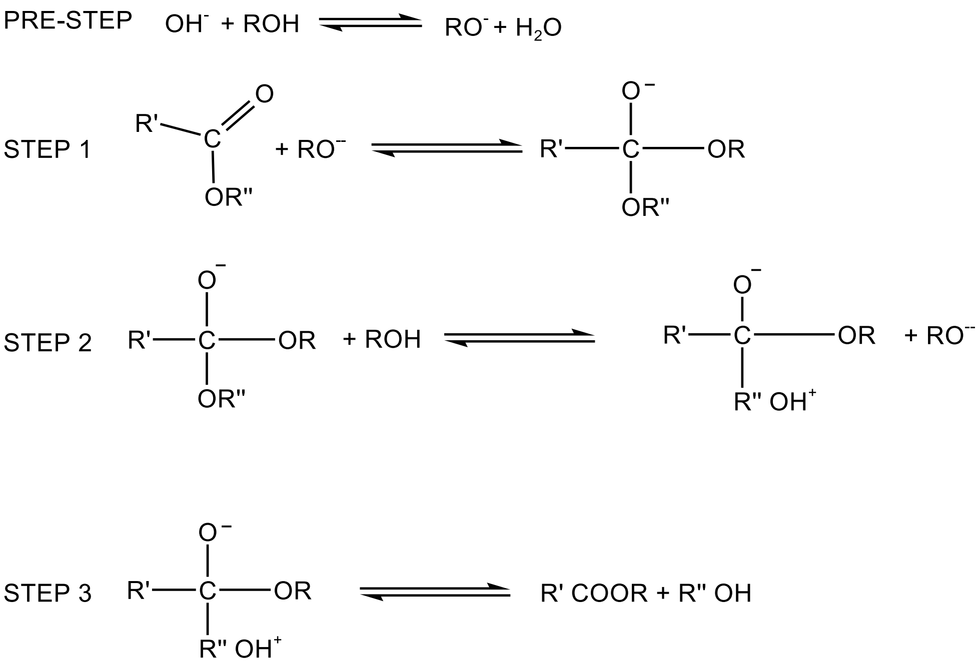
Triacylglyceride (TAG) is an abbreviation of the oil used for producing biodiesel. Covalent bonds between alcohol and carboxylic acid result in the formation of TAGs. Three fatty acid molecules bound by covalent bonds to a glycerol molecule combine to create TAG, which is an ester in this context. In contrast to glycerol, which contains three hydroxyl groups, fatty acids have carboxylic groups, which are combined can produce ester or TAGs. In the chemical process of transesterification, one carboxylic acid ester is changed into other carboxylic acid esters. Depending on whether an acid or a base is employed as a catalyst, fatty acids react differently. Heterogeneous, homogeneous alkali catalysts which include potassium hydroxide and sodium hydroxide have a frequent application in industrial transesterification since they accelerate reaction at below average temperatures as well.

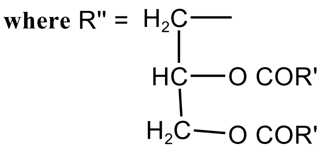
3.8. Homogeneous catalyst

Catalysts that are homogeneous can also be categorised into homogeneous acids and homogeneous bases. It is usual practise to use homogeneous base catalysts for triglyceride transesterification. The most often utilised homogeneous base catalysts include carbonates, alkaline metal hydroxide, and alkoxides. Because alkoxide is hygroscopic by nature, it is more challenging to handle than hydroxide. In consequence of the existence of the hydroxide ion, which serves as a type of contaminant within alkoxide, it is impossible for alkoxide to produce soap from triglyceride saponification. The free fatty acid concentration shouldn't rise by more than 0.5% by weight while employing an alkaline catalyst; else, soap formation would occur and hinder the generation of biodiesel. According to several publications, the methanolysis and ethanolysis of coconut and palm oil yield 90% when potassium hydroxide and boiler ashes are used, and alkaline catalyst NaOH outperforms sodium methoxide (NaOMe) in these processes. When alkaline catalysts (NaOH, KOH, KOMe, and NaOMe) were tested, it was shown that potassium-based catalysts provide greater yields than sodium-based catalysts. This is because the concentration of NaOMe is somewhat higher than NaOH in order to get a higher yield. Higher yield is produced by methoxide-based catalysts than by hydroxide-based catalysts in this case.

3.8.1. Transesterification catalyzed by a base

The majority of bases that act as catalysts have applications in the transesterification of oils produced from numerous vegetable seeds. Acid-catalyzing agents are employed to minimize the development of soap whenever the triglyceride has FFA or a large quantity of water. Although base catalytic transesterification is 4000 times quicker compared with acidic substances triggered transesterification, it may only employed whenever the triglyceride contains a maximum of two percent free fatty acids. Both potassium and sodium hydroxide are primarily used in industry. Base catalysts are commonly utilized when the chemical process occurs at relatively low temperature and pressure of 60°C and 20 psi respectively, yielding an excellent yield of around 98%. Still, there are particular drawbacks, such as the significant amount of energy necessary to extract the catalytic agent from the medium following transesterification, the difficulty in recovering glycerol after the process, and the formation of soap comprising free fatty acids.



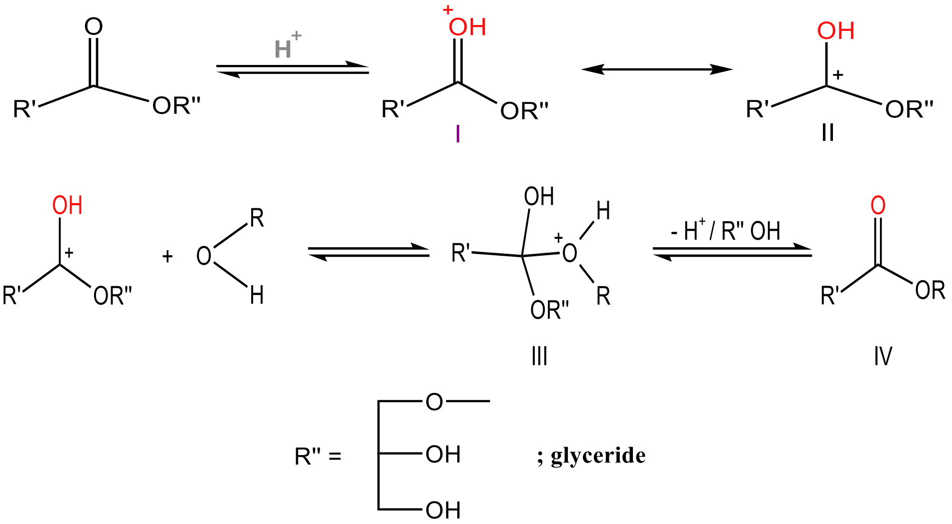


R' = **carbon chain of fatty acid**
R = **alkyl group of alcohol**

Figure 3.9: Base catalysed transesterification

3.8.2. Transesterification catalyzed by an acid

At low to moderate temperature level, acid catalyzed transesterification is a preferred choice. In this process high concentrations of acid catalyst is used to obtain best yields within reasonable time limit. For sunflower oil with 200:1 molar ratio at low temperature and pressure, it takes 4 hrs to complete the reaction. Although acid catalysts are insensitive to the presence of FFA in the feedstock but owing to a huge drawback of slow reaction time they are ignored.



R' = **carbon chain of the fatty acid**
R = **alkyl group of the alcohol**

Figure 3.10: Acid catalysed transesterification

3.9. Heterogeneous catalyst

Heterogeneous catalysts are developed because homogeneous catalysts are difficult to remove from the reaction mixture. The benefits of heterogeneous catalysts include the elimination of corrosion issues, high temperature and pressure reactions, and the formation of soap by triglyceride saponification.

They function poorly compared to homogeneous catalysts, and since there is less surface contact, the catalyst cannot effectively participate in the process and must instead be in a porous condition. The heterogeneous catalyst's surface has to have a hydrophobic character in order to adsorb triglycerides and prevent the adsorption of polar byproducts like water and glycerol. Amongst those which are most frequently implemented catalysts that are solid are the following: earth oxide of alkaline nature, organic bases of solid state, oxides encouraged bases, basic zeolite, insoluble metal salts and hydroxides, basic metal oxide, hydrotolerite, and hetropolyacids.

Comparatively speaking, homogeneous acid catalysts are more efficient in transesterifying free fatty acids (FFAs) than base catalysts. Base-catalyzed reactions move 4000 times faster than acid-catalyzed ones. Acid-catalyzed processes, on the other hand, are less sensitive to moisture and don't produce any soap. Where oil has a greater FFA content, acid catalysts are utilised. Soap will be produced if base catalyst is employed. Esterification occurs in the midst of a catalyst that is acidic in nature in the preliminary cycle of an acid-catalyzed reaction, whereas the reaction occurs without the presence of an acid catalyst in the later or secondary step of transesterification.

3.10. Catalyst from waste aluminium foil

Aluminium foil is emerging out as one of the most consumed aluminium products across the world due to its versatility and multiple uses. The demand of Aluminium foil may rise to 6.4 MMT by 2020. As per aluminium foils market intelligence, aluminium foil is also used in the packaging of beverages and other liquids whose demand is also expected to grow at 4-5 percent by 2022. Growth in the consumption of aluminium foil is increasing due to the change in lifestyle and food habits. The high demand for ready to eat confectionaries and pharmaceutical products is also a reason for the growth of the aluminium foils industry. As per aluminium association, out of total aluminium foil produced in the world, more than 75% is used for food packaging which ends up in the local landfills. The aluminium producing industries are optimistic to recover and recycle aluminium from the used foil. They want to get back all of the used metal at the expense of less energy than that is required to produce the pure metal.

Industries are making significant efforts and investment to explore the different ways to recycle the aluminium from foil over several years. There are many companies in the world supplying the thin foils, used as laminates in drinks cartons. They have carried a feasibility study that shows the possibility to remove the aluminium from laminating material and return the used aluminium to produce more foil. Aluminium oxide (Al_2O_3) known as alumina, is a compound of aluminium and oxygen. The oxides of aluminium are good in hardness, chemical inertness, high melting point, resistance to oxidation and corrosion, so broadly used in making ceramic refractory. The alumina as a catalyst is used in many chemical reactions. Aluminium oxide because of its properties like high dielectric constant, favourable thermal conductivity, excellent stability, low refractive index, and high hardness is used in electric batteries, thermal machinery, optics, and microelectronics.

There are seven phases of polymorphs of transition alumina identified till now. These are named as phase γ (gamma), η (eta), δ (delta), θ (theta), χ (chi), κ (kappa), and ρ (rho). The phase-γ of transition alumina has a larger surface area in comparison to other phases and also has better structural stability. So it is widely used in industries for catalysis processes. The phase γ-alumina is formed conventionally by thermal dehydration of aluminium oxy-hydroxide above 450°C temperature. The γ-phase is a metastable phase which upon heating will form αphase. The phase-γ of alumina is used as a catalyst in various processes due to its high surface area and mesoporous properties. Phase γ-alumina is usually used as a catalyst in various chemical processes such as ammonia synthesis, production of hydrogen gas, hydrogenation of oils, refining of petroleum, emissions control of automotive, etc.

3.10.1. Preparation of $AlCl_3 .6H_2O$ from waste aluminium foil

Waste aluminium foil was cleaned using water and detergent to remove any dirt or oil. Dried foil was dissolved in a hydrochloric acid (HCl) solution containing 6 moles of hydrochloric acid. As aluminium being amphoteric, so can react with acid as well as with base. While reacting with acids, aluminium dissolves in hydrochloric acid to form solution of aluminium chloride and colourless gas hydrogen.

$2Al + 6HCl = 2AlCl_3 + 3H_2$

Aluminium chloride solution is kept for drying under desiccators to form crystalline aluminium chloride hexahydrate ($AlCl_3.6H_2O$). These crystals were dissolved in distilled water and the solution is filtered to remove any soluble chloride. The solution is again dried to re-crystallize. The process of purifying the crystal is done 2-3 times. The complete removal of chloride was verified by adding a drop of silver nitrate ($AgNO_3$) in the sample of the solution. The presence of any chloride content will be indicated by a turbid solution. Finally, pure $AlCl_3.6H_2O$ is obtained.

3.10.2. Preparation of catalyst γ-Al_2O_3 from $AlCl_3$.$6H_2O$

The obtained precipitate of aluminium chloride hexahydrate was dispersed in distilled water. The pH value of the solution was regulated with ammonia solution NH_4OH drop by drop. The solution was stirred continuously at a speed of 200 rpm with a magnetic stirrer.

$AlCl_3(aq) + 3NH_4OH(aq) \rightarrow Al(OH)_3(s) + 3NH_4Cl\ (aq)$

At 120 degrees Celsius, the pale off-white precipitate of Al(OH)3 has been filtered, washed, and dried. The substance was subsequently calcined in a muffle furnace for 2 hours at 500 degrees Celsius and 600 degrees Celsius to produce γ-Al_2O_3.

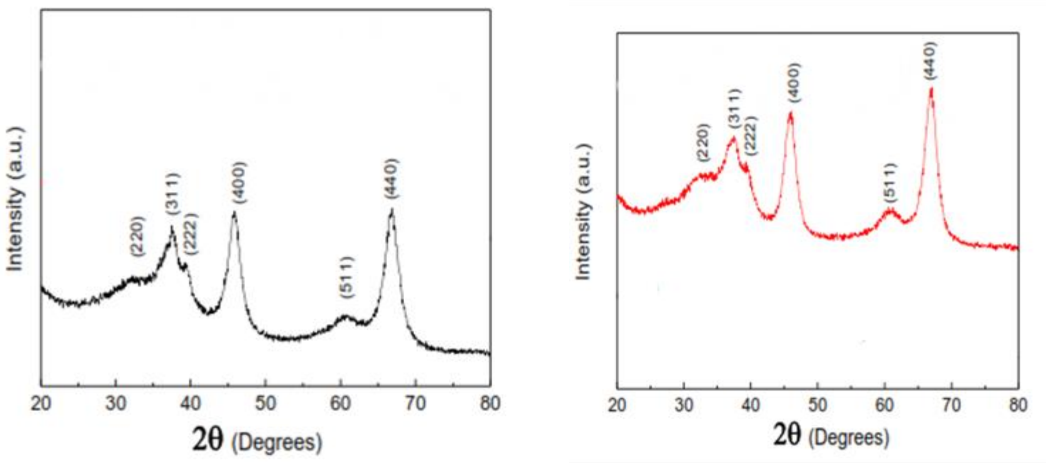
3.10.3. Preparation of catalyst γ-Al_2O_3 /NaOH/Na

The catalyst was prepared by employing the impregnation method using phase γ-Al2O3 and a solution of sodium hydroxide. The phase γ-Al2O3 was impregnated with 15 ml of sodium hydroxide solutions (50 wt%). This impregnated γ-Al_2O_3 was then dried at a temperature of 120°C. The catalyst was then calcined at 650°C for 2 hours.

3.10.4. X-Ray Diffraction analysis

The crystalline phase was verified using X-ray diffraction (XRD) patterns, obtained with CuK radiation of $\lambda = 1.54$ A° in 2θ range varying from 20° to 80° at room temperature as shown in Figure 4 and Figure 5. The diffractograms for the samples at a calcination temperature of

500°C and 600°C have shown three distinctive reflections of 311, 400 and 440 at angle 2θs of 37.2°, 45.6°, and 66.8° respectively. These reflections are found in line with the gamma Al_2O_3 standards. The further increase in the calcination temperature did not have any effect in the position of peaks, therefore indicating no further change in the structure of γ-Al_2O_3.



(a) 500 °C **(b) 600 °C**

Figure 3.11: XRD pattern of γ-Al_2O_3 calcined at

3.11. Process parameters/ Factors affecting transesterification

3.11.1. Reaction temperature

Among indispensable parameters, the temperature of the reactants or the reaction temperature observed to be a significant parameter, since rate of reaction is directly proportional to it. The rate of reaction increases because the viscosity reduces with an increment in temperature and hence the reacting fluid mixes rapidly. But certainly, the temperature rise is limited by the boiling point of the alcohol, it evaporates, leaving the mixture with noteworthy decrease in the biodiesel yield. Another cause of limiting the maximum temperature is saponification of triglycerides. Hence, the range of optimal temperature of trans-esterification varies between 50ºC- 60ºC according to type of fat or oil. It is to be noted that, at room temperature, there is up to 78% conversion in an hour.

