iJRASET
International Journal For Research in
Applied Science and Engineering Technology

# INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

**Volume:** 11 **Issue:** VI **Month of publication:** June 2023

**DOI:** https://doi.org/10.22214/ijraset.2023.53741

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
*ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538*
*Volume 11 Issue VI Jun 2023- Available at www.ijraset.com*

# Experimental and Simulation Analysis to Predict the Optimum Biodiesel Yield from Two Different Feedstock Using Heterogenous Base Catalyst

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***Abstract: Biodiesel can be easily used an alternative fuel. Using the Soxhlet apparatus, the oil waas extracted, and centrifuged to separate the oil. The physical characteristics of oil was determined. A number of >3 indicates an acid, while a value < 3 indicates a base. If the FFA content is >0.5, the substance is an acid, and if it's less than 0.5, a base. Suitable solvent based on its physicochemical characteristics was selected. In this study two types of seeds were used. A mixed heterogeneous base catalyst containing CaO prepared from waste egg shells andcockle shells. The egg shells and cockle shells were dried in microwave oven for 24 hours at 120◦C and placed in Muffle furnace at 900 ◦C and mixed in 1:1 ratio. SEM, EDAX and FTIR were used to characterize the catalyst. The two feedstock used in this study was Solanum Torvum and Murraya Koenigii. Double Step Esterification was preferred. The oil was treatedwith the strong acid H2SO4 . Then oil along with methanol was treated with both acid and base catalyst separately to give Fatty Acid Methyl Ester. Finally optimum condition was found for maximum yield of FAME, was obtained using RSM.***

***Keywords: Solanum torvum, Murraya Koenigii , CaOcatalyst, methanol, SEM, TEM, EDAX, FTIR,Biodiesel, RSM.***

## I. INTRODUCTION

Automotive vehicles that rely on petroleum, diesel, gasoline, or petro-fuels as their combustion fuel are one of the main contributors to greenhouse gas emissions that result fromthe use of fossil fuels. However, there are relatively few fossilised matter deposits, and thus, there are very few fossilised matter-derived goods like petroleum and diesel, etc (Hannah Ritchie et al. 2020). Environmental pollution caused by fuel combustion's byproducts is severe (Barbir, F. et al. 1990). Investigating and learning about new, clean renewable energy sources is urgently needed.

In the past century, the transportation, industries have been developed in India and other parts of the world. Also, the change in lifestyle of people, population growth leads to more energy consumption. This has caused the depletion of fossil fuels. The use fossil fuels also lead to degradation of environment causing global warming, acidification, climate change etc…The excessive nitrogen oxides and carbon monoxide emission can lead impairment of health in human beings. Since the fossil fuels have many disadvantages, it is important to develop a fuelwhich is non-toxic, environmentally friendly, readily available, cost effective etc. Due to the increased transportation and industries in. past decade the energy demands have been increasedseveral folds. It is apparent that the energy demands further increases in future due to the growing population and increased transportation facilities. However, the fossil fuel available currently may diminish within 100 years and also 'the price of fossil fuels increases every dayand their usage also cause adverse environmental and health effects. Due to this increased price,scarcity of diesel fuel and detrimental environmental effects, biofuels emerge as an ultimate solution to overcome this problem. They are environmentally friendly, cost effect and has similar energy content as petroleum fuels.

India is a diverse country that promotes the use of renewable energy sources and offers incentives for the creation of energy sources based on non-conventional green fuels, like gobargas, biomass fuels, and biofuels. In order to create a B20 mixed blend (petroleum diesel oil is blended with 20% biodiesel), India also promotes the blending of biodiesel with petroleum diesel.

Bio-fuels cause lesser pollution, leading to lower chances of acid rain and reducing the greenhouse gases emission into the atmosphere. Biodiesel consists of monoalkyl methyl esters that are derived from animal or plant sources. Biodiesel causes very negligible pollution and isa promising alternative to petroleum and diesel. It exhibits greater heating value than coal but slightly lesser than that of diesel, petro-diesel, or gasoline and it can be directly used in automotive engines configured for diesel as the combustion fuel. (Biodiesel: A Realistic Fuel Alternative for Diesel Engines, 2008).

These biofuels are non-toxic because they emit less amount of greenhouse gases like carbon monoxide, hydrocarbons, particulate matter and smoke. Although the energy production has improved the economy, it has adversely affected the environment at local, regional and global level by the emission of greenhouse gases, acid rain, drought, ozone depletion etc. Hence now the focus is to produce fuels which is environmentally friendly and economical. These methods can help us to develop sustainable earth.

Rapeseed, palm, and soybean oils are the most often used oils for biodiesel synthesis, althoughthey are less profitable as biofuels due to their high production costs as well as significant demand for their raw materials in the cosmetics and pharmaceutical industries. Vegetable sources are generally favoured because of their widespread availability and the ease of growing and harvesting them (Issariyakul et. al. 2014). Although edible oils can be used to produce biodiesel since they eventually put a strain on agricultural lands through repetitive cultivation.The fact that they may be produced easily without the aid of fertilisers to increase production is perhaps the most essential benefit of all. This lowers manufacturing costs overall and enablesproducers to use biodiesel.

Transesterification is the next step in the process of biodiesel synthesis once the triglycerides have been extracted from the oil. Alcohols including methanol, ethanol, and iso-propanol can be used in the transesterification process. Alkaline catalysts and methanol do not react poorly,making it simple to separate the substance (H. Sanli et al. 2008). An acidic or basic homogeneous or heterogeneous catalyst can be used to carry out the transesterification process(Meena, R. et al. 2014).

It is reasonable to choose homogeneous catalysts over heterogeneous catalysts since they havea faster rate of reaction. Additionally, homogeneous catalysts are exceedingly expensive and challenging to separate from the reaction mixture (Ma and Hanna, 1999; Du et al. 2004). The lipid FFA% is crucial for determining which acid and alkali catalyst to use (Free Fatty Acid). Acid esterification is followed by an alkaline transesterification process if the FFA content is greater than 3% and Base esterification is followed by an alkaline transesterification process ifthe FFA content is lesser than 3% (Lopez et.al 2006; Dossin et. al 2006; Sharma et. al. 2009). Contrarily, soap production is a significant obstacle to using base catalysts, particularly when that are derived from animal or plant sources. Biodiesel causes very negligible pollution and isa promising alternative to petroleum and diesel. It exhibits greater heating value than coal butslightly lesser than that of diesel, petro-diesel, or gasoline and it can be directly used in automotive engines configured for diesel as the combustion fuel. (Biodiesel: A Realistic Fuel Alternative for Diesel Engines, 2008). These biofuels are non-toxic because they emit less amount of greenhouse gases like carbon monoxide, hydrocarbons, particulate matter and smoke. Although the energy production has improved the economy, it has adversely affected the environment at local, regional and global level by the emission of greenhouse gases, acid rain, drought, ozone depletion etc. Hence now the focus is to produce fuels which are environmentally friendly and economical. These methods can help us to develop sustainable earth.

## II. SINGLE STEP BASE TRANSESTERIFICATION

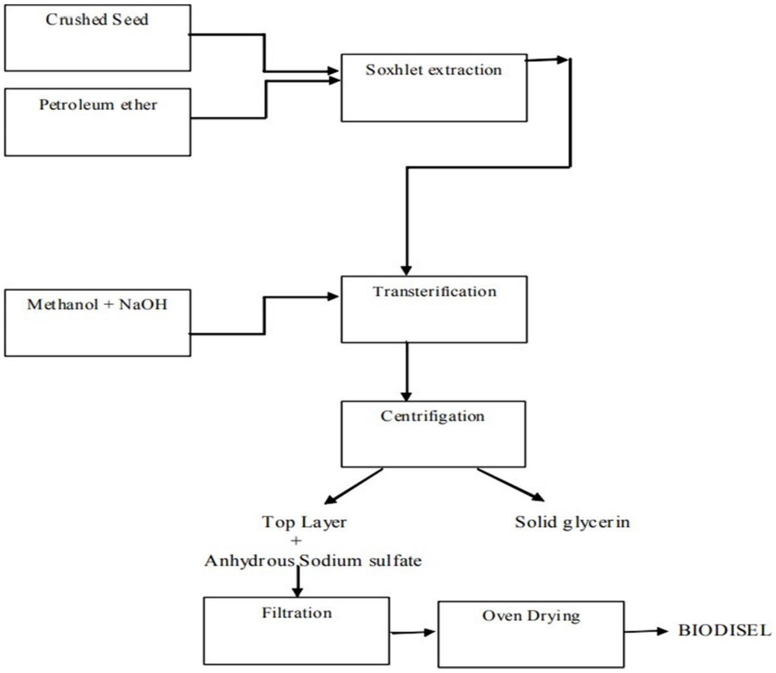


Figure 1.2. Block diagram for base transesterification method using NaOH catalyst

## III. TWO STEP TRANSESTERIFICATION

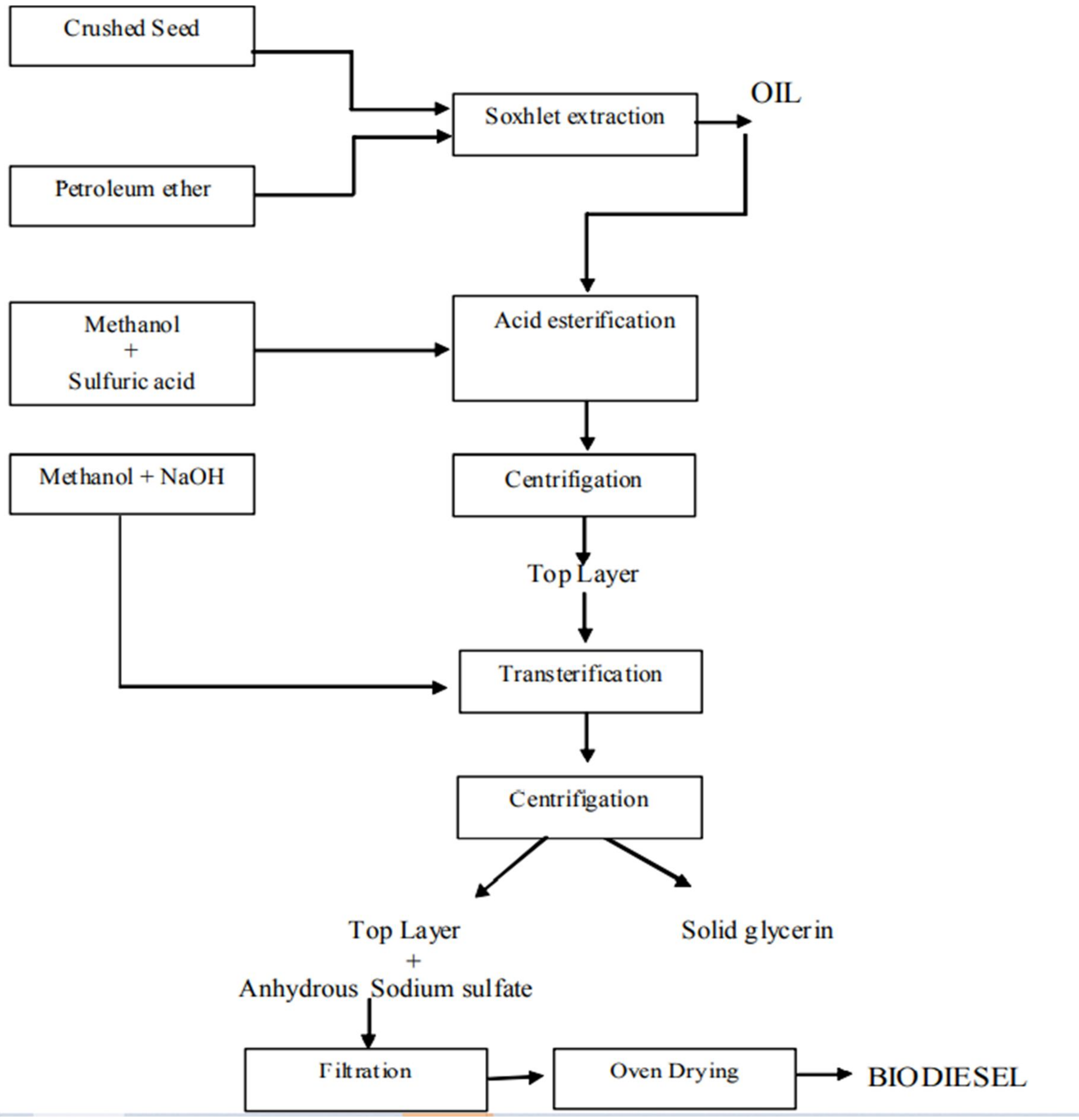


Figure1.3. Block diagram for two-step transesterification method using sulphuric acidand NaOH catalyst

