

SYNTHESIS OF BIODIESEL FROM SILK- COTTON SEED OIL USING FULLER'S EARTH CATALYST

Submitted in partial fulfillment of the requirements for the award of Bachelor
of Technology in Chemical Engineering.

by

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SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(Deemed to be university U/S 3 of UGC Act 1956)

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the Bonafide work of **Srayansh Kashyap(40190031)** and **Chirag.K(40190005)** who have done the Project work as a team who carried out the project entitled "**Synthesis of Biodiesel from silk-cotton seed oil using Fuller's earth catalyst**" under my supervision from November 2023 to April 2024.

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DECLARATION

We, SRAYANSH KASHYAP AND CHIRAG.K hereby declare that the Project Report entitled "SYNTHESIS OF BIODIESEL FROM SLIK-COTTON SEED OIL USING FULLER'S EARTH CATALYST." done by us under the guidance of DR.D.VENKATESAN, M.Tech,Ph.D is submitted in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering.

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ABSTRACT

The global demand for clean and sustainable energy sources has driven extensive research into renewable fuels, with biodiesel emerging as a promising alternative to traditional fossil fuels. This paper delivers the study of production of biodiesel through Transesterification reaction using silk-cotton seed oil, fuller's earth catalyst and methanol with the help of microwave. Use silk cotton seed oil for biodiesel could promote sustainable agriculture and reduce dependence on traditional fossil fuels. Silk cotton seed oil is not mainly used for food production so using it for biodiesel would not compete directly with food crops potentially decreasing concerns about food security. Biodiesel produced from this feedstock is biodegradable, which can be beneficial in decreasing the environment impact in case of spills or leaks. The silk-cotton seed oil has a free fatty acid content of 6.87% and the density of silk-cotton seed oil was found to be 0.91g/cm^3 . Fuller's earth has been chosen as a catalyst in this project, it is also the novelty of this project. The fuller's earth catalyst was characterized using modern techniques such as Fourier Transform-Infrared Spectroscopy (FT-IR), which indicated presence of essential bonds. FESEM & EDX which confirmed the presence of active sites and elemental composition. From BET analysis the surface area was found to be $7.176\text{ m}^2/\text{g}$ and pore volume was 0.008 cc/g . The biodiesel was hence obtained with a yield of 67%. The biodiesel obtained characterized using Gas chromatography-Flame Ionization Detection (GC-FID) and Gas chromatography Mass Spectrometry (GC-MS) indicating presence of the Fatty Acid Methyl Ester (FAME). Following this result RSM was conducted for optimization and optimum condition was found. After the property test density was 863g/cc , Viscosity was $2.22\text{ mm}^2/\text{s}$. Overall this paper contributes to the ongoing efforts to develop sustainable and economically viable biodiesel production processes.

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LIST OF ABBREVIATIONS

FAME	Fatty Acid Methyl Esters
RSM	Response Surface Methodology
D.Time	Duration of Reaction Time
FTIR	Fourier Transform Infrared Spectroscopy
FESEM	Field Emission Scanning Electron Microscope
EDX	Energy Dispersive X-Ray
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
GCFID	Gas Chromatography Flame Ionization Detection
GCMS	Gas Chromatography Mass Spectroscopy
ANOVA	Analysis of Variance
A	Alcohol to oil molar ratio
B	Catalyst wt%
C	Microwave Power
D	Time
AB	Alcohol to oil molar ratio * Catalyst wt. %
AC	Alcohol to oil molar ratio * Microwave Power
AD	Alcohol to oil molar ratio * Time
BC	Catalyst wt. % * Microwave Power
BD	Catalyst wt. % * Time
CD	Microwave Power * Time
A ²	Alcohol to oil molar ratio ²
B ²	Catalyst wt. % ²
C ²	Microwave Power ²
D ²	Time ²

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1. INTRODUCTION

The worldwide chase of sustainable and environment friendly source of energy has lead to deep and detailed research and innovation dedicated in the area of biofuels. Among all the biofuels, biodiesel has shown a promising alternate to crude fossil fuels, which offers a clean and more prolonged sustainable energy source(Rahul Soosai *et al.*, 2022). Biodiesel manufactured using a reversible process named as transesterification. In process triglycerides from renewable sources like oils are transformed into biodiesel and glycerol, with the little help of a catalyst. While we choose the catalyst for production of biodiesel we should always go for heterogenous catalyst as it is the best option in order to use the catalyst again and again so that it will positively impact the economy(Chouhan and Sarma, 2011) . This project provides detailed insight on the manufacturing of biodiesel by using silk-cotton seed oil for renewable feedstock, methanol for the alcohol component, and Fuller's Earth as catalyst for accelerating the reaction. This very unique reaction mixture not only focuses on the issue of the need for renewable energy option but also discovers the capability of not very conventional feedstock and innovative catalysts to make the biodiesel production process more convinient and a lot more easier. Moreover, the project focuses on advanced and unique methodology such as microwave irradiation, which help increasing reaction speed for the transesterification reaction and contributes to making a more efficient process. The successful production of biodiesel from silk-cotton seed oil shows a promising sustainable and eco-friendly energy source, which reduces the human impact on precious environment related with traditional fossil fuels(Rahul Soosai *et al.*, 2022). Futhermore, the innovative use of Fuller's Earth catalyst, together with other efficient analytical techniques, which increases the quality and efficiency of the biodiesel production process. By going through some rigorous tests and analysis, our project ensures that the biodiesel we have produced meets the necessary quality standard and is viable for use as a biofuel.

1.1 BIODIESEL

Biodiesel is a renewable and sustainable alternate option for use in place of conventional fossil diesel fuel(Okoduwa *et al.*, 2024). It is produced from multiple feedstocks, which includes vegetable oils, animal fats, and even wasteover oil left after cooking(Novita *et al.*, 2024). It is mainly from a chemical process known as transesterification, where triglycerides present in different feedstocks are transformed to fatty acid methyl esters (FAME)(Balat and Balat, 2010) .One feedstock with notable potential is silk-cotton seed oil, which is formed from seeds of the silk-cotton plant (*Ceiba pentandra*). Biodiesel synthesis from silk-cotton seed oil undergoes a transesterification process where triglycerides present in the oil are transformed to fatty acid methyl esters (FAME) by the use of methanol and a catalyst, such as Fuller's Earth.

1.1.1 Advantages of Biodiesel

Biodiesel, as a renewable and sustainable energy source alternative to traditional fossil fuels. Some of the key benefits of biodiesel include:-

- Renewable feedstock: Biodiesel synthesis from renewable source such as vegetable oils, animal fats, and also algae. This can be used in place of finite fossil fuel resources, making biodiesel a sustainable and clean and green energy option(Uttamrao, 2018).
- Lowered Greenhouse Gas Emissions: Biodiesel generally has lower carbon emissions in comparison to fossil diesel. It can notably reduce greenhouse gas emission including CO₂ and particulate matter
- Biodegradable and Non-Toxic: Biodiesel decomposes at a faster rate, it has a less environment impact as compared to conventional fuel in the factors of spills or leaks. Moreover, it is safer for the environment than traditional diesel because it is less toxic.
- Better engine performance: Due to Biodiesel being more lubricious as compared to conventional diesel fuel, it can keep engines running efficiently and for long- term.

- Availability: A large no of local resources can be used to produce biodiesel, decreasing dependency on imported oil, and increasing energy security.

1.1.2 Disadvantages of Biodiesel

Biodiesel, inspite of its many advantages, it also comes with certain disadvantages that should be :-

- Feedstock Competition: Biodiesel production competes on land and resources with food and also feed production, which raises concerns about food security and land use conflicts(Li, Chen and Nie, 2023).
- Limited Feedstock Availability: The availability of viable feedstocks for biodiesel manufacturing can be restricted by factors such as climate, soil conditions, and land available.
- Energy Consumption: The procedure of growing feedstock crops and transforming into biodiesel can be energy-intensive,, offsetting of some of the environment advantages(Falowo *et al.*, 2021).
- Unsuitable Weather Performance: Biodiesel could have issues with cold weather performance, which includes potential gelling or solidification at lower temperatures, which can possibly affect engine operation.
- Variance in Quality: The quality of biodiesel can be diffrentiated depending upon the feedstock source and production methodology, that requires careful quality control.

1.2 TRANSESTERIFICATION

Transesterification is traditionally used chemical process at the heart of biodiesel production. It is a unique reaction that transforms triglycerides , mainly to be found among vegetable oils and animal fats, to fatty acid methyl esters (FAME), which makes up the core of biodiesel. The transesterification process can be carried out in a variety of ways, such as microwave, ultrasonic, and conventional heating(Bizualem and Nurie, 2024). Since ultrasonic heating converts electrical energy into heat energy, it functions similarly to microwave heating(Mohd Johari *et al.*, 2024).The transesterification process includes the breaking of chemical bonds in the triglycerides, in which three fatty acids are attached to a glycerol molecule. In presence of an alcohol, mainly methanol or ethanol, and a catalyst, such as our Fuller's Earth, these bonds are cleaved, leading to glycerol formation as a

byproduct and the creation of FAME molecules. The result is a cleaner-energy fuel that shows properties familiar to diesel but with lowered emissions of greenhouse gases, sulfur compounds, and particulate matter. Transesterification not only crucial for converting renewable feedstocks into a practical energy source but also plays a crucial role in decreasing the environment impact of transportation and promoting sustainable energy solutions.

1.3 SILK-COTTON SEED OIL

Silk-cotton seed oil is the primary raw material used by us in production of biodiesel. Made from seeds of the silk-cotton tree (*Ceiba pentandra*), this oil serves as the source of triglycerides we need for the transesterification process (Muhammad, 2018).

The oil constitutes of a mixture of triglycerides, which are made of fatty acid chains which are bonded to a glycerol compound. Triglycerides like these are the raw materials that undergo transesterification, a chemical reaction where the glycerol is replaced with methanol (or another alcohol), resulting in the production of methyl esters of fatty acid, which make up biodiesel.

The use of silk-cotton seed oil as a feedstock is significant for biodiesel production. It is considered a non-food feedstock, i.e., it doesn't compete with food production and also concerns related to the diversion of food crops for fuel synthesis. This feature contributes to sustainability and reduce the ethical and economic dilemmas related with food-based biodiesel feedstocks (Muhammad, 2018).

Moreover, the chemical composition of oil silk-cotton seed, including the types and amount of fatty acids present, it can vary depending on factors like the geographical origin of the tree from which silk-cotton seed is obtained and the oil extraction procedures. Understanding and optimizing the use of silk-cotton seed oil is essential for achieving the desired biodiesel yield percentage, quality, and overall process efficiency (Oil, 2020).

1.4 FULLER'S EARTH

Fuller's Earth is a naturally found in nature, it appears to be a clay-like substance known for its advanced adsorbent properties and multiple industrial applications. It is mainly made up of hydrated aluminum silicates and differs in mineral compositions, depending on its source (Rekunge, Indalkar and Chaturbhuj, 2016).

In the reference of biodiesel production, Fuller's Earth can be used as a catalyst for the transesterification process in the following ways:-

- Fuller's Earth is viable as a catalyst in the reaction of transesterification, a crucial step in biodiesel synthesis. It plays a most important part in the transformation of triglycerides (which is found in plant oils or animal fats) into fatty acid methyl esters (FAME), which make up biodiesel. Fuller's Earth's catalytic abilities arises from its surface properties, which provides significant no of active sites in order to carry out transesterification process(Ulakpa *et al.*, 2022).
- In the presence of methanol or other alcohol, Fuller's Earth increses the reaction kinetics by decreasing the activation energy required for the reaction. It, functions by breaking down the chemical bonds present in the triglycerides, efficiently splitting them into glycerol and methyl esters. This process allows for the transformation of the feedstock, like silk-cotton seed oil, into biodiesel.
- The advantage of Fuller's Earth use as catalyst in biodiesel synthesis includes it's, low price, natural abundance, and also the fact that it can be regenerated and reused again and again multiple times. These characteristics make Fuller's Earth a most viable and sustainable choice for biodiesel manufacturers. However, the efficiency of Fuller's Earth depends upon factors such as the quality of the feedstock, conditions of the reaction, and the category of Fuller's Earth used, making optimization and fine-tuning essential to achieve the desired biodiesel yield and quality(Banga and Pathak, 2023).

1.5 PROPERTIES OF BIODIESEL

Biodiesel, a renewable and sustainable substitute for traditional diesel fuel and a clean source of energy, shows very distinctive properties that affects its suitability for use in diesel engines(Ude *et al.*, 2022). Here are the basic characteristics of biodiesel:-

- Viscosity: Biodiesel generally have a higher viscosity, or thickness, if compared to conventional diesel. This influences fuel atomization and combustion in engines.

- **Cetane Number:** Biodiesel mostly has a higher cetane number, which signifies better ignition quality and a shorter delay in ignition. These characteristics lead to improved engine performance and reduced emissions.
- **Density:** Biodiesel's density is one higher than that of traditional diesel, although the difference is not of any major importance. This deflection in density has minimal effect on the engine performance and fuel storage.
- **Flash Point:** Biodiesel shows a higher flash point, which makes it safer for handling and transportation purposes as it is very less susceptible to ignition when exposed to an open flame or spark.
- **Cloud Point:** Biodiesel does have a higher cloud point, which indicates the temperature at which it starts to solidify. This aspect can be of a concern in colder climates and may necessitate mixing in with fossil diesel for use in such conditions.
- **Pour Point:** Like cloud point, pour point of biodiesel signifies the temperature at which it becomes too viscous to flow i.e. it has a higher resistance in flow. This property can be higher as compared to fossil diesel and may require the addition of cold-flow additives in lower temperature regions.

1.6 APPLICATIONS

Biodiesel, a renewable and eco-friendly alternative to conventional diesel fuel, serves as a lot of practical applications in various divisions (Li, Tian and Xu, 2012). Here are some primary uses of biodiesel:-

- **Transportation:** Biodiesel is mainly used in place of diesel fuel in transportation vehicles such as cars, trucks, buses, and trains. It can also be mixed with diesel fuel, allowing pre-existing diesel engines to operate with less carbon emissions, contributing to cleaner and greener air (Adepoju *et al.*, 2022).
- **Agriculture and Farming:** Biodiesel can be used to power farm equipments like tractors and other agricultural vehicles. This supports environment-friendly agricultural methods, reduces agriculture's impact on the environment, and supports ecological goals.

- Maritime Industry: Biodiesel is a viable fuel source for marine engines i.e engines running on water, fueling in boats and ships. Adoption lowers emissions in the marine industry, reducing the environmental effect of transportation by water.
- Electricity Generation: Diesel generators that produces power use biodiesel, mainly in isolated or off-grid areas. It gives a dependable power supply while limiting pollution.
- Aviation: Even though biodiesel are rare in commercial aviation, a few smaller and experimental aircraft have been modified to operate on biodiesel, which helps reduce emissions in this industry(Das and Chowdhury, 2023).
- Heating Oil: Biodiesel could be mixed with conventional heating oil, offering an eco-friendly option for residential and commercial space heating. It helps reducing the carbon emissions of heating systems.

1.7 BY-PRODUCT UTILIZATION

As a byproduct, around 100 kg of glycerol are produced for every ton of biodiesel. Centrifugation or gravity separation are the two primary methods used in the conventional removal of glycerol(Neubauer *et al.*, 2023). This process produces crude glycerol, which may still include ppm levels of contaminants such methanol, oil, soap, salt, and other organic compounds. To increase the biodiesel industry's economic sustainability and lessen the negative environmental effects of the waste produced, it is critical to use crude glycerol efficiently. The removal or reduction of these contaminants can improve the use and value of crude glycerol. Therefore, it's critical to create a process that would raise crude glycerol's marketable and economic worth(Abdul Raman, Tan and Buthiyappan, 2019).This project presents a different approach to purify glycerol i.e by acidification and activated charcoal treatment. Consequently, we may increase glycerol's application in creating value-added goods by glycerol purification, which would increase income for the biodiesel sector.

2. LITERATURE REVIEW

- **Michael Rahul Soosai et al.(2022)** have published a research paper title **“Integrated global optimization and process modelling for biodiesel production from non-edible silk-cotton seed oil by microwave-assisted transesterification with heterogeneous calcium oxide catalyst,”**. In the paper the possibility of oil from silk cotton seed as a feedstock for biodiesel synthesis is examined by the authors in their research study, with a particular emphasis on the use of heterogeneous catalysts and microwave-assisted transesterification. In comparison to traditional approaches, the study shows that microwave irradiation may effectively speed up the transesterification process, resulting in larger yields of biodiesel within shorter reaction times. Furthermore, the use of heterogeneous catalysts improves the reaction's efficiency and selectivity and makes it easier for catalysts to be reused, which lowers costs and has a less environmental effect. Overall, the study emphasizes the potential of silk cotton seed oil in the synthesis of biodiesel and stresses the value of heterogeneous catalysis and microwave-assisted transesterification as long-term strategies for raising the productivity of biodiesel manufacturing processes.
- **Chouhan et al.(2011)** have published a review paper on the title **“Modern heterogeneous catalysts for biodiesel production: A comprehensive review,”**. The review study examines the function of diverse catalysts in the biodiesel synthesis process in detail, examining several types of catalysts such as solid acids and metal oxides. In order to clarify catalyst performance, efficiency, and influence on biodiesel output and quality, study findings are synthesized. Catalyst activity, stability, and recyclability—all essential for real-world use and financial sustainability—are assessed severely in this review. It focuses on catalyst design and optimization techniques to improve performance and get around constraints, providing industry and research with direction. The study is a useful tool for pushing biodiesel production toward sustainability and the use of renewable energy sources by looking at current developments and new trends.