3.11.2. Duration of the reaction

With an escalation in the duration of the reaction during the process of transesterification, the transformation of oil into biodiesel takes place at a faster rate. The agitation among the reactants which decides the reaction rate observed to the relatively sluggish during the commencement of transesterification for the reason that the mingling and dissemination action of the alcohol and oil proceeds slowly. As soon as the progression of involvement of oil and alcohol finishes the rate of reaction escalates promptly. If the duration of the reaction increased further, there is no increment in the biodiesel yield but if the reaction time further increases, this can leads to the drop in the biodiesel yield owing to the converse reaction of transesterification, causing soap formation.

3.11.3. Molar ratio (methanol: oil ratio)

Methanol along with triglycerides react with one another to produce glycerine. The total quantity of alcohol that is used in the process of transformation of oil into biodiesel is directly proportional to the precise quantity of oil utilized. One of the important parameters that affects the creation of biodiesel happen to be the molar ratio of methanol to triglyceride which is 3:1in a chemically correct mixture for transesterification. It yields 3 moles of the fatty acid methyl esters and 1 mole of glycerol. It fluctuates between 3:1 and 7.5:1. With enhancing the quantity of methanol, the state of equilibrium of the reaction shifts from left to right, boosting biodiesel generation.

3.11.4. Catalyst concentration

Alkali and acidic catalysts constitute the two main categories of catalysts. Enzymes fall under a different group of biological catalysts. Transesterification that is catalysed by alkalis moves more quickly than reactions that are catalysed by acids. Vegetable oily substances experiencing high levels of FFA and substantial moisture content, on the other hand, transesterify more effectively with catalysts that are acidic. Alkaline catalysts are advantageous for commercial application because of their quick esterification and less damaging effects on industrial equipment [1].

3.11.5. Varying input power

The energy used to heat up the samples, partially generate the effect of dielectric heating. Therefore, it is an important process parameter to be observed. The sample's rate of heating is also impacted by changes in input power, which directly affects how much heat is produced in the sample per unit of time. At each input power, the hot spots develop in the sample. These hot spots, would first approach the boiling point[108].

3.11.6. Change in turntable speed

The discriminatory heating of microwave ovens is one drawback when using them for cooking or heating. They have "hot spots" that show where the concentration of microwaves is. Turntables are frequently seen inside microwave ovens to aid in cooking food more uniformly. To assist avoid hot spots, a turntable of variable speed must be affecting the reaction rate. Hence, the change in turntable speed was identified as another process parameter to be observed.

3.11.7. Change in cooling fan speed

In general, it is dangerous to operate a microwave without a cooling fan within the system or with a defective one. The microwave's cooling fan is in charge of expelling heat from the device; without it, the microwave risks getting quite hot. This may result in a fire or other types of harm to sample or the equipment. The fan is also in charge of eliminating steam and moisture, which, if removed improperly, might harm the microwave. For the safety of the equipment and sample the change in cooling fan speed might improve the performance of the magnetron of the microwave. Therefore, it is considered as another process parameter to be observed.

3.12. Transesterification through microwave irradiation

As it is already mentioned in chapter 1, there are multiple techniques that combine different reactants to produce biodiesel, including mechanical stirring, which involves using a motor-driven mechanical stirrer to mix oil and alcohol. The most popular traditional approach is this one. The hydrodynamic cavitation approach, involves mixing of two phases of reactants under cavitation situations shaped by changing the pressure conditions, resulting which a change is noticed in the system's geometry to make velocity difference, has limits related to greater reaction times and capacities. The approach is often complicated and limited to small-scale biodiesel production. Another technique is called ultrasonic cavitation, which works by irradiating a liquid with enough ultrasonic energy to cause cavities to form in it (oil and alcohol are immiscible with one another). Thus, tiny, fine bubbles are created, and as they collapse at various locations throughout the reactor, they unsettle the phase boundary between two immiscible liquids, causing the mixture to emulsify. However, this method is also found to be time-consuming and is only suitable for small-scale biodiesel production. Several researchers have previously used industrial microwave irradiation to treat various feedstocks. Additionally, some people have experimented with trans-esterifying various feedstocks using their domestic microwave.

3.13. Generation of biodiesel from cottonseed oil

Processes including filtering, FFA measurement, alkaline catalyzed, microwave assistance, washing and drying process are applied to derive biodiesel from cotton seed oil.

3.13.1. Oil filtration

Cotton seed oil included more moisture and several additional impurities. It has been cleaned up and refined to remove contaminants. The specimen has been preheated in order to reduce its moisture content. The oil from a clean raw material is sampled to determine its FFA concentration.

3.13.2. Determination of FFA

Titration has been used to determine the amount of FFA present in the sample. Alcohol (methanol) is first combined with the cotton seed oil. Then, water and sodium hydroxide (NaOH) are added to the alcohol and oil mixture. This procedure is repeated until all of the FFA has reacted. The mixture's pH can be used to demonstrate this. It is confirmed that all of the FFA has been reacted when the pH arrives at around 9. A 0.1% NaOH solution made up of one gram of NaOH was added to one liter of distilled water. To determine the end point, or when the reaction was complete, phenolphthalein solution, an indicator, was utilized. 10ml of methanol and 1ml of cotton seed oil are combined in a smaller beaker. The combination was gently swirled until all of the oil had completely dissolved in the alcohol and the liquid had become transparent.

Utilizing a burette, two or three drops of the indicator solution were added to the combination of oil, alcohol, and indicator. The 0.1% NaOH solution was then added drop by drop to the mixture, stirring constantly, until the solution became pink. The amount of NaOH to be used is determined by the number of ml of 0.1% NaOH solution. According to the preceding data, cottonseed oil contains up to 0.64 percent free fatty acids, which is below the 2-percent threshold and indicates that the process is one step. The transesterification of cottonseed oil to get biodiesel from it doesn’t involve acid esterification.

3.13.3. Mixing of Methanol and Catalyst

When the reaction is complete, a new compound called methoxide is formed by combining the catalyst (KOH) with the alcohol (methanol). The amount of alcohol used should be equal to 20% of the amount of cotton seed oil. The hazardous chemical substances alcohol and KOH are, but methoxide is more harmful. Skin contact with these chemicals is not recommended. Vapour inhalation should not be done. When working, gloves are used. When dealing with these compounds, the area must always have enough ventilation.

3.13.4. Microwave irradiated trans-esterification of cotton seed oil

A 23-liter household microwave oven, with base power input of 230V, 50 Hz supply, microwave output power of 800W, 2.45 GHz and power ingestion equals to 1250W have been rationalised suitably for experimental events. Potassium hydroxide (KOH) and methanol were combined. The catalyst and methanol mixture was then added to the beaker containing the cottonseed oil. The beaker containing the cottonseed oil was placed within the microwave's cavity for microwave irradiation. Using the turn table's various speeds, the reaction solution swirled itself while the trans-esterification process was underway.

3.13.5. Removal of Glycerol

Allowing sufficient time period for the glycerol to get settle down at the bottom of the jar after trans-esterification is necessary. Because glycerol weighs more than biodiesel, this occurs. Even though the settling will start right away, the mixture has to be allowed for at least eight to twelve hours. Glycerol then begins to settle, and biodiesel begins to float above it. The biodiesel is then glycerol-free.

3.13.6. Biodiesel wash

Biodiesel cleansing or its washing can be defined as a process employed to get rid of scattered impurities in it. These impurities incudes remains of different reactants such as catalyst salt, glycerin, as well as methanol. Because these contaminants have been heavier in comparison to the biodiesel they settle during separation, providing the washed biodiesel. The process described here may result in the loss of some amount of biodiesel.

In the present work, the cleansing to biodiesel was successfully carried out through the addition of three quarters of distilled warm water to contaminated or raw biodiesel and carefully shaking the mixture. The layer on top had been washed biodiesel, with unwanted substances collecting in the base layer.

3.13.7. Biodiesel drying process

Subsequent to the washing with water, the produced biodiesel had been dried to 110°C to evaporate any remaining water residue before filtration and character analysis.

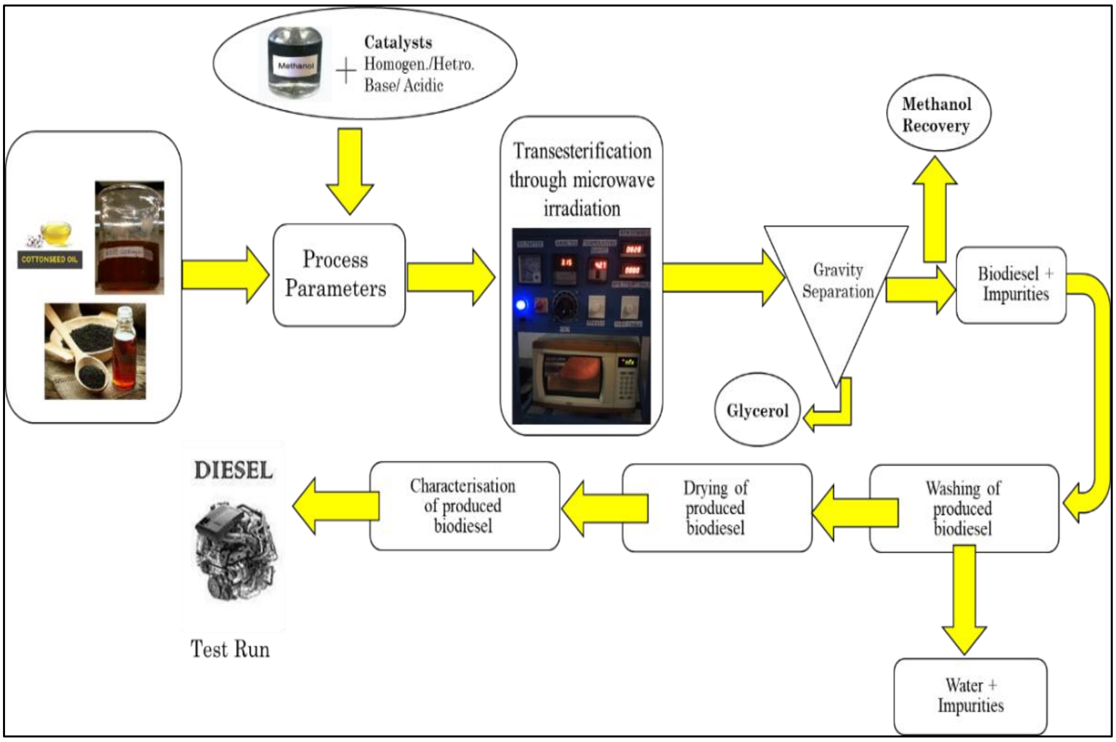


Figure 3.12: Schematic diagram for microwave irradiated trans-esterification

3.14. Generation of biodiesel from kalonji oil

The process of generating biodiesel from another feed stock i.e. kalonji oil is more or less similar to the earlier described for the cottonseed oil. However, it includes the FFA determination of kalonji oil, which is found to be 4.7%. Transesterification in two steps has been suggested for such feedstocks. The same microwave reactor as described earlier had been used for this experimental procedure also.

In the course of the initial phase of the reaction known as esterification, the crude kalonji oil, methanol, and the sulfuric acid solution were mingled together and agitated gradually just for 15 minutes in a customized microwave under the specified parameters such as its temperature (60°C to 65°C) as well as atmosphere pressure. The resulting mixture was subsequently put to the separating funnel for 10-15 minutes to extract the glycerin from it. The subsequently separated oil came to be known as "pretreated oil," and the FFA amount was found to be less than 2% i.e. 1.7%.

The kalonji oil had been thereafter transesterified approximately for a period of five minutes at a stable temperature of somewhere 60°C -65°C using

KOH as a catalyst for the reaction in an appropriate molar ratio of oil and methanol. The methanol to oil ratio was established to 9:1 for the purpose of the transesterification phase, with a catalyst that had a concentration equal to 1.5 wt%. Furthermore, the pretreated oil was retained in the separating funnel for approximately 10 minutes to separate biodiesel from glycerol.

Before filtering, the biodiesel washed off with luke warm water followed by its drying out at 110°C in order to remove any remaining water residue from the resulting biodiesel. Figure 3.12 depicts the process run illustration.

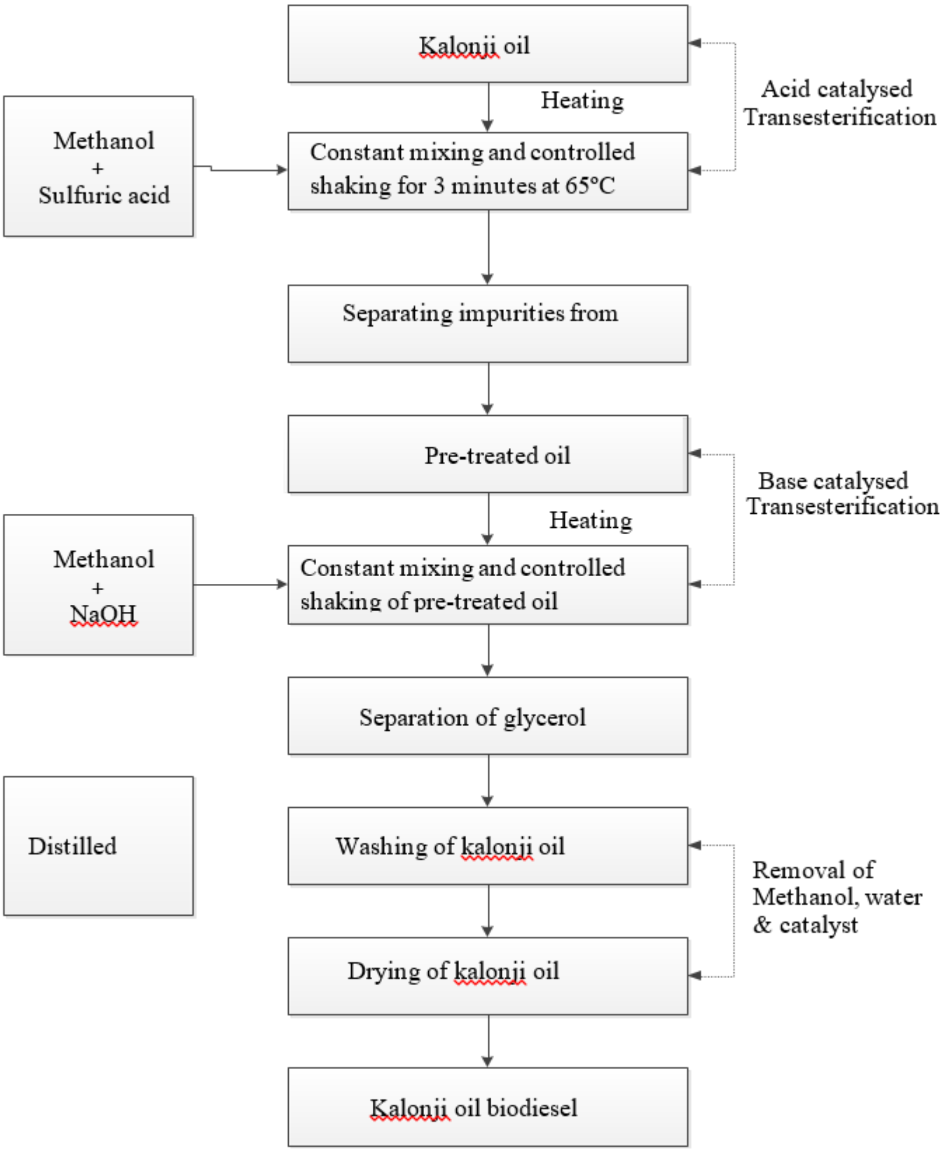


Figure 3.13: The process flow for kalonji oil's microwave irradiated transesterification

3.15. Generation from waste cooking oil

i. WCO sample was collected in a glass beaker. To eliminate any solid contaminants, it was filtered using a clean cloth. To eliminate any moisture existing in the oil, the oil was heated to 105°C. The oil was then allowed to cool to 60°C before being treated further.

ii. The catalyst (γ-$Al2O_3$ / NaOH) powder (3 % by weight of oil) was stirred in a molar ratio of 15:1 with heated methyl alcohol (CH_3OH) and poured into the glass beaker carrying oil.

iii. 100 gm WCO sample was transferred to a round bottom flask. It has been treated with a CH_3OH solution and the prepared catalyst. The reactant mixture in the flask was treated in a microwave reactor. The reaction temperature was recorded at 60°C. Throughout the reaction, the solution was constantly agitated. The speed of the turntable stirring was kept constant at 5 rpm throughout the process.

iv. To avoid alcohol boiling, the reaction temperature of the solution was not allowed to increase over 60°C. The procedure was repeated for 60 minutes.

v. The reaction catalyst was removed from the reaction product. The remaining solution was allowed to settle in a separate funnel. The reaction products, bio-diesel, methyl alcohol, and glycerin, were separated based on their specific weight.

vi. Because biodiesel and methyl alcohol are lighter in weight, they settled on top of the solution, leaving glycerin at the bottom. The distillation procedure was used to extract the methanol from the biodiesel.