## IV. ADVANTAGES OF BIODIESEL

1) Improved fuel performance and lubricity.
2) The biodiesel can be used as such or it can be blended with diesel in different proportionwithout any modification of diesel engine.
3) Biodiesel is non-toxic because it does not emit sulphur and other aromatic compounds.
4) It has high flash point than diesel fuel hence it is safe to store and handle.
5) It also allows complete combustion of hydrocarbons.
6) It improves air quality and and the environment and provides safety benefits.
7) Help in reducing the carbon footprint of transportation and other industries.
8) It is domestically produced, clean burning, renewable substitute for petroleumbiodiesel.

## V. FEEDSTOCK FOR BIODIESEL PRODUCTION

The biodiesel can be produced from animal fats, vegetable oils and used cooking oils. The animal fats cannot be used directly for biodiesel because the distribution of fats is a major disadvantage. The biodiesel can be produced from waste cooking oils too. However, usually, vegetable oils are used in biodiesel production. There are different vegetable oils which are used for biodiesel production. Some of them are soyabean oil, sunflower oil, cotton seed oil, jojoba oil etc…
In the present study oil extracted from berries of *Solanum torvum*(turkey berry) is used for biodiesel production.

## VI. EXTRACTION OF OIL

There are different methods involved in extraction of oil from seed. They are catalytic cracking, blending, solvent extraction, enzymatic extraction etc. In the mechanical pressing process the oil extracted requires further treatment of filtering and degumming. The enzymatic process uses enzyme to extract oil which increases the cost and is a long process. Thus, solvent extraction using Soxhlet extraction technique is the one with less disadvantages. Hence it is used in this study to extract oil.

## VII. CATALYSTS FOR BIODIESEL PRODUCTION

The catalysts are used in biodiesel production process so as to initiate and speed up the formation of Fatty Acid Methyl Ester compounds. The catalysts can be homogeneous, heterogeneous catalyst.

### *A. Heterogeneous Catalysts*

Heterogeneous catalysts are developed recently. Generally, calcium oxide derived from bio- based substance is used as catalyst. These are easy to separate from the reaction mixture after the reaction is over.

### *B. Homogeneous Catalyst*

If the catalyst is soluble in one of the reactants or if the reactants and catalysts are in same phase then it is called homogeneous catalyst. These are generally used in commercial production of biodiesel owing to the faster reaction rates provided by them. The homogeneouscatalysts can be further classified as base catalysts and acid catalysts.

### *C. Acid Catalysts*

Sulfuric Acid
It is a strong corrosive acid with pungent smell. It is colourless to slight yellow in colour.so inthis study sulphuric acid is used as acid catalyst.

### *D. Base Catalysts*

Sodium Hydroxide Catalyst
Initially sodium hydroxide was produced by reaction between sodium carbonate and calcium hydroxide. However, with the arrival of chlor-alkali industry, nowadays sodium hydroxide is produced from brine using an electrolytic process. The formula for Sodium hydroxide is NaOH.This catalyst is used for biodiesel production from olden days.
In this study, sodium hydroxide is used as catalyst for biodiesel production.

## VIII. INFLUENCE OF DIFFERENT PARAMETERS ON BIODIESEL PRODUCTION

*1)* Temperature
*2)* Speed of agitation
*3)* Mole ratio of methanol to oil
*4)* Reaction time
*5)* Catalyst concentration

## IX. PROPERTIES OF BIODIESEL

### *A. Cetane Number*

Cetane number indicates the combustion speed of diesel fuel. Higher cetane ratings lead to maximum engine power and efficiency. The cetane number is an indication of fuel readiness to auto-ignite when it has been injected into the diesel engine.

Diesel fuels are required to havea cetane number higher than 40 and most refineries produce diesel with cetane numbers between 40 and 45. Biodiesel has a higher cetane number between 46 and 60 (depending on the feedstock used) which shortens the ignition delay in the engine and improves the combustion characteristics.

### *B. Flash Point*

The flashpoint of a fuel is the temperature at which the vapor above the fuel becomes flammable. Petroleum-based diesels have flashpoints of 50 ºC to 80 ºC which makes them intrinsically safe. Biodiesel has a flashpoint of over 160 ºC which means that the fire hazard associated with transportation, storage and usage of biodiesel is much less than with other commonly used.

### *C. Viscosity*

Plants and waste oils have higher viscosities compared to diesel fuel; transesterification of these oils reduces the viscosity to a large extent in converting to biodiesels. Viscosity measurement is a basic parameter reflecting the extent of the reaction.

### *D. Melt Point Or Pour Point*

Melt or pour point refers to the temperature at which the oil in solid form starts to melt or pour. In cases where the temperatures fall below the melt point, the entire fuel system including all fuel lines and fuel tank will need to be heated.

### *E. Cloud Point*

The temperature at which an oil starts to solidify is known as the cloud point. While operating an engine at temperatures below an oil's cloud point, heating will be necessary in order to avoidwaxing of the fuel.

### *F. Density*

It is the weight per unit volume. Oils that are denser contain more energy. For example, petrol and diesel fuels give comparable energy by weight, but diesel is denser and hence gives more energy per litre. (Fuel Density). The aspects listed above are the key aspects that determine the efficiency of a fuel for diesel engines. There are other aspects/characteristics which do not have a direct bearing on the performance, but are important for reasons such as environmental impactetc.

### *G. Calorific Value, Heat Of Combustion*

Heating Value or Heat of Combustion, is the amount of heating energy released by the combustion of a unit value of fuels. One of the most important determinants of heating value is moisture content. Air-dried biomass typically has about 15-20% moisture, whereas the moisture content for oven-dried biomass is negligible.

Moisture content in coals vary in the range 2-30%. However, the bulk density of most biomass feedstocks is generally low, even after densification between about 10 and 40% of the bulk density of most fossil fuels. Liquid biofuels however have bulk densities comparable to those for fossil fuels.

### *H. Simulation Analysis*

The simulation software used here was Design of expert version 13. Response surface methodology was performed using the software.

Effect of process parameters such as Temperatures , Reaction time, Methanol /oil ratio and catalyst loading on biodiesel production was analysed using Response surface methodology(RSM)

## X. MATERIALS AND METHODS

### *A. Materials*

#### *1) Equipments*

Extraction thimble, hot air oven, magnetic stirrer with hot plate, measuring jar, conical flask, beakers, centrifuge, Sohxlet apparatus, Round bottom flask, condenser etc.

#### *2) Reagents*

*Solanum torvum* and *Murraya koenigii* seeds were purchased from world export Madurai. Methanol, petroleum ether, sulfuric acid.

### 3) *Collection and Preparation of Weed Sample*

### a) *Turkey Berry Seeds*

Seeds of *solanum torvum* were collected from all over the world. The common name of *solanum torvum* is turkey berry. It is a bushy, erect and spiny perennial plant. The plant is usually 2 or 3 m in height and 2 cm in diameter. It is also an edible part and have lot of medicinalvalues. It seeds also used in extraction of biodiesel. The seeds were sundried to remove any external moisture and then dried in a hot air oven for 8 hours at 150 °c and weighed. The seedswere crushed using an electric grinder till a consistency similar to ground powder were obtained and sieved to 60 micrometre sieve. The seeds of *solanum torvum* is shown in figure.

Figure 3.1. seeds of *solanum torvum*

Table 3.1. Classification of *solanum torvum*

| | |
|---|---|
| Kingdom | Plantae |
| Order | Solanales |
| Family | Solanaceae |
| Genus | Solanum |
| Species | Solanumtorvum |
| Habitat | Forests, plantation crops, waste areas. |

### b) *Curry Seeds*

Curry berries are the fruits of Curry tree which grows in clusters. The clusters consists of 32- 85 small curry berries. The white bloom present in Curry tree produce tiny fruits which is initially green in colour and ripens to shiny black in colour. About 50% of the fruit constitutesof flesh and ethereal blue is with juicy texture. Each of this fruit consists of 1-2 seeds which are in deep green colour. These seeds are toxic and not for consumption. Curry berries are nota seasonal fruit, available in all the seasons. Large amount of vitamin C is present in curry berries .It also contains anthocyanins, minerals like calcium, phosphorous, magnesium and iron. The edible part of curry tree plays an important role in Indian cooking and also in Ayurvedic medicine. The curry tree is the native of Asia. The curry tree is small in size of 4 to6m in height and 40cm diameter. The leaves are used in many dishes in Indian subcontinent.

Figure 3.2. seeds of *Murraya Koenigii*

Table 3.2. Classification of *Murraya Koenigii*

| | |
|---|---|
| Kingdom | Plantae |
| Order | Sapindales |
| Family | Rutaceae |
| Genus | Murraya |
| Species | *Murraya Koenigii* |
| Habitat | Grown throughout India |

### *B. Properites*

### *1) Petroleum Ether*

Table 3.3. Properties of petroleum ether

| | |
|---|---|
| Vapor pressure | 31 kPa at 20°C |
| Molar mass | 82.2 g/mol |
| Appearance | Volatile, clear, colorless, non-fluorescent liquid |
| Density | 0.653 g/ml |
| Viscosity | 0.46 m Pa s |
| Boiling point | 42-62°C |

Uses and safety measures: It is used as a solvent for dissolving fat, oil and it helps in the extraction of oil. It is used in the manufacture of pharmaceutical compounds. It is extremely volatile, since it has low flash point and it acts as an allergen to skin.

### *2) Sulphuric Acid*

Table 3.4. Properties of Sulphuric acid

| | |
|---|---|
| Molecular formula | H2SO4 |
| Molar mass | 98.097 g/mol |
| Density | 1.83 g/ml |
| Boiling point | 337°C |
| Melting point | 10.31°C |

Uses and safety measures: It is used for fertilizer manufacturing and it is also used for manufacturing printing ink, enamels. It can cause severe burns and it can cause permanent blindness. When it is swallowed, internal organs are affected and cause death. So safety measures should be taken.