- **Rekunge et al(2016).** have published a research paper titled “**Activated Fuller’s earth as an inexpensive, eco-friendly, efficient catalyst for the synthesis of 5-aryl 1-H-tetrazole via [3+2] cycloaddition of nitriles and sodium azide,**” The study investigates Fuller's earth in activated state as a very effective heterogeneous catalyst for the synthesis of biodiesel. Numerous benefits occur with this catalyst, such as affordability, stability, simplicity of recovery, reusability, and ecological advantages. Its practical application is further enhanced by the fact that it functions under moderate reaction conditions and permits a high product yield. The study emphasizes how important Fuller's earth is to catalyzing the transesterification reaction, which allows feedstock to be converted into biodiesel with exceptional efficiency. Utilizing these advantageous properties, the catalyst shows great promise for large-scale biodiesel production. Furthermore, its eco-friendly qualities support sustainable production methods, which is in line with the increased focus on eco-friendly technology and renewable energy sources.
- **Balat et al.(2010).**have published a review paper titled “**Progress in biodiesel processing,**” This review article offers an in-depth review of the developments in the manufacture of biodiesel from a variety of edible and non-edible oils. It summarizes the advancements made in the use of a range of feedstocks, including conventional and non-conventional sources such waste oils and algae. In addition, the paper explores modern techniques used in the production of biodiesel, emphasizing cutting-edge methods such as heterogeneous catalysts, and transesterification reaction. Through an analysis of various techniques' efficiency, yield, and sustainability, the report provides insightful information on how the biodiesel manufacturing industry is changing. Researchers and industry experts looking to improve biodiesel production methods, encourage resource diversification, and accelerate the shift to sustainable energy solutions may find this brief overview of study findings to be a helpful reference.
- **Mohd Johari et al.(2024).** have had researched and presented a paper titled“**Optimization and kinetic studies for biodiesel production from**

dairy waste scum oil via microwave assisted transesterification,”. The study looks at the use of microwave-assisted heating in transesterification to produce biodiesel, and it finds a number of significant things. The technology efficiently shortens response times and overcomes activation energy barriers by utilizing microwave energy, which speeds up the production of biodiesel. This creative method improves overall process efficiency while also quickening the feedstock's conversion to biodiesel. The study emphasizes how important microwave-assisted heating is for accelerating reaction kinetics because of its capacity to heat reaction components individually and promote molecular interactions. The research presents convincing evidence of the revolutionary effects of microwave-assisted transesterification through rigorous testing and analysis, providing knowledge that has the potential to completely change the processes involved in the manufacturing of biodiesel. This study increases productivity, decreases resource consumption, and optimizes industrial processes to advance sustainable energy solutions.

- **Muhammad et al.(2018).**have researched on silk-cotton seed oil and presented paper titled“**Biodiesel Production from Ceiba pentandra Seed Oil Using CaO Derived from Snail Shell as Catalyst,**”. The study investigates the unrealized potential of non-food oil from silk cotton seed as a possible feedstock for the manufacturing of biodiesel. The study shows that using silk cotton seed oil as a renewable supply-chain for biodiesel production is feasible via considerable testing and analysis. In order to determine if oil from silk cotton seed is a viable resource for the biodiesel synthesis the research evaluates important factors such oil composition, yield, and quality of biodiesel produced. Additionally, the study emphasizes the benefits—both financial and ecological—of utilizing non-food oils, such as silk cotton seed oil, which expands the range of feedstock alternatives and encourages environmentally friendly methods in the energy industry. All things considered, the results highlight the importance of non-food oils, especially silk cotton seed in enhancing biodiesel production and using it as a renewable energy source.

- **Ude et al.(2022)** published a paper titled **“Prediction of some thermo-physical properties of biodiesel using ANFIS and ANN cum sensitivity analysis,”** The study indicate uses modern techniques to comprehensively investigate the biodiesel properties like kinematic viscosity and cetane number. Using advanced techniques like spectroscopy and chromatography, the research explores the physical and chemical properties essential to biodiesel efficiency. The research clarifies the quality, effectiveness, and environmental impact of biodiesel by closely examining relationships among these attributes. Regulations and biodiesel production optimization are guided by such observations. The research advances our understanding of biodiesel and promotes its viability and effectiveness as a substitute fuel.
- **Tian et al(2012).**made a comprehensive study and presented a paper titled **“Application of Biodiesel in Automotive Diesel Engines,”**. The study examines the numerous applications of biodiesel in diesel engines and offers a thorough examination of its efficiency, emissions, and engine compatibility. The research assesses the effectiveness of biodiesel as a sustainable fuel substitute using practical investigations and theoretical assessments. It compares its emission profiles, engine operating parameters, and combustion properties to those of standard diesel fuels. The influence of biodiesel mixes on engine longevity, lubrication, and fuel system integrity are also examined in this article. It provides insightful information on viability of biodiesel and potential obstacles in diesel engine applications by combining the results of many research. The study advances our knowledge of how biodiesel helps to mitigate environmental pollution from internal combustion engines and lessen reliance on fossil fuels.
- **Umar et al.(2020).**have done a reseach and published a paper titled **“The Effects of Temperature and Catalyst Concentration on Transesterification of Oil Extracted from Ceiba Pentandra (Silk Cotton),”**. This study examines temperature and catalyst concentration effects on the transesterification of Ceiba pentandra (silk cotton) seed oil using a two-phase process that includes alkali-catalyzed transesterification

and acid esterification. The research investigation clarifies their impact on biodiesel output and quality by methodical experimentation with different catalyst concentrations and temperature ranges. The study identifies ideal parameters for increasing biodiesel production efficiency while lowering expenses and environmental effect by examining reaction kinetics and product features. In order to optimize the synthesis of biodiesel from Ceiba pentandra seed oil, the results provide insightful information on the complex interaction between process variables and reaction outcomes. In order to improve transesterification efficiency and advance sustainable biodiesel manufacturing methods, this research emphasizes the significance of controlling catalyst concentration and temperature.

- **Uttamrao et al.(2018).**have had published paper titled “**Production of Biodiesel From Refined Cotton Seed,**”. The study examines the best circumstances for producing biodiesel and evaluates the benefits and drawbacks of doing so. To enhance biodiesel output and quality, critical factors including temperature, catalyst type, and reaction time are tuned through methodical testing. The goal of the research is to minimize resource consumption and environmental effect while increasing the efficiency of biodiesel manufacturing processes by determining these ideal circumstances. The study also emphasizes the benefits of biodiesel, such as its renewable nature, lower greenhouse gas emissions, and potential for energy independence. It also covers disadvantages including feedstock rivalry with food crops, land use issues, and variations in biodiesel quality. By means of an extensive evaluation, the study offers significant perspectives for promoting the production of sustainable biodiesel and guides the process of deciding whether or not to use it as a substitute fuel source.

3. AIM & SCOPE

3.1 AIM

- To synthesize the Fuller's Earth catalyst and study the characteristics of catalyst.
- To produce Biodiesel from silk-cotton seed oil and fuller's earth catalyst.
- Examine the characteristics of Biodiesel produced.
- Analyse the properties of produced Biodiesel.

3.2 SCOPE

- Sustainable development in the field of fuels by biodiesel synthesis from silk-cotton seed oil and fuller's earth.
- Discover the application of fuller's earth as catalyst in transesterification reaction.
- Optimization to enhance the efficiency and yield of biodiesel production through silk-cotton seed oil.

4. MATERIALS AND METHOD

4.1 EQUIPMENTS AND CHEMICALS

Equipments used in this project are:-

- Magnetic Stirrer
- Sonicator
- Hot Air Oven
- Microwave Oven
- Seperating Funnel

Chemicals used for this project are:-

- Fuller's Earth
- Sodium Hydroxide
- Silk-Cotton Seed Oil
- Methanol

4.2 CATALYST PREPARATION

The synthesis of our catalyst using Fuller's Earth is a crucial step in the biodiesel production process. The catalyst is essential for facilitating the transesterification reaction, converting feedstock oils into biodiesel. Here is step by step procedure of preparation of catalyst:-

1. **Mixing Fuller's Earth with Distilled Water:-** In this first step, distilled water and Fuller's Earth are combined with a magnetic stirrer to create a slurry. The Fuller's Earth particles are thoroughly dispersed throughout the water by the magnetic stirrer, resulting in an uniform combination. The catalyst is built on top of this slurry.
2. **Addition of NaOH for activation:-** Adding NaOH to solution of Fuller's Earth and water is next step. NaOH is a typical catalyst used in transesterification reactions. After that we have stirred the solution for 2 hours helping the NaOH mixture dissolve into the solution, which leads to the catalytic solution formation.

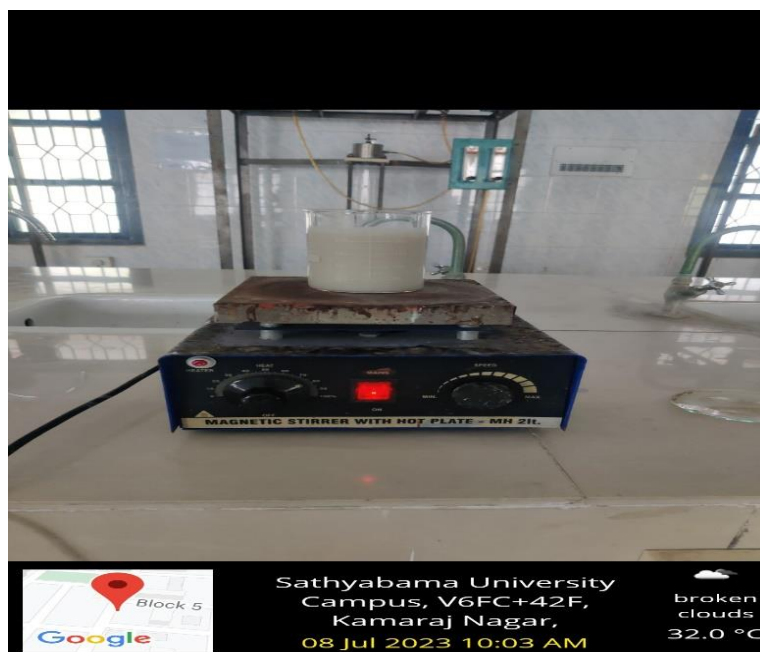


Fig 4.1:-Magnetic stirrer

3. **Sonication:-** The solution is thereafter sonicated for 20 minutes at 40°C temperature. High-frequency sound waves are used in sonication to increase agitation and mixing. By making sure Fuller's Earth and NaOH are well distributed, this step serves to enhance catalytic activity.



Fig 4.2:-Sonicator

4. **Stirring:-** The solution is agitated for two hours after sonication. This extra stirring promotes the catalyst's component interaction and helps to keep everything consistent.
5. **Filtering the Solution:-** The solution is filtered to extract the liquid from the slurry. The transesterification reaction requires the catalyst particles, which is the solid slurry left after extracting the liquid.
6. **Drying in a Hot Air Oven at 70°C (1 hour):-** After filtering, the solid slurry is dried for an hour at 70°C in a hot air oven. By removing any last traces of moisture, this stage guarantees that the catalyst is dry and active and prepared for use in the transesterification reaction.

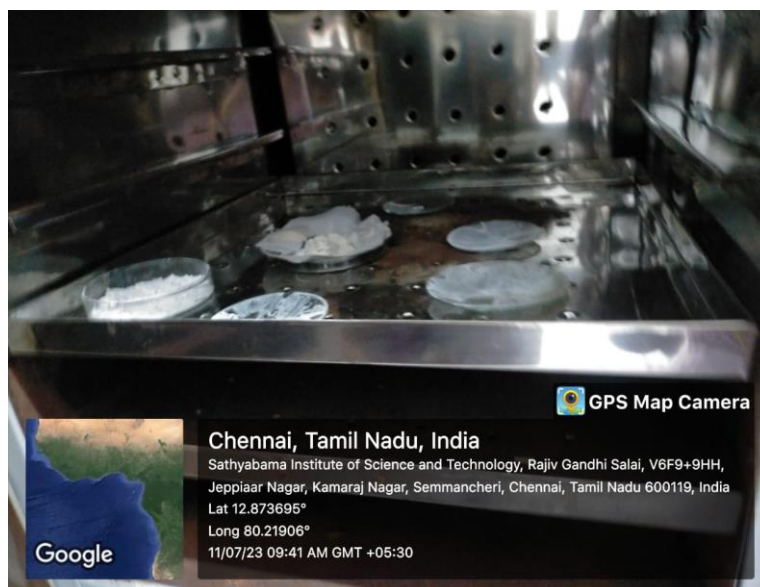


Fig 4.3:-Drying in Hot Air Oven

To produce a highly effective catalyst, a number of precisely regulated procedures are involved in the catalyst production process. Fuller's Earth and NaOH combine to form the resultant catalyst, which is essential for starting and accelerating the transesterification reaction and enabling the effective synthesis of biodiesel from feedstock oils such as silk-cotton seed oil. This carefully designed catalyst guarantees that triglycerides are converted to glycerol and biodiesel, helping to produce a premium biodiesel product.

4.3 SYNTHESIS OF BIODIESEL

The biodiesel synthesis from silk-cotton seed oil, methanol, and a catalyst by microwave irradiation is a novel and effective process. The transesterification process gets sped up using this approach by heating the reaction mixture quickly and precisely. This is the detailed process that was used to create biodiesel. Below is the step by step procedure followed for biodiesel production:-

1. **Microwave Irradiation:-** In this stage, the reaction mixture—which consists of 3.04 grams of catalyst, 31.08 milliliters of methanol, and 30 milliliters of silk-cotton seed oil in the molar ratio of 1:27—is heated in a microwave oven. The microwave has a 300W power supply and a 270-second reaction period with an on and off cycle. This cycle keeps the temperature from rising too high while enabling effective heating. The transesterification step, which turns the triglycerides in the oil into glycerol and biodiesel, is started by the microwave irradiation.



Fig 4.4:-Microwave irradiation

2. **Separation:-** The reaction mixture is allowed to cool and split into two different phases followed microwave irradiation. Fatty acid methyl esters, or FAME, the biodiesel, are found in the top phase, while glycerol and any unreacted methanol or reagents are found in the lower phase. In order to

obtain the biodiesel product from the reaction mixture requires separation and is carried out in a separating funnel.



Fig 4.5:-Separation

3. **Yield calculation:-**It was determined that the biodiesel yield was 67%. The proportion of the theoretical maximum amount of biodiesel that was successfully generated from the raw ingredients is represented by this yield. A 67% output suggests that a significant portion of the feedstock oil has been synthesized into biodiesel.

$$\text{Yield\%} = \frac{\text{Weight of biodiesel}}{\text{Weight of Oil}} \times 100$$

4. **Density measurement:** The biodiesel product's density was determined to be 788 kg/m³. A crucial attribute that tells us about the fuel's weight and volume is its density. It serves to evaluate the biodiesel product's quality.
5. **Water Washing:** In order to purify the biodiesel further and get rid of any residual impurities, the biodiesel is sent for water washing. In this process the biodiesel is mixed with hot water and left for some time to let the impurities settle down. Water washing serves to increase the quality of the final biodiesel product.



Fig 4.6:-Water washing

The process mentioned above is a quick and effective way to use microwave irradiation to make biodiesel. By using this method, reaction durations are reduced, transesterification proceeds more quickly, and 67% biodiesel with a density of 788 kg/m³ is produced. The biodiesel is further purified in the next stage by washing it with water, which guarantees that it satisfies quality requirements to be used as fuel.

4.4 PROCESS OPTIMIZATION WITH RSM.

Response Surface Methodology (RSM) which is a statistical and mathematical technique utilized to optimize processes and experiments. In order to identify the ideal condition of input factors that will result in best outcome or response, it is used to investigate the correlations between several variables and responses. RSM's objective is to design a mathematical model that can predict a process' reaction based on the values of its input variables.

Table 4.1: Coded values of Experimental matrix

Factor	Name	Level	Low Level	High Level
1	oil: alcohol ratio	27	24	30
2	catalyst wt%	4	3	5
3	Microwave power	300W	270W	360
4	D.Time	270s	180s	270s

Oil to Alcohol Ratio: This factor tells how much oil there is compared to alcohol in the reaction mixture. This is an important step in the transesterification process, which produces biodiesel. The biodiesel experiment had a low level of 24; a high level of 30, and a mid-level of 27.

Catalyst Weight %: The yield and reaction rate are greatly influenced by the catalyst's weight % in the reaction mixture. This factor has three levels: 3% for the low level, 5% for the high level, and 4% for the mid-level.

Microwave Power: Reaction kinetics and heating efficiency depends upon the power we supply, microwave power is a crucial factor in chemical reactions performed by microwaves. This power at mid-level was set at 300W, its high level at 360W, and its low level at 270W.

Duration of Reaction Time (D.Time): The amount of time taken to perform the reaction i.e, the time duration for which the reaction mixture was inside the microwave. In this instance, the duration at lower level was for 180 seconds with mid and high level to be same at 270 seconds.

Table 4.2:RSM Response Table(Yield percentage)

		Factor 1	Factor 2	Factor 3	Factor 4				Response
Std	Run	A:Alcohol to oil molar ratio	B:Catalyst wt. %	C: Microwave Power	D: Time	Oil	Alcohol	Catalyst	Biodiesel Yield
				W	s	mL	mL	g	%
1	28	24	3	270	270	30	27.63	1.47	80
2	6	30	3	270	270	30	34.53	1.64	82
3	17	24	5	270	270	30	27.63	2.46	86
4	15	30	5	270	270	30	34.53	2.73	83
5	26	27	4	180	180	30	31.08	2.08	87
6	4	27	4	360	180	30	31.08	2.08	86
7	11	27	4	180	360	30	31.08	2.08	89
8	14	27	4	360	360	30	31.08	2.08	87
9	23	24	4	270	180	30	27.63	1.96	84
10	3	30	4	270	180	30	34.53	2.18	82
11	5	24	4	270	360	30	27.63	1.96	84
12	12	30	4	270	360	30	34.53	2.18	83
13	1	27	3	180	270	30	31.08	1.55	80
14	16	27	5	180	270	30	31.08	2.59	83
15	21	27	3	360	270	30	31.08	1.55	82
16	29	27	5	360	270	30	31.08	2.59	84
17	10	24	4	180	270	30	27.63	1.96	87
18	9	30	4	180	270	30	34.53	2.18	85
19	7	24	4	360	270	30	27.63	1.96	83
20	18	30	4	360	270	30	34.53	2.18	87
21	22	27	3	270	180	30	31.08	1.55	89
22	27	27	5	270	180	30	31.08	2.59	87
23	20	27	3	270	360	30	31.08	1.55	84
24	24	27	5	270	360	30	31.08	2.59	80
25	13	27	4	270	270	30	31.08	2.08	92
26	19	27	4	270	270	30	31.08	2.08	92.5
27	25	27	4	270	270	30	31.08	2.08	92.4
28	2	27	4	270	270	30	31.08	2.08	92
29	8	27	4	270	270	30	31.08	2.08	92

Above table is the designed mathematical model to find the ideal response for biodiesel synthesis by use of Response Surface Methodology. After a continuous 30 trials the following is the response derived through experiments. Oil to alcohol ratio of 1:27 with catalyst weight percentage of 3 had the best response of 92.2 yield percent.