3.16. Taguchi method of process optimization

3.16.1. Background

The technique of writing out the parameters of a study that include numerous components was first put forward by Sir R. A. Fisher and was known as factorial design of experiments. Because the majority of studies usually utilize a significant number of variables, a high number of trials have been identified to be required in this procedure. To reduce the number of trials to a manageable level, just a small portion of the entire available alternatives will be selected, and Taguchi creates an outstanding collection of fundamental design guidelines for factorial experiments that span a wide range of situations.

3.16.2. Definition

Taguchi developed an unusual think about arranging trials according to predefined principles. The approach made use of an orthogonal set of matrices. The standard matrix specified ways to perform the smallest amount of tests necessary for offering comprehensive information on all factors influencing the performance. The crux regarding the orthogonal matrix approach was selecting the levels of the input design variables before every trial.

3.16.3. An illustration of an orthogonal array

An orthogonal array acts as a basis for orthogonal array testing. Orthogonal array testing can be defined as a technique that enables the minimization of test runs in an activity having multiple factors and interactions. An orthogonal array is a mathematical model which ensures balanced and uniform coverage of all possibilities.

3.16.4. Assumptions of the Taguchi method

The model primarily operates under the assumption that there are no dependencies between the distinct variables and products. This indicates that the initial influence of the distinct output parameter doesn’t rely on any other level adjustments other than individual elements, and vice versa.

3.16.5. Designing of an experiment

The design of an experiment contains the subsequent steps

1. Independent variables selection
2. Level setting numerical being chosen for every variable that

is independent

3. Orthogonal array selection
4. Giving every single column a variable that is independent
5. Performing the experiments
6. Analysis of data
7. Interpretation

3.16.6. Independent variables selection

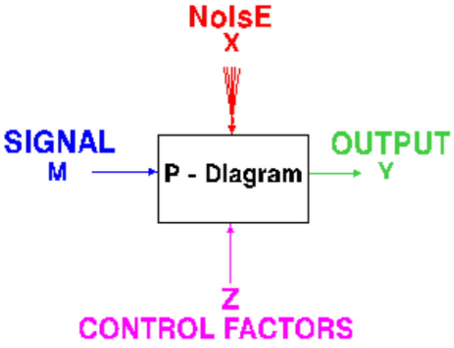
The very initial stage is to identify the variables/ priority factors impacting the end result. To compile the list of these factors, test entry is used.

3.16.7. Choosing the number of levels

Post- selection of individual variable, no. of levels are determined. Selection of the levels are defined based on the performance parameters because of the impact of different levels of adjustments. The level correction frequency needs to be 2 if the effectiveness variable reflects a linear function of the independent variable. However, in case it appears that if the independent variable is not a linear relationship then 3, 4, or higher levels can be achieved. The selection of the higher number of levels depends on whether the connection is quadratic, cubic or higher. In case no correlation between a variable parameter and independent performance is found, two-level configuration can be selected. Experimental data is analysed, based on it, it is decided to make changes in the variable levels needed or not, appears to be the function of percentage of contributions as well as calculation error.

3.17. Robust Design

The existing production processes are still not suitable for mass production owing to weak production yield. Temperature and humidity fluctuations, alterations to raw materials, as well as process parameter deviation are examples of industrialized production variations. These factors are also termed as that are very difficult to manage or too expensive to be controlled.



P- Diagram for Dynamic problems

Figure 3.14: Signal to noise diagram of Taguchi method

The primary objective is to analyse process limitations and extreme ways to control them, without affecting the quality of products or process. One of the goal of this design is to ascertain the values variable for the appropriate design attributes, but not for the ungovernable noise attributes. In order to accomplish this, however, the systematize factors, additionally referred to as inner array characteristics, has been through analysis adjusted in accordance with the inner orthogonal array's predefined range.

3.18. Experimental Data

3.18.1. Design of experiment for this study

In addition to among the most often utilized process variables of input power, reaction time, and oil:methanol molar ratio, the speed of the turn table and cooling fan speed have been taken into account in the present study. Minitab 17 has been employed to carry out the design of experiment using the Taguchi method. A four-level design with 2 to 5 elements has been chosen. The various input power levels were chosen to be 200, 300, 400, and 500W. Four levels of duration of reaction 4, 6, 8, and 11 minutes were chosen, as well as oil:methanol molar ratios of 1:4.5, 1:6, 1:9, and 1:12. The newly incorporated turntable speed and cooling fan speed parameters were set to 10, 20, 30, 40 rpm and 800, 1000, 1200, and 1500 rpm, respectively. The software suggested a Taguchi Orthogonal array design with L16 (4^5) and 5 factors with 4 levels each. In all, 16 runs have been suggested and are listed in table 3.7 below.

Table3.7: Experimental data for cottonseed oil

Input Power (W)	Reaction Time (minutes)	Oil: Methanol Molar Ratio	Turntable Speed (RPM)	Cooling Fan Speed (RPM)
200	4	1:4.5	10	800
300	6	1:4.5	20	1000
400	8	1:4.5	30	1200
500	11	1:4.5	40	1500
200	11	1:6	20	1200
300	8	1:6	10	1500
400	6	1:6	40	800
500	4	1:6	30	1000
200	6	1:9	30	1500
300	4	1:9	40	1200
400	11	1:9	10	1000
500	8	1:9	20	800
200	8	1:12	40	1000
300	11	1:12	30	800
400	4	1:12	20	1500
500	6	1:12	10	1200

Simultaneously, for the second experimentation with kalonji oil having FFA content more than 2% thereby recommended two step transesterification similar approach was followed but with 4 process parameters including the reaction temperature. Suggested runs are listed in table 3.8 below.

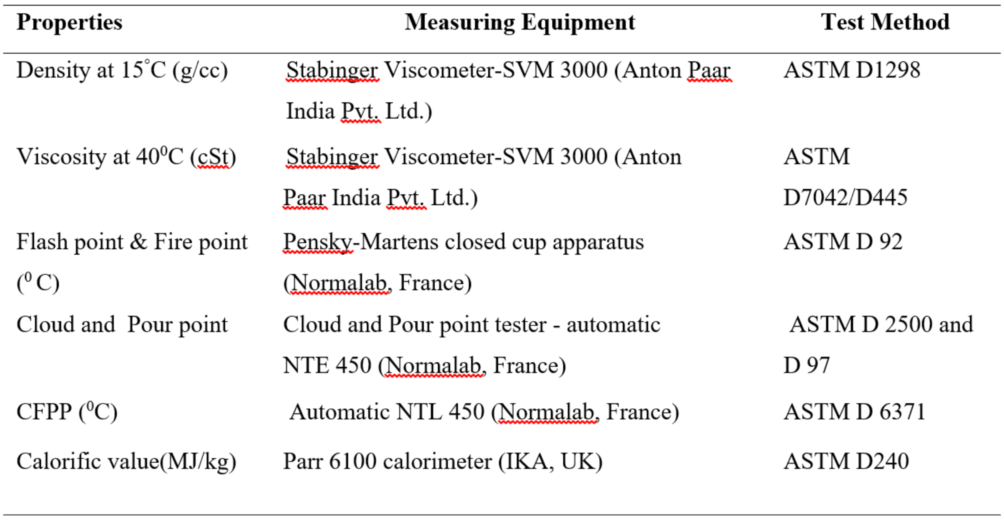
Table 3.8: Experiment sets showing suggested runs for kalonji oil

Experiment No.	Variable 1 Methanol: oil molar ratio	Variable 2 Catalyst (wt%)	Variable 3 Temp. (ºC)	Variable 4 Time (min)
1	3:1	1	63	37
2	3:1	1	62	35
3	3:1	1	65	35
4	3:1	1.5	60	32
5	3:1	1.5	59	33
6	3:1	1.5	61	32
7	3:1	0.75	58	38
8	3:1	0.75	60	37
9	3:1	0.75	60	39
10	6:1	0.75	62	32
11	6:1	0.75	65	31
12	6:1	0.75	58	30
13	6:1	1	59	31
14	6:1	1	64	30
15	6:1	1	62	31
16	6:1	1.5	65	28
17	6:1	1.5	60	26
18	6:1	1.5	55	29
19	9:1	0.75	61	25
20	9:1	0.75	60	26
21	9:1	0.75	62	23
22	9:1	1	61	24
23	9:1	1	57	23
24	9:1	1	59	25
25	9:1	1.5	59	21
26	9:1	1.5	62	20
27	9:1	1.5	63	19

3.19. Physio-chemical properties analysis and equipment

The combustion of the diesel-air mixture, exhaust emission, quality of fuel, and calorific value, ignition characteristics, and engine starting in cold weather conditions (cloud point, pour point, and cold filter clogging point) are all significantly influenced by physio-chemical attributes. The attributes of lubricity, cleaning effect, and viscosity are connected to wear of engine components. The qualities of the materials used to make the fuel system are quite important. Table 3.9 lists the parameters, tested in accordance with several standards, including viscidness, density, flash point, cloud point, pour point, cold filter plugging point, calorific value, and oxidation stability. It also illustrates the many equipment used to determine these qualities.

Table 3.9: Equipment and test procedures for evaluating properties



Properties	Measuring Equipment	Test Method
Density at 15°C (g/cc)	Stabinger Viscometer-SVM 3000 (Anton Paar India Pvt. Ltd.)	ASTM D1298
Viscosity at 40^{0}C (cSt)	Stabinger Viscometer-SVM 3000 (Anton Paar India Pvt. Ltd.)	ASTM D7042/D445
Flash point & Fire point (^{0} C)	Pensky-Martens closed cup apparatus (Normalab, France)	ASTM D 92
Cloud and Pour point	Cloud and Pour point tester - automatic NTE 450 (Normalab, France)	ASTM D 2500 and D 97
CFPP (^{0}C)	Automatic NTL 450 (Normalab, France)	ASTM D 6371
Calorific value(MJ/kg)	Parr 6100 calorimeter (IKA, UK)	ASTM D240

CHAPTER 4

MICROWAVE ASSISTED BIODIESEL PRODUCTION

4.1. Introduction

In this Chapter biodiesel is produced from microwave assisted transesterification and conventional magnetic stirring method. This chapter contains the details of biodiesel production by using cottonseed oil, kalonji oil and waste cooking oil emphasizing more on assistance of microwave irradiation than conventional magnetic stirring method and results of these methods are relatively compared. The properties of cottonseed oil, kalonji oil and waste cooking oil has already been tabulated and compared in table 3.2, 3.5 and 3.6 respectively.

4.2. Cottonseed biodiesel production

The characterization of feedstock, experimental setup etc. has already been discussed in the previous chapter. In the present work, the cottonseed oil's conversion to biodiesel was accomplished using methanol of purity-99%, potassium hydroxide pellets (Fischer scientific, purity-99.5%). The experimental set-up involved a modified microwave reactor in this study. To carry out experimental work, a 23.0-liter stainless steel domestic microwave oven with the specifications as- Make-SANYO, Model-EM-G3686WY, Power/source 230V, 50Hz, Output Microwave power 800W, 2.45 GHz, Power Consumption-1250W was modified suitably. The updated and modified domestic microwave oven specifications for this work are presented in table 3.1. The corresponding range of experimental values was taken from 200W to 500W, 4 to 11minutes, 1:4.5 to 1:12, 10-40 rpm, and 800-1500 rpm. For all experiments in this analysis, the volume of the catalyst was kept constant as 1 percent.

During Primary investigations there were incomplete reactions, low yield, poor biodiesel quality and equipment overheating were observed at the values below the minimum values of parameters considered. Therefore, the configured values for different experimental parameters were considered as the parameter's lower value. Figure 4.1 shows the schematic diagram of this process. Different runs for conversion from cottonseed oil to biodiesel were conducted with a predefined volume of cottonseed oil, methanol, and catalyst. Specific reaction time slots were set for the reactions in the microwave oven for each cycle and the samples were allowed to cool and phase-separation occurred at the end of each cycle in separating funnel. It was also experienced that, the separation in the process took place inside the microwave cavity itself during some experimentations. The bio-diesel was drained and washed with lukewarm water and then dried up.

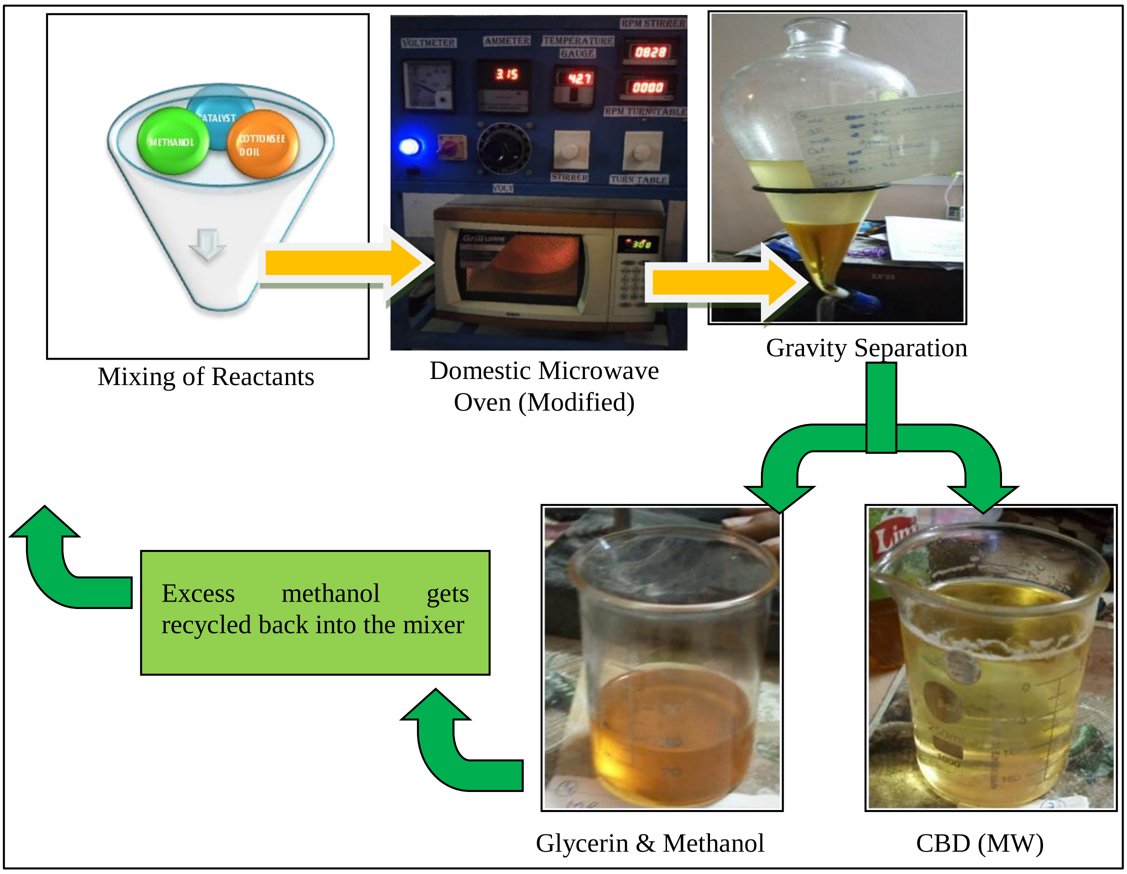


Figure 4.1: Schematic diagram of Cottonseed biodiesel production using microwave

Experiments with oil to methanol ratios ranging from 1:3 to 1:12 [109] for a catalyzed esterification reaction in the solid-phase alkali were performed by various researchers. In this study, the primary selection of 1:4.5 oil: methanol ratio was chosen. The software suggested trial runs were made to achieve the maximum yield from the cottonseed oil and the result are tabulated in table 4.1.

Table 4.1: Experimental data for cottonseed oil

Input Power (W)	Reaction Time (minutes)	Oil: Methanol Molar Ratio	Turntable Speed (RPM)	Cooling Fan Speed (RPM)	Yield (%)
200	4	1:4.5	10	800	91.40
300	6	1:4.5	20	1000	96.34
400	8	1:4.5	30	1200	98.79
500	11	1:4.5	40	1500	99.50
200	11	1:6	20	1200	94.65
300	8	1:6	10	1500	97.63
400	6	1:6	40	800	98.60
500	4	1:6	30	1000	98.65
200	6	1:9	30	1500	92.50
300	4	1:9	40	1200	95.85
400	11	1:9	10	1000	98.91
500	8	1:9	20	800	99.34
200	8	1:12	40	1000	93.15
300	11	1:12	30	800	97.96
400	4	1:12	20	1500	98.12
500	6	1:12	10	1200	99.10

The process flow for kalonji oil’s microwave irradiated transesterification has been summarized in figure 3.12. Total 27 runs were made to transesterify the kalonji oil and obtain biodiesel from it. The obtained biodiesel yield is tabulated in table 4.2.