### *3) Methanol*

Methanol also known as methyl alcohol among others, is a chemical with the formula CH3OH.Methanol acquired the name "wood alcohol" because it was once produced chiefly as a byproduct of the destructive distillation of wood. Today, industrial methanol is produced in a catalytic process directly from carbon monoxide, carbon dioxide, and hydrogen. Methanol is the simplest alcohol being only a methyl group linked to a hydroxyl group. It is a light, volatile,colorless, flammable liquid with a distinctive odor very similar to that of ethanol (drinking alcohol). However, unlike ethanol, methanol is highly toxic and unfit for consumption. At room temperature, it is a polar liquid, and is used as an antifreeze, solvent, fuel, and as a denaturant for ethanol. It is also used for producing biodiesel via transesterification process. Oils and fatsare included in the ester family. When they react with methanol or ethanol, they make methylor ethyl esters and a new alcohol called glycerol or, more commonly, glycerin. The vegetable oils and animal fats used to make biodiesel can come from virtually any source.

Table 3.5. Properties of methanol

| | |
|---|---|
| Molecular formula | $CH_3OH$ |
| Density | 0.792 g/ml |
| Boiling point | 64.7°C |
| Solubility | Miscible and soluble in water |
| Appearance | Colourless liquid |

### 4) *Methyl Ester*

Fatty acid methyl esters are a type of fatty acid ester that is derived by transesterification of fats with methanol. The molecules in biodiesel are primarily FAMEs, usually obtained from vegetable oils by transesterification.

Table 3.6. Properties of methyl ester

| | |
|---|---|
| Molecular formula | C14-C24 |
| Density | 0.860-0.849 g/ml |
| Boiling point | >475 |
| Solubility | Insoluble in water |
| Appearance | Light to dark yellow, clear liquid |

Uses and safety measures: In leather chemical industry, it is used to produce the leather fat- liquoring agent and sulfonic-succinate acyclic acyl ether disodium, serving as a promising textile detergent.

Fatty acid methyl ester can also serve as a raw material of bio-diesel which is a newenvironment-friendly energy source. For safety measures avoid contact to the eyes because it may cause eyes irritation and skin irritation. Vapors or mist may be irritating to mucous membranes and cause irritation and dizziness.

### 5) *Glycerol*

Glycerol is a simple polyol compound. It is a Colorless, odorless, viscous liquid that is widelyused in pharmaceutical formulations. Glycerol has three hydroxyl groups that are responsible for its solubility in water and its hygroscopic nature. IUPAC Name: propane-1, 2, 3-triol. Othernames: Glycerin, propanetriol, 1, 2, 3-trihydroxypropane.

Table 3.7. Properties of Glycerol

| | |
|---|---|
| Molecular formula | $C_3H_8O_3$ |
| Molecular mass | 92.09382 g/mol |
| Density | 0.126 g/ml |
| Boiling point | 290 ° C |
| Melting Point | 17.8 ° C |
| Appearance | Colorless, liquid, hygroscopic |

## C. *Methods*

### 1) *Preparation of seed*

The dry seeds of *solanum torvum* and *Murraya koenigii* were obtained from world life export.They were dried in the sunlight .Dried in a Hot air oven at 130 ° C for 8 hours and at $105^0$C for2 hours respectively until it is dried well. The seeds are then crushed and stored in air-tight container.

*2) Extraction of Oil*

The crushed seeds were used for oil extraction by Soxhlet extraction. Petroleum ether and n- hexane were used as two different solvents for extraction. The ratio of solvent to dried seed powder was varied for process optimization. In brief the extraction apparatus was washed withparticular solvent and a cotton plug was placed at bottom and ground. The crushed seed powder(feed) was placed above the cotton plug. Appropriate solvent was filled in the round bottom flask. The entire apparatus was placed on a heating mantle. The extraction was done for 3 – 4 hours. The contents from the round bottom flask were distilled to separate solvent andextract. The volume and weight of extract and solvent was noted.

The yield of oil was calculated using the following formula:

$$\%\ \text{Yield of oil} = \frac{Amount of oil obtained}{Amount of crushed berries used} \text{ X } 100$$

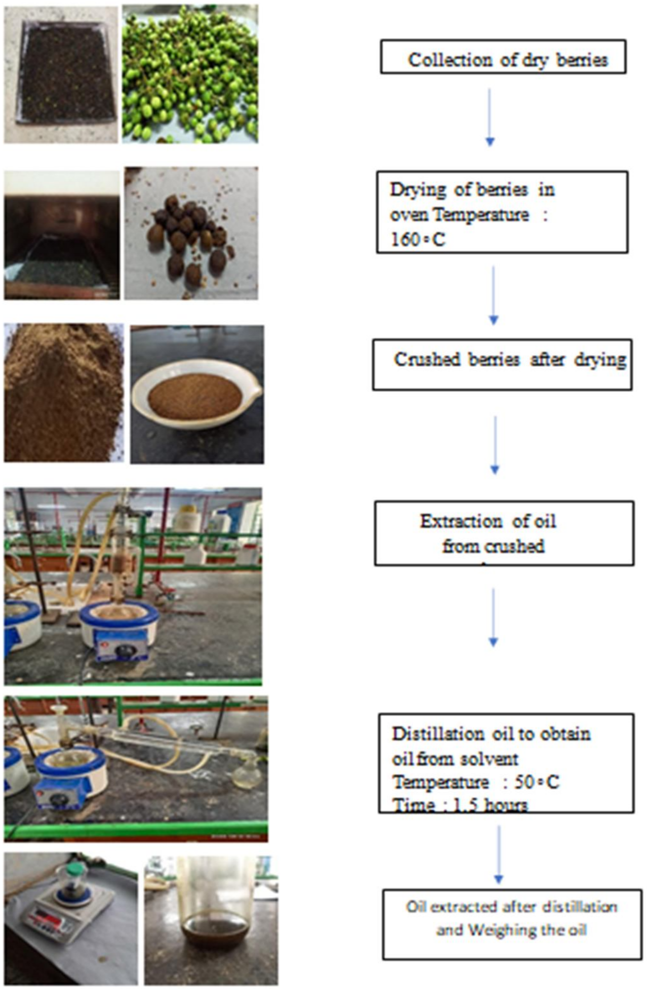


Figure 3.3. Extraction of oil

## XI. CHARACTERIZATION OF EXTRACTED OIL

The physicochemical properties of extracted oil were estimated using standard methods(AOCS, 1998; Vicente et al., 2004). FTIR spectral analysis was done to identify the functionalgroups and chemical interactions using FTIR spectrophotometer (Bruker, USA). GCMSanalysis of the oil was performed using Gas Chromatography Mass spectrophotometer CH- GCMSMS-02 and the obtained data was compared against the data available in the Mass spectralibrary of the National Institute of Standards and Technology and Wiley FTIR analysis handbook. From the GC-MS and FTIR analysis, the oil's fatty acid composition and existing functional groups were analyzed. The FFA (%) of each end product from each trial was measured using toluene solvent (AS S ilitonga et al, 2013).

### *A. Catalyst Preparation*

#### *1) Base Catalyst*

The waste eggshell was collected from the hotels near by and cockle shell was also collected. Both the eggshell and cockle shell were thoroughly cleaned with warm water to remove impurities make it free from any dirties. The eggshell and cockle shell were kept in daylight for 2-3 days separately. After this both the eggshell and cockle shells were heated in oven for 4-6-hours separately at a temperature 100℃-150℃. The eggshell and cockle shell were crushed using mortar machine and sieved separately for 100-150micron sieve tray. The product obtained were in fine powder form. These powders were filled in crucible and kept in muffle furnace for calcination. The eggshell powder were kept at 700-800 whereas cockle shell powder were kept at 800-900. After calcination the product was allowed to cool. The both the eggshell powder and cockle shell powder were in the ratio of 1:1 which served as a base catalystin this project.

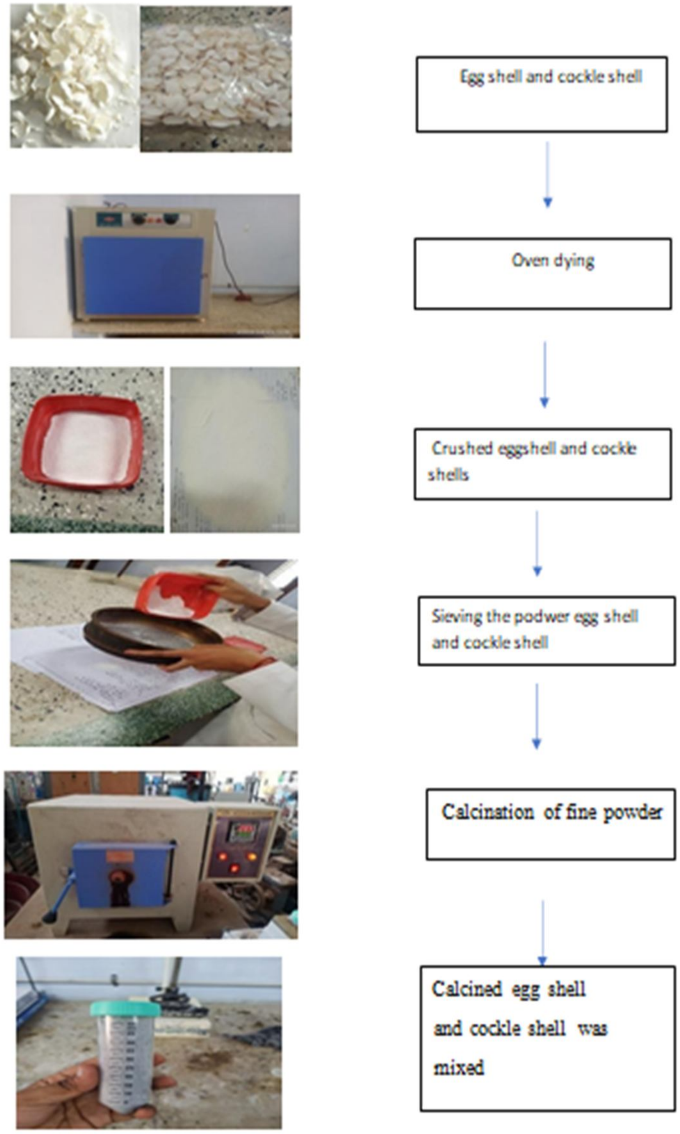


Figure 3.4. Steps involved in preparation of mixed CaO catalyst

### *B. Esterification Process*

#### *1) First step acid esterification using Sulphuric catalyst*

The oil had a pretreatment step in the first esterification reaction of the double step esterification process in order to lower the acid value. Three parts oil to one part solvent made up the oil to solvent ratio. In accordance with the above technique, numerous experimental tests were carried out to optimise the process factors.