4.5 GLYCEROL PURIFICATION

- Checking glycerol pH: Measure the pH of the glycerol sample to assess its initial acidity or alkalinity.
- Acidify glycerol by adding HCl: Adding hydrochloric acid (HCl) to the glycerol to acidify it. This step protonates any remaining hydroxyl groups on the glycerol molecules.
- Check pH after acidification: Measure the pH of the acidified glycerol to confirm the effectiveness of the acidification process. Typically, this step will yield an acidic pH due to the addition of HCl. We have to allow the solution to settle down and transfer it into a separating funnel allowing for phase separation.
- Collecting glycerol: After settling, we have to collect the glycerol layer, which should be the lower layer in the separating funnel.
- Neutralize glycerol with NaOH: Add sodium hydroxide (NaOH) to the collected glycerol to neutralize any remaining acid and adjust the pH to a basic level.
- Heat the solution and allow salt to settle down: Heat the neutralized glycerol solution to 110 degrees Celsius to evaporate any water and allow any dissolved salts to settle down.
- Addition of activated charcoal: Transfer the glycerol solution to another container and add activated charcoal to adsorb any remaining impurities.
- Filtering the solution: Filter the glycerol solution using filter paper to remove the activated charcoal and any impurities adsorbed by it, resulting in purified glycerol.



Fig 4.7 Glycerol Purification

5. RESULT

5.1 TEST ANALYSIS

To demonstrate that the catalyst synthesized had sufficient capabilities to promote a transesterification process and that the biodiesel thus produced could be effectively used in place of conventional diesel, an examination of the catalyst particle and biodiesel is required. Here are some characterizations we carried out to demonstrate the catalyst and biodiesel sample viability.

5.2 FOURIER TRANSFORM INFRARED SPECTROSCOPY(FTIR) FOR FULLER'S EARTH CATALYST.

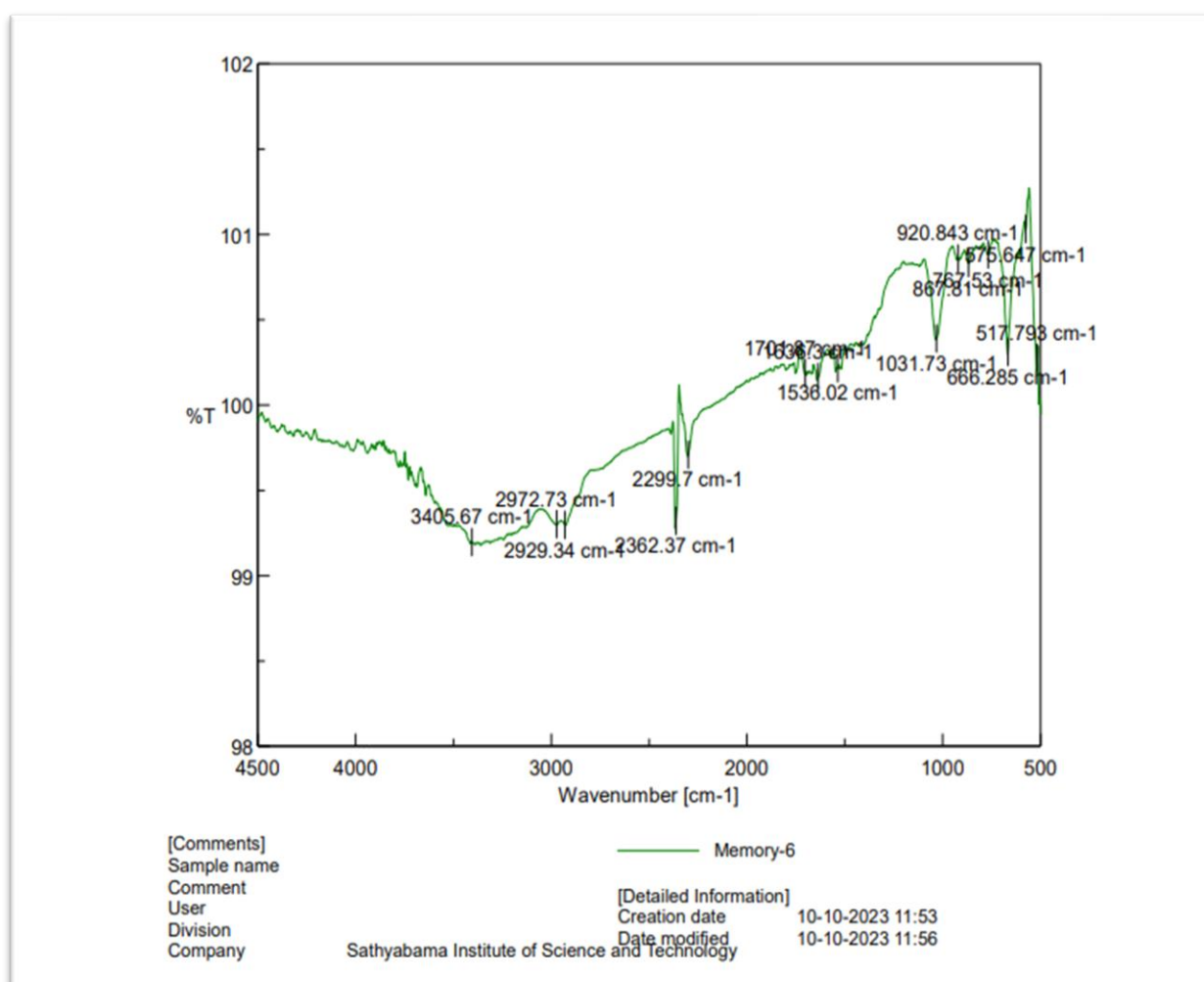


Fig 5.1 FT-IR for catalyst

Based on an infrared absorption spectrum, FTIR is a potent analytical method used to detect and describe chemical substances. FTIR may be used to evaluate functional groups that are present in the catalyst material in the context of the catalyst (Fuller's Earth) study. It offers details on the structure and chemical makeup of the catalyst. It can help validate the identify of the catalyst by pointing out the existence of certain bonds and chemical groups that are typical of Fuller's Earth. The catalyst's suitability for its intended usage in the transesterification process and its proper chemical form are both confirmed by FTIR analysis.

Sample preparation, instrument setup, and background measurement are all part of FTIR analysis. After that, the sample was put into the device for being analyzed. The process of acquiring data began with the collection of interferograms, which are then Fourier-transformed to produce the infrared spectrum. Characteristic absorption bands were identified by analysis, and the spectrum was compared to references for identification. Moreover, quantitative analysis may be carried out. Reports were created from the results. Instrument cleaning and shutdown are included in instrument maintenance. The procedure, which includes measurement, preparation, analysis, and maintenance, allowed materials to be identified and characterized according to their infrared absorption characteristics.

The Fourier Transform Infrared Spectroscopy (FTIR) test results revealed specific peaks at various wavenumbers, indicating the presence of functional groups and molecular vibrations in the analyzed sample. Here's an explanation of the observed peaks:-

C-H Alkane Peaks (2972.73, 2929.34): The presence of C-H (Carbon-Hydrogen) alkane functional groups is indicated by these peaks. Alkane groups are characteristic of hydrocarbon chains in the sample, which are essential for biodiesel.

C=O Peaks (1701.87, 1636.3): The peaks at the certain wavenumbers signify the presence of C=O (Carbon-Oxygen double bond) functional group. This is mainly associated with carbonyl compounds and is indicative of ester bonds.

O-H Stretch Peaks (3405.67, 2362.37, 2299.7): The peaks in this region suggest the presence of O-H (Oxygen-Hydrogen) stretching vibrations.

Major Peaks (575.64, 517.79, 666.255): These prominent peaks indicate characteristic vibrations associated with specific functional groups.

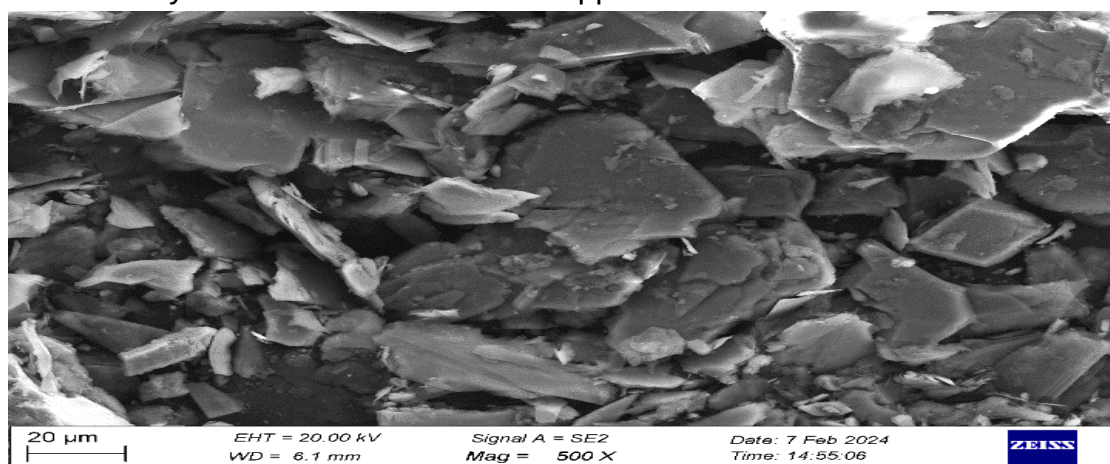
Therefore, the test results indicate the presence of hydrocarbon chains (alkanes) and ester bonds (C=O) in the biodiesel sample, which aligns with the expected chemical composition that are essential for production of biodiesel.

5.3 FIELD EMISSION SCANNING ELECTRON MICROSCOPE (FESEM) FOR FULLER'S EARTH CATALYST

Scanning Electron Microscopy is a very crucial analysis in scientific inquiry, demonstrates a lot of capabilities for understanding and analysing the surface of material with more precision. It employs a focused electron beam at the particles. It reveals the unique surface morphology and composition of particles, which operates by magnifying the images of particles in nanoscale.

Its features is that it zooms deep into the surface of particles and elaborates minute details about the particle, in this case my catalyst. It shows in detail about the surface morphology of the catalyst, its composition and its performance. It also shows the particle size and the arrangement of the particles.

Vacuum chamber evacuation, equipment setup, and sample preparation are all steps for scanning electron microscopy (SEM). After being mounted on a conductive substrate, the sample is put inside the SEM chamber. The voltage and current of an electron beam are set. By using the electron beam to scan the sample surface and find secondary or backscattered electrons, images were obtained. After that elemental analysis was done through energy-dispersive X-ray spectroscopy (EDX). Surface shape, elemental composition, and other features were examined in the data analysis process. Reports were created from the results. High-resolution imaging and elemental analysis of materials are made possible by SEM, which helps with a variety of scientific and industrial applications.



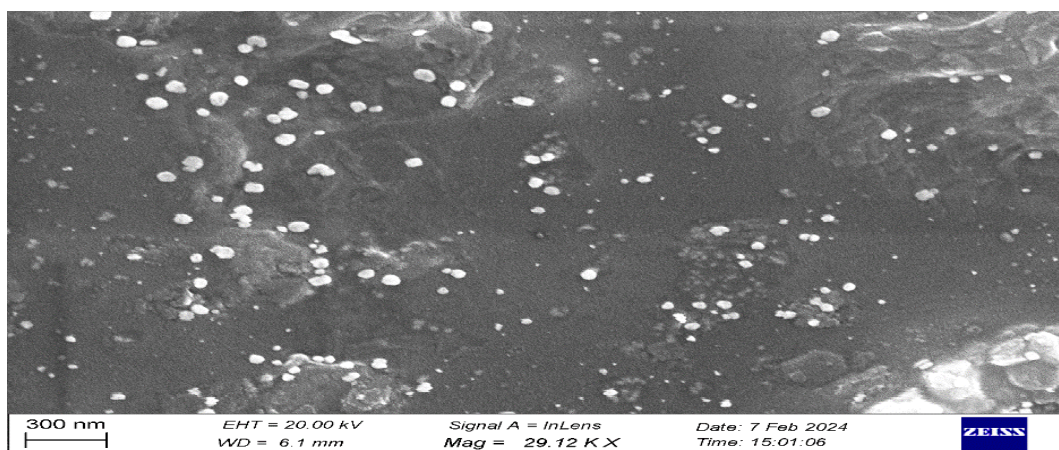
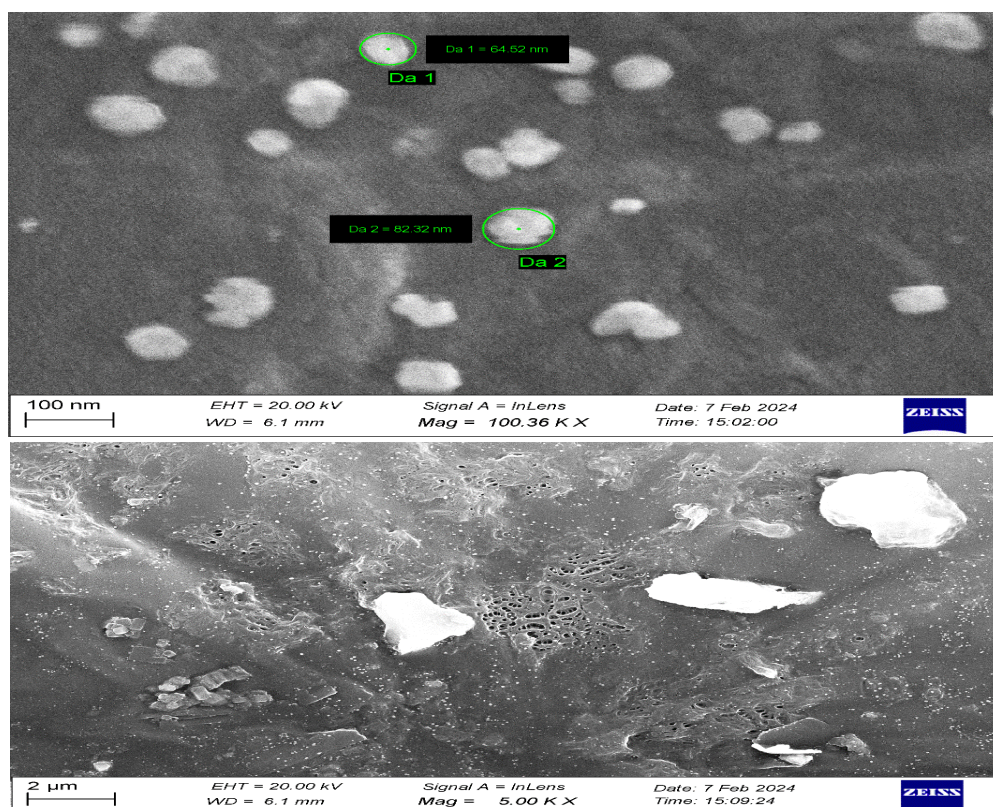


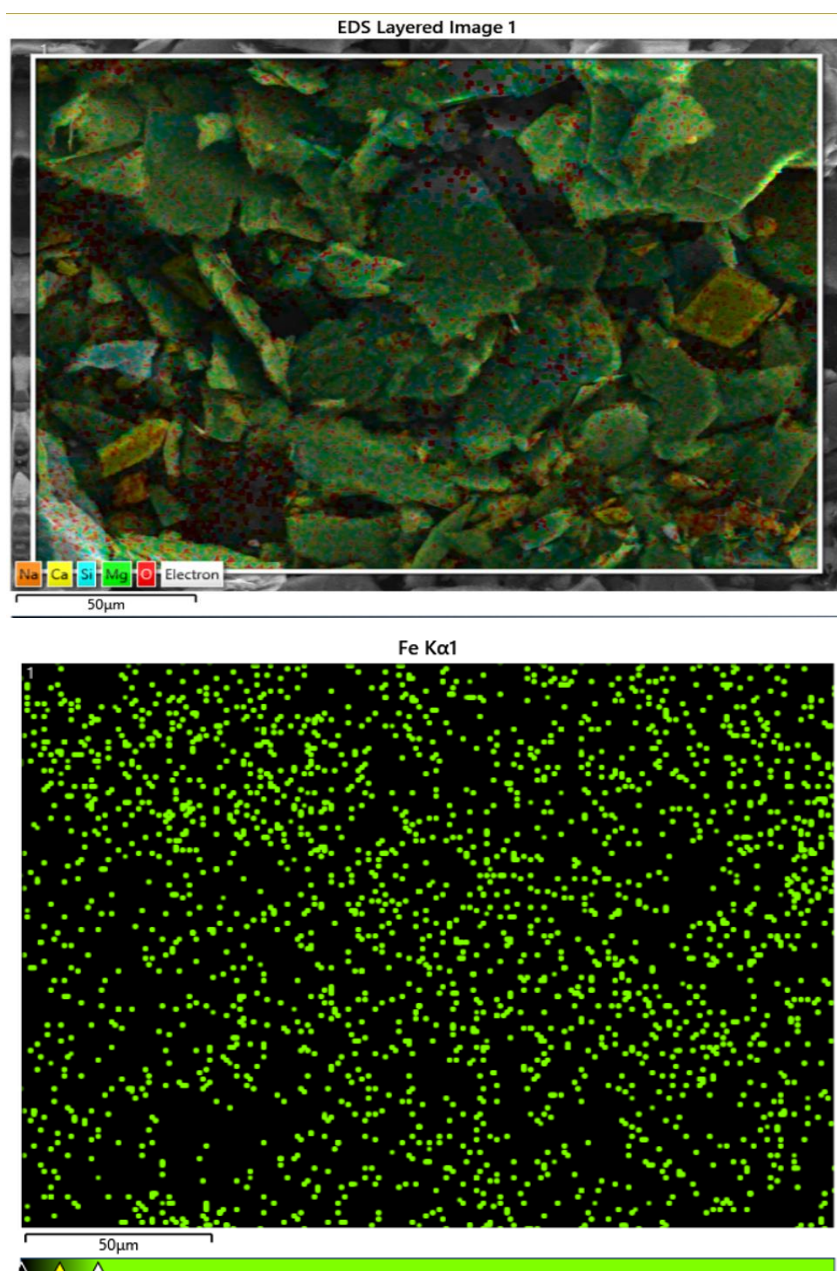
Fig 5.2 SEM for catalyst

Upon analyzing our catalyst sample of Fuller's Earth there were quite good revelations we had a good no of pores and particle diameters were ranging from 64.4 nm to 84.2 nm which indicated its potential efficiency in biodiesel production. The characterization denoted that the catalyst had a high surface area and optimum particle size distribution, which is viable for enhancing catalytic activities and providing efficient reactant diffusion while performing the transesterification process.