Table 4.2: Experiment sets showing comparable yields for kalonji oil

Experiment No.	Variable 1 Methanol: oil molar ratio	Variable 2 Catalyst (wt%)	Variable 3 Temp. (ºC)	Variable 4 Time (min)	Yield (%)
1	3	1	63	37	63.585
2	3	1	62	35	62.362
3	3	1	65	35	63.445
4	3	1.5	60	32	66.995
5	3	1.5	59	33	65.568
6	3	1.5	61	32	68.995
7	3	0.75	58	38	64.238
8	3	0.75	60	37	63.951
9	3	0.75	60	39	63.124
10	6	0.75	62	32	81.417
11	6	0.75	65	31	81.138
12	6	0.75	58	30	79.721
13	6	1	59	31	83.562
14	6	1	64	30	83.399
15	6	1	62	31	83.725
16	6	1.5	65	28	87.064
17	6	1.5	60	26	88.531
18	6	1.5	55	29	88.089
19	9	0.75	61	25	90.765
20	9	0.75	60	26	91.231
21	9	0.75	62	23	91.101
22	9	1	61	24	93.475
23	9	1	57	23	92.695
24	9	1	59	25	92.755
25	9	1.5	59	21	96.423
26	9	1.5	62	20	95.425
27	9	1.5	63	19	95.159

4.3. Biodiesel Characterization (CBD (MS & MW))

Transesterification reactions were performed and the yield was measured by three consecutive readings. The average of the three was taken as a final reading. The yield was computed as per the formulae given below.

$$\text{CBD Yield (\%)} = (\text{wt. of the CBD produced} \div \text{wt. of the cottonseed oil}) \text{ x}100$$

Also, the GC-FID technique was used to assess the purity of CBD (MS & MW) based on a relative concentration of methyl ester is shown here in figure 4.2.

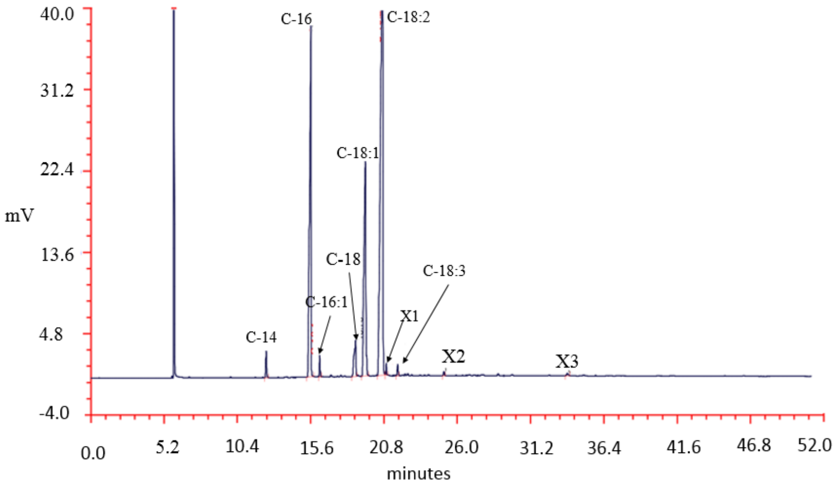


Figure 4.2: GC-FID plot of CBD obtained in the present study

The compositions of obtained values of CBD (MS & MW) are tabulated and compared in table 4.3.

Table 4.3: Composition of methyl esters (%) in CBD (MS& MW) in the present study

Compound	Peak Area		Retention Time (minutes)		Conc.	
	MS	MW	MS	MW	MS	MW
C-14	7510.3	11503.2	12.656	12.437	0.975	0.968
C-16	187602.8	288547.1	15.797	15.616	24.353	24.277
C-16:1	5470.4	8402.1	16.453	16.240	0.710	0.707
C-18	23590.3	36245.1	18.933	18.779	3.062	3.049
C-18:1	143529.6	221978.3	19.659	19.493	18.632	18.676
C-18:2	393540.9	607539.9	20.864	20.720	51.085	51.115
X1	3034.5	4745.0	21.147	20.960	0.394	0.399

C-18:3	3666.2	5793.1	21.984	21.781	0.476	0.487
X2	1187.2	1796.4	25.264	25.056	0.154	0.151
X3	1227.4	2024.9	34.021	33.813	0.159	0.17

Different physicochemical properties of CBD (MS & MW) were measured and are tabulated in table 4.4. The table exhibits the comparison of different physicochemical properties of CBD (MS & MW) in the present study.

Table 4.4: Comparison of different physicochemical properties of CBD (MS& MW)

Property	Test Method	CBD	
		MS	MW
Relative Density at 25 °C (g/cm3)	D 1298	0.9121	0.8837
Kinematic viscosity at 40°C (mm^2/s)	D 445	4.87	4.71
Dynamic Viscosity (at 40°C)- mPa*s	Cal.	44.41	41.62
Heating Value (MJ/kg)		38.94	39.49
Lubricity (HFRR: µm)	Ball wear/scar method	140.2	138.7
Oxidation stability	EN 14112	3.9 hrs	4.1 hrs
Flash point (°C)	D 93	96.7	98.4

4.4. Biodiesel's (kalonji) characterisation

The characteristics of biodiesel derived from specimen oils were measured and summarized in Table 4.5.

Table 4.5: Values of different physico-chemical properties of kalonji bio-diesel

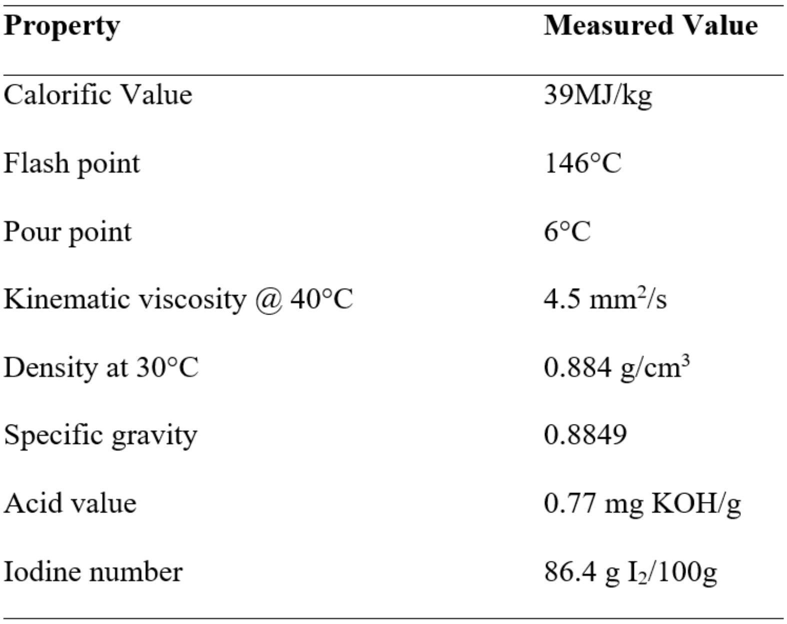
Parameter	Standard	Sample1	Sample2	Sample3	Sample4
Saturated Fatty Acid (%)	EN 14103	30.15	32.6	30.43	33.12
Poly Unsaturated Fatty Acid (%)	EN 14103	38.85	37.23	38.45	30.2
Saponification Value (mg of KOH//g oil)	ISO 1385-1:1977	204.268	205.586	200.942	190.736
Iodine Value (Wij's)	ISO 1385-1:1977	100.156	95.168	96.375	81.535
Cold Filter Plugging Point (°C)	D6371	0.878	5.267	5.367	2.524

Cloud Point (°C)	IS: 1448 [P: 10]: 1970	5.838	6.349	4.229	5.938
Pour Point (°C)	IS: 1448 [P: 10]: 1970	-0.483	0.071	-2.23	-0.375
Oxidation Stability	EN 14112	5.777	5.758	5.658	6.495
Calorific value (CV)	D240	39.408	39.669	38.874	36.658
Viscosity (mm^2/s)	D445	3.721	3.797	3.695	3.445
Density (g/ml)	D1298	0.877	0.882	0.864	0.814

4.5. Waste cooking oil biodiesel characteristics and their significance

The characteristics of biodiesel are mostly determined by the quality of the feedstock and the amount of free fatty acid present in it. In the laboratory of Delhi Technological University, the biodiesel made from WCO has been tested for its chemical and physical attributes. As demonstrated in table 4.4, the sample satisfies the ASTM D6751 and EN14214 standards.

Table 4.6: Characteristics of biodiesel produced from waste cooking oil



Property	Measured Value
Calorific Value	39MJ/kg
Flash point	146°C
Pour point	6°C
Kinematic viscosity @ 40°C	4.5 mm^2/s
Density at 30°C	0.884 g/cm^3
Specific gravity	0.8849
Acid value	0.77 mg KOH/g
Iodine number	86.4 g I_2/100g

The flash point would be a critical factor in the safe storage and handling of bio-diesel for fire safety. The minimal temperature at which the vapour of a liquid fuel forms a combustible structure when combines with oxygen present in the air and produces a short flash in the presence of a minor flame has been defined as the flash point. The flashpoint of bio-diesel produced in this study was determined to be 146°C, which is greater than the flashpoint of petro-diesel fuel (52 to 96°C) [110]. The pour point of fuel determines its ability to flow in the engine at lower temperatures. Fuels with a high pour point should not be used in engines at low temperatures. When subjected to external forces, a fluid's resistance to flow is caused by internal friction. The viscosity of the fuel influences its atomization (spray properties and droplet volume) [111]. For the fuel injection system to function properly, the kinematic viscosity of the fuel should be close to that of petro-diesel [112]. At 40°C, the kinematic viscosity of the bio-diesel produced in this investigation was determined to be 4.5mm2/s. Bio-diesel with a higher density consumes more fuel, resulting in higher nitrogen oxide emissions. The higher the acid value of the fuel, the more likely it is to induce corrosion in the engine's fuel supply system. The acid value in this investigation was 0.77 mg KOH/g, which was close to the ASTM D-6751 norm of 0.8 mg KOH/g. The iodine number represents the occurrence of double bonds in bio-diesel, indicating the extent of unsaturation [113]. The iodine number has an effect on the oxidation stability of bio-diesel.

4.6. Results and Discussion

For a sample size of 250 mg of cottonseed oil, the dissipated energy (Qth) estimated to be 20.59 Wh (@ 600W), with traditional MS process, whereas the supplied energy (Q_{MS}) was found to be 250Wh (25 minutes) for a yield of 153 mg (61.23 percent). However, with the proposed method of modified domestic microwave oven energy supplied (Q_{MW}) was 91.65 Wh in 11 minutes to achieve a yield of 248 mg (99.5 percent). The dissipated energy (Qth) remained the same for both.

In this experimental study, for the generation of 1 kg of biodiesel, the power requirement was calculated as 1.635kWh/ kg of biodiesel with a traditional MS system and 0.369 kWh/ kg BD using the proposed system of the modified domestic microwave oven. The energy efficiency observed for the proposed method found to be substantially higher than that of the conventional MS method.

4.6.1. Effect of varying input power

In microwave heating, the reactants intrigue microwave energy and convert it into heat due to dipole reorientation [35], [114]. Initially, the outermost particles intermingle with microwaves to raise the temperature of the substance. Because of the microwave field, the outermost particles arrange themselves in a manner very much similar to the iron shavings arranges themselves with the presence of nearby magnetic fields. Throughout this experimental analysis, it was found that the yield increased along with the increase in power level up to 500W, but further increase in the power level adversely affected the reaction [42]. In microwave-assisted processes, a large amount of kinetic energy transformed into frictional energy, resulting in the mixing of reactants and heating [115]. It was also seen that within a very short time the temperature of the reactor cavity increased to the optimum level. This resulted in increased methanol evaporation, reducing the potency of the mixture to convert into the bio-diesel. The highest yield of 99.5 percent was achieved in this experimental work at 500W input power with 40 rpm of the turntable and 1500 rpm of cooling fan speed in 11 minutes.

4.6.2. Effect of reaction time

In the proposed process, the minimum reaction time needed for completing transesterification was found to be 4 minutes. Incomplete reactions, lower yield and poor quality of bio-diesel were observed in lesser periods. Therefore the reaction time range for the proposed method was chosen from 4- minutes. Figure 4.3 showed that the current study attained a yield of 61.23 percent in 25 minutes with the popular MS system and a yield of 99.5 percent with the microwave reactor in 11 minutes. Figure 4.4 provided a direct relationship that displayed the linear trend between percentage yield and power of MW. Nevertheless, this relationship appeared to be asymptotic close to 100 percent for higher MW power, and an increase in both (reaction time and MW power) be expected to deliver enhanced performance, but also significantly boost production costs. Therefore an optimum value of MW power and reaction time was obtained that minimized the cost of output.

For transesterification of different samples of WCO, the reaction time ranged from 5 to 90 minutes. It was discovered that with a reaction period of 60 minutes, a maximum yield of 98 % was achieved catalysing with heterogeneous catalyst using microwave's assisted method. There was no apparent increase in yield afterwards.

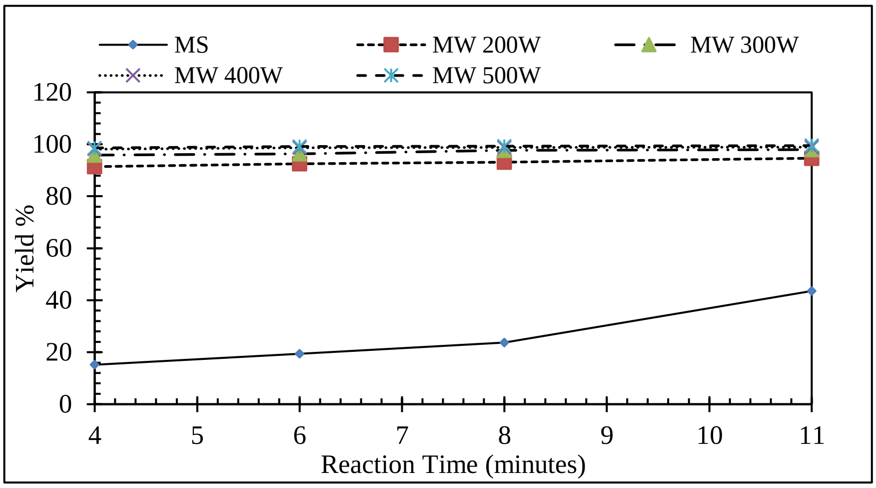


Figure 4.3: Plot for Yield % V Reaction time (Magnetic stirrer (MS) & Microwave (MW) Reactor)

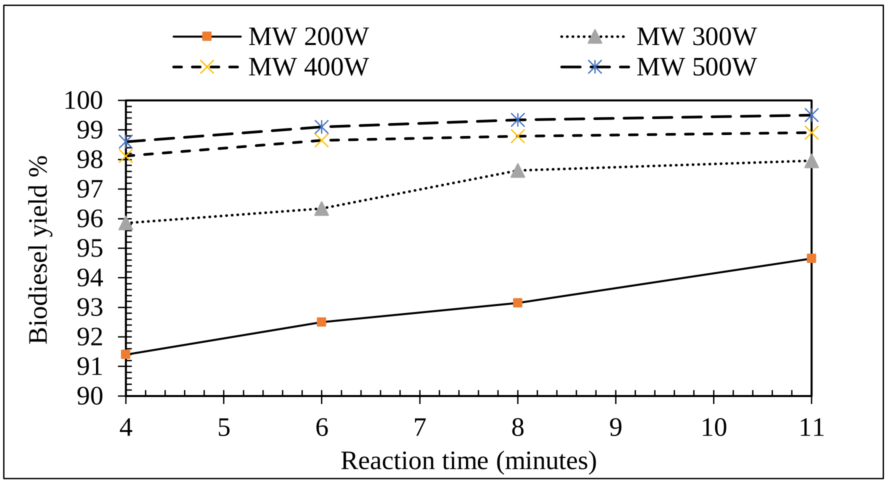


Figure 4.4: Plot for Yield % V Reaction time (MW Reactor)

4.6.3. Effect of methanol: oil molar ratio

For a 20 minute reaction period, figure 4.5 compared the percentage of bio-diesel obtained through the MS and MW operation. As stated in earlier study[109] , which was confirmed in this study, an increase in yield was observed in the MS cycle with an increase in a molar ratio. The MW method has been investigated to have much better performance than the MS method, which in the medium to long term will develop it as a possible candidate for small scale bio-diesel production.