### *a) First step esterification process for Solanum torvum oil*

Oil was measured, transferred to a 50 ml eppendorf tube, and then methanol and catalyst wereadded. Thermo-mixer settings for the tube's reaction time, temperature, and agitation speed were made. After the reaction was finished, the tube's contents were transferred to a separation funnel, where the mixture instantly split into two different layers. The top and bottom layers were tested. The top layer was found to have methanol predominantly and the bottom layer was found to have the treated oil. From this treated oil alone taken to find the optimum. Testing was done on the top and bottom layers. Methanol was discovered to be predominatelypresent in the top layer, whereas treated oil was discovered to be present in the bottom layer. To obtain the best results, only use this prepared oil. The following criteria were taken into account during the initial trials: Methanol to oil ratios of 3:1, 6:1, 9:1, and 15:1 were used, as well as catalyst loadings of 0.1- 0.5. The reaction temperature was 45°C-55°C. A significant reduction was only seen for the methanol :oil ratios of 3:1 at a reaction temperature of 55°C for 0.1ml catalyst loading , for the reaction time of 3 hours. This oil was produced in bulk volume to carry out second step esterification.

Table 3.8. First step esterification process for *Solanum torvum* oil

| Methanol / Oil | Temperature | Catalyst Loading | Time | Acid value | FFA |
|---|---|---|---|---|---|
| 3:1 | 55 | 0.1 | 3 | 0.76 | 0.38 |
| 3:1 | 55 | 0.2 | 3 | 0.9 | 0.45 |
| 6:1 | 55 | 0.1 | 3 | 1.06 | 0.51 |
| 6:1 | 55 | 0.2 | 3 | 1.2 | 0.6 |
| 9:1 | 55 | 0.1 | 3 | 1.6 | 0.8 |
| 9:1 | 55 | 0.2 | 3 | 1.78 | 0.89 |
| 12:1 | 55 | 0.1 | 3 | 3.6 | 1.8 |
| 12:1 | 55 | 0.2 | 3 | 4.2 | 2.1 |

### *b) First step esterification process for Murraya koenigii oil*

Oil was measured, transferred to a 100ml beaker, and then methanol and catalyst were added.The sample was placed in magnetic stirrer with varying time ,temperature, catalyst. After the reaction was finished, the beakers's contents were transferred and centrifuged and then transferred to separation funnel, where the mixture instantly split into two different layers. Thetop and bottom layers were tested. The bottom layer was found to have methanol predominantlyand the top layer was found to have the treated oil. From this treated oil alone taken to find theoptimum.

Testing was done on the top and bottom layers. Methanol was discovered to be predominatelypresent in the bottom layer, whereas treated oil was discovered to be present in the top layer. To obtain the best results, only use this prepared oil. The following criteria were taken into account during the initial trials: Methanol to oil ratios of 3:1, 6:1, 9:1, and 15:1 were used, as well as catalyst loadings of 0.1- 0.5. The reaction temperature was 45°C-55°C. A significant reduction was only seen for the methanol :oil ratios of 3:1 at a reaction temperature of 50°C for 0.2ml catalyst loading , for the reaction time of 1.5 hours. This oil was produced in bulk volume to carry out second step esterification.

Table 3.9. First step esterification process for *Murraya Koenigii*

| Methanol / Oil | Temperature | Catalyst Loading | Time | Acid value | FFA |
|---|---|---|---|---|---|
| 3:1 | 50 | 0.1 | 3 | 0.96 | 0.48 |
| **3:1** | **50** | **0.2** | **1.5** | **0.84** | **0.42** |
| 6:1 | 50 | 0.1 | 3 | 1.18 | 0.59 |
| 6:1 | 50 | 0.2 | 1.5 | 1.1 | 0.55 |
| 9:1 | 50 | 0.1 | 3 | 1.86 | 0.93 |
| 9:1 | 50 | 0.2 | 1.5 | 1.8 | 0.9 |
| 12:1 | 50 | 0.1 | 3 | 7.6 | 3.8 |
| 12:1 | 50 | 0.2 | 1.5 | 7 | 3.75 |

### *2) Second step acid esterification using CaO catalyst*

The oil had a pretreatment step in the first esterification reaction of the double step esterification process in order to lower the acid value. The optimum oil was prepared in bulk to perform second step esterification. In accordance with the above technique, numerous experimental tests were carried out to optimise the process factors.

### *a) Second step acid esterification – Solanum torvum*

The oil that produced in bulk volume from optimum of first step esterification is used for the second step esterification process. As the FFA (%) value fall down below 0.5, it was chosen to continue the esterification process using the base catalyst. Oil was measured, transferred to a 50 ml eppendorf tube, and then methanol and catalyst (CaO) were added. Thermo-mixer settings for the tube's reaction time, temperature, and agitation speed were made. After the reaction was finished, the tube's contents were transferred to a separation funnel, where the mixture instantly split into two different layers. The qualities of the top and bottom layers wereexamined. The testing revealed that the top layer primarily contained methanol, catalyst, and other contaminants, while the bottom layer had a dark, oil-like substance that was only slightlymiscible in distilled water. The substance from the bottom layer was centrifuged and stored.

Methanol to oil ratios was maintained from 3:1, 6:1, 9:1, and 15:1 Catalyst loadings of 0.1-0.5 was maintained. The reaction temperature was 45°C-55°C. A significant reduction was only seen for the methanol :oil ratios of 3:1 at a reaction temperature of 55°C for 0.2grams catalyst loading , for the reaction time of 2 hours. The oil was given for biodiesel test

The yield was calculated by the formula,

$$Yield\ (\%) = \left(\frac{Weightofbiodieselobtained}{Weightofoilusedinreaction}\right) \times 100$$

Table 3.10. Second step esterification process for *Solanum torvum* oil

| RUN | OIL / METHANOL RATIO (mL) | WEIGHTOF OIL TAKEN (grams) | CATALYST LOADING (grams | TEMP(°C) | TIME (hours) | WEIGHT OF PRODUCT OBTAINED (grams) | ACID | FFA | YIELD(%) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 3 | 1.5 | 0.1 | 55 | 3 | 1.285 | 0.32 | 0.16 | 85.65 |
| 2 | 6 | 1.5 | 0.2 | 50 | 2 | 1.220 | 0.40 | 0.20 | 81.33 |
| 3 | 6 | 1.5 | 0.5 | 45 | 2 | 1.342 | 0.46 | 0.23 | 89.46 |
| 4 | 6 | 1.5 | 0.5 | 55 | 3 | 0.828 | 0.62 | 0.31 | 55.20 |
| 5 | 9 | 1.5 | 0.2 | 50 | 2 | 1.312 | 0.50 | 0.25 | 87.46 |
| 6 | 9 | 1.5 | 0.1 | 50 | 3 | 1.237 | 0.40 | 0.20 | 82.46 |
| 7 | 3 | 1.5 | 0.1 | 50 | 3 | 1.150 | 0.38 | 0.19 | 76.66 |
| 8 | 3 | 1.5 | 0.2 | 55 | 2 | 1.402 | 0.24 | 0.12 | 93.46 |
| 9 | 6 | 1.5 | 0.2 | 55 | 3 | 0.913 | 0.58 | 0.29 | 60.86 |
| 10 | 9 | 1.5 | 0.3 | 50 | 2 | 1.328 | 0.62 | 0.31 | 88.53 |
| 11 | 15 | 1.5 | 0.5 | 55 | 3 | 0.832 | 0.74 | 0.37 | 55.46 |
| 12 | 3 | 1.5 | 0.5 | 50 | 3 | 1.282 | 0.38 | 0.19 | 85.46 |
| 13 | 6 | 1.5 | 0.1 | 50 | 2 | 1.343 | 0.48 | 0.24 | 89.53 |
| 14 | 6 | 1.5 | 0.1 | 55 | 3 | 0.955 | 0.64 | 0.32 | 63.66 |
| 15 | 6 | 1.5 | 0.3 | 45 | 3 | 1.268 | 0.54 | 0.27 | 84.53 |
| 16 | 9 | 1.5 | 0.5 | 50 | 2 | 1.314 | 0.48 | 0.24 | 87.60 |
| 17 | 9 | 1.5 | 0.2 | 55 | 3 | 0.808 | 0.42 | 0.21 | 53.86 |
| 18 | 9 | 1.5 | 0.3 | 55 | 2 | 0.954 | 0.36 | 0.18 | 63.45 |
| 19 | 3 | 1.5 | 0.5 | 55 | 3 | 0.762 | 0.38 | 0.19 | 50.80 |
| 20 | 15 | 1.5 | 0.1 | 50 | 3 | 1.209 | 0.54 | 0.27 | 80.60 |
| 21 | 6 | 1.5 | 0.2 | 55 | 3 | 0.826 | 0.64 | 0.32 | 55.06 |
| 22 | 3 | 1.5 | 0.1 | 45 | 3 | 1.354 | 0.28 | 0.14 | 90.26 |
| 23 | 3 | 1.5 | 0.5 | 55 | 2 | 0.890 | 0.64 | 0.32 | 59.33 |
| 24 | 3 | 1.5 | 0.3 | 50 | 3 | 1.087 | 0.46 | 0.23 | 72.46 |

*b) Second step acid esterification – Murraya Koenigii*

The oil that produced in bulk volume from optimum of first step esterification is used for the second step esterification process. As the FFA (%) value fall down below 0.5, it was chosen to continue the esterification process using the base catalyst. Oil was measured, transferred to a 50 ml eppendorf tube, and then methanol and catalyst (CaO) were added. Magnetic stirrer settings for the tube's reaction time, temperature, and agitation speed were made. After the reaction was finished, the tube's contents were transferred to a separation funnel, where the mixture instantly split into two different layers. The qualities of the top and bottom layers were examined. The testing revealed that the bottom layer primarily contained methanol, catalyst, and other contaminants, while the top layer had a dark, oil-like substance that was only slightly miscible in distilled water. The substance from the bottom layer was centrifuged and stored.

Methanol to oil ratios was maintained from 3:1, 6:1, 9:1, and 15:1 Catalyst loadings of 0.1- 0.5 was maintained. The reaction temperature was 45°C-55°C. A significant reduction was only seen for the methanol :oil ratios of 6:1 at a reaction temperature of 50°C for 0.5grams catalyst loading , for the reaction time of 2 hours. The oil was given for biodiesel test

The yield was calculated by the formula,

$$Yield\ (\%) = \left(\frac{Weight of biodiesel obtained}{Weight of oil used in reaction}\right) \times 100$$