5.4 ENERGY DISPERSIVE X-RAY(EDX) FOR FULLER'S EARTH CATALYST.

An effective method for analysing and for figuring out a material's elementary composition is Energy Dispersive X-ray Spectroscopy , which is used in junction with Scanning Electron Microscopy. With SEM, a sample's surface is bombarded with a concentrated electron beam, producing different X-rays. To determine which elements are present in the sample and how much of each is present relative to the others i.e the amount of it present,It examines these X-rays and gives a detailed information on elements and it's compositions.



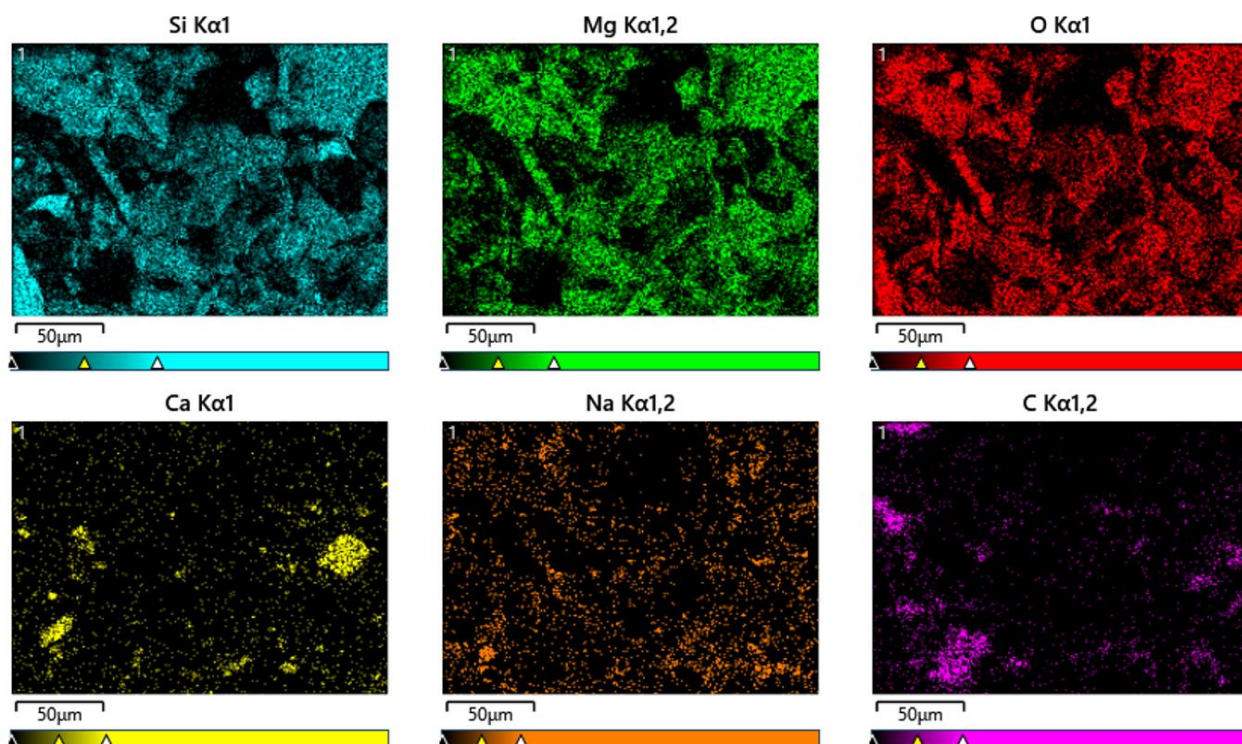


Fig 5.3 EDX for catalyst

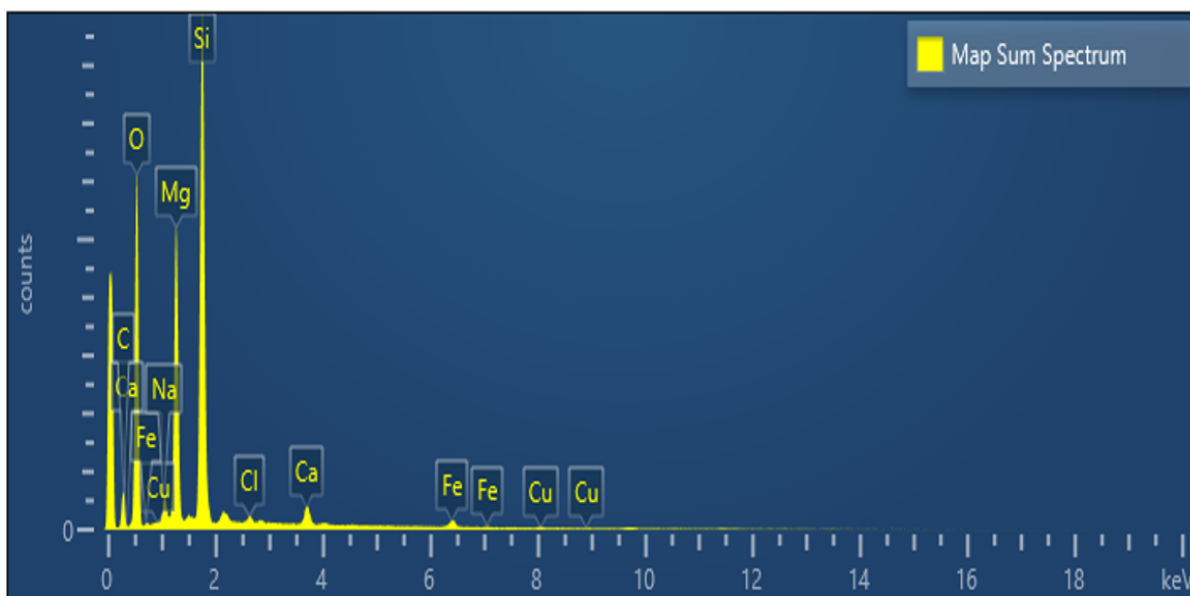


Fig 5.4 Elemental composition of catalyst

Table 5.1 :Elemental composition of catalyst

Map Sum Spectrum Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	22.61	0.58	31.91
O	K series	45.34	0.39	48.03
Na	K series	1.28	0.06	0.95
Mg	K series	11.27	0.12	7.86
Si	K series	17.40	0.17	10.50
Ca	K series	1.07	0.04	0.45
Fe	K series	1.03	0.06	0.31
Total		100.00		100.00

The EDX analysis of the sample indicated the existence of carbon (C), oxygen (O), sodium (Na), magnesium (Mg), silicon (Si), calcium (Ca), and iron (Fe). Among these elements that we found, oxygen has the highest weight percentage at 45.34%, second is carbon at 22.61%. The findings of these elements suggests the sample is mainly made up of organic compounds, along with materials such as polymers or organic coatings.

Furthermore, silicates or other inorganic compounds present due to the significant concentrations of silicon (17.40%) and magnesium (11.27%). The point that these elements can be found in glasses and minerals suggests the fact that composition of our catalyst is heterogeneous, meaning it contains both organic and inorganic elements.

We also found that iron, calcium, and salt together make up less than 2% of the sample's weight, it can be more likely that these elements are mostly additions or contaminants than main components. However, their existence might still affect the catalyst characteristics and uses, especially when it comes to reactivity or structural stability.

Therefore the EDX analysis provided an overview on the elements and its concentration by weight percentage also highlighting the organic and the inorganic compounds. The analysis and information provided through the analysis is very crucial if we want to understand the catalyst properties its application etc.

5.5 BRUNAUER-EMMETT-TELLER(BET) FOR FULLER'S EARTH CATALYST.

BET is a popular technique for figuring out the specific surface area of materials with pores, like catalysts, adsorbents, and nanoparticles. It gives information about surface area, porosity of materials and the methodology is based on measurements of gas adsorption, usually nitrogen adsorption at low temperatures.

BET is used along with BJH in order to calculate pore size distribution of the catalyst particle. The BET method gives information on the catalyst material's total surface area, but it does not tell us specific information about distribution of pore sizes. By offering data on the distribution of pore sizes which is a crucial component for calculating the porosity and functionality of the catalyst material the BJH approach enhances the BET analysis.

The total analysis of a porous material takes place in three simple steps.

- **Adsorption Isotherm Calculation:** Adsorption isotherms are produced by gas adsorption studies, usually across a range of relative pressures (P/P_0). Amount of gas adsorbed onto the material as a function of relative pressure is described by the isotherm. For this step mainly nitrogen gas is used.
- **BET Analysis:** After the adsorption isotherm calculation BET analysis is performed on this isotherm data. This analysis is done on BET equation which is mainly a plot of $P/V(P_0-P)$ v/s P/P_0 where P_0 is saturation pressure
- **BJH Desorption Analysis:** After BET analysis, the desorption of the adsorption isotherm takes place. It analyses the release of gas from pores as pressure decreases. This method is then used to calculate pore size distribution data.

Adsorption Isotherm Calculation.

Parameters for Data Reduction:-

- Temperature 77.350K
- Molec. Wt.: 28.013
- Cross Section: 16.200 Å²
- Liquid Density: 0.808

Adsorbate :-Nitrogen.

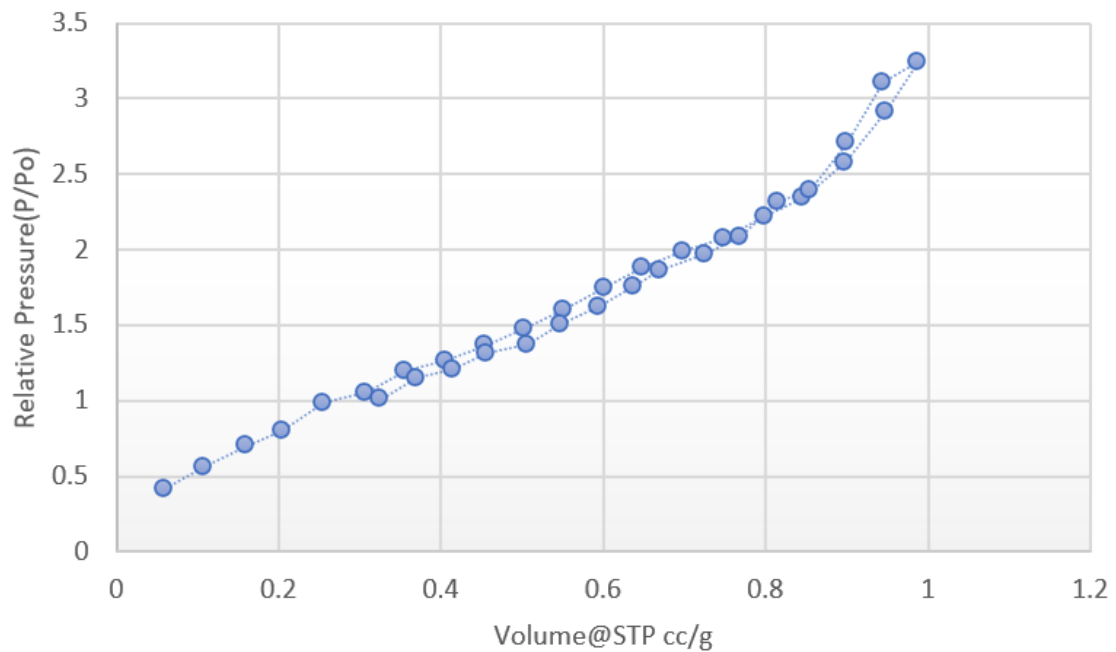


Fig 5.5 Adsorption Desorption Graph.

- ❖ The above graph of relative pressure v/s pore volume represents the adsorption and the desorption curve, it helps in determination of surface area and pore diameter.
- ❖ The top line is the desorption line and the bottom one is adsorption.

BET summary

- Slope = 863.102 1/g
- Intercept = 8.019e+01 1/g
- Correlation coefficient, $R^2 = 0.999564$
- C constant = 11.763
- Surface Area = 3.692 m²/g

Surface Area: The catalyst surface area is was calculated to be at 3.692 m²/g. It indicates a significant active surface area for catalytic reactions. A higher surface area generally correlates with more available active sites for reactions, potentially leading to enhanced catalytic activity.

Slope: The slope value come within a certain range which depends upon the adsorption characteristics of the catalyst material and the experimental conditions at which experiments were performed. Generally, higher slopes indicate more favorable adsorption behavior. The slope value of 863.102 1/g suggests a reasonable and indication of a material with viable adsorption capacity.

Intercept: The intercept value tells us the amount of adsorbate adsorbed at low relative pressures (approx close to zero). It has to be a positive value, but mainly its magnitude depends on conditions such as the surface properties of the material and the adsorbate we have used. The calculated intercept value of 8.019e+01 1/g comes to be within an acceptable range.

Correlation Coefficient (R^2): A significantly high correlation coefficient value which was found to be 0.999564 tells us that there is a strong linear relationship between the relative pressure and the quantity of adsorbate. This shows that the data fits well to the BET model.

Constant C: The role of constant C in the BET equation is to transform the experimental data into surface area values. Its results depends on the specific adsorption procedure and experimental conditions. The calculated value of 11.763 comes under a typical range for this constant.

BJH Pore Size Distribution Desorption Summary

Surface Area = 7.176 m²/g

Pore Volume = 0.008 cc/g

Pore Diameter Dv(d) = 2.120 nm

5.6 CATALYST REUSABILITY

Fuller's Earth catalysts can be reused several times depending on the reaction type, catalyst stability, and catalyst recovery method, among other variables. Depending upon microwave assisted reaction and filtration we were able to recover a significant amount of catalyst for approx 2 grams. The reusability of a catalyst can be cost-effective and sustainable solution. Fuller's Earth catalysts are very promising for producing biodiesel because they can be reused, which offers aid to financial and environment factors. Fuller's earth can be very useful for bulk production because it can maximize production efficiency and reduce costs by utilizing Fuller's Earth's long-lasting and reuseability catalytic characteristics by adhering to strict regeneration procedures and keeping performance in check. The long use of Fuller's Earth catalysts can be extended by regular optimization efforts and following maintenance procedures, guaranteeing its continuous contribution to environmentally friendly biodiesel production methods.



Fig 5.6 Recovered Catalyst

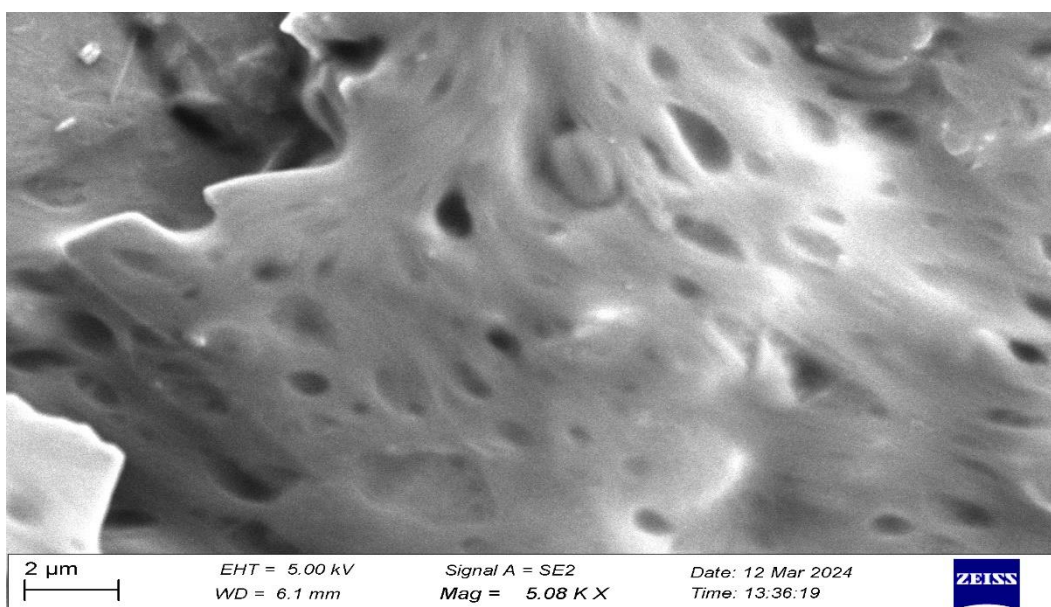
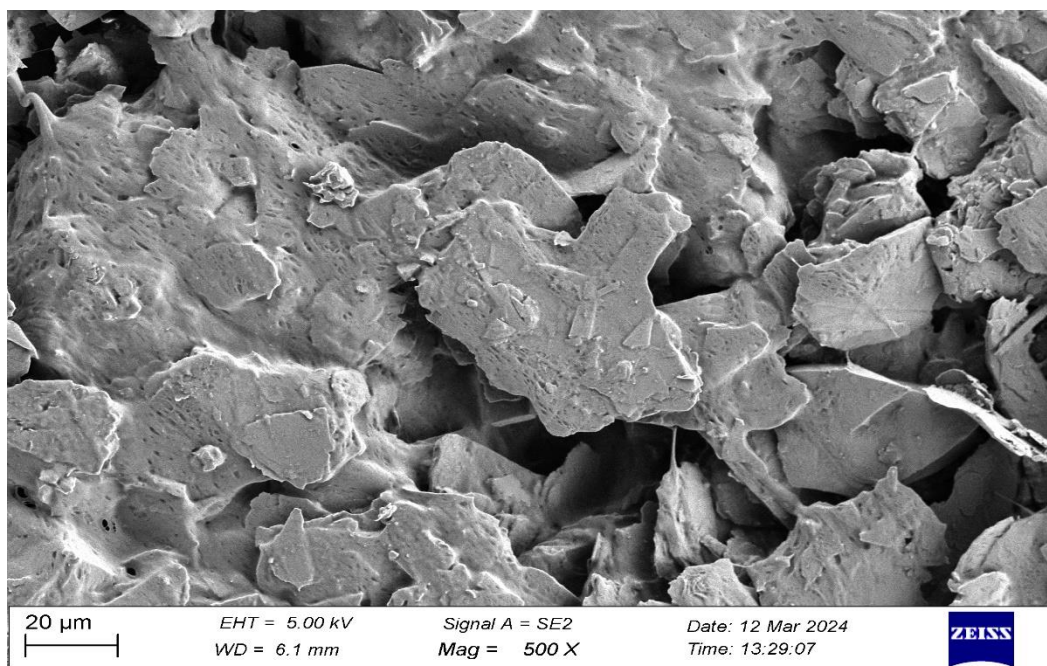


Fig 5.7 SEM images for recovered catalyst

The catalyst's surface morphology, structural integrity, and deposition of impurities or reaction byproducts can all be examined using after-use SEM analysis. After using a catalyst, SEM pictures have shown changes to the surface topography of the catalyst, such as the existence of pores or surface roughness due to catalytic reactions and regeneration processes. SEM analysis can also help assist in finding contaminants or organic residues on the catalyst's surface, which will affect its catalytic activity and require renewal.