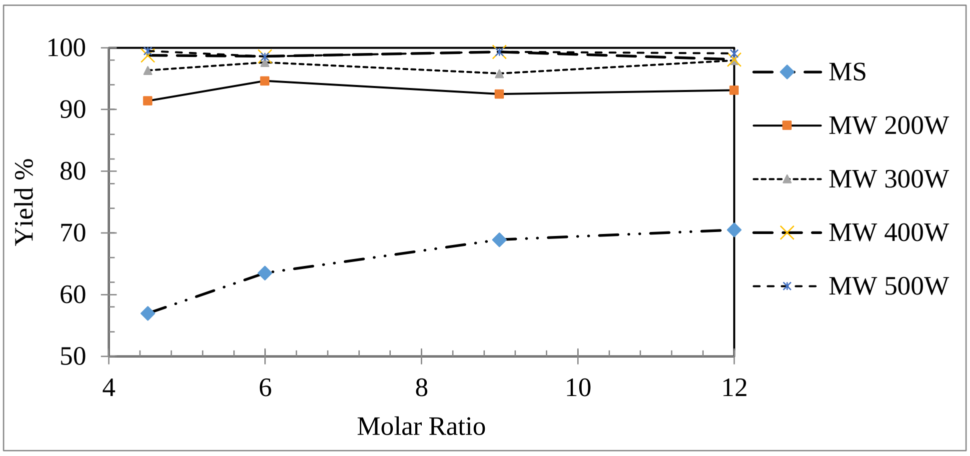


Figure 4.5: Plot for Yield % Vs Molar ratio (MS & MW Reactor)

It was also found that the yield increased linearly with the molar ratio for 200W and 300 W and then resumed to decrease and was almost constant afterward. The decrement with higher molar ratios and no significant change in the yields at higher input power was observed because the methanol had already detached the interaction between triglyceride and fatty acid, and the increase had already been obtained in smaller response time. Methanol consumption was found to be lower and the yield found to be enhanced by 28.5 percent. This resulted in the reduction of the material cost and the methanol recovery expenses.

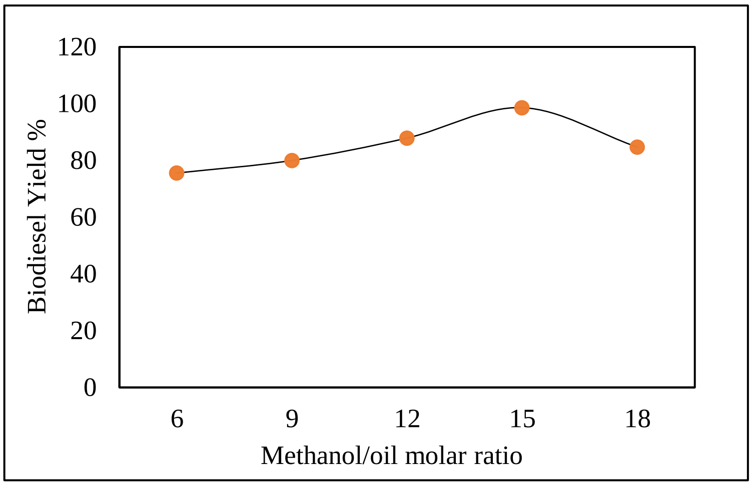


Figure 4.5(a): Plot for Yield % V Molar ratio

While transesterifying the discarded culinary oil (WCO) taking the assistance of modified microwave reactor and using the catalyst made from aluminium foil, it has observed that maximum biodiesel was obtained at methanol: oil molar ratio of 15:1 (Figure 4.5(a)). The excess amount of methanol may be due to non-uniformity in structure of the WCO (being a mixture of so many oils).

4.6.4. Effect of change in turntable speed

Previous research work [38] discussed non-uniform heating in microwaves as one of the main issues to tackle. A turntable system found to be an easy and fairly efficient way of ensuring that the microwaves to be well distributed and thus provide better yield. The effect of different turntable speeds on yield produced at different input power was established in the present study in figure 4. The uniform heating effect was generated with higher energy input, which tends to bring the yield towards the peak. Nevertheless, a comparable CBD (MW) yield of 94 percent was obtained at a lower energy input of 200 W, fan speed of 1200 rpm and turntable rotational speed of 20 rpm. It suggests the potential for substantial energy savings relative to those with higher energy inputs between 400 W and 500 W.

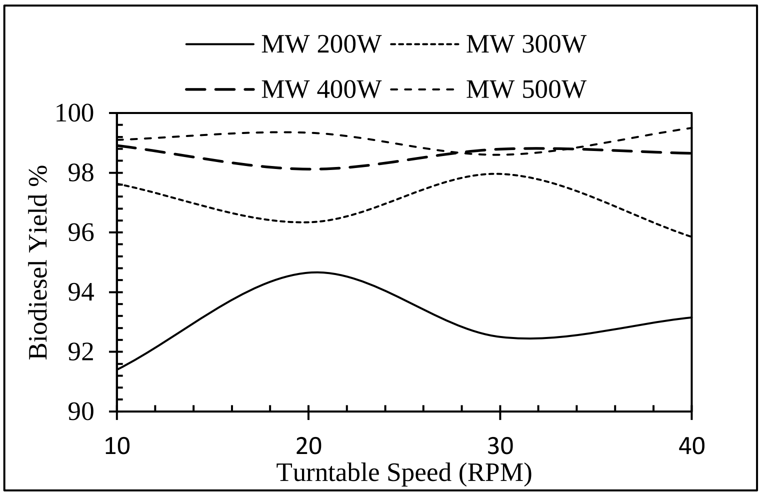


Figure 4.6: Plot for Yield % Vs Turntable Speed

In figure 4.6, yield fluctuation with turntable speed was observed at lower input frequency, which indicates that the microwaves could not produce the uniform effect of temperature across various oil layers at some rpm. Thus turntable rpm established to be one of the crucial parameters for reducing CBD (MW) processing costs.

4.6.5. Effect of change in cooling fan’s speed

The cooling fan of the microwave helps to keep its power components protected from overheating by blowing outside air over the hot magnetron and minimizing condensation chances inside the oven. Like turntable, the blown air also helped in the circulation of the microwaves inside the oven's cavity and uniform heating. In this experimental study, it was observed (figure 4.7) that the yield increased first, then decreased at 200W with a proliferation in the cooling fan speed. Whereas it was studied as contrasting in nature for 300W. This rise and fall found to be the net result of the presence and absence of hot spots, which in some regions helped to complete the reaction and in other locations, the reaction left incomplete. That aggravated the overall reactor performance. The burning of samples was also observed in this experimental study when the device was operated at standard cooling fan speed between 500 and 700 rpm. Which posed a greater risk of accidents. Effective temperature regulation, therefore, has been recognized as an essential safety measures. The effect found to be similar at higher input powers but was not so substantial.

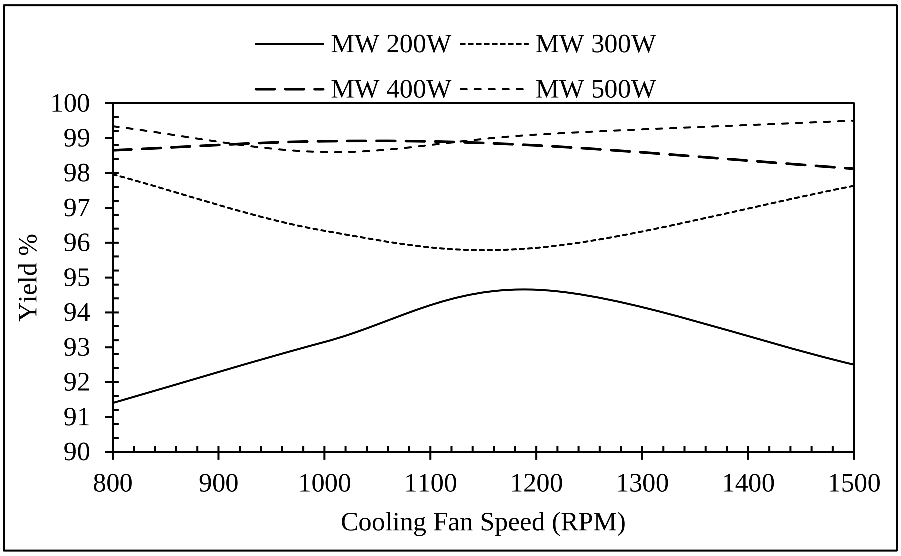


Figure 4.7: Plot for Yield % Vs Cooling fan Speed

4.6.6. Impacts of kalonji biodiesel's too much saturated fatty acid level

The fourth sample had the largest saturated fatty acid amount of any of the samples tested. Biodiesel made from such seeds which has a high content of saturated fatty acids appears to have higher oxidation stability and a higher heating value, but its cold flow qualities are poorer than those of other biodiesels due to the fatty acids solidifying at high temperatures. The higher the concentration of these kinds of acidic substances, the higher the freezing point and the higher the CFPP and CP, resulting in the process of solidification of the biodiesel and the likelihood of its usage in hotter climate regions only[116].

4.6.7. Impacts of kalonji biodiesel's major un-saturated fatty acids level

In the present research, the quantity of unsaturated fatty acids in the sample was revealed in the amounts of 38.85%, 37.23, 38.45, and 30.2%, respectively. It was found that the first sample had the greatest concentration of polyunsaturated fatty acids. It was found from the previous studies that the biodiesel containing the greatest amount of unsaturated fatty acids discharges higher than normal nitrogen oxides as well as has inferior thermodynamic endurance when compared with biodiesel having a greater number of saturated acids. It was found that no noticeable differences in hydrocarbons and smoke emissions throughout the biodiesel fuels tested [117], [118].

4.6.8. Acid number determination in kalonji biodiesel

In this case, the titration approach has been adopted to figure out the acid number of the biodiesel. A beaker was filled with 0.1-0.5 mL of biodiesel. 50 cc solvent mix (95% ethanol and diethyl ether in similar amounts) has been added and carefully stirred. Such solvent-oil combination was titrated with 0.1M KOH using 1% phenolphthalein as an indicator.

4.6.9. Kalonji biodiesel’s calorific value

Bomb calorimeter (Parr 6100) was the instrument utilized to figure out the calorific value of the kalonji biodiesel. 0.5 g of biodiesel specimen was positioned in a container within the device, and an 80-mm cotton thread suspended from an 80-mm nichrome wire was dipped into the biodiesel. The combustible was loaded with oxygen at 400 psi pressure. The insulated container carrying distilled water in it was then placed inside. The fuse wires were strategically placed at the bomb's position. The nichrome wire was attached to a pair of sticks that were linked to the fuse's wires. The starting temperature was recorded, after which it was lowered to zero degrees Celsius. The fire trigger was the device used to create a short circuit on the nichrome string to start the ignition.

4.6.10. Cold filter plugging point and cloud point

The CFPP indicates the lowest achievable temperature to which an identified amount of biodiesel that is 100% pure will continue to transfer across a criterion-referenced filtering system in a minute's time.

4.6.11. The influence of microwave power

The steady increase in microwave power and the methanol to oil molar ratio contributed in lowering the free fatty acid concentration to a crucial level below which it dropped. Excessive microwave intensity may potentially evaporate the alcohol, resulting in insufficient esterification. As the entire power of the microwave rises, so does the actual temperature of the reaction; however, the duration of the interval as well as the chances to be reverse rate both increase as a result, lowering the yield. A significant methanol to oil molar ratio will use the heat from the microwave, resulting in a decrease in activating radiation as dipolar amplification becomes larger. Higher microwave power used over a longer period of time leads in a reduction in yield as well as rapid reaction that can be reversed development.

4.6.12. The effect of catalyst amount

The impact of catalyst loading on the reaction has been investigated. The method was evaluated by adjusting the amount of catalyst from 1 to 5 (wt%) of oil weight, and the effect on biodiesel yield is shown in figure 4.8. The yield increased when the catalyst was increased from 1 to 3. (wt%).

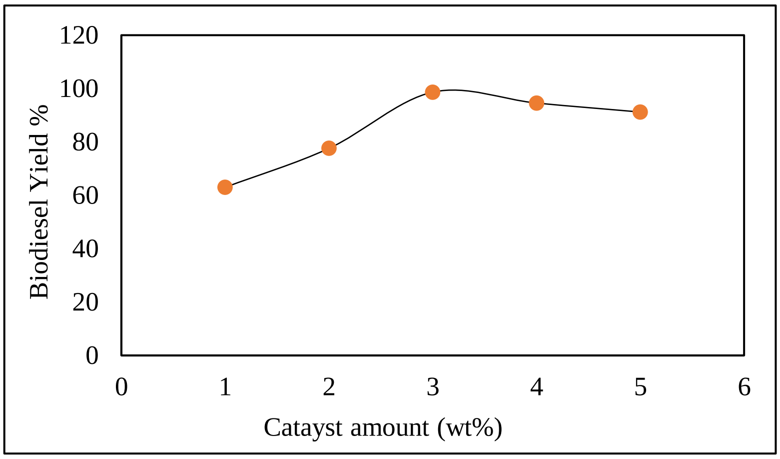


Figure 4.8: Impact of the catalyst on yield

A further increase in the amount of catalyst has lowered the yield. The addition of catalyst apparently made the liquid viscous enough to minimise the likelihood of reactants coming together for the reaction.

4.6.13. Influence of reaction temperature

The transesterification reaction gets influenced by the reaction temperatures. The temperature of the reaction may vary depending on the type of alcohol and catalyst used. Figure 4.9 illustrates an example of the mixture prepared at five different temperatures. It can be noticed that the yield was decreased at a lower temperature range of 50-55°C due to mass transfer and solubility issues.

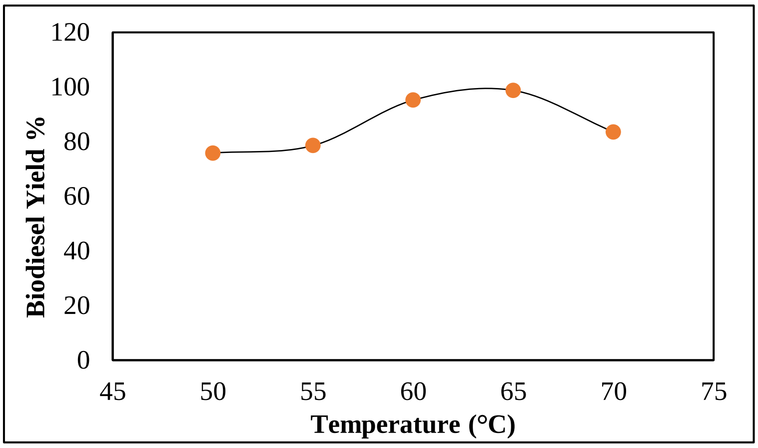


Figure 4.9: Impact of reaction temperature on bio-diesel yield

The viscosity of the mixture was found to be reduced at higher temperatures, which is sufficient to increase the kinetic energy of the reactant and provide good mass transfer between oil and methanol to improve yield. The highest yield was found at reaction temperatures ranging from 60 to 65°C. Methanol began to boil at around 65°C, and methanol evaporate from the mixture, lowering the molar ratio and resulting in a reduced bio-diesel yield.

4.6.14. Reusability of catalyst

The cost of producing bio-diesel has been a determining element in bio-diesel manufacturing technologies. The cost of production can be lowered to some extent by reusing the same catalyst. The recovered catalyst γ-Al2O3 / NaOH has been washed with n-hexane and calcined again at 650°C temperature. In the same experiment, the utilised and rejuvenated catalyst was employed in four successive transesterification reactions with similar regeneration strategies.

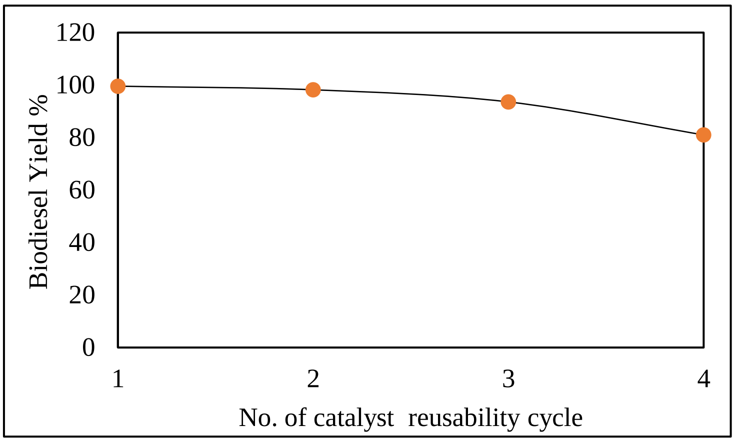


Figure 4.10: Reusability of catalyst

No substantial loss of catalytic activity was seen in the first three consecutive uses of the catalyst, while catalytic activity was observed to be lowered in the fourth run of the experiment, as shown in figure 4.10. This reduction of catalytic activity could be owing to the absorbed organic molecule blocking active sites on the catalyst surface, resulting in less surface area accessible for the reaction site.