Table 3.11. Second step esterification process for *Murraya Koenigii*

| RUN | OIL / METHANOL RATIO (mL) | WEIGHT OF OIL TAKEN (grams) | CATALYST LOADING (grams | TEMP (°C) | TIME (hours) | WEIGHT OF PRODUCT OBTAINED (grams) | ACID | FFA | YIELD(%) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 3 | 1.84 | 0.1 | 50 | 3 | 1.482 | 0.70 | 0.35 | 80.54 |
| 2 | 6 | 1.84 | 0.2 | 45 | 2 | 1.538 | 0.42 | 0.21 | 83.57 |
| 3 | 6 | 1.84 | 0.5 | 50 | 2 | 1.734 | 0.36 | 0.18 | 94.23 |
| 4 | 6 | 1.84 | 0.5 | 55 | 3 | 1.081 | 0.48 | 0.24 | 58.75 |
| 5 | 9 | 1.84 | 0.2 | 45 | 2 | 1.664 | 0.80 | 0.40 | 90.43 |
| 6 | 9 | 1.84 | 0.1 | 50 | 3 | 1.460 | 0.68 | 0.34 | 78.34 |
| 7 | 3 | 1.84 | 0.1 | 55 | 2 | 1.572 | 0.74 | 0.37 | 85.43 |
| 8 | 3 | 1.84 | 0.5 | 50 | 3 | 1.607 | 0.58 | 0.29 | 87.33 |
| 9 | 6 | 1.84 | 0.2 | 55 | 3 | 1.118 | 0.48 | 0.24 | 60.76 |
| 10 | 9 | 1.84 | 0.2 | 50 | 2 | 1.648 | 0.72 | 0.36 | 89.56 |
| 11 | 15 | 1.84 | 0.5 | 55 | 3 | 1.059 | 0.78 | 0.39 | 57.55 |
| 12 | 3 | 1.84 | 0.5 | 50 | 2 | 1.650 | 0.50 | 0.25 | 89.67 |
| 13 | 6 | 1.84 | 0.1 | 50 | 2 | 1.680 | 0.52 | 0.26 | 91.30 |
| 14 | 6 | 1.84 | 0.1 | 55 | 3 | 1.106 | 0.60 | 0.30 | 60.09 |
| 15 | 6 | 1.84 | 0.3 | 50 | 3 | 1.611 | 0.38 | 0.19 | 87.55 |
| 16 | 9 | 1.84 | 0.5 | 45 | 2 | 1.660 | 0.76 | 0.38 | 90.21 |
| 17 | 9 | 1.84 | 0.2 | 55 | 3 | 0.975 | 0.82 | 0.41 | 52.98 |
| 18 | 9 | 1.84 | 0.3 | 55 | 2 | 1.114 | 0.74 | 0.37 | 60.54 |
| 19 | 3 | 1.84 | 0.5 | 55 | 3 | 1.024 | 0.62 | 0.31 | 55.65 |
| 20 | 15 | 1.84 | 0.1 | 50 | 3 | 1.427 | 0.82 | 0.41 | 77.55 |
| 21 | 6 | 1.84 | 0.3 | 55 | 2 | 1.092 | 0.76 | 0.38 | 59.34 |
| 22 | 3 | 1.84 | 0.2 | 45 | 3 | 1.575 | 0.44 | 0.22 | 85.58 |
| 23 | 3 | 1.84 | 0.5 | 55 | 2 | 1.141 | 0.54 | 0.27 | 62.01 |
| 24 | 3 | 1.84 | 0.1 | 50 | 3 | 1.381 | 0.48 | 0.24 | 75.05 |

## XII. RESULT AND DISCUSSION

### *A. Selection Of Solvent For Extraction Of Oil*

The trials for oil extraction using the proven solvent method with petroleum ether and N- hexane yielded a higher amount of triglycerides . The crushed seed to solvent ratio was maintained as 1:4 and the extraction was carried out using three solvents. The extraction with this solvent was carried out for 3-4 hours It seen that yield of petroleum ether of 40% and using n-hexane an yield of 20% oil is obtained for *Solanum torvum* and it is seen that an yieldof 43.25% was obtained by using petroleum ether and using n-hexane an yield of oil of about 12% is obtained for *Murraya koenigii*. Thus, the solvent petroleum ether shows the best result,as it provides higher yield, relatively lower cost and enables easier separation process than n- hexane. The extraction was carried for same ratio of 1:4 feed to petroleum ether at to petroleumether at different time and the Table 4.1 shows the optimisation for the time of oil extraction for both feedstocks.

Table 4.1. Optimization of Solvent Seed Ratio – *Solanum torvum*

| S.NO | Amount of seeds in grams | Solvent used | Yield of oil in % |
|---|---|---|---|
| 1. | 20 | Petroleum ether | 6.7 |
| 2. | 20 | Chloroform | - |
| 3. | 20 | N-hexane | 4.5 |

Table 4.2 Optimization of Solvent Seed Ratio – *Murraya Koenigii*

| S.NO | Amount of seeds in grams | Solvent used | Yield of oil in % |
|---|---|---|---|
| 1. | 40 | Petroleum ether | 17.4 |
| 2. | 40 | Chloroform | - |
| 3. | 40 | N-hexane | 4.9 |

### *B. Characterization Of Base Catalyst*

The FTIR spectra of calcined eggshell powder, calcined cockle shell powder, mixed calcined eggshell powder and calcined cockle shell powder revealed several bands in range of 500cm $^{-1}$ -2500 cm $^{-1}$. Visible differences were observed in the band aspect of the FTIR spectra difference between the calcined eggshell powder (Figure 4.1), calcined cockle shell powder (Figure 4.2), mixed calcined eggshell powder and calcined cockle shell powder (Figure 4.3).

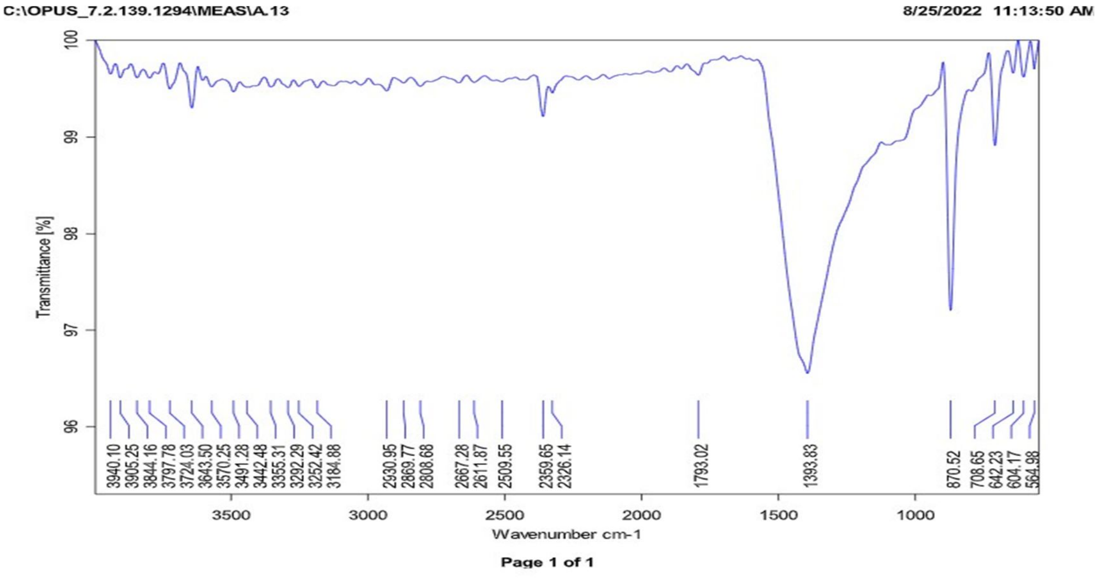


FIGURE A.

Figure 4.1 FTIR Spectra of Calcined eggshell

The prominent absorption peak at 1500 cm -1 absorbed in Figure 4.1. is associated with the presence of Cao in calcined eggshell powder. The prominent absorption peak at 1393.93 cm -1 absorbed in Figure A is associated with the presence of Cao in calcined eggshellpowder.

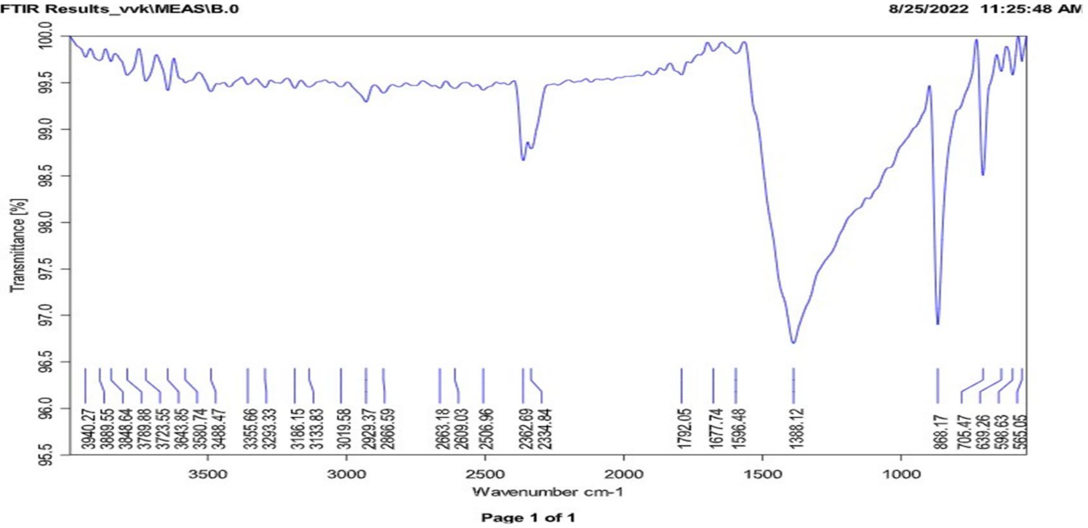


FIGURE B

Figure 4.2 FTIR Spectra of Calcined cockleshell

The prominent absorption peak at 1388.12 cm -1 absorbed in Figure 4.2. is associated with the presence of Cao in calcined cockleshell powder.

Figure 4.3 FTIR Spectra of Calcined eggshell and calcined cockleshell

The prominent absorption peak at 1388.27 cm -1 absorbed in Figure 4.3. is associated withthe presence of Cao in mixed calcined eggshell powder and calcined eggshell powder.

## *C. Physicochemical Properties Of Oil*

Physicochemical Properties of oil

The oil properties such as moisture content, specific gravity, acid value, and FFA% were determined using standard methods. The oil has naturally high antioxidant properties which isan added advantage for bio-fuels. The physicochemical properties of the oil showed very interesting inferences are shown in Table 4.2

Table 4.3. Physicochemical properties of the *Solanum tovum* oil

| PROPERTIES | VALUES |
|---|---|
| Specific gravity | 0.76 |
| Flash point | $113^0$ C |
| Fire point | $117^0$ C |
| Pour point | $13^0$ C |
| Viscosity | 3.92 CST |
| Acid value | 9.3 |
| Free fatty acid | 4.65 |

Table 4.4 Physicochemical properties of the *Solanum tovum* oil

| PROPERTIES | VALUE |
|---|---|
| Specific Gravity | 0.6839 |
| Viscosity | 5.94CST |
| Flash point | 202°C |
| Fire point | 252°C |
| Cloud point | 13.6°C |
| Pour point | 8.4°C |
| Acid Value | 13.4 |
| FFA | 6.7 |

### *D. Fatty Acid Profile of Oil*

From the GC-MS analysis, the *Solanumtorvum* and *Murraya koenigii* has shown a high percentage of Linoleic acid (methyl ester) with an overall fatty acid constitution as shown in Figure 4.4 and Figure 4.5. It was clear that the oil has contained considerable levels of methylesters and fatty acids.