5.7 GAS CHROMATOGRAPHY FLAME IONIZATION DETECTION(GCFID)FOR BIODIESEL

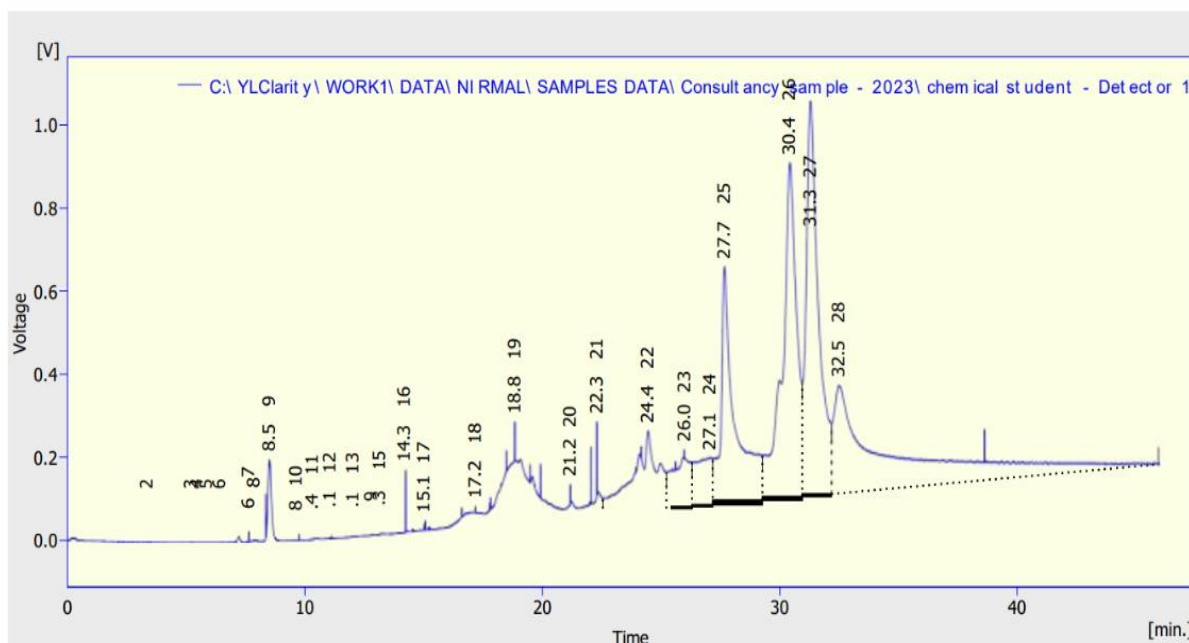


Fig 5.8 GCFID graph for Biodiesel

GC-FID is a technology that works well for assessing biodiesel samples since it can be used to extract and quantify specific components in a complicated mixture: The different elements contained in the biodiesel sample are separated and quantified using GC-FID. The presence of methyl esters, which are the main ingredients of biodiesel, is indicated in this instance. It allows the production and purity of biodiesel to be determined by quantifying the amount of methyl esters. This data is essential for evaluating the biodiesel's quality and appropriateness as a fuel. In order to make sure the biodiesel satisfies quality requirements, GC-FID analysis may also identify any contaminants or undesirable molecules that may have developed during the transesterification procedure.

The steps that were involved in Gas Chromatography with Flame Ionization Detection (GC-FID) included sample preparation, instrument setup, and GC column installation. During chromatographic runs, samples were injected and sorted according to their affinity for the stationary phase. Organic chemicals that elute from the column were burned in flame ionization detection (FID), creating ions and a current proportional to their concentration. Compounds were then identified and quantified by acquiring and analyzing data. Quantification were aided by calibration curves that are derived from standards. Reports are created from the results,

analyzing the substances and concentrations found. Instrument performance is guaranteed by maintenance chores.

In this analysis, the results are presented in terms of retention time, area, and height for different compounds, along with their percentages. Here's an explanation of the observed results:-

- Heptadecanoic Acid, Methyl Ester (Compound 1): This compound is detected at a retention time of 10.427 minutes with an area of 33.185 mV.s. It makes up a small fraction of the total, with a percentage of 2.370%.
- Stearic Acid, Elaidic Acid, Methyl Ester (Compound 2): Detected at a retention time of 11.113 minutes with an area of 2.578 mV.s. It also constitutes a small portion of the sample, with a percentage of 6.333%.
- Linolenic Acid, Methyl Ester (Compounds 3 and 4): Two peaks at retention times 12.113 and 12.903 minutes correspond to this compound. They have relatively small areas and percentages (1.448% and 1.594%, respectively).
- Arachidic Acid, Methyl Ester (Compound 5): Detected at 13.260 minutes with an area of 43.124 mV.s. It represents a small portion of the sample, with a percentage of 2.620%.
- cis-8,11,14-Eicosatrienoic Acid, Methyl Ester (Compound 6): This compound shows a significantly larger area of 72.479 mV.s, representing 150.462% of the total. This may be due to an anomaly in the analysis or the presence of an abundant compound.
- cis-5,8,11,14,17-Eicosapentaenoic Acid, Methyl Ester (Compound 7): Detected at 15.077 minutes with an area of 113.770 mV.s. It constitutes a small fraction of the sample, with a percentage of 24.376%.
- Lignoceric Acid, Methyl Ester (Compound 8): This compound is detected at 17.187 minutes with a notably large area of 2407.856 mV.s, accounting for 48.227% of the total.
- cis-4,7,10,13,16,19-Docosahexaenoic Acid, Methyl Ester (Compound 9): Detected at 18.843 minutes with a substantial area of 15627.608 mV.s, representing 242.453% of the sample. This high percentage may indicate the presence of an abundant compound.

- Capric Acid, Methyl Ester (Compound 10): Detected at 21.183 minutes with an area of 1108.170 mV.s. It makes up a relatively small percentage of 79.701%.
- Undecanoic Acid, Methyl Ester (Compound 11): Detected at 22.283 minutes with an area of 2123.266 mV.s. It represents 224.992% of the total.
- Lauric Acid, Methyl Ester (Compound 12): Detected at 24.447 minutes with a substantial area of 13972.118 mV.s, accounting for 191.898% of the sample.
- Tridecanoic Acid, Methyl Ester (Compound 13): Detected at 25.973 minutes with an area of 6531.097 mV.s. It constitutes 138.992% of the total.
- Myristic Acid, Methyl Ester (Compound 14): Detected at 27.067 minutes with an area of 5735.785 mV.s, representing 113.638% of the sample.
- Myristoleic Acid, Methyl Ester (Compound 15): Detected at 27.663 minutes with a substantial area of 23536.503 mV.s, accounting for 570.606% of the total.
- Pentadecanoic Acid, Methyl Ester (Compounds 16 and 17): Two peaks at retention times 30.420 and 31.277 minutes correspond to this compound. They have significantly large areas and percentages (806.432% and 951.237%, respectively).
- cis-10-Pentadecenoic Acid, Methyl Ester (Compound 18): Detected at 32.477 minutes with an area of 43771.535 mV.s, representing 259.254% of the sample.

The total area for all compounds is 186004.728 mV.s, and the total height is 4064.804 mV. These results provide a detailed breakdown of the compounds present in the analyzed sample, with some compounds exhibiting notably high percentages.

5.8 GAS CHROMATOGRAPHY MASS SPECTROSCOPY FOR BIODIESEL

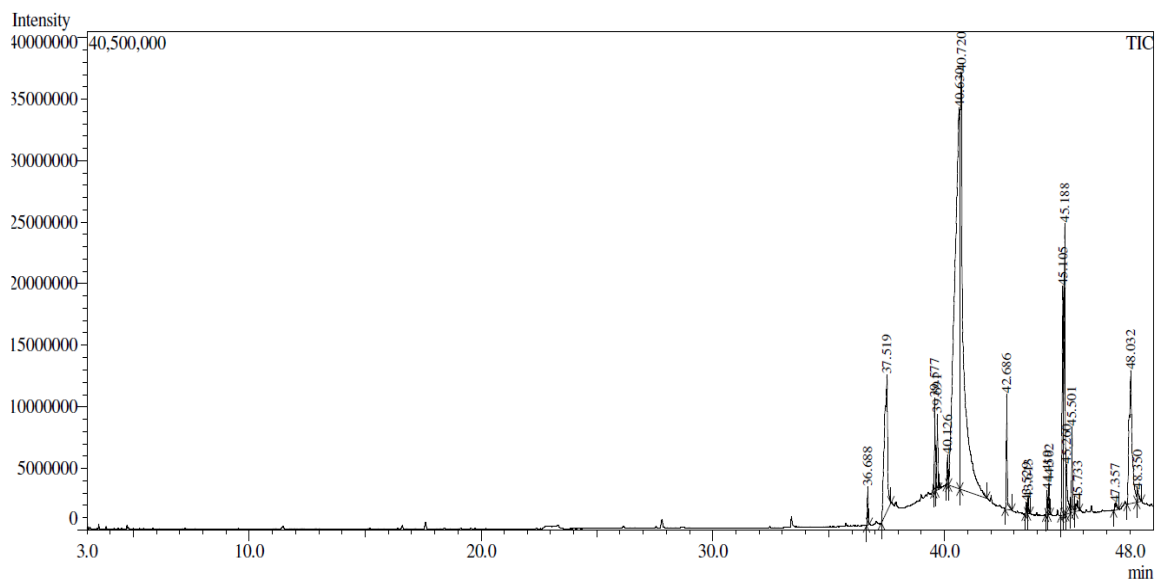


Fig 5.9 GCMS graph for biodiesel

Table 5.2: GCMS graph interpretation table

S.No	Compound Composition	Compound Name	Category
1	0.53	Palmitic acid,Methyl ester	Saturated
2	8.08	Linoleic acid,Methyl ester	Saturated
3	1.4	9,12-octadecadienoic acid,Methyl ester	poly-unsaturated
4	1.41	9-octadecenoic acid,Methyl ester	mono-unsaturated
5	0.43	Heptadecanoic acid,Methyl ester	Saturated
6	34.68	9,12-octadecadienoic acid,Methyl ester	poly-unsaturated
7	28.05	9-octadecenoic acid	mono-unsaturated
8	1.99	Hexadecanoic acid,2-Hydroxy,3 Propanediyl ester	Saturated
9	0.18	6,9-Octadecadienoic acid,Methyl ester	poly-unsaturated
10	0.24	9-octadecenamide	mono-unsaturated
11	0.38	6,9-Octadecadienoic acid,Methyl ester	poly-unsaturated
12	0.48	9-octadecenoic acid,1,2,3-propanetriyl ester	Saturated
13	5.09	9,12,15-octadecatrienoic acid, methyl acid	mono-unsaturated
14	5.85	9-octadecenoic acid,2,3-dihydroxypropyl ester	poly-unsaturated
15	1.42	14-methyl-8-Hexadecyn-1-ol	mono-unsaturated
16	1.41	Hexadecanoic acid,3 Propanediyl ester	Saturated
17	0.4	14-methyl-8-Hexadecyn-1-ol	Saturated
18	0.23	Linalol oxide,trimethylsiyl ester	-----
19	7.39	9-octadecenoic acid,9-Hexadecenyl ester	Saturated
20	0.36	Octadecanoic acid,2,3-dihydroxypropyl ester	Saturated

This gas chromatography-mass spectrometry (GCMS) analysis of my biodiesel sample indicates multiple composition of Fatty Acid Methyl Esters (FAMES) and other compounds.

The compounds which were present in ample quantity were saturated fatty acids like Palmitic acid ,methyl ester, Linoleic acid , Heptadecanoic acid, and Hexadecanoic acid. These compounds help in maintaining the stability and viscosity of the biodiesel.

The methyl esters that was found in the biodiesel sample are of great importance due to their crucial roles in biodiesel quality, it's performance, and environment impact it will cause. These esters make up the bulk of biodiesel composition, and impact crucial fuel properties such as viscosity, combustion characteristics, and engine compatibility. These methyl esters found in the sample also indicates that the given sample is biodiesel confirmed.

Besides from methyl esters, polyunsaturated fatty acids as 9,12-Octadecadienoic acid and 6,9-octadecadienoic acid were also found in the test, providing detailing into the oxidative stability potential and cold flow properties of the biodiesel.

Mono-unsaturated fatty acids, along with 9-octadecenoic and 9-octadecenamide, were found in considerable proportions, influencing the biodiesel's combustion characteristics and oxidative stability.

More importantly, minor components such as 9-octadecenoic acid, 2,3-dihydroxypropyl ester, and 14-methyl-8-hexadecyn-1-ol make up and form the overall composition, which also affects the biodiesel's lubricating potential and thermal stability.

Also, the compounds present like hexadecanoic acid, 2-hydroxy, 3-propanediyl ester, and linalol oxide, trimethylsilyl ester shows the occurrence of esterification reaction and oxidation reaction during the synthesis of biodiesel.

Therefore, the GCMS analysis provides a detailed information into the chemical constituents that make up the biodiesel, it also helps in understanding the performance and quality of biodiesel, moreover it shows the potential of biodiesel for use in energy sector as a green source of energy.

5.9 PROCESS ANALYSIS WITH RSM

A statistical method for improving processes such as the manufacture of biodiesel is called response surface methodology, or RSM. It involves developing tests, usually using a Central Composite Design (CCD), to collect information on the effects of input variables (such as temperature and catalyst concentration) on response variables (such as yield and quality). By fitting a model to this data, design expert software makes it possible to forecast responses for other component combinations. Optimization, which frequently makes use of numerical methods or response surface visualization, seeks to identify circumstances that maximize yield or quality. By methodically determining the ideal circumstances, RSM improves the production of biodiesel and eventually lowers costs by increasing yields, improving quality, and increasing resource efficiency.

5.9.1 Analysis of Variance

Response Surface Methodology (RSM)'s ANOVA (Analysis of Variance) assesses the importance of variables affecting biodiesel output or quality in the context of biodiesel production. ANOVA evaluates the effects of each component and their interactions on the generation of biodiesel by planning experiments and gathering data on variables such as temperature, catalyst concentration, and reaction time. It directs process optimization by assisting in the identification of important variables influencing yield or quality. ANOVA in RSM assesses how well regression models fit the experimental data to ascertain whether the model can account for variance in the response variable. In the end, ANOVA helps researchers identify crucial variables for increasing output, improving productivity, and lowering expenses in the procedures involved in producing biodiesel.

Table 5.3 RSM response value.

Response 1: Biodiesel Yield

Source	Sum of squares	df	Mean Square	F-value	P-value	
Model	323.77	14	23.13	3.35	0.0153	significant
A-Alcohol to oil molar ratio	0.333	1	0.333	0.0483	0.8292	
B-Catalyst wt%	3.00	1	3.00	0.4349	0.5203	
C-Microwave Power	0.3333	1	0.3333	0.0483	0.8292	
D-Time	5.33	1	5.33	0.7731	0.3941	
AB	6.25	1	6.25	0.9060	0.3573	
AC	9.00	1	9.00	1.30	0.2725	
AD	0.2500	1	0.2500	0.0362	0.8518	
BC	0.2500	1	0.2500	0.0362	0.8518	
BD	1.00	1	1.00	0.1450	0.7091	
CD	0.2500	1	0.2500	0.0362	0.8518	
A ²	141.67	1	141.67	20.54	0.0005	
B ²	190.78	1	190.78	27.66	0.0001	
C ²	55.43	1	55.43	8.04	0.0132	
D ²	46.36	1	46.36	6.72	0.0213	
Residual	96.58	14	6.90			
Lack of Fit	96.33	10	9.63	155.38	<0.0001	significant

Pure Error	0.2480	4	0.0620			
Cor Total	420.35	28				

Factor coding is **Coded**.

Sum of squares is **Type III - Partial**

The **Model F-value** of 3.35 implies the model is significant. There is only a 1.53% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A², B², C², D² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 155.38 implies the Lack of Fit is significant. There is only a 0.01% chance that a Lack of Fit F-value this large could occur due to noise. Significant lack of fit is bad -- we want the model to fit.

Table 5.4 Fit Statistics

Std Dev.	2.63	R²	0.9702
Mean	85.69	Adjusted R²	0.9545
C.V.%	3.07	Predicted R²	0.9321

A negative **Predicted R²** implies that the overall mean may be a better predictor of your response than the current model. In some cases, a higher order model may also predict better.

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 6.183 indicates an adequate signal. This model can be used to navigate the design space.

5.9.2 Final Equation In Terms of Actual Factors

$$\text{Biodiesel Yield} = -430.50 + 27.90111A + 57.38667B + 0.062481C + 0.176370D - 0.416667AB + 0.005556AC + 0.000926AD - 0.002778BC - 0.005556BD - 0.000031CD - 0.519259A^2 - 5.42333B^2 - 0.000361C^2 - 0.000330D^2$$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

5.9.3 Validation of Model

It is essential to distinguish between projected (anticipated) and actual responses when using Response Surface Methodology (RSM). Statistical models predict the response, which informs the best values for the input variables. As this is going on, real answers from trials help to develop the model and confirm its predictions. Confirmation of the model's predictive power is achieved by doing validation trials at software-proposed optimal parameters. When the findings of these trials match the predicted reactions, the model's predictions are likely accurate and resilient. By improving decision-making and problem-solving skills, this validation procedure guarantees dependability when using the model in real-world situations. Thus, in addition to streamlining procedures, RSM's dual emphasis on anticipated and actual

reactions also strengthens the statistical rigor necessary for successful testing and analysis.