4.7. Summary

It has been concluded from this chapter that cottonseed oil transesterification into biodiesel using a household microwave oven was effective. A significant parameter that was effectively used to alleviate the sample burning bottleneck appears to be variation in turntable speed. The paradigm change in the fan cooling's speed strengthened the stirring and addressed the safety concerns, making it a significant new parameter. The testing findings showed that, with an input power of 500W, a molar ratio of 1:4.5, catalyst at 1%, a turntable speed of 40 rpm, and a cooling fan speed of 1500 rpm, a high-quality yield of 99.5% was achieved in 11 minutes. There was a time savings of almost 56 % and a yield that was about 63 % greater than the traditional MS method when CBD (MW) was converted from cottonseed oil.

For kalonji oil, parameters includes system wattage, reaction time, stirrer speed, methanol to oil ratio and catalyst loading. The optimum values for different parameters were found to be 500W, 20 minutes, 30 rpm, 9:1 and 1.5(%w/w) respectively, for this study, and the obtained bio-diesel yield was recorded as 95.47 percent.

The prepared catalyst NaOH/γ-Al_2O_3 (3% by wt. of oil) has been used to transesterify waste cooking oil (WCO) in combination to methyl alcohol having a molar ratio of 15:1 for a reaction time of 1 hour in microwave based processor. The bio-diesel yield has been recorded as 98%.

The suggested method was determined to be an energy-efficient and time-saving alternative to conventional methods, offering higher yields, lower costs, and suitability for small-scale biodiesel production.

CHAPTER 5

TEST RUN OF CI ENGINE WITH BIODIESEL BLENDS

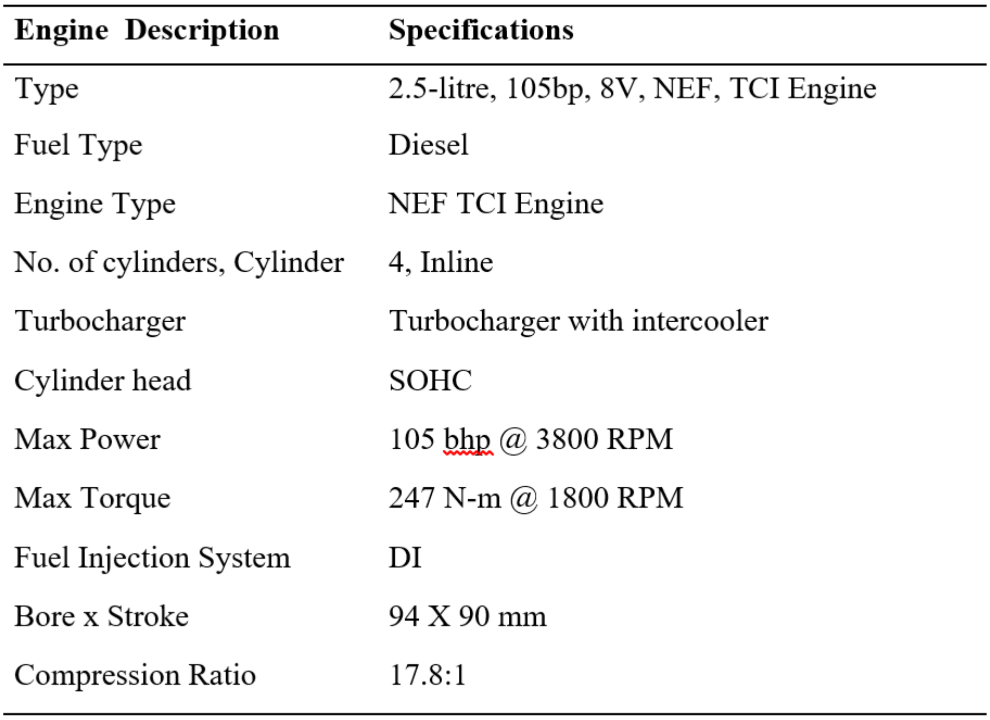
5.1. Introduction to test run on CI engine

This section covers the working effectiveness and engine exhaust testing performed on the blend of biodiesel produced from cottonseed oil utilizing the microwave irradiation process, which was detailed in earlier sections of this report. The section includes information on the engine, the process, and numerous performance and emission characteristics.

5.2. Test Rig

A four-cylinder, four-stroke diesel engine is connected to an eddy current type dynamometer for applying different load on the engine.

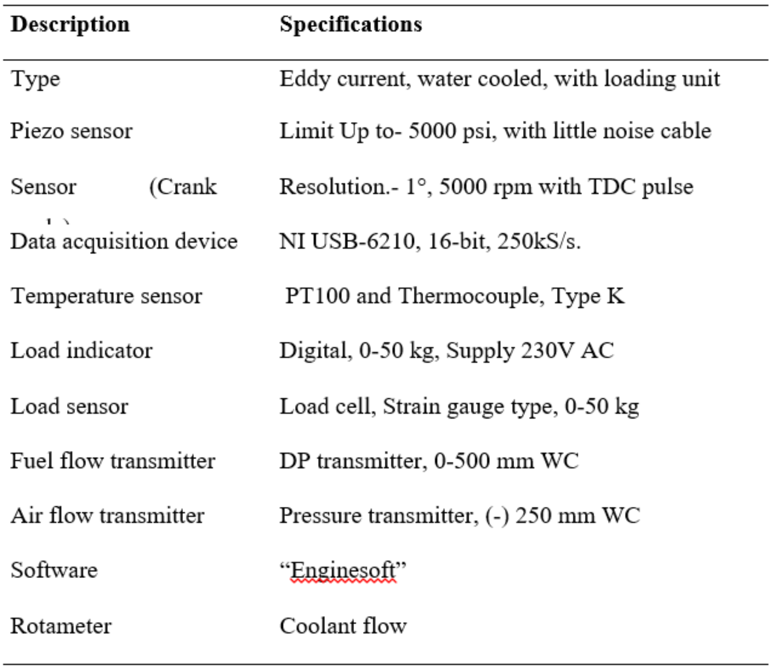
Table 5.1: Technical Specifications of the test engine



Engine Description	Specifications
Type	2.5-litre, 105bp, 8V, NEF, TCI Engine
Fuel Type	Diesel
Engine Type	NEF TCI Engine
No. of cylinders, Cylinder	4, Inline
Turbocharger	Turbocharger with intercooler
Cylinder head	SOHC
Max Power	105 bhp @ 3800 RPM
Max Torque	247 N-m @ 1800 RPM
Fuel Injection System	DI
Bore x Stroke	94 X 90 mm
Compression Ratio	17.8:1

The test rig was equipped with the equipment needed to determine crank angle and combustion pressure. The ability to interface airflow, fuel flow, temperature, and load monitoring was also provided. A standalone panel box with an air box, fuel tank, manometer, and arrangement for measurement of supplied fuel quantity, sensors to monitor fuel and oxygen intake, devices to display load and rpm found to be part of the arrangement. Water cooling was handled by rotameters, and water flow was measured by calorimeter. In order to monitor the engine's smoke opacity when running on a given fuel, a smoke meter was also connected to the engine. This system's design, provides the engine's assessments of performance including engine power (brake as well as indicated), mean bmep, imep, bsfc and bte. For online performance evaluation, the Windows-based engine performance analysis software package "Engine soft" has been made available. The table 5.2 below contains the particular details for the individual components.

Table 5.2: Technical Specifications of the dynamometer



Description	Specifications
Type	Eddy current, water cooled, with loading unit
Piezo sensor	Limit Up to- 5000 psi, with little noise cable
Sensor (Crank	Resolution.- 1°, 5000 rpm with TDC pulse
Data acquisition device	NI USB-6210, 16-bit, 250kS/s.
Temperature sensor	PT100 and Thermocouple, Type K
Load indicator	Digital, 0-50 kg, Supply 230V AC
Load sensor	Load cell, Strain gauge type, 0-50 kg
Fuel flow transmitter	DP transmitter, 0-500 mm WC
Air flow transmitter	Pressure transmitter, (-) 250 mm WC
Software	“Enginesoft”
Rotameter	Coolant flow

The major goal of this experiment was to examine how mixing biodiesel produced through microwave methodology of transesterification with conventional fossil -diesel fuel affects the performance of a diesel engine under various operating conditions. Pictures of the engine configuration that was chosen for performance and emission testing (Test run) are displayed in figure 5.1. Figure 5.2 depicts the schematic diagram of the test set up.

Figure 5.1: Actual engine set-up for test run

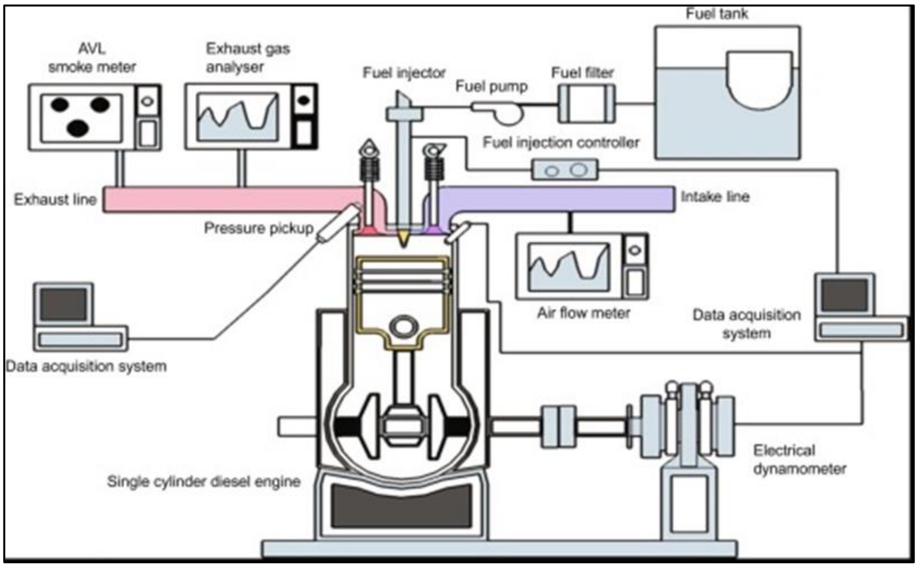
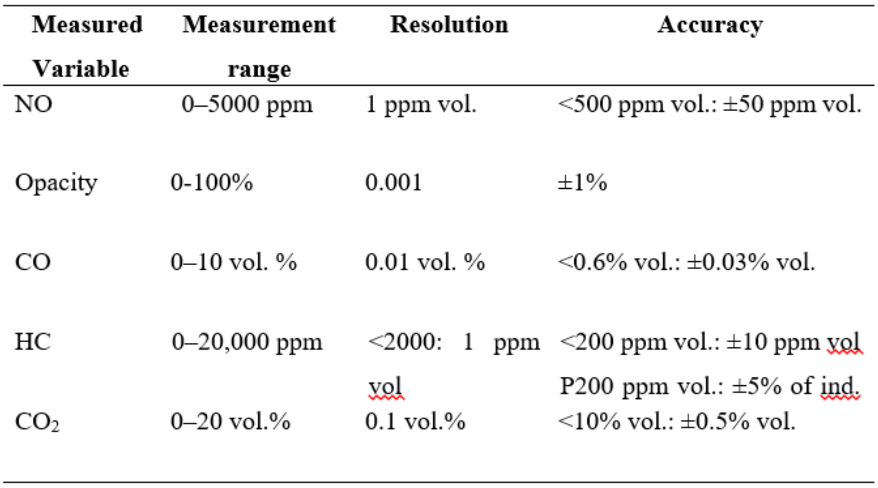


Figure 5.2: Schematic diagram of specified engine set-up for test run

5.3. Multi gas analyser

A multigas analyzer (AVL digass 444) that was awarded ARAI certification was used to perform the emission analysis. Analysis of CO_2, hydrocarbons, carbon monoxide, NO, and O_2 is aided by it. While other emissions were assessed electrochemically, hydrocarbon, CO, and CO_2 emissions were measured with the employment of Nondispersive infrared (NDIR) gas analyzer. The amount of carbon monoxide and carbon dioxide were measured in vol.%. Whereas, to found the concentrations of hydrocarbons and oxides of nitrogen p unit of parts per million of hexane equivalent was preferred. Table 5.3 provides a technical description of the multigas analyzer. Figure 5.3 displays the multigas analyzer in its actual form. All of the information was communicated through the RS232C electronics communication bus, which collected it over a 2 minutes duration having 20 seconds intervals in it. Which, for each instance of the engine run, stored the discharge information throughout a traverse of 2 minutes in 20 seconds regular intervals, it was found to be more significant when compared to a period of reaction of 15 seconds. Before entering the analyzer, the gasses from the exhaust tail pipe are tapped in for analysis and cooling. Leak testing was done before the experiments began. Prior to commencing the experiments, the analyzer had been calibrated and positioned using the essential calibrated gases recommended by the manufacturer.

Table 5.3: Specifications for the AVL DIX exhaust detection equipment



Measured Variable	Measurement range	Resolution	Accuracy
NO	0–5000 ppm	1 ppm vol.	<500 ppm vol.: ±50 ppm vol.
Opacity	0-100%	0.001	±1%
CO	0–10 vol. %	0.01 vol. %	<0.6% vol.: ±0.03% vol.
HC	0–20,000 ppm	<2000: 1 ppm vol	<200 ppm vol.: ±10 ppm vol P200 ppm vol.: ±5% of ind.
CO_2	0–20 vol.%	0.1 vol.%	<10% vol.: ±0.5% vol.

The exhaust gas analyser's sensor had been 45 mm in diameter and made of selenium photocells. The exhaust emissions tests were carried out by inserting a probe into the engine's tailpipe in addition to activating the spinning ball gate. A leakage inspection inside the electronic gas analyzer must be performed prior to executing the test for emission to guarantee that no remaining gases are retained inside the system by mechanically closing the probe's nozzle.

Figure 5.3: Real multigas analyzer set-up

5.4. Statistics acquisition regarding engine effectiveness

5.4.1 Brake thermal efficiency (BTE)

Three pertinent pieces of information have been collected with the explicit objective of calculating the brake thermal efficiency (BTE):

i. Total time calculation for 20 cc of fuel utilization

ii. Calorific value (CV) of the diesel and biodiesel blends and

iii. Density of diesel and its blends.

The equation used to compute the BTE, is given below:

$$BTE = (BP \times 360000/ (Vol.\ fuel\ flow\ rate/ hour \times \rho \times CV)). \qquad (5.1)$$

Brake specific fuel consumption (BSFC)

The equation utilized to determine the BSFC is specified under:

$$BSFC = (vol.\ fuel\ flow\ rate/hour * \rho / BP) \qquad (5.2)$$

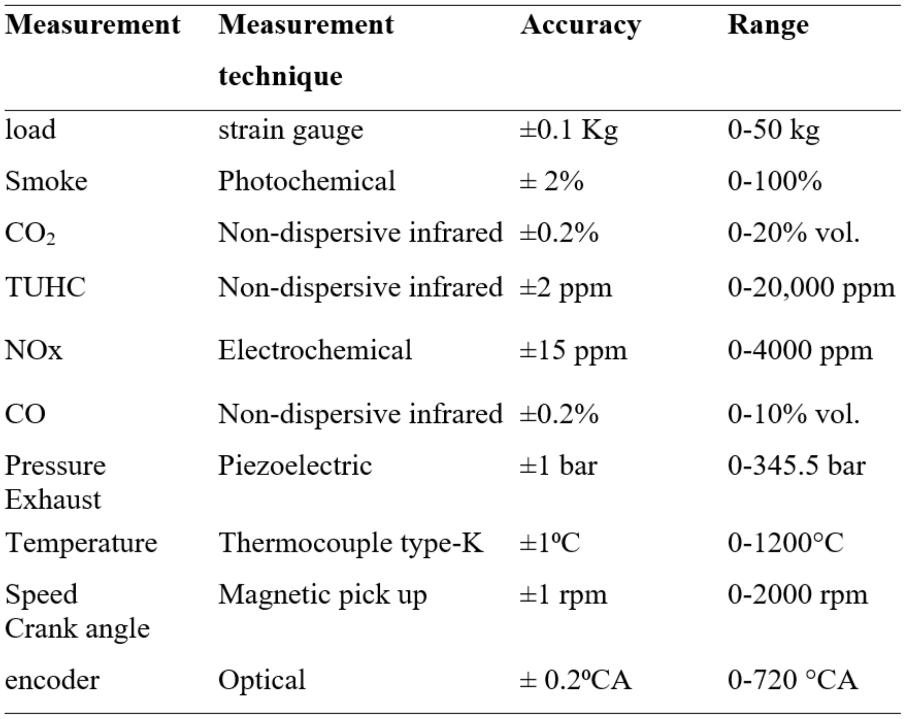
The both BSFC and BTE data was calculated with the help excel sheet by data obtained from data acquisition system utilizing the previously explained equations.