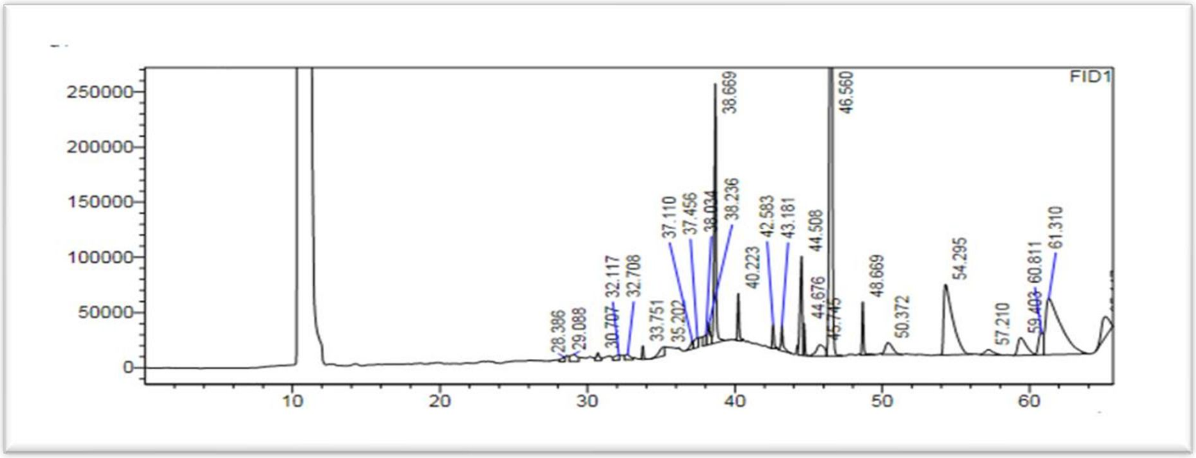


Figure 4.4. GCMS spectrum of Turkey berry oil

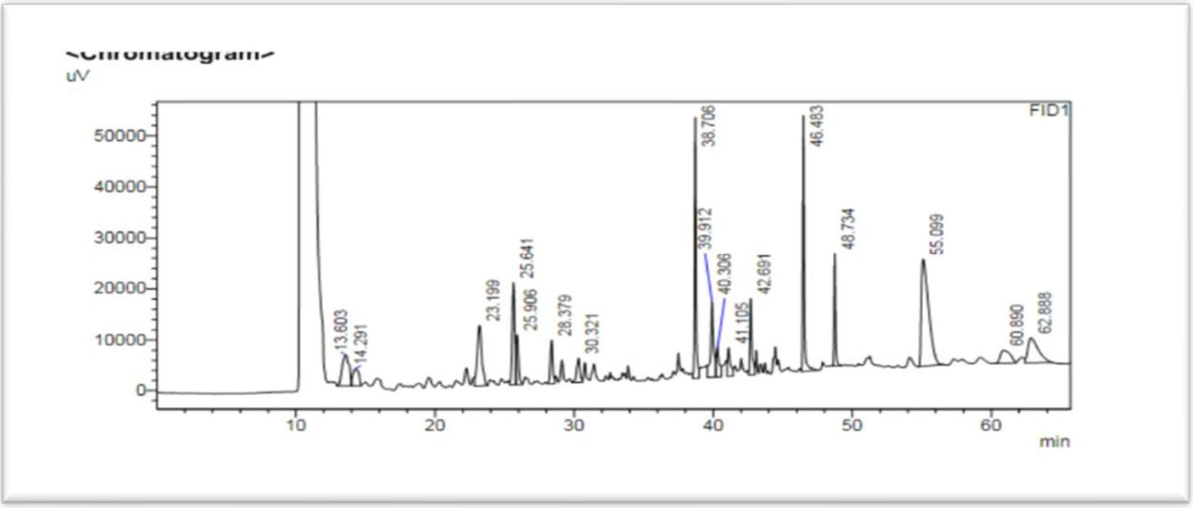


Figure 4.5. GCMS spectrum of curry oil

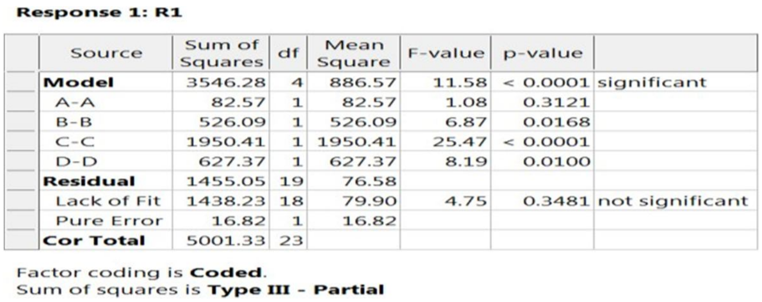
### *E. Simulation Analysis Of Second Step Esterification*

The simulation software used here was Design of experts version 13. Response surface methodology was performed using the software. Effect of process parameters such as Temperatures, Reaction time, Methanol /oil ratio and catalyst loading on biodiesel production was analysed using Response surface methodology (RSM). It compress of a group of mathematical and statistical techniques that are based on the fit of empirical models to the experimental data. Numeric factors considered for simulation are A,B,C,D and R where,

A- Methanol / oil ratio
B- Catalyst Loading
C- Temperature
D- Time R – Yield

#### *1) Analysis of Variance*

Analysis of variance is the collection of statistical models and their associated estimation procedure used to analyze the differences among means . ANOVA is based on the law of total variance where the observed variance in a particu lar variable is partitioned into components attributable to different sources of variations. ANOVA statistical test of whether two or more population means are equal. ANOVA is used to test the difference between twoor more means.

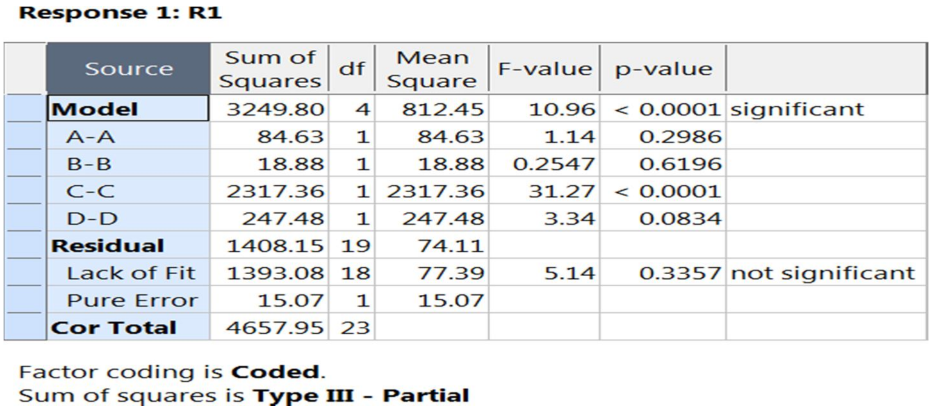


**Response 1: R1**

| Source | Sum of Squares | df | Mean Square | F-value | p-value | |
|---|---|---|---|---|---|---|
| **Model** | 3546.28 | 4 | 886.57 | 11.58 | < 0.0001 | significant |
| A-A | 82.57 | 1 | 82.57 | 1.08 | 0.3121 | |
| B-B | 526.09 | 1 | 526.09 | 6.87 | 0.0168 | |
| C-C | 1950.41 | 1 | 1950.41 | 25.47 | < 0.0001 | |
| D-D | 627.37 | 1 | 627.37 | 8.19 | 0.0100 | |
| **Residual** | 1455.05 | 19 | 76.58 | | | |
| Lack of Fit | 1438.23 | 18 | 79.90 | 4.75 | 0.3481 | not significant |
| Pure Error | 16.82 | 1 | 16.82 | | | |
| **Cor Total** | 5001.33 | 23 | | | | |

Factor coding is **Coded**.
Sum of squares is **Type III - Partial**

Figure 4.6. ANOVA for *solanum torvum*



**Response 1: R1**

| Source | Sum of Squares | df | Mean Square | F-value | p-value | |
|---|---|---|---|---|---|---|
| **Model** | 3249.80 | 4 | 812.45 | 10.96 | < 0.0001 | significant |
| A-A | 84.63 | 1 | 84.63 | 1.14 | 0.2986 | |
| B-B | 18.88 | 1 | 18.88 | 0.2547 | 0.6196 | |
| C-C | 2317.36 | 1 | 2317.36 | 31.27 | < 0.0001 | |
| D-D | 247.48 | 1 | 247.48 | 3.34 | 0.0834 | |
| **Residual** | 1408.15 | 19 | 74.11 | | | |
| Lack of Fit | 1393.08 | 18 | 77.39 | 5.14 | 0.3357 | not significant |
| Pure Error | 15.07 | 1 | 15.07 | | | |
| **Cor Total** | 4657.95 | 23 | | | | |

Factor coding is **Coded**.
Sum of squares is **Type III - Partial**

Figure 4.7. ANOVA for *Murraya koenigii*

The model is significant for experimental values of *Solanum torvum* and *Murraya koenigii.* p-value for model should be 0.05 or below. P-value measures the probability of obtaining the observed results assuming that the null hypothesis is true. Here p- value for the model is less than 0.05.For *Solanum torvum* the terms B,C,D are significant. For *Muarraya koenigii* the terms C, D are significant. f-value is calculated by dividing the two mean squares.

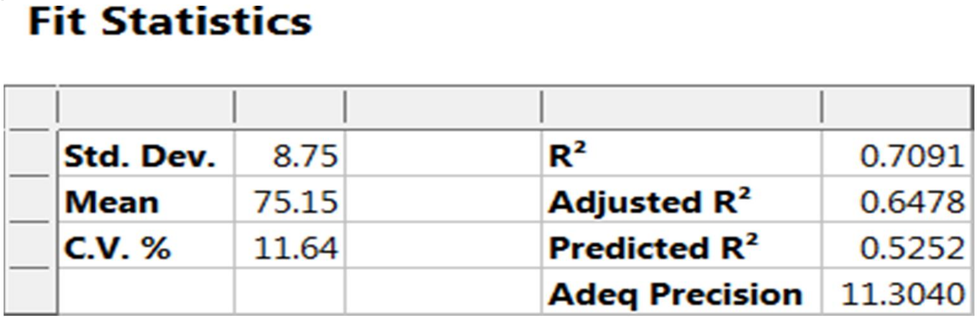
### *F. Lack Of Fit For Linear*

Lack of fit sum of squares for the linear model. The F-valuecompares the variation of the differences in the average responses at the design points, and the corresponding estimated responses using the linear model, with the expected experimental variation as estimated from replicated design points (Pure Error). It is the mean square for the linear model lack-of-fit divided by the mean square for pure error.

The lack-of-fit tests compare the residual error to the pure error from replicated design points. A lack-of-fit error significantly larger than the pure error indicates that something remains in the residuals that can be removed by a more appropriate model. If you see significant lack-of-fit (Prob>F value 0.10 or smaller) then don't use the model as a predictor of the response.

### *G. Fit Statistics*

The predicted $R^2$ value should be reasonable agreement with adjusted $R^2$ value that is the difference is less than 0.2. Adequate precision means measures the signal to noise ratio. It compares the range of predicted value at the design point to the average prediction error. A ratio greater than 4 is desirable.