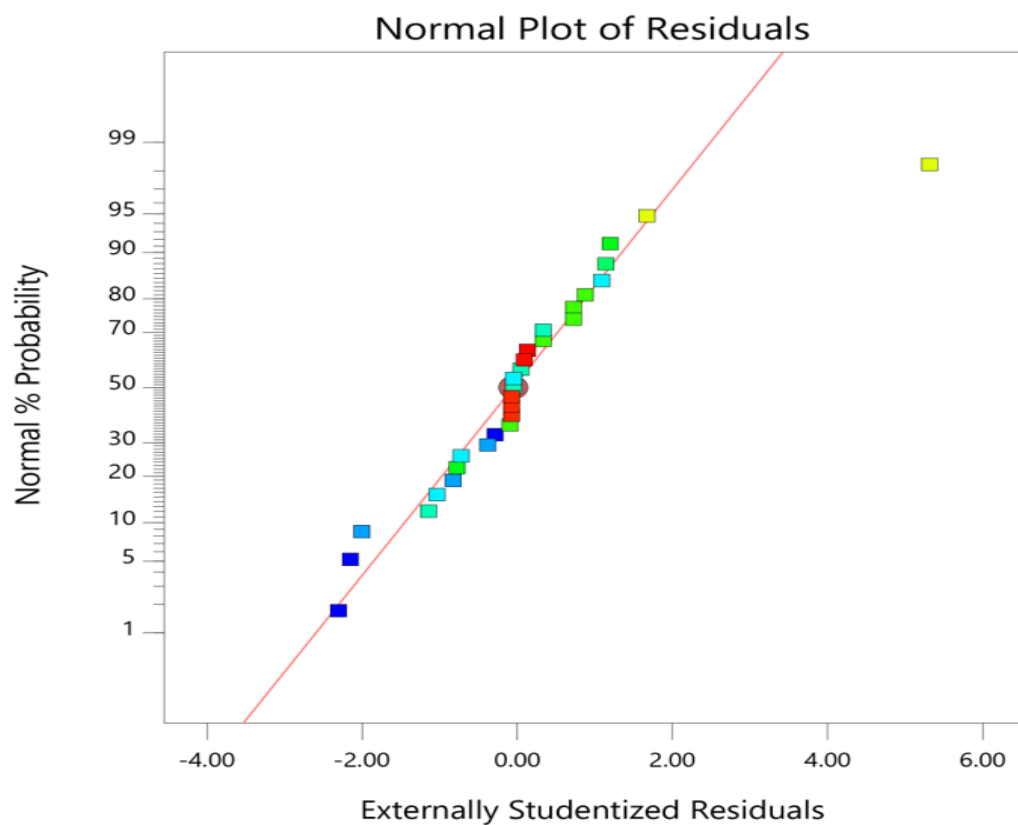


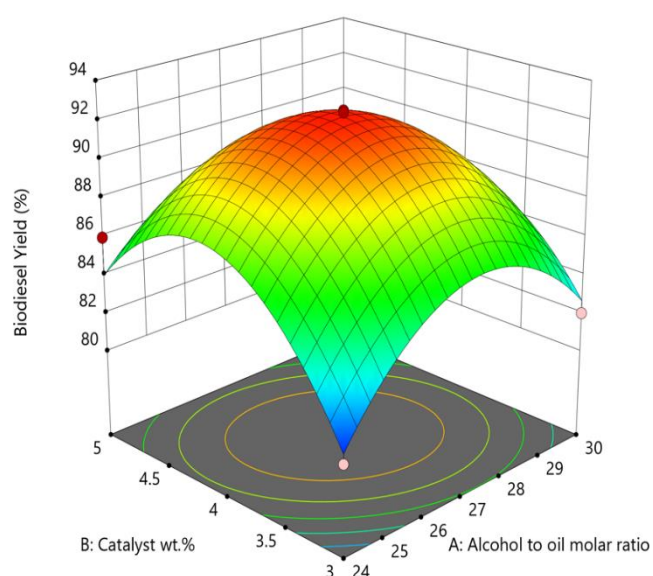
Fig 5.10 Validation of Model

Table 5.5 Validation of Results

Actual value	Predicted value	Standard order
80.00	83.25	13
92.00	92.18	28
82.00	85.08	10
86.00	87.33	6
84.00	84.08	11
82.00	82.67	2
83.00	83.08	19
92.00	92.18	29
85.00	83.08	18
87.00	86.42	17
89.00	86.33	7
83.00	84.25	12
92.00	92.18	25
87.00	85.50	8
83.00	81.17	4
83.00	84.75	14
86.00	84.00	3
87.00	85.75	20
92.50	92.18	26
84.00	83.42	23
82.00	83.42	15
89.00	83.75	21
84.00	85.92	9
80.00	83.42	24
92.40	92.18	27
87.00	87.17	5
87.00	85.75	22
80.00	80.50	1
84.00	83.92	16

5.9.4 3-D Surface Plots

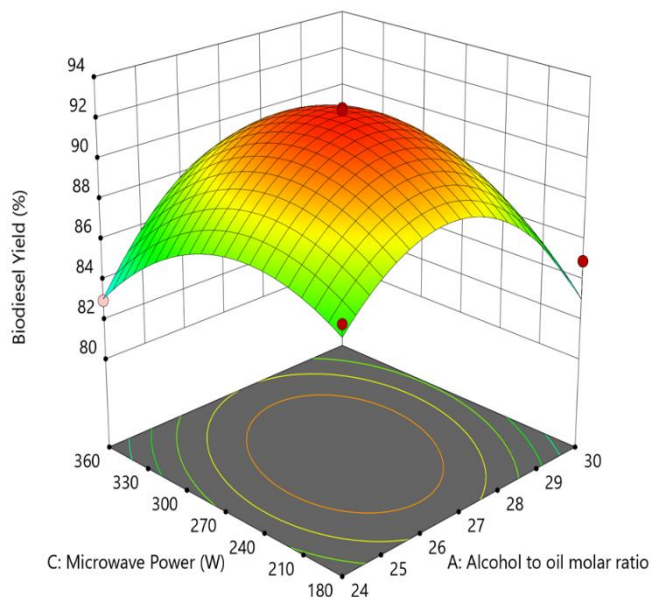
When displaying the findings of Response Surface Methodology (RSM) experiments with several input variables, three-dimensional (3-D) graphs are essential. When the number of input variables exceeds two, 3-D graphs show the response variable (biodiesel yield, for instance) as a function of the first two input variables, with the vertical axis representing the third input variable. These graphs give researchers a thorough understanding of how the response variable varies with respect to various combinations of input factors, making it possible to pinpoint ideal circumstances. Researchers may better understand the intricate linkages and interactions among the input elements by visualizing the response surface in three dimensions. This helps to optimize the processes involved in producing biodiesel, resulting in increased output and efficiency.



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = A: Alcohol to oil molar ratio
 X2 = B: Catalyst wt. %

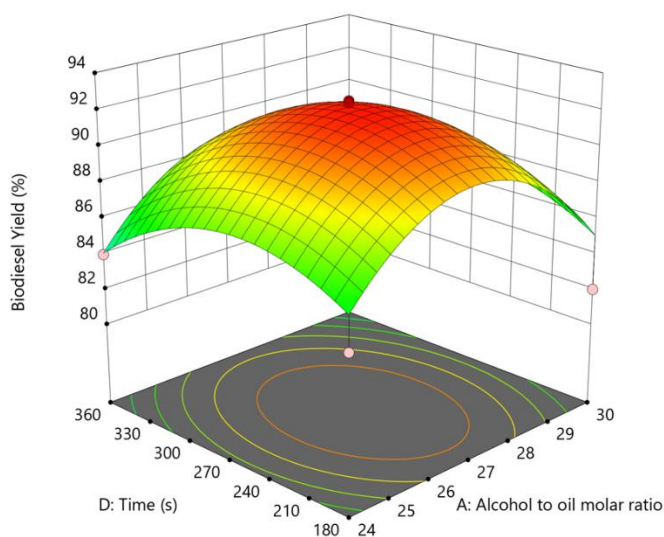
Actual Factors
 C: Microwave Power = 270
 D: Time = 270



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = A: Alcohol to oil molar ratio
 X2 = C: Microwave Power

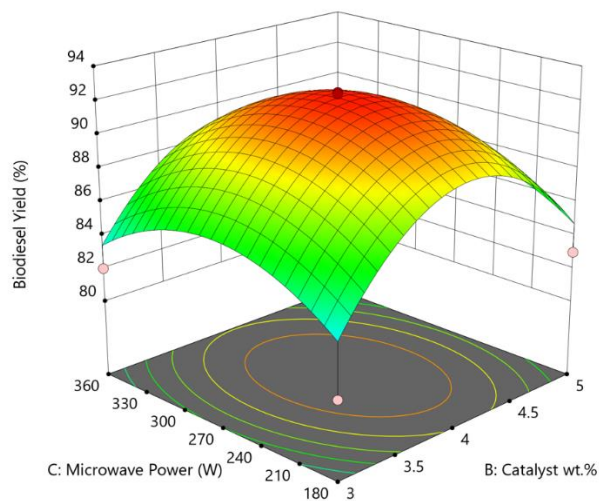
Actual Factors
 B: Catalyst wt. % = 4
 D: Time = 270



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = A: Alcohol to oil molar ratio
 X2 = D: Time

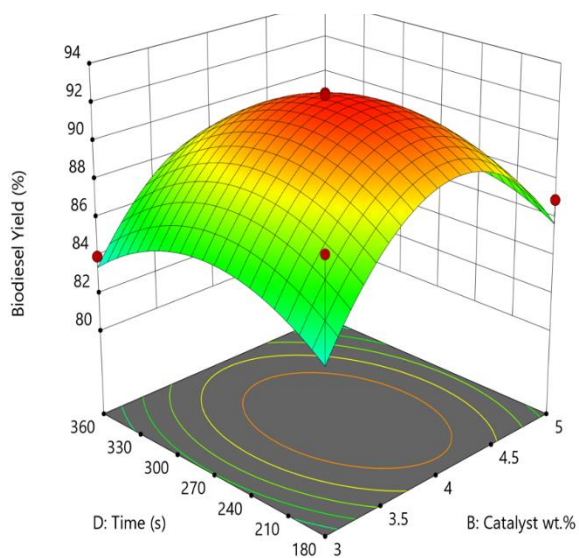
Actual Factors
 B: Catalyst wt.% = 4
 C: Microwave Power = 270



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = B: Catalyst wt.%
 X2 = C: Microwave Power

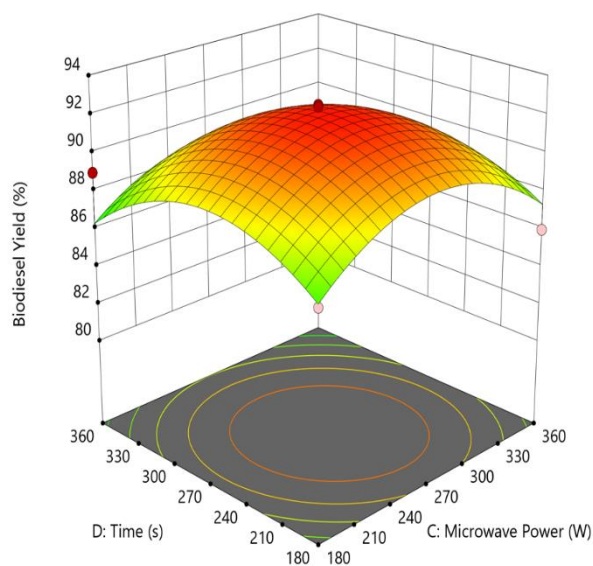
Actual Factors
 A: Alcohol to oil molar ratio = 27
 D: Time = 270



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = B: Catalyst wt.%
 X2 = D: Time

Actual Factors
 A: Alcohol to oil molar ratio = 27
 C: Microwave Power = 270



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = C: Microwave Power
 X2 = D: Time

Actual Factors
 A: Alcohol to oil molar ratio = 27
 B: Catalyst wt.% = 4

Fig 5.11- 3-D Surface Plots

5.10 PROPERTIES OF BIODIESEL

5.10.1 Density

Mass of biodiesel per unit volume is expressed as density (g/cc). Biodiesel has a density of 0.863 g/cc, which is lower than that of water, which normally has a density of 1 g/cc. Reduced density suggests that biodiesel is simpler to handle and carry since it is lighter and less dense than water. Density can also impact engine emissions performance and combustion efficiency; for example, biodiesel with a lower density may burn and atomize fuel more effectively.

5.10.2 Viscosity

Viscosity evaluates a fluid's resistance to flowing. Biodiesel flows more readily than fluids with a greater viscosity, as seen by its comparatively low viscosity of 2.22 mm²/s. Biodiesel with a lower viscosity is preferred because it improves fuel atomization and mixing in engines, resulting in more effective combustion and less wear on the engines. Additionally, it guarantees simpler biodiesel handling and pumping, particularly in colder climates.

5.10.3 Calorific Value

Calorific value, often referred to as heating value or energy content, is the quantity of energy produced upon full combustion of one unit mass of biodiesel. With a high calorific value of 37.3 MJ/kg, burning biodiesel releases a sizable quantity of energy per unit mass. Biodiesel with a higher calorific value produces more energy, which boosts engine power and fuel economy.

5.10.4 Flash Point

The lowest temperature at which biodiesel releases sufficient vapor to catch fire when an ignition source is present is known as the flash point. Biodiesel with a flash point of 168°C is considered to have a moderately high flash point, which is advantageous for handling, storage, and transportation safety. Biodiesel with a higher flash point lowers the possibility of fire risks and unintentional ignition.

5.10.5 Fire Point

The temperature at which biodiesel may continue to burn after it is ignited is known as the "fire point." Biodiesel has a fire point of 188°C, meaning it can burn at

temperatures higher than its flash point. An extra safety margin offered by a higher fire point lowers the possibility of fire dangers during handling and use.

5.10.6 Pour Point

The lowest temperature at which biodiesel can still flow and remain fluid is known as the pour point. Biodiesel can still flow at relatively low temperatures, as indicated by its pour point of 3°C, which is advantageous for its use in cold climes. Lower pour point biodiesel ensures continued fuel flow and engine performance even in subfreezing temperatures since it is less prone to gel or freeze in cold weather.

Table 5.6 Properties of Biodiesel

Property	Biodiesel	Standard value as per ASTM Std
Density (g/cc)	863	860-900
Viscosity (mm ² /s)	2.22	1.9-6.0
Calorific value (MJ/Kg)	37.3	35-40
Flash point(°C)	168	60-190
Fire point(°C)	188	93-180
Pour point(°C)	3	-5 - 1

All in all, these attributes point to a high-quality biodiesel that has the qualities that are needed for safe operation of engines, good quality, and adaptability to different environmental settings.

6.CONCLUSION

The biodiesel synthesis from silk-cotton seed oil, methanol, and a Fuller's Earth catalyst represents an efficient and innovative process for producing a sustainable alternative fuel. The step-by-step procedure, which incorporates microwave irradiation and subsequent testing, demonstrates the viability of this method for biodiesel production.

The catalyst preparation involved meticulous steps, from mixing Fuller's Earth and NaOH, to sonication and drying. These steps ensure the catalyst's optimal performance in promoting the transesterification reaction. The resulting biodiesel product exhibited yield of 67% and the conditions were further optimized through RSM to get a higher yield, which meets key quality criteria.

The analysis of both the catalyst and biodiesel through modern testing techniques confirmed the presence of essential chemical groups and methyl esters in the biodiesel, validating its suitability as a fuel. These analytical techniques provide insights into the chemical composition and purity of the catalyst and biodiesel.

In summary, this biodiesel synthesis method, combining microwave irradiation, catalyst preparation, and rigorous testing, offers a rapid and efficient approach to biodiesel production. It underscores the importance of catalyst preparation, quality testing, and process optimization to ensure the viability and sustainability of biodiesel as a renewable fuel source. This research and methodology contribute to the ongoing pursuit of cleaner and more sustainable energy alternatives, decreasing our dependence on fossil fuels and mitigating environment impact.

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ABSTRACT

The global demand for clean and sustainable energy sources has driven extensive research into renewable fuels, with biodiesel emerging as a promising alternative to traditional fossil fuels. This paper delivers the study of production of biodiesel through Transesterification reaction using silk-cotton seed oil, fuller's earth catalyst and methanol with the help of microwave. Use silk cotton seed oil for biodiesel could promote sustainable agriculture and reduce dependence on traditional fossil fuels. Silk cotton seed oil is not mainly used for food production so using it for biodiesel would not compete directly with food crops potentially decreasing concerns about food security. Biodiesel produced from this feedstock is biodegradable, which can be beneficial in decreasing the environment impact in case of spills or leaks. The silk-cotton seed oil has a free fatty acid content of 6.87% and the density of silk-cotton seed oil was found to be 0.91g/cm³. Fuller's earth has been chosen as a catalyst in this project, it is also the novelty of this project. The fuller's earth catalyst was characterized using modern techniques such as Fourier Transform-Infrared Spectroscopy (FT-IR), which indicated presence of essential bonds. FESEM & EDX which confirmed the presence of active sites and elemental composition. From BET analysis the surface area was found to be 7.176 m²/g and pore volume was 0.008 cc/g. The biodiesel was hence obtained with a yield of 67%. The biodiesel obtained characterized using Gas chromatography-Flame Ionization Detection (GC-FID) and Gas chromatography Mass Spectrometry (GC-MS) indicating presence of the Fatty Acid Methyl Ester (FAME). Following this result RSM was conducted for optimization and optimum condition was found. After the property test density was 863g/cc, Viscosity was 2.22 mm²/s. Overall this paper contributes to the ongoing efforts to develop sustainable and economically viable biodiesel production processes.