5.5. Estimation of uncertainty

Despite careful testing, when establishing any value, the results always differ from the real values. The accuracy and uncertainty associated with various measurements are shown in table 5.4. All of the experiments demonstrated improved accuracy, it was discovered. To determine the repeatability accuracy of the measurements, all tests were carried out three times and were determined to be adequately near. Numerous measured parameters, including density, viscosity, calorific value (CV), flash point, acid value, fatty acid profile, etc., were noted to be examined three times, with the average result being used for analysis. Because the equipment used had excellent accuracy and were frequently calibrated, the values obtained were approximately similar. As a result, the instruments' uncertainties were relatively minimal.

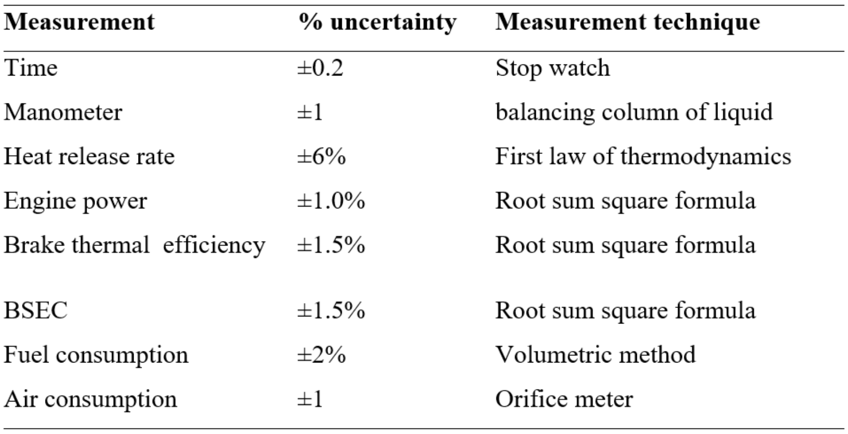
Table 5.5 demonstrates that the few characteristics' uncertainty. As a result, there were relatively few uncertainties in these equipment. The level of uncertainty for viscosity, density, calorific value, and cold flow plugging point was less than 0.3% and performed extremely successfully.

Table 5.4: Accuracies and uncertainties of measurements



Measurement	Measurement technique	Accuracy	Range
load	strain gauge	±0.1 Kg	0-50 kg
Smoke	Photochemical	± 2%	0-100%
CO_2	Non-dispersive infrared	±0.2%	0-20% vol.
TUHC	Non-dispersive infrared	±2 ppm	0-20,000 ppm
NOx	Electrochemical	±15 ppm	0-4000 ppm
CO	Non-dispersive infrared	±0.2%	0-10% vol.
Pressure Exhaust	Piezoelectric	±1 bar	0-345.5 bar
Temperature	Thermocouple type-K	±1°C	0-1200°C
Speed Crank angle	Magnetic pick up	±1 rpm	0-2000 rpm
encoder	Optical	± 0.2°CA	0-720 °CA

Table 5.5: Calculated values of accuracies and uncertainties of measurements



Measurement	% uncertainty	Measurement technique
Time	±0.2	Stop watch
Manometer	±1	balancing column of liquid
Heat release rate	±6%	First law of thermodynamics
Engine power	±1.0%	Root sum square formula
Brake thermal efficiency	±1.5%	Root sum square formula
BSEC	±1.5%	Root sum square formula
Fuel consumption	±2%	Volumetric method
Air consumption	±1	Orifice meter

5.6. Preparation of biodiesel blends

Three different cottonseed biodiesel with fossil -diesel blends B10 (10 % biodiesel + 90 % fossil -diesel), B20 (20 % biodiesel + 80 % fossil -diesel), B30 (30 % biodiesel + 70 % fossil -diesel) stood arranged. Each mix holds an aggregate volume of one litre. These mixes were created specifically for cottonseed biodiesel.

Calorific value of petroleum diesel = 43000 kJ/kg

Calorific value of biodiesel = 39542 kJ/kg.

Density of petro diesel = 821 kg/m^3

Density of biodiesel = 883.70 kg/m^3 [tested in laboratory]

The calorific value and densities of biodiesel blends have been calculated by volume fraction and are shown in table 5.6.

Table 5.6: Description of different blends of biodiesel

Blend designation	**Biodiesel quantity (ml)**	**Fossil–diesel quantity (ml)**	**Calorific value (kJ/kg)**	**Density (kg/m^3)**
Diesel	0	1000	43000.27	821.2
B10	100	900	42570.81	822.7
B20	200	800	42065.67	831.4
B30	300	700	41162.00	839.9
B100	1000	0	39542.95	883.7

5.7. Performance data of load test

5.7.1 Biodiesel blends from cottonseed oil biodiesel

Table 5.7 to 5.12 shows the observations for load test for B0 i.e. fossil -diesel, different blends of cottonseed biodiesel with fossil -diesel (B10, B20, and B30). The proposed average speed remained fairly constant throughout all of the measurements, changing in an extremely small range of 1500 to 1600 rpm. The emitted gas temperatures vary between 150°C at low load conditions to 220°C when under heavy load. In the investigation of all fuel blends, the anticipated rate of mass flow boosts as there is a rise in load. The water circulation rates between the engine and calorimeter were kept nearly constant at 200 llitre per hour and 75 litre per hour, respectively. Emissions data indicate that whenever the biodiesel quantity in the blends goes up, the CO, smoke opacity, and UHC go down, with B100 yielding the best emission result. All of the biodiesel blending percentages had lower quantities of carbon monoxide, total hydrocarbons, and smoke opacity than the fossil -diesel. Tabled below are a few of the performance characteristics observed for various blends:

Table 5.7: BSFC Vs Engine Load%

Load%	B0	B10	B20	B30
100	228.6100	245.2023	253.3032	258.10
91	232.1031	248.1104	257.4015	262.35
83	235.7104	252.1301	261.2701	266.11
69	245.8020	263.2032	273.3830	278.51
55	255.9036	274.2061	284.1023	289.41
41	291.2036	312.2031	323.2124	329.10
28	326.6120	349.6120	362.5123	369.20

Table 5.8: BSEC Vs Engine Load%

Load%	B0	B10	B20	B30
100	10.4013	10.8297	11.0887	11.0669
91	10.5628	10.9979	11.2640	11.2384
83	10.7243	11.1661	11.4393	11.4100
69	11.1839	11.6445	11.9653	11.9248
55	11.6434	12.1230	12.4474	12.3966
41	13.2518	13.7977	14.1567	14.1124
28	14.8603	15.4724	15.8660	15.8282

Table 5.9: BTE Vs Engine Load%

Load%	B0	B20	B30	B10
100	30.9021	27.8100	25.0290	29.3550
91	32.5621	29.2500	26.3250	30.8750
83	33.7420	30.3300	27.2971	32.0152
69	32.2350	28.8000	25.9215	30.4145
55	29.5063	26.5557	23.9001	28.0310
41	23.1433	20.8290	18.7461	21.9861
28	15.4083	13.8675	12.4807	14.6379

Table 5.10: Emission parameters for CO Vs Engine Load%

Load%	B0	B10	B20	B30
100	52.90	50.00	43.21	37.02
91	62.91	56.70	54.13	44.03
83	72.93	69.30	62.71	51.01
69	117.54	102.23	97.65	82.35
55	162.22	152.00	142.91	113.57
41	272.33	252.00	250.73	190.63
28	382.51	352.40	342.02	267.72

Table 5.11: Emission parameters for THC Vs Engine Load%

Load %	B0	B10	B20	B30
100	42.61	38.31	36.23	34.12
91	43.00	38.70	36.65	34.47
83	43.42	39.09	36.96	34.72
69	49.57	44.57	42.13	39.63
55	55.65	50.05	47.32	44.52
41	66.12	59.50	56.23	52.93
28	76.63	68.96	65.11	61.32

Table 5.12: Emission parameters for NO Vs Engine Load%

Load %	B0	B10	B20	B30
100	245.02	262.13	311.03	294.05
91	215.18	230.17	290.43	258.12
83	185.32	198.22	250.12	222.33
69	155.83	166.70	210.34	186.91
55	126.34	135.17	170.54	151.62
41	122.72	131.28	165.68	147.23
28	119.12	127.39	160.72	142.93

5.8. Results and Discussion

The data from table 5.7 to 5.12 are used to plot graphs for various performance and emission parameters to understand the characteristics of CI engine fuelled with cottonseed biodiesel blends are discussed below:

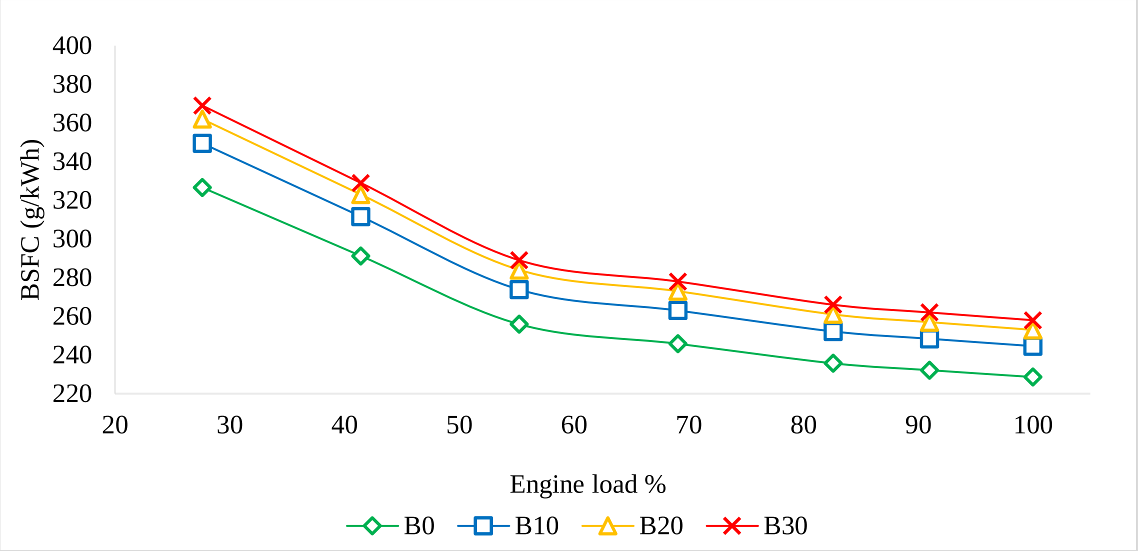


Figure 5.4: BSFC Vs Engine load %

Figure 5.5 shows the change in brake specific fuel consumption (BSFC) in relation to engine load %. With majority of the studied fuels, it appeared that the BSFC fell off with the increment in load%. The Brake Specific Fuel Consumption (BSFC) can be defined as the ratio of input energy to the useful work. It minimizes when the load increases. This condition appears because the braking power of the CI engine increases with load by a larger percentage than fuel consumption does. It is clear from the graph that the BSFC had the lowest load at B0 and highest at B30 blend. The heating value of the test fuel falls as the amount of biodiesel proportion in blends rises. As a result, the biodiesel concentration of the fuel blends increases the BSFC. Compared to diesel fuel, the BSFC of biodiesel and its blends is greater. This can be explained by the petro-diesel's reduced viscosity, lower density, and higher heating value.

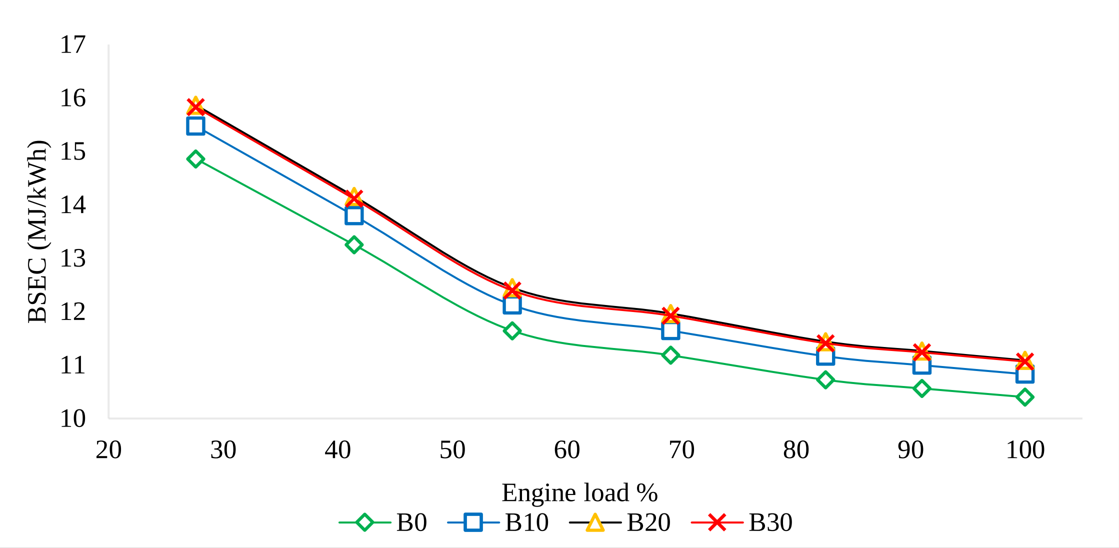


Figure 5.5: BSEC Vs Engine load %

Figure 5.6 shows the variance in BSEC for all tested biodiesel blends with different engine loads at the recommended speed. For all loads, the engine running on cottonseed oil biodiesel consumes more energy compared with one running on fossil diesel. This could be caused by biodiesel's increased density and reduced calorific value. Fossil-diesel performed the least well out of all the tested fuels, and it was noted that the BSEC climbed as the biodiesel component % grew. The B10 mixture offers the lowest BSEC in comparison with several of the other biodiesel blends, this trend indicates that the neat biodiesel will have the greatest BSEC across the engine load range. The BSEC is greater under lower engine load circumstances and steadily drops as load rises.

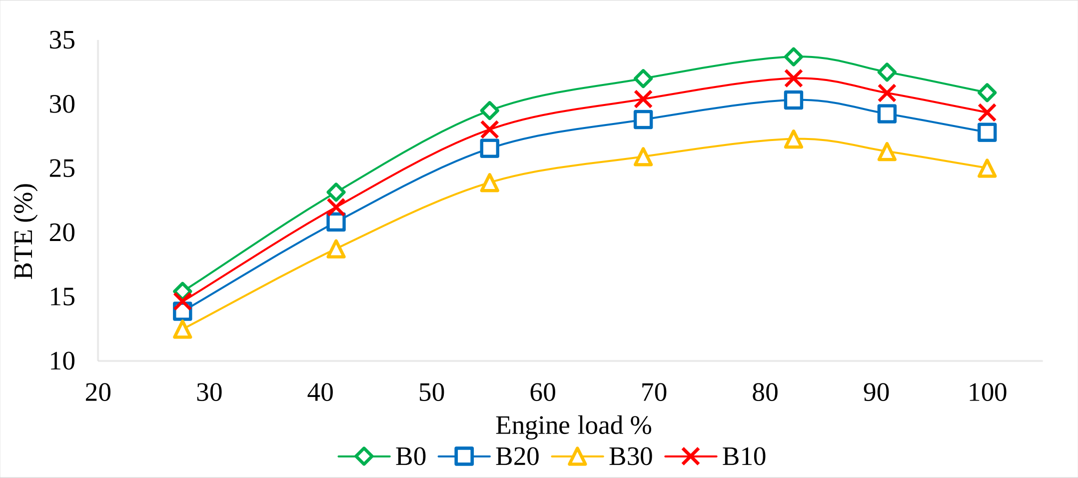


Figure 5.6: BTE Vs Engine load %

The ratio between power generation and energy given by the injection of the combustible fuel is known as brake thermal efficiency (BTE). So, BSFC can be described as the opposite of BTE. BTE is a better way for evaluating the effectiveness of various fuels than only their heating values. According to Figure biodiesel and its mixes with diesel fuel have lower BTE values than pure diesel fuel. The BTE of biodiesel blends is halfway between diesel and biodiesel. However, the proportion between the drop in BTE and the rise in biodiesel content is not equal. The increased effectiveness might be attributed to the blends' superior lubricating qualities as compared to the pure components.