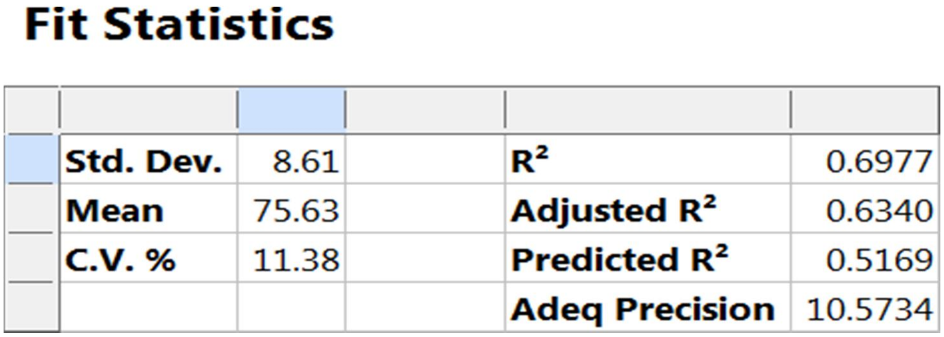


**Fit Statistics**

| | | | | | |
|---|---|---|---|---|---|
| | **Std. Dev.** | 8.75 | | **R²** | 0.7091 |
| | **Mean** | 75.15 | | **Adjusted R²** | 0.6478 |
| | **C.V. %** | 11.64 | | **Predicted R²** | 0.5252 |
| | | | | **Adeq Precision** | 11.3040 |

Figure 4.8. Fit statistics for *solanum torvum*

The predicted $R^2$ of 0.5252 is reasonable agreement with adjusted $R^2$ of 0.6478 that is the difference is less than 0.2. Adequate precision ratio value is 11.3040 indicates an adequate signal. This model can be used to navigate the design space.



**Fit Statistics**

| | | | | | |
|---|---|---|---|---|---|
| | **Std. Dev.** | 8.61 | | **R²** | 0.6977 |
| | **Mean** | 75.63 | | **Adjusted R²** | 0.6340 |
| | **C.V. %** | 11.38 | | **Predicted R²** | 0.5169 |
| | | | | **Adeq Precision** | 10.5734 |

Figure 4.9. Fit statistics for *Murraya koenigii*

The predicted $R^2$ value should be reasonable agreement with adjusted $R^2$ value that is the difference is less than 0.2. Adequate precision ratio value is 10.5734 indicates an adequate signal. This model can be used to navigate the design space.

### *H. General Model Equation*

$$y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4$$

Table 4.5. General model equation table

| Coefficient | *Solanum Torvum* | *Murraya koenigii* |
|---|---|---|
| β0 | +252.88203 | +242.53082 |
| β1 | -0.541962 | -0.550029 |
| β2 | -30.11232 | -5.52811 |
| β3 | -2.66034 | -2.82425 |
| β4 | -10.84782 | -6.69592 |

Model Equation for *Solanum torvum*

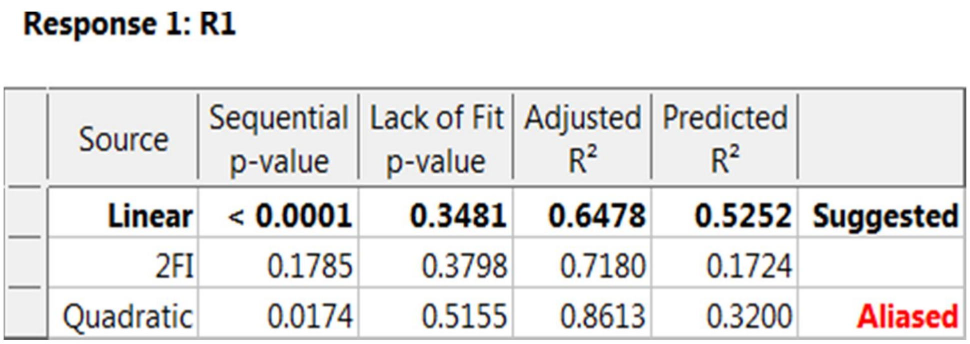
$$\mathbf{R_1} = +252.88203-0.541962\ \mathbf{X_1} -30.11232\ \mathbf{X_2} -2.66034\ \mathbf{X_3} -10.84782\ \mathbf{X_4}$$

Model Equation for *Murraya koenigii*

$$\mathbf{R_1} = +242.53082 -0.550029\ \mathbf{X_1} -5.52811\ \mathbf{X_2} -2.82425\ \mathbf{X_3} -6.69592\ \mathbf{X_4}$$

*I. Fit Summary*

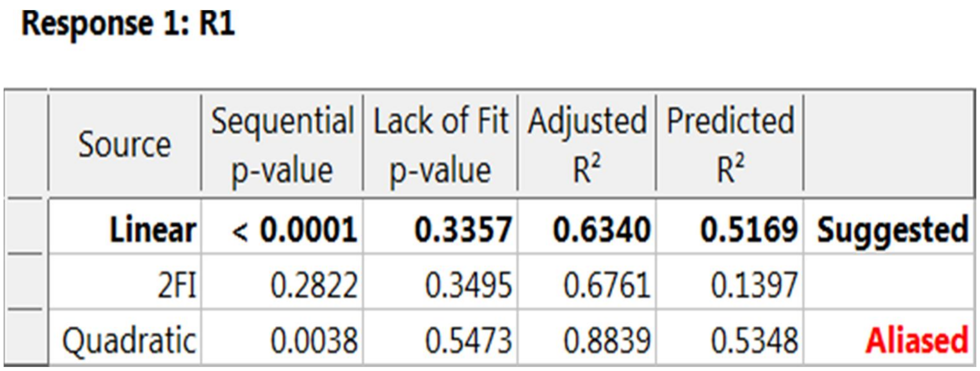
Fit summary conveys which model is best fit for given data. The fit summary collects the important statistics used to select the correct starting point for the final model. The suggested model should be considered a good stating point for the model fitting. For given experimental values linear model was suggested. Figure 4.10 and Figure 4.11 shows that linear model was suggested by the software.



**Response 1: R1**

| Source | Sequential p-value | Lack of Fit p-value | Adjusted R² | Predicted R² | |
|---|---|---|---|---|---|
| **Linear** | **< 0.0001** | **0.3481** | **0.6478** | **0.5252** | **Suggested** |
| 2FI | 0.1785 | 0.3798 | 0.7180 | 0.1724 | |
| Quadratic | 0.0174 | 0.5155 | 0.8613 | 0.3200 | **Aliased** |

Figure 4.10. Fit Summary for *solanum torvum*



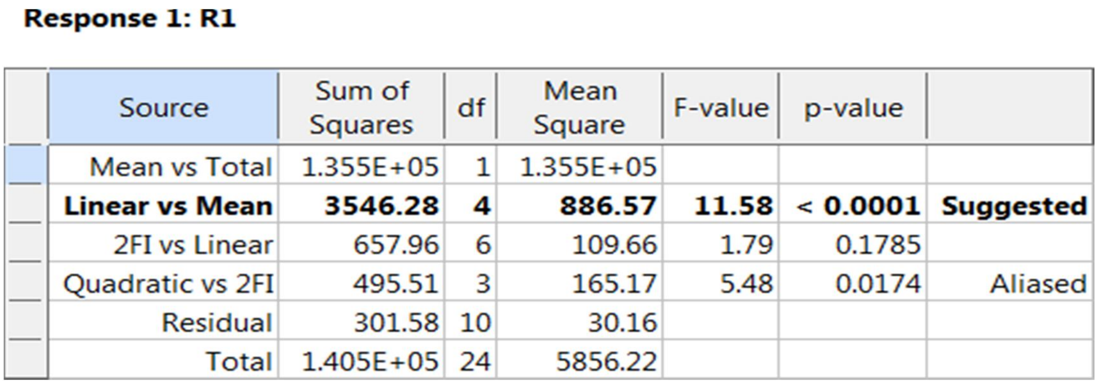
**Response 1: R1**

| Source | Sequential p-value | Lack of Fit p-value | Adjusted R² | Predicted R² | |
|---|---|---|---|---|---|
| **Linear** | **< 0.0001** | **0.3357** | **0.6340** | **0.5169** | **Suggested** |
| 2FI | 0.2822 | 0.3495 | 0.6761 | 0.1397 | |
| Quadratic | 0.0038 | 0.5473 | 0.8839 | 0.5348 | **Aliased** |

Figure 4.11. Fit Summary for *Murraya koenigii*

### J. *Sequential Model Sum Of The Square*

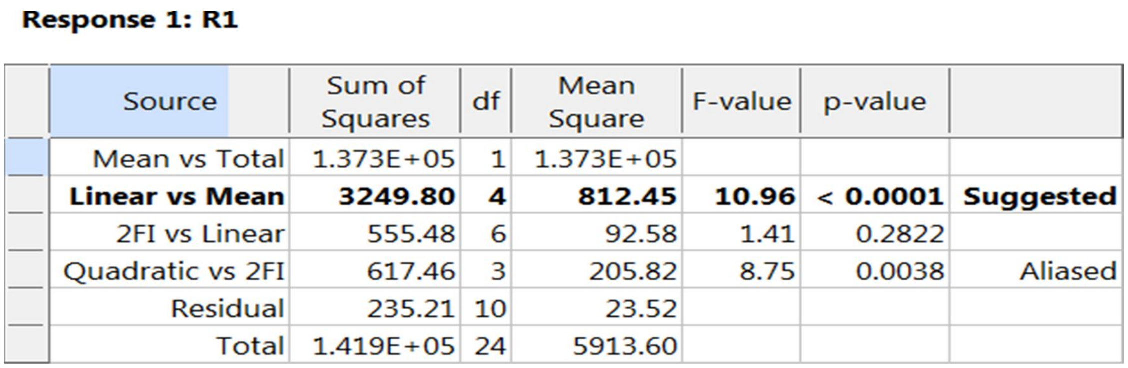
It is the reduction in error sum of squares when one or more predicted variables are added to the model. Or it is the increase in the regression sum of the squares when one or more predictedvariables are added to the model



**Response 1: R1**

| Source | Sum of Squares | df | Mean Square | F-value | p-value | |
|---|---|---|---|---|---|---|
| Mean vs Total | 1.355E+05 | 1 | 1.355E+05 | | | |
| **Linear vs Mean** | **3546.28** | **4** | **886.57** | **11.58** | **< 0.0001** | **Suggested** |
| 2FI vs Linear | 657.96 | 6 | 109.66 | 1.79 | 0.1785 | |
| Quadratic vs 2FI | 495.51 | 3 | 165.17 | 5.48 | 0.0174 | Aliased |
| Residual | 301.58 | 10 | 30.16 | | | |
| Total | 1.405E+05 | 24 | 5856.22 | | | |

Figure 4.12. Sequential model for *solanum torvum*



**Response 1: R1**

| Source | Sum of Squares | df | Mean Square | F-value | p-value | |
|---|---|---|---|---|---|---|
| Mean vs Total | 1.373E+05 | 1 | 1.373E+05 | | | |
| **Linear vs Mean** | **3249.80** | **4** | **812.45** | **10.96** | **< 0.0001** | **Suggested** |
| 2FI vs Linear | 555.48 | 6 | 92.58 | 1.41 | 0.2822 | |
| Quadratic vs 2FI | 617.46 | 3 | 205.82 | 8.75 | 0.0038 | Aliased |
| Residual | 235.21 | 10 | 23.52 | | | |
| Total | 1.419E+05 | 24 | 5913.60 | | | |

Figure 4.13. Sequential model for *Murraya koenigii*

- *Mean:* The sum of squares for the effect of the mean.
- *Linear:* Sequential sum of squares for the linear terms. The F-value tests the significance of adding linear terms to the intercept and block effects. A small p-value (Prob>F) indicates thatadding linear terms has improved the model.