1. INTRODUCTION

The worldwide chase of sustainable and environment friendly source of energy has lead to deep and detailed research and innovation dedicated in the area of biofuels. Among all the biofuels, biodiesel has shown a promising alternate to crude fossil fuels, which offers a clean and more prolonged sustainable energy source(Rahul Soosai *et al.*, 2022). Biodiesel manufactured using a reversible process named as transesterification. In process triglycerides from renewable sources like oils are transformed into biodiesel and glycerol, with the little help of a catalyst. While we choose the catalyst for production of biodiesel we should always go for heterogenous catalyst as it is the best option in order to use the catalyst again and again so that it will positively impact the economy(Chouhan and Sarma, 2011) . This project provides detailed insight on the manufacturing of biodiesel by using silk-cotton seed oil for renewable feedstock, methanol for the alcohol component, and Fuller's Earth as catalyst for accelerating the reaction. This very unique reaction mixture not only focuses on the issue of the need for renewable energy option but also discovers the capability of not very conventional feedstock and innovative catalysts to make the biodiesel production process more convinient and a lot more easier. Moreover, the project focuses on advanced and unique methodology such as microwave irradiation, which help increasing reaction speed for the transesterification reaction and contributes to making a more efficient process. The successful ²⁵ production of biodiesel from silk-cotton seed oil shows a promising sustainable and eco-friendly energy source, which reduces the human impact on precious environment related with traditional fossil fuels(Rahul Soosai *et al.*, 2022). Futhermore, the innovative use of Fuller's Earth catalyst, together with other ⁶ efficient analytical techniques, which increases the quality and efficiency of the biodiesel production process. By going through some rigorous tests and analysis,our project ensures that the biodiesel we have produced meets the necessary quality standard and is viable for use as a biofuel.

1.1 Biodiesel

Biodiesel is a renewable and sustainable alternate option for use in place of conventional fossil diesel fuel (Okoduwa *et al.*, 2024). It is produced from multiple feedstocks, which includes vegetable oils, animal fats, and even waste oil left after cooking (Novita *et al.*, 2024). It is mainly from a chemical process known as transesterification, where triglycerides present in different feedstocks are transformed to fatty acid methyl esters (FAME) (Balat and Balat, 2010). One feedstock with notable potential is silk-cotton seed oil, which is formed from seeds of the silk-cotton plant (*Ceiba pentandra*). Biodiesel synthesis from silk-cotton seed oil undergoes a transesterification process where triglycerides present in the oil are transformed to fatty acid methyl esters (FAME) by the use of methanol and a catalyst, such as Fuller's Earth.

1.1.1 Advantages of Biodiesel

Biodiesel, as a renewable and sustainable energy source alternative to traditional fossil fuels. Some of the key benefits of biodiesel include:-

- **Renewable feedstock:** Biodiesel synthesis from renewable source such as vegetable oils, animal fats, and also algae. This can be used in place of finite fossil fuel resources, making biodiesel a sustainable and clean and green energy option (Uttamrao, 2018).
- **Lowered Greenhouse Gas Emissions:** Biodiesel generally has lower carbon emissions in comparison to fossil diesel. It can notably reduce greenhouse gas emission including CO₂ and particulate matter
- **Biodegradable and Non-Toxic:** Biodiesel decomposes at a faster rate, it has a less environment impact as compared to conventional fuel in the factors of spills or leaks. Moreover, it is safer for the environment than traditional diesel because it is less toxic.
- **Better engine performance:** Due to Biodiesel being more lubricious as compared to conventional diesel fuel, it can keep engines running efficiently and for long-term.

- Availability: A large no of local resources can be used to produce biodiesel, decreasing dependency on imported oil, and increasing energy security.

1.1.2 Disadvantages of Biodiesel

Biodiesel, inspite of its many advantages, it also comes with certain disadvantages that should be :-

- Feedstock Competition: Biodiesel production competes on land and resources with food and also feed production, which raises concerns about food security and land use conflicts(Li, Chen and Nie, 2023).
- Limited Feedstock Availability: The availability of viable feedstocks for biodiesel manufacturing can be restricted by factors such as climate, soil conditions, and land available.
- Energy Consumption: The procedure of growing feedstock crops and transforming into biodiesel can be energy-intensive,, offsetting of some of the environment advantages(Falowo *et al.*, 2021).
- Unsuitable Weather Performance: Biodiesel could have issues with cold weather performance, which includes potential gelling or solidification at lower temperatures, which can possibly affect engine operation.
- Variance in Quality: The quality of biodiesel can be differentiated depending upon the feedstock source and production methodology, that requires careful quality control.

1.2 Transesterification

Transesterification is traditionally used chemical process at the heart of biodiesel production. It is a unique reaction that transforms triglycerides , mainly to be found among ³⁷vegetable oils and animal fats, to fatty acid methyl esters (FAME), which makes up the core of biodiesel. The transesterification process can be carried out in a variety of ways, such as microwave, ultrasonic, and conventional heating(Bizualem and Nurie, 2024). Since ultrasonic heating converts electrical energy into heat energy, it functions similarly to microwave heating(Mohd Johari *et al.*, 2024).The transesterification process includes the breaking of chemical bonds in the triglycerides, in which three fatty acids are attached to a glycerol molecule. In presence of an alcohol, mainly methanol or ethanol, and a catalyst, such as our Fuller's Earth, these bonds are cleaved, leading to glycerol formation as a

byproduct and the creation of FAME molecules. The result is a cleaner-energy fuel that shows properties familiar to diesel but with lowered emissions of greenhouse gases, sulfur compounds, and particulate matter. Transesterification not only crucial for converting renewable feedstocks into a practical energy source but also plays a crucial role in decreasing the environment impact of transportation and promoting sustainable energy solutions.

1.3 Silk-Cotton seed Oil

Silk-cotton seed oil is the primary raw material used by us in production of biodiesel. Made from seeds of the silk-cotton tree (*Ceiba pentandra*), this oil serves as the source of triglycerides we need for the transesterification process (Muhammad, 2018).

The oil constitutes a mixture of triglycerides, which are made of fatty acid chains which are bonded to a glycerol compound. Triglycerides like these are the raw materials that undergo transesterification, a chemical reaction where the glycerol is replaced with methanol (or another alcohol), resulting in the production of methyl esters of fatty acid, which make up biodiesel.

The use of silk-cotton seed oil as a feedstock is significant for biodiesel production. It is considered a non-food feedstock, i.e., it doesn't compete with food production and also concerns related to the diversion of food crops for fuel synthesis. This feature contributes to sustainability and reduce the ethical and economic dilemmas related with food-based biodiesel feedstocks (Muhammad, 2018).

Moreover, the chemical composition of oil silk-cotton seed, including the types and amount of fatty acids present, it can vary depending on factors like the geographical origin of the tree from which silk-cotton seed is obtained and the oil extraction procedures. Understanding and optimizing the use of silk-cotton seed oil is essential for achieving the desired biodiesel yield percentage, quality, and overall process efficiency (Oil, 2020).

1.4 Fuller's Earth

Fuller's Earth is a naturally found in nature, it appears to be a clay-like substance known for its advanced adsorbent properties and multiple industrial applications. It

is mainly made up of hydrated aluminum silicates and differs in mineral compositions, depending on its source (Rekunge, Indalkar and Chaturbhuj, 2016). In the reference of biodiesel production, Fuller's Earth can be used as a catalyst for the transesterification process in the following ways:-

- Fuller's Earth is viable as a catalyst in the reaction of transesterification, a crucial step in biodiesel synthesis. It plays a most important part in the transformation of triglycerides (which is found in plant oils or animal fats) into fatty acid methyl esters (FAME), which make up biodiesel. Fuller's Earth's catalytic abilities arise from its surface properties, which provides significant no. of active sites in order to carry out transesterification process (Ulakpa *et al.*, 2022).
- In the presence of methanol or other alcohol, Fuller's Earth increases the reaction kinetics by decreasing the activation energy required for the reaction. It functions by breaking down the chemical bonds present in the triglycerides, efficiently splitting them into glycerol and methyl esters. This process allows for the transformation of the feedstock, like silk-cotton seed oil, into biodiesel.
- The advantage of Fuller's Earth use as catalyst in biodiesel synthesis includes its low price, natural abundance, and also the fact that it can be regenerated and reused again and again multiple times. These characteristics make Fuller's Earth a most viable and sustainable choice for biodiesel manufacturers. However, the efficiency of Fuller's Earth depends upon factors such as the quality of the feedstock, conditions of the reaction, and the category of Fuller's Earth used, making optimization and fine-tuning essential to achieve the desired biodiesel yield and quality (Banga and Pathak, 2023).

1.5 Properties of Biodiesel

Biodiesel, a renewable and sustainable substitute for traditional diesel fuel and a clean source of energy, shows very distinctive properties that affect its suitability for use in diesel engines (Ude *et al.*, 2022). Here are the basic characteristics of biodiesel:-

- Viscosity: Biodiesel generally have a higher viscosity, or thickness, if compared to conventional diesel. This influences fuel atomization and combustion in engines.
- Cetane Number: Biodiesel mostly has a higher cetane number, which signifies better ignition quality and a shorter delay in ignition. These characteristics leads to improved engine performance and reduced emissions.
- Density: Biodiesel's density is one higher than that of traditional diesel, although the difference is not of any major importance. This deflection in density has minimal affect on the engine performance and fuel storage.
- Flash Point: Biodiesel shows a higher flash point, which makes it safer for handling and transportation purposes as it is very less susceptible to ignition when exposed to an open flame or spark.
- Cloud Point: Biodiesel does have a higher cloud point, which indicates the temperature at which it starts to solidify. This aspect can be of a concern in colder climates and may necessitate mixing in with fossil diesel for use in such conditions.
- Pour Point: Like cloud point,, pour point of biodiesel signifies the temperature at which it becomes too viscous to flow i.e it has a higher resistance in flow. This property can be higher as compared to fossil diesel and may require the addition of cold-flow additives in lower temperature regions.

1.6 Applications

Biodiesel, a renewable and eco-friendly alternative to conventional diesel fuel, serves as a lot of practical applications in various divisions(Li, Tian and Xu, 2012). Here are some primary uses of biodiesel:-

- Transportation: Biodiesel is mainly used in place of diesel fuel in transportation vehicles such as cars, trucks, buses, and trains. It can also be mixed with diesel fuel, allowing pre existing diesel engines to operate with less carbon emissions, contributing to cleaner and greener air(Adepoju *et al.*, 2022).
- Agriculture and Farming: Biodiesel can be used to power farm equipments like tractors and other agricultural vehicles. This supports environment

friendly agricultural methods, reduces agriculture's impact on the environment, and supports ecological goals.

- **Maritime Industry:** Biodiesel is a viable fuel source for marine engines i.e engines running on water, fueling in boats and ships. Adoption lowers emissions in the marine industry, reducing the environmental effect of transportation by water.
- **Electricity Generation:** Diesel generators that produces power use biodiesel, mainly in isolated or off-grid areas. It gives a dependable power supply while limiting pollution.
- **Aviation:** Even though biodiesel are rare in commercial aviation, a few smaller and experimental aircraft have been modified to operate on biodiesel, which helps reduce emissions in this industry(Das and Chowdhury, 2023).
- **Heating Oil:** Biodiesel could be mixed with conventional heating oil, offering an eco-friendly option for residential and commercial space heating. It helps reducing the carbon emissions of heating systems.

1.7 By-Product Utilization

As a byproduct, around 100 kg of glycerol are produced for every ton of biodiesel. Centrifugation or gravity separation are the two primary methods used in the conventional removal of glycerol(Neubauer *et al.*, 2023). This process produces crude glycerol, which may still include ppm levels of contaminants such as methanol, oil, soap, salt, and other organic compounds. To increase the biodiesel industry's economic sustainability and lessen the negative environmental effects of the waste produced, it is critical to use crude glycerol efficiently. The removal or reduction of these contaminants can improve the use and value of crude glycerol. Therefore, it's critical to create a process that would raise crude glycerol's marketable and economic worth(Abdul Raman, Tan and Buthiyappan, 2019).This project presents a different approach to purify glycerol i.e by acidification and activated charcoal treatment. Consequently, we may increase glycerol's application in creating value-added goods by glycerol purification, which would increase income for the biodiesel sector.

2. LITERATURE REVIEWS

- **Michael Rahul Soosai et al.(2022)** .In the paper the possibility of oil from silk cotton seed as a feedstock for biodiesel synthesis is examined by the authors in their research study, with a particular emphasis on the use of heterogeneous catalysts and microwave-assisted transesterification. In comparison to traditional approaches, the study shows that microwave irradiation may effectively speed up the transesterification process, resulting in larger yields of biodiesel within shorter reaction times.Furthermore, the use of heterogeneous catalysts improves the reaction's efficiency and selectivity and makes it easier for catalysts to be reused, which lowers costs and has a less environmental effect. Overall, the study emphasizes the potential of silk cotton seed oil in the synthesis of biodiesel and stresses the value of heterogeneous catalysis and microwave-assisted transesterification as long-term strategies for raising the productivity of biodiesel manufacturing processes.
- **Chouhan et al.(2011)** . The review study examines the function of diverse catalysts in the biodiesel synthesis process in detail, examining several types of catalysts such as solid acids and metal oxides. In order to clarify catalyst performance, efficiency, and influence on biodiesel output and quality, study findings are synthesized. Catalyst activity, stability, and recyclability—all essential for real-world use and financial sustainability—are assessed severely in this review. It focuses on catalyst design and optimization techniques to improve performance and get around constraints, providing industry and research with direction. The study is a useful tool for pushing biodiesel production toward sustainability and the use of renewable energy sources by looking at current developments and new trends.
- **Rekunge et al(2016)**. The study investigates Fuller's earth in activated state as a very effective heterogeneous catalyst for the synthesis of biodiesel. Numerous benefits occur with this catalyst, such as affordability, stability,

simplicity of recovery, reusability, and ecological advantages. Its practical application is further enhanced by the fact that it functions under moderate reaction conditions and permits a high product yield. The study emphasizes how important Fuller's earth is to catalyzing the transesterification reaction, which allows feedstock to be converted into biodiesel with exceptional efficiency. Utilizing these advantageous properties, the catalyst shows great promise for large-scale biodiesel production. Furthermore, its eco-friendly qualities support sustainable production methods, which is in line with the increased focus on eco-friendly technology and renewable energy sources.

- **Balat et al.(2010).** This review article offers an in-depth review of the developments in the manufacture of biodiesel from a variety of edible and non-edible oils. It summarizes the advancements made in the use of a range of feedstocks, including conventional and non-conventional sources such as waste oils and algae. In addition, the paper explores modern techniques used in the production of biodiesel, emphasizing cutting-edge methods such as heterogeneous catalysts, and transesterification reaction. Through an analysis of various techniques' efficiency, yield, and sustainability, the report provides insightful information on how the biodiesel manufacturing industry is changing. Researchers and industry experts looking to improve biodiesel production methods, encourage resource diversification, and accelerate the shift to sustainable energy solutions may find this brief overview of study findings to be a helpful reference.
- **Mohd Johari et al.(2024).** The study looks at the use of microwave-assisted heating in transesterification to produce biodiesel, and it finds a number of significant things. The technology efficiently shortens response times and overcomes activation energy barriers by utilizing microwave energy, which speeds up the production of biodiesel. This creative method improves overall process efficiency while also quickening the feedstock's conversion to biodiesel. The study emphasizes how important microwave-assisted heating is for accelerating reaction kinetics because of its capacity to heat reaction components individually and promote molecular interactions. The research presents convincing evidence of the revolutionary effects of microwave-

assisted transesterification through rigorous testing and analysis, providing knowledge that has the potential to completely change the processes involved in the manufacturing of biodiesel. This study increases productivity, decreases resource consumption, and optimizes industrial processes to advance sustainable energy solutions.

- **Muhammad et al.(2018).** The study investigates the unrealized potential of non-food oil from silk cotton seed ⁴¹ as a possible feedstock for the manufacturing of biodiesel. The study shows that using silk cotton seed oil as a renewable supply-chain for biodiesel production is feasible via considerable testing and analysis. In order to determine if oil from silk cotton seed is a viable resource for the biodiesel synthesis the research evaluates important factors such oil composition, yield, and quality of biodiesel produced. Additionally, the study emphasizes the benefits—both financial and ecological—of utilizing non-food oils, such as silk cotton seed oil, which expands the range of feedstock alternatives and encourages environmentally friendly methods in the energy industry. All things considered, the results highlight the importance of non-food oils, especially silk cotton seed in enhancing biodiesel production and using it as a renewable energy source.
- **Ude et al.(2022).**The study indicate uses modern techniques to comprehensively investigate the biodiesel properties like kinematic viscosity and cetane number. Using advanced techniques like spectroscopy and chromatography, the research explores the physical and chemical properties essential to biodiesel efficiency. The research clarifies the quality, effectiveness, and environmental impact of biodiesel by closely examining relationships among these attributes. Regulations and biodiesel production optimization are guided by such observations. The research advances our understanding of biodiesel and promotes its viability and effectiveness as a substitute fuel.
- **Tian et al(2012).**⁵⁸made a comprehensive study and presented a paper titled “Application of Biodiesel in Automotive Diesel Engines,”. The study

examines the numerous applications of biodiesel in diesel engines and offers a thorough examination of its efficiency, emissions, and engine compatibility. The research assesses the effectiveness of biodiesel as a sustainable fuel substitute using practical investigations and theoretical assessments. It compares its emission profiles, engine operating parameters, and combustion properties to those of standard diesel fuels. The influence of biodiesel mixes on engine longevity, lubrication, and fuel system integrity are also examined in this article. It provides insightful information on viability of biodiesel and potential obstacles in diesel engine applications by combining the results of many research. The study advances our knowledge of how biodiesel helps to mitigate environmental pollution from internal combustion engines and lessen reliance on fossil fuels.

- **Umar et al.(2020).** This study examines temperature and catalyst concentration effects on the transesterification of Ceiba pentandra (silk cotton) seed oil using a two-phase process that includes alkali-catalyzed transesterification and acid esterification. The research investigation clarifies their impact on biodiesel output and quality by methodical experimentation with different catalyst concentrations and temperature ranges. The study identifies ideal parameters for increasing biodiesel production efficiency while lowering expenses and environmental effect by examining reaction kinetics and product features. In order to optimize the synthesis of biodiesel from Ceiba pentandra seed oil, the results provide insightful information on the complex interaction between process variables and reaction outcomes. In order to improve transesterification efficiency and advance sustainable biodiesel manufacturing methods, this research emphasizes the significance of controlling catalyst concentration and temperature.
- **Uttamrao et al.(2018).** The study examines the best circumstances for producing biodiesel and evaluates the benefits and drawbacks of doing so. To enhance biodiesel output and quality, critical factors including temperature, catalyst type, and reaction time are tuned through methodical testing. The goal of the research is to minimize resource consumption and environmental effect while increasing the efficiency of biodiesel

manufacturing processes by determining these ideal circumstances. The study also emphasizes the benefits of biodiesel, such as its renewable nature, lower greenhouse gas emissions, and potential for energy independence. It also covers disadvantages including feedstock rivalry with food crops, land use issues, and variations in biodiesel quality. By means of an extensive evaluation, the study offers significant perspectives for promoting the production of sustainable biodiesel and guides the process of deciding whether or not to use it as a substitute fuel source.