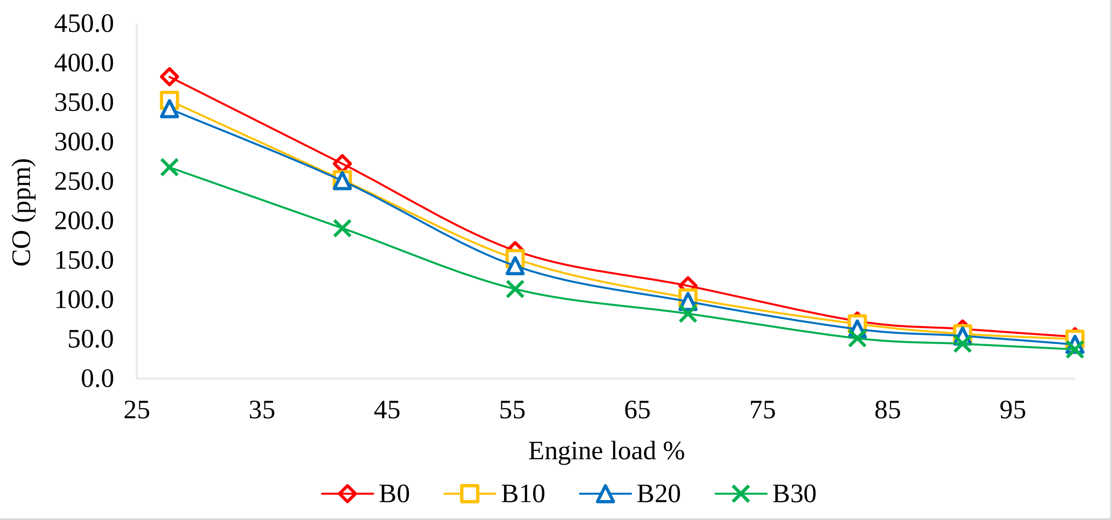


Figure 5.7: CO Vs Engine load %

The extra oxygen present in the biodiesel fuel, boosting combustion in the cylinder, may be the cause of the decrease in Carbon monoxide emissions from biodiesel and its mixes. Additionally, biodiesel happens to be less compressible compared to diesel fuel and possesses a greater cetane number. An increased cetane rating for biodiesel fuel also reduces the likelihood of energy-rich regions emerging and improved injection timing. These all lead to a reduction in the ignition delay, an extension of combustion, and an increase in the areas of full combustion response. Less compressible fuels start the injection process sooner and result in longer combustion times. The results shown agree with the majority of those reported in the literature.

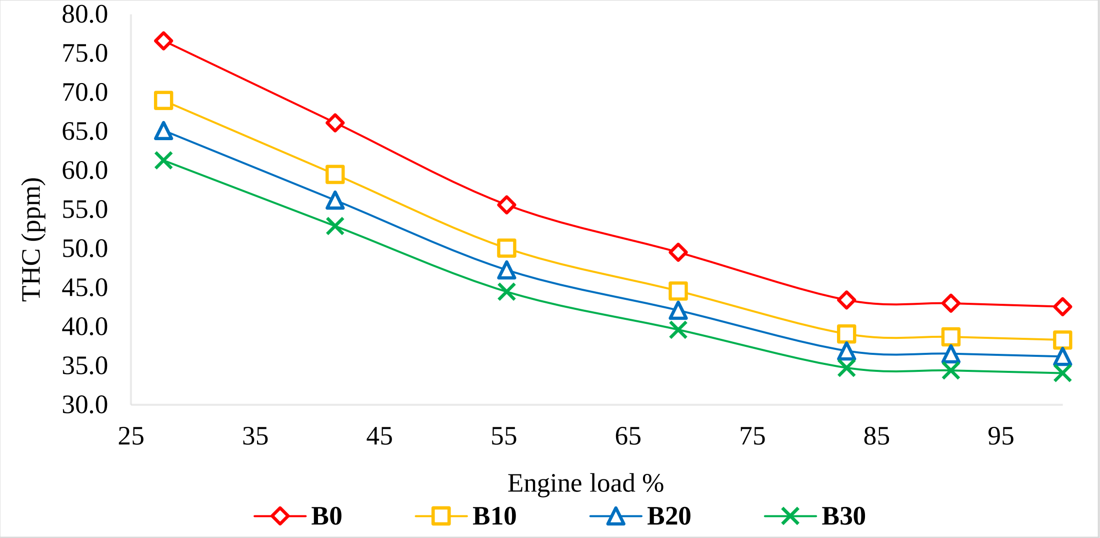


Figure 5.8: THC Vs Engine load %

Because biodiesel comprises more oxygen in it, it promotes a better, thorough and full combustion, which reduces the pollutants of THC. More cetane number of this biodiesel blended diesel fuel has a shorter delay time, which results in fewer THC emissions. As a result, compared to fossil - diesel fuel, biodiesel-fossil diesel fuel blends tend to have lower THC pollutants due to their greater cetane number and levels of oxygen.

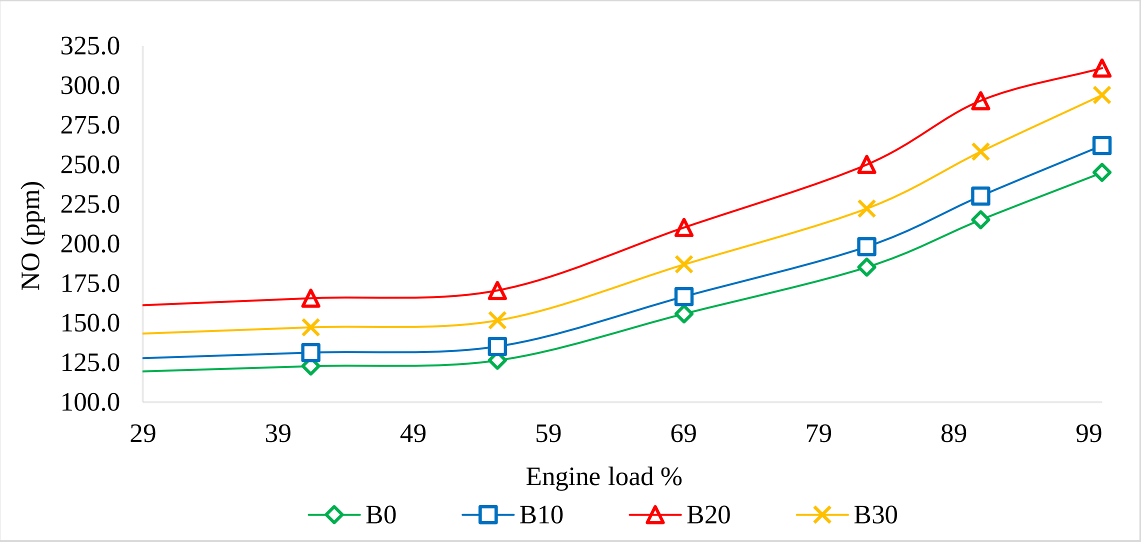


Figure 5.9: NO Vs Engine load %

Total oxygen in the combustion chamber, temperature, pressure, compressibility, and sound velocity are the main variables that influence nitrogen oxide production. There is always some amount of oxygen bound to the chemical structures of biodiesel. Therefore, the creation of nitrogen oxide may have been brought on by the oxygen content in cottonseed biodiesel fuel. Since biodiesel is an oxygenated fuel, it may facilitate the generation of NO by adding oxygen to the air already present in the combustion chamber. As more of the combustion of cottonseed biodiesel and its blends is finished before TDC due to shorter initiation of combustion, it is possible that higher combustion temperatures will result from enhanced combustion.

5.9. Summary

In this chapter, it has been summed up that the potential for using biodiesel as an alternative fuel in diesel engines is quite high because it is made from cottonseed oil using a microwave aided reactor. When fueled with biodiesel and its mixtures with diesel fuel, a multi-cylinder diesel engine performs quite similarly to one that is fueled with straight fossil -diesel. Here are the investigation's vital outcomes: As the percentage of biodiesel in the fuel mixes increased so did the BSFC. When contrasted with diesel, biodiesel and its blends have a poorer brake thermal efficiency. As the amount of biodiesel in fuel increased the amount of smoke, THC, and CO exhaust emissions fell while NO emissions raises. The research findings provide preliminary evidence in favour of the claim that biodiesel may successfully replace fossil diesel fuel in current diesel engines without requiring changes, although this will need further testing.

CHAPTER 6

ECONOMIC COST ANALYSIS

6.1. Introduction

The definition of “Life Cycle Assessment” refers to evaluating something's whole ecological sustainability across all stages of its life, sometimes known as from “cradle to grave”. Other words used to define can be given as life cycle analysis and eco-balance. When discussing fuels, the right phrase is “Well to Wheel Analysis”. There are five stages those help in studying the entire cycle of a transportation fuel[119]. These five stages are enlisted below:

i. Production of the feedstock

ii. Transportation of the feedstock

iii. Production of fuel from the feedstock

iv. Distribution of the obtained fuel

v. Usability in engine

The aforementioned stages are capable of being categorized more thoroughly into Well to Tank and Tank to Wheel components that make up the Well to Wheel assessment. The well to tank study takes into account the whole fuel supply chain, particularly extraction from resources with transmission to the motor vehicle's reservoir, including production of the feedstock, logistics, production of the biodiesel, and distribution of the fuel. The tank to wheel study evaluates the efficiency of fuel consumption, or the usage of biodiesel by engines. It can be concluded that that the well to wheel study amalgamates well to tank and tank to wheel.

6.2. Economy analysis of the present study

The price of making biodiesel using cottonseed oil in a closed loop microwave reactor within optimum circumstances has been assessed in the present investigation in order to evaluate the financial viability of the manufacturing of biodiesel. This pilot study of biodiesel manufacturing revealed that a high-quality yield of 99.5 % was obtained in 11 minutes with an input power of 500W, a molar ratio of 1:4.5, catalyst-1%, turntable and the cooling fan speed of 40 rpm and 1500 rpm respectively. During the conversion of CBD (MW) from cottonseed oil, there was a saving of around 56% in the time taken including a yield nearly 63% higher than the conventional MS process.

The estimation of cost production of biodiesel was based on the following assumptions:

i. The manufacturing facility's capability to generate biodiesel is projected to be 0.5 tonne or 500 kilogram daily.

ii. The operating hours were anticipated to be 8 hours each day.

iii. Virgin cottonseed oil, which is used to make biodiesel, had no evidence of contaminants as well as water content.

iv. Table 6.1 provides the approximate price for each of the chemicals, including cotton seed oil, catalyst, and the solvent.

v. The shipping charge, insurance, and tax, which are expected to be 15% of the expenses.

Table 6.1: The purchase price of the basic components needed in the production of biodiesel

Product	Quantity	Price(INR)
Cottonseed oil	1 kg	70.00
Methanol	1 kg	35.00
Catalyst (potassium hydroxide)	1 kg	300.00

The charges for production of biodiesel from cottonseed oil is calculated per day for a facility that utilizes 0.5 tonne or 500 kg of feedstock each day. Table 6.2 shows the calculations for calculating the processing cost of any feedstock having FFA content less than 2% with the help of microwave irradiation assisted reactor.

Table 6.2: Calculations for calculating the processing cost with the help of microwave irradiation assisted reactor

Fixed Cost

Account	**Amount in Rs.**
Cost of microwave processor etc. is 500000, (Assuming life 10 years	5,00,000
Expanses on fixed cost (500000(1+15*10/100))	12,50,000
Equipment Cost/Day	500
Miscellaneous expenses towards labour, rent and maintenance	2500
Total expenses/ day	**3000**

Operating/ Variable cost

Account	Amount in Rs
Raw Material & Chemicals	
Expenses on methanol (@ 35/ lit*110.5 lit/day)	3867.5
Expenses on catalyst (KOH) (@ 300/kg*5kg/ day)	1,500
Total expenses on raw materials/ day	**5367.5**
Expenditure towards use of electrical energy Assumptions: (Unit Price@ 7.75/ kWh (commercial application)) Specific heat of water= 4.18 kJ/kgK escaping out heat in microwave radiation @1%	
a) to agitate the reactants with an electric motor of 10HP for time duration 10 minutes and pre heating of oil	100
b) for transesterification using microwave working power of microwave 100W, Duration- 0.333 hours (for 0.4 kg) energy consumption needed to perform microwave	355.21

irradiated transesterification of 0.5 tonne =P*time (100*165000 J) =45.833 kWh Expenses incurred for consumption of electricity per day (45.833*7.75)	
c) for heating of water from 20 degree C to 50 degree C for washing the biodiesel Requirement of water for washing of 500kg of oil = 200 kg (approx. 40% of oil) Heat energy required for the heating Q= m*cp*dT/ efficiency (200×4.18*(50-20)/0.99) = 25333.33 kJ = 7.037 kWh Charges for consumption of electricity per day (7.037*7.75)	54.53
d) to provide finishing heat up of biodiesel to a temperature of 110 degrees Celsius in order to remove moisture (in the event that exist) The microwave processor's wattage is kept steady at 180W. Energy requirement to raise the temperature of the oil in the range of 25ºC -110ºC =P * time(180*3750 J)= 18.75 kWh Charges for electricity consumed per day =(18.75 *7.75)	145.31
Total charges on electrical energy/ day	**655.05**
Total Variable cost	**6022.5**
Grand Total (Expenses/ day)	**9022**
Income from glycerin produced 20 kg/day @300/ kg	6000
Net expenses/ day	3022
Quantity of biodiesel produced/ day (500*0.96) (yield efficiency 96%) =480 liter	
Conversion cost of biodiesel from "X" oil of FFA<2 (using microwave method) = 3022/480	**6.29/ kg of oil

The conversion cost of biodiesel from “X” oil of FFA<2 (using **conventional mechanical stirring method**)	8.50/ kg of oil

**** Expenses on ‘X’ oil of FFA<2 (500kg/day))**

6.3. Summary

The current study used microwave assisted transesterification, a crucial technique in the development of biodiesel production, to investigate the expenses of the conversion of cottonseed oil to biodiesel and the earnings from the by-products. The costs of converting feedstock into biodiesel would include capital expenses and variable costs. Initial equipment purchases are susceptible to capital expenditures as well as variable expenses such chemicals, labour, power, and ongoing maintenance. The estimated processing has been measured for a 500 litre/day output. Any feedstock having FFA<2 has been suggested in this study. The unit is operational for 8 hours, with a 15% cost for transportation, insurance, and taxes, the conversion cost for producing pure biodiesel using a microwave-assisted method is around INR 6.29/litre.

CHAPTER 7

CONCLUSION & FUTURE SCOPE

7.1. Introduction

The microwave (MW)-assisted transesterification process was effectively demonstrated in this work through single step transesterification and two step transesterification to obtain biodiesel from cottonseed oil, waste cooking oil and kalonji oil using different cataysts. The produced biodiesel was successfully tested to run a CI engine. The conclusion were drawn as under:

7.1.1. Biodiesel Production using microwave assisted

i. The microwave (MW)-assisted transesterification process was effectively demonstrated in this work to obtain biodiesel from CSO and WCO under KOH and alumina powder catalysed conditions. (Single step transesterification)

ii. In this experiment, the MW-assisted transesterification process was used to successfully produce biodiesel from kalonji oil using base as well as acid catalysed conditions.(Two step transesterification)

iii. Using the design of experiment under Taguchi approach, optimal operating variables for recently recommended process variables of turn table speed and cooling fan speed, in addition to the conventional ones, were determined.

iv. When contrasted to the traditional (mechanical stirring) approach, the assistance of microwave in transesterification practice substantially speeds up the transesterification process's pace, drastically decreasing the duration of the reaction.

v. The physicochemical parameters of the generated biodiesel meet the ASTM D6751 fuel standards.

7.1.2. Test Run on engine

The test run of the CI engine powered with a combination of diesel and cottonseed biodiesel demonstrated that the operating characteristic and exhaust traits of cottonseed biodiesel outperformed those of diesel fuel.

7.2. Key points for Future Scope

i. The current study can be expanded by employing the MW in a loop for continuous production.

ii. The frequency of microwaves can be altered and their effects can be studied.

iii. Hybrid transesterification methods can be developed in which one source can be MW irradiation.

iv. A detailed study on economic and environmental concerns would help this approach to evolve.

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