### K. *Graphs Interpretation*

#### 1) *Normal Probability plot*

It shows that residual lies close to the diagonal line. The diagonal line represents the ideal normal distribution line from this data. The experimental values lies close to the ideal normal distribution line which is shown in the figure 4.14

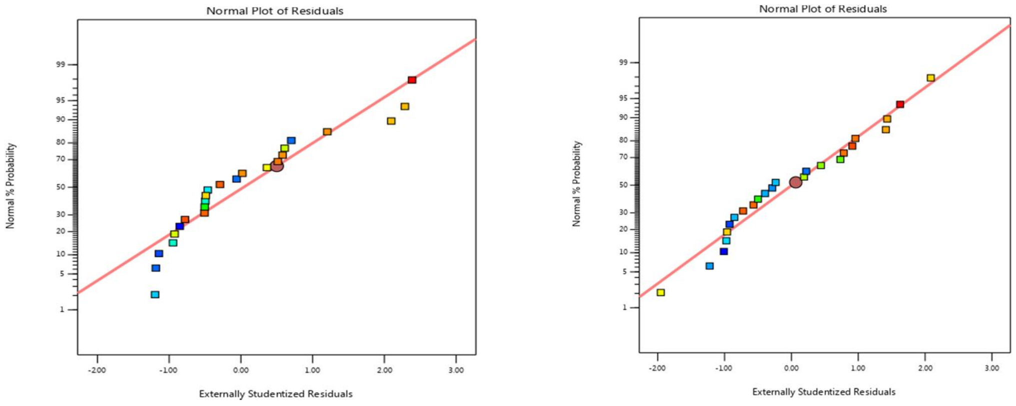


Figure 4.14. Normal Probability plot for *Solanum torvum* and *Murraya koenigii*

### *2) Residual Vs Predicted*

Middle line represents the least square regression, the distance between the least square regression line. Some of residuals are positive and negative. The residuals lines which is above the regression line is called positive residuals whereas the residuals lines which is below the regression line is called negative residuals. Here the pattern follows random scattering because even no of point are above and below the regression line. Random scattering signifies the residuals do not contradict the linear assumptions

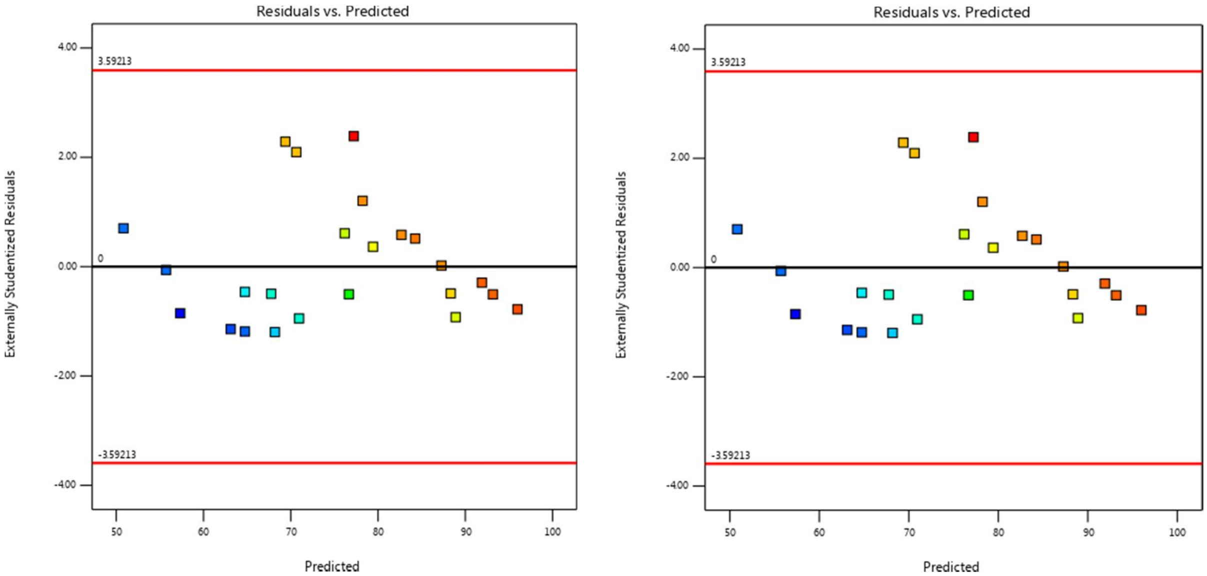


Figure 4.15 Residual Vs predicted plot for *Solanum torvum* and *Murraya koenigii*

### *3) Contour Plot*

Contour plot are the way to show a three dimensional surface on a two dimensional plane. It graphs two predictor numeric factors like A,B,C,D on the y-axis and the R1 on the Z- contour.

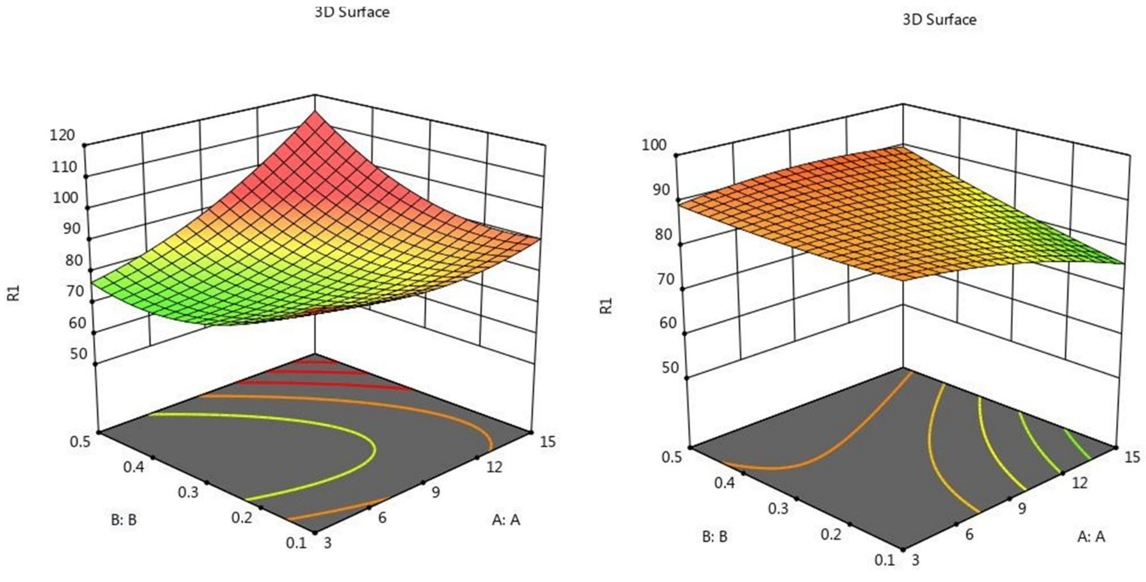


Figure 4.16. Contour plot for *Solanum torvum* and *Murraya koenigii*

## XIII. CONCLUSION

The findings revealed that utilising petroleum ether as the solvent seed ratio, the seed oil had a40% yield. Esters and carboxylic acids were present, as determined by the FTIR analysis. Theresults of the GCMS analysis revealed a significant amount of linoleic acid (methyl ester) anda general fatty acid composition. The free fatty acid content of the oil was decreased to 1.2% at 55°C, a reaction period of 2 hours, a 3:1 Methanol Oil ratio, and a catalyst loading of 0.2grams of CaO for *Solanum torvum*. The free fatty acid content of the oil was decreased to 1.8% at 50°C, a reaction period of 2 hours, a 3:1 Methanol Oil ratio, and a catalyst loading of 0.5 grams of CaO for *Murraya koenigii*. The experimental values were simulated using Design of expert software version 13 following RSM. Linear model was suggested for set of both experimental values.

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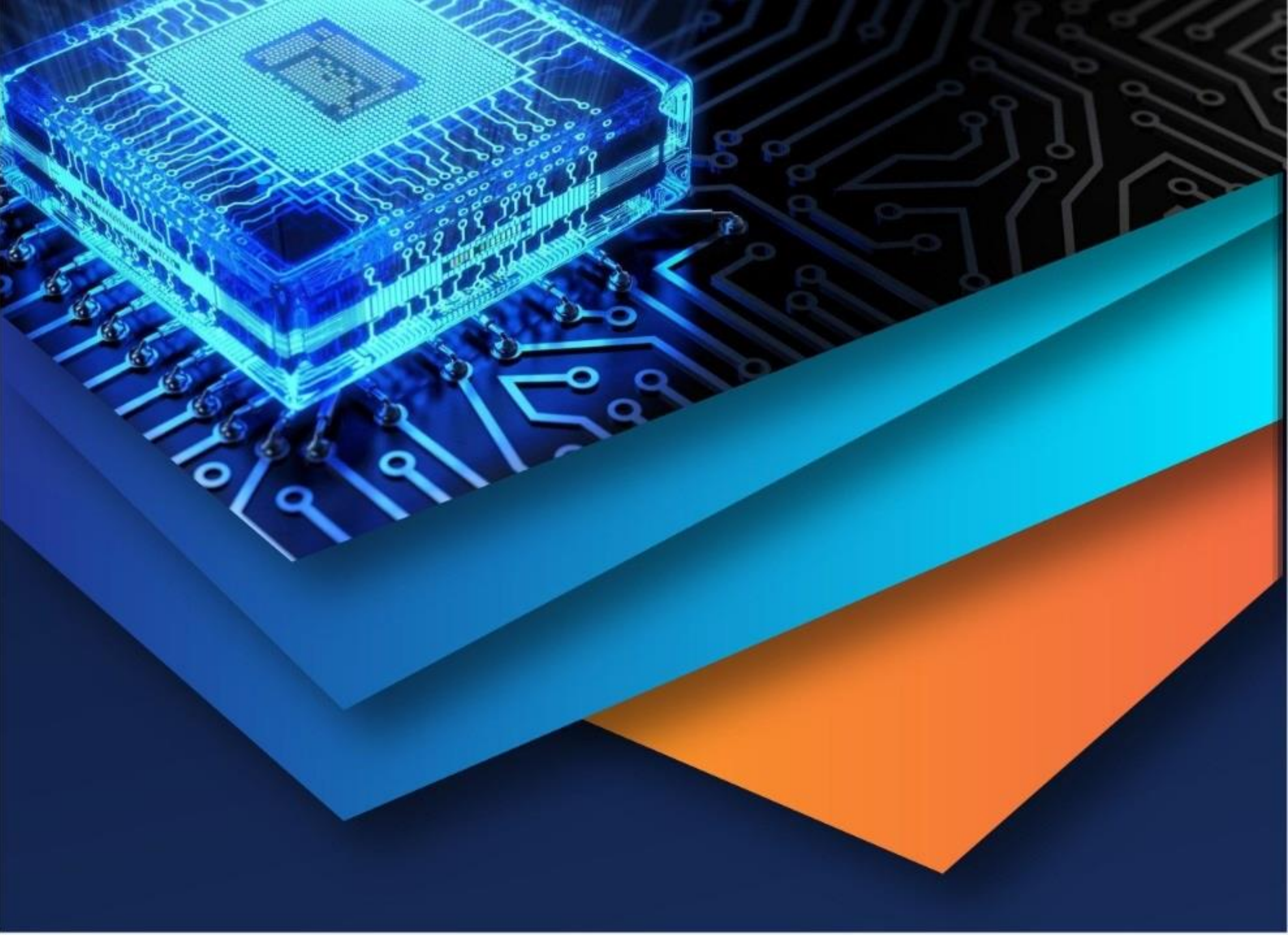

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