3. AIM & SCOPE

3.1 Aim

- To synthesize the Fuller's Earth catalyst and study the characteristics of catalyst.
- To produce Biodiesel from silk-cotton seed oil and fuller's earth catalyst.
- Examine the characteristics of Biodiesel produced.
- Analyse the properties of produced Biodiesel.

3.2 Scope

- Sustainable development in the field of fuels by biodiesel synthesis from silk-cotton seed oil and fuller's earth.
- Discover the application of fuller's earth as catalyst in transesterification reaction.
- Optimization to enhance the efficiency and yield of biodiesel production through silk-cotton seed oil.

4. MATERIALS AND METHOD

4.1 Equipments and Chemicals

Equipments used in this project are:-

- Magnetic Stirrer
- Sonicator
- Hot Air Oven
- Microwave Oven
- Seperating Funnel

Chemicals used for this project are:-

- Fuller's Earth
- Sodium Hydroxide
- Silk-Cotton Seed Oil
- Methanol

4.2 Catalyst Preparation

The synthesis of our catalyst using Fuller's Earth is a crucial step in the biodiesel production process. The catalyst is essential for facilitating the transesterification reaction, converting feedstock oils into biodiesel. Here is step by step procedure of preparation of catalyst:-

1. **Mixing Fuller's Earth with Distilled Water:-** In this first step, distilled water and Fuller's Earth are combined with a magnetic stirrer to create a slurry. The Fuller's Earth particles are thoroughly dispersed throughout the water by the magnetic stirrer, resulting in an uniform combination. The catalyst is built on top of this slurry.
2. **Addition of NaOH for activation:-** Adding NaOH to solution of Fuller's Earth and water is next step. NaOH is a typical catalyst used in transesterification reactions. After that we have stirred the solution for 2 hours helping the NaOH mixture dissolve into the solution, which leads to the catalytic solution formation.



Fig 4.1:-Magnetic stirrer

3. **Sonication:-** The solution is thereafter sonicated for 20 minutes at 40°C temperature. High-frequency sound waves are used in sonication to increase agitation and mixing. By making sure Fuller's Earth and NaOH are well distributed, this step serves to enhance catalytic activity.



Fig 4.2:-sonicator

4. **Stirring:-** The solution is agitated for two hours after sonication. This extra stirring promotes the catalyst's component interaction and helps to keep everything consistent.
5. **Filtering the Solution:-** The solution is filtered to extract the liquid from the slurry. The transesterification reaction requires the catalyst particles, which is the solid slurry left after extracting the liquid.
6. **Drying in a Hot Air Oven at 70°C (1 hour):-** After filtering, the solid slurry is dried for an hour at 70°C in a hot air oven. By removing any last traces of moisture, this stage guarantees that the catalyst is dry and active and prepared for use in the transesterification reaction.



Fig 4.3:-Drying in Hot Air Oven

To produce a highly effective catalyst, a number of precisely regulated procedures are involved in the catalyst production process. Fuller's Earth and NaOH combine to form the resultant catalyst, which is essential for starting and accelerating the transesterification reaction and enabling the effective synthesis of biodiesel from feedstock oils such as silk-cotton seed oil. This carefully designed catalyst guarantees that triglycerides are converted to glycerol and biodiesel, helping to produce a premium biodiesel product.

4.3 Synthesis of Biodiesel

The biodiesel synthesis from silk-cotton seed oil, methanol, and a catalyst by microwave irradiation is a novel and effective process. The transesterification process gets sped up using this approach by heating the reaction mixture quickly and precisely. This is the detailed process that was used to create biodiesel. Below is the step by step procedure followed for biodiesel production:-

1. **Microwave Irradiation**:- In this stage, the reaction mixture—which consists of 3.04 grams of catalyst, 31.08 milliliters of methanol, and 30 milliliters of silk-cotton seed oil in the molar ratio of 1:27—is heated in a microwave oven. The microwave has a 300W power supply and a 270-second reaction period with an on and off cycle. This cycle keeps the temperature from rising too high while enabling effective heating. The transesterification step, which turns the triglycerides in the oil into glycerol and biodiesel, is started by the microwave irradiation.



Fig 4.4:-Microwave irradiation

2. **Separation**:- The reaction mixture is allowed to cool and split into two different phases followed microwave irradiation. Fatty acid methyl esters, or FAME, the biodiesel, are found in the top phase, while glycerol and any unreacted methanol or reagents are found in the lower phase. In order to

obtain the biodiesel product from the reaction mixture requires separation and is carried out in a separating funnel.



Fig 4.5:-Separation

3. **Yield calculation:-**It was determined that the biodiesel yield was 67%. The proportion of the theoretical maximum amount of biodiesel that was successfully generated from the raw ingredients is represented by this yield. A 67% output suggests that a significant portion of the feedstock oil has been synthesized into biodiesel.

$$\text{Yield\%} = \frac{\text{Weight of biodiesel}}{\text{Weight of Oil}} \times 100$$

4. **Density measurement:** The biodiesel product's density was determined to be 788 kg/m³. A crucial attribute that tells us about the fuel's weight and volume is its density. It serves to evaluate the biodiesel product's quality.
5. **Water Washing:** In order to purify the biodiesel further and get rid of any residual impurities, the biodiesel is sent for water washing. In this process the biodiesel is mixed with hot water and left for some time to let the impurities settle down. Water washing serves to increase the quality of the final biodiesel product.



Fig 4.6:-Water washing

The process mentioned above is a quick and effective way to use microwave irradiation to make biodiesel. By using this method, reaction durations are reduced, transesterification proceeds more quickly, and 67% biodiesel with a density of 788 kg/m^3 is produced. The biodiesel is further purified in the next stage by washing it with water, which guarantees that it satisfies quality requirements to be used as fuel.

4.4 Process Optimization with RSM.

³⁹ Response Surface Methodology (RSM) which is a statistical and mathematical technique utilized to optimize processes and experiments. In order to identify the ideal condition of input factors that will result in best outcome or response, it is used to investigate the correlations between several variables and responses. RSM's objective is to design a mathematical model that can predict a process' reaction based on the values of its input variables.

Table 4.1: Coded values of Experimental matrix

Factor	Name	Level	Low Level	High Level
1	oil: alcohol ratio	27	24	30
2	catalyst wt%	4	3	5
3	Microwave power	300W	270W	360
4	D.Time	270s	180s	270s

Oil to Alcohol Ratio: This factor tells how much oil there is compared to alcohol in the reaction mixture. This is an important step in the transesterification process, which produces biodiesel. The biodiesel experiment had a low level of 24; a high level of 30, and a mid-level of 27.

Catalyst Weight %: The yield and reaction rate are greatly influenced by the catalyst's weight % in the reaction mixture. This factor has three levels: 3% for the low level, 5% for the high level, and 4% for the mid-level.

Microwave Power: Reaction kinetics and heating efficiency depends upon the power we supply, microwave power is a crucial factor in chemical reactions performed by microwaves. This power at mid-level was set at 300W, its high level at 360W, and its low level at 270W.

Duration of Reaction Time (D.Time): The amount of time taken to perform the reaction i.e, the time duration for which the reaction mixture was inside the microwave. In this instance, the duration at lower level was for 180 seconds with mid and high level to be same at 270 seconds.

Table 4.2:RSM Response Table(Yield percentage)

		Factor 1	Factor 2	Factor 3	Factor 4				Response
Std	Run	A:Alcohol to oil molar ratio	B:Catalyst wt. %	C: Microwave Power	D: Time	Oil	Alcohol	Catalyst	Biodiesel Yield
				W	s	mL	mL	g	%
1	28	24	3	270	270	30	27.63	1.47	80
2	6	30	3	270	270	30	34.53	1.64	82
3	17	24	5	270	270	30	27.63	2.46	86
4	15	30	5	270	270	30	34.53	2.73	83
5	26	27	4	180	180	30	31.08	2.08	87
6	4	27	4	360	180	30	31.08	2.08	86
7	11	27	4	180	360	30	31.08	2.08	89
8	14	27	4	360	360	30	31.08	2.08	87
9	23	24	4	270	180	30	27.63	1.96	84
10	3	30	4	270	180	30	34.53	2.18	82
11	5	24	4	270	360	30	27.63	1.96	84
12	12	30	4	270	360	30	34.53	2.18	83
13	1	27	3	180	270	30	31.08	1.55	80
14	16	27	5	180	270	30	31.08	2.59	83
15	21	27	3	360	270	30	31.08	1.55	82
16	29	27	5	360	270	30	31.08	2.59	84
17	10	24	4	180	270	30	27.63	1.96	87
18	9	30	4	180	270	30	34.53	2.18	85
19	7	24	4	360	270	30	27.63	1.96	83
20	18	30	4	360	270	30	34.53	2.18	87
21	22	27	3	270	180	30	31.08	1.55	89
22	27	27	5	270	180	30	31.08	2.59	87
23	20	27	3	270	360	30	31.08	1.55	84
24	24	27	5	270	360	30	31.08	2.59	80
25	13	27	4	270	270	30	31.08	2.08	92
26	19	27	4	270	270	30	31.08	2.08	92.5
27	25	27	4	270	270	30	31.08	2.08	92.4
28	2	27	4	270	270	30	31.08	2.08	92
29	8	27	4	270	270	30	31.08	2.08	92

Above table is the designed mathematical model to find the ideal response for biodiesel synthesis by use of Response Surface Methodology. After a continuous 30 trials the following is the response derived through experiments. Oil to alcohol ratio of 1:27 with catalyst weight percentage of 3 had the best response of 92.2 yield percent.

4.5 Glycerol Purification

- Checking glycerol pH: Measure the pH of the glycerol sample to assess its initial acidity or alkalinity.
- Acidify glycerol by adding HCl: Adding hydrochloric acid (HCl) to the glycerol to acidify it. This step protonates any remaining hydroxyl groups on the glycerol molecules.
- Check pH after acidification: Measure the pH of the acidified glycerol to confirm the effectiveness of the acidification process. Typically, this step will yield an acidic pH due to the addition of HCl. We have to allow the solution to settle down and transfer it into a separating funnel allowing for phase separation.
- Collecting glycerol: After settling, we have to collect the glycerol layer, which should be the lower layer in the separating funnel.
- Neutralize glycerol with NaOH: Add sodium hydroxide (NaOH) to the collected glycerol to neutralize any remaining acid and adjust the pH to a basic level.
- Heat the solution and allow salt to settle down: Heat the neutralized glycerol solution to 110 degrees Celsius to evaporate any water and allow any dissolved salts to settle down.
- Addition of activated charcoal: Transfer the glycerol solution to another container and add activated charcoal to adsorb any remaining impurities.
- Filtering the solution: Filter the glycerol solution using filter paper to remove the activated charcoal and any impurities adsorbed by it, resulting in purified glycerol.



Fig 4.7 Glycerol Purification

5. RESULT

5.1 Test Analysis

To demonstrate that the catalyst synthesized had sufficient capabilities to promote a transesterification process and that the biodiesel thus produced could be effectively used in place of conventional diesel, an examination of the catalyst particle and biodiesel is required. Here are some characterizations we carried out to demonstrate the catalyst and biodiesel sample viability.

5.2 Fourier Transform Infrared Spectroscopy(FTIR) for Fuller's earth catalyst

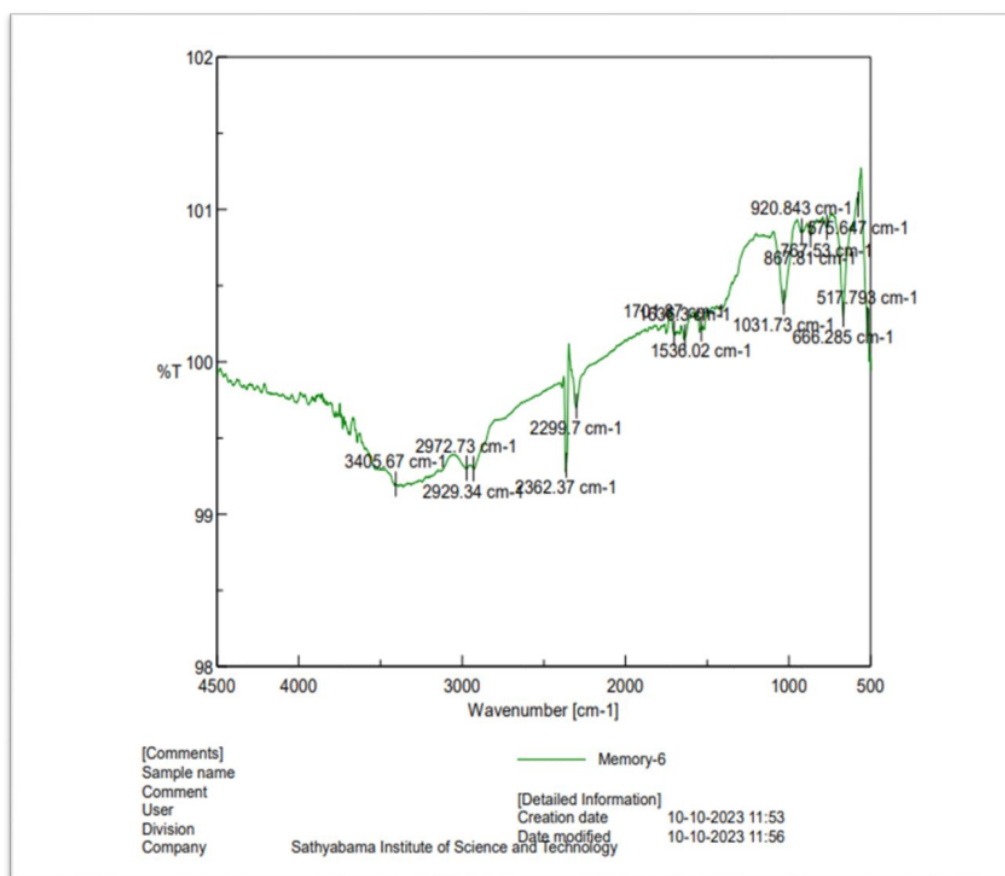


Fig 5.1 FT-IR for catalyst

Based on an infrared absorption spectrum, FTIR is a potent analytical method used to detect and describe chemical substances. FTIR may be used to evaluate functional groups that are present in the catalyst material in the context of the catalyst (Fuller's Earth) study. It offers details on the structure and chemical makeup of the catalyst. It can help validate the identify of the catalyst by pointing out the existence of certain bonds and chemical groups that are typical of Fuller's Earth. The catalyst's suitability for its intended usage in the transesterification process and its proper chemical form are both confirmed by FTIR analysis.

Sample preparation, instrument setup, and background measurement are all part of FTIR analysis. After that, the sample was put into the device for being analyzed. The process of acquiring data began with the collection of interferograms, which are then Fourier-transformed to produce the infrared spectrum. Characteristic absorption bands were identified by analysis, and the spectrum was compared to references for identification. Moreover, quantitative analysis may be carried out. Reports were created from the results. Instrument cleaning and shutdown are included in instrument maintenance. The procedure, which includes measurement, preparation, analysis, and maintenance, allowed materials to be identified and characterized according to their infrared absorption characteristics.

The Fourier Transform Infrared Spectroscopy (FTIR) test results revealed specific peaks at various wavenumbers, indicating the presence of functional groups and molecular vibrations in the analyzed sample. Here's an explanation of the observed peaks:-

C-H Alkane Peaks (2972.73, 2929.34): The presence of C-H (Carbon-Hydrogen) alkane functional groups is indicated by these peaks. Alkane groups are characteristic of hydrocarbon chains in the sample, which are essential for biodiesel.

C=O Peaks (1701.87, 1636.3): The peaks at the certain wavenumbers signify the presence of C=O (Carbon-Oxygen double bond) functional group. This is mainly associated with carbonyl compounds and is indicative of ester bonds.

O-H Stretch Peaks (3405.67, 2362.37, 2299.7): The peaks in this region suggest the presence of O-H (Oxygen-Hydrogen) stretching vibrations.

Major Peaks (575.64, 517.79, 666.255): These prominent peaks indicate characteristic vibrations associated with specific functional groups.

Therefore, the test results indicate the presence of hydrocarbon chains (alkanes) and ester bonds (C=O) in the biodiesel sample, which aligns with the expected chemical composition that are essential for production of biodiesel.

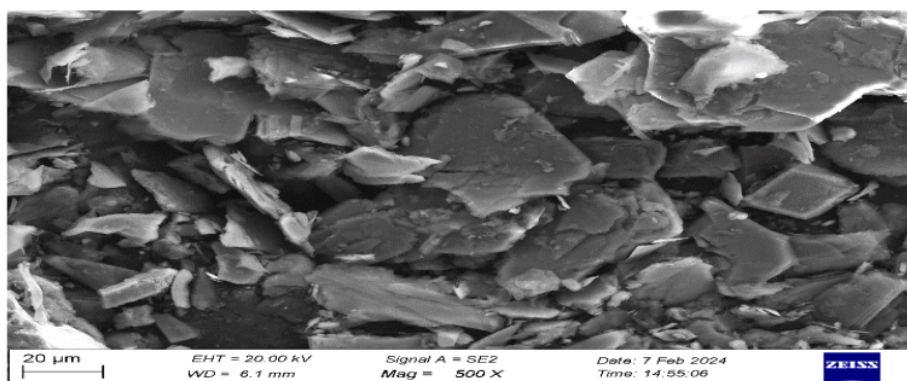
45

5.3 Field Emission Scanning Electron Microscope (FESEM) for Fuller's earth catalyst

Scanning Electron Microscopy is a very crucial analysis in scientific inquiry, demonstrates a lot of capabilities for understanding and analysing the surface of material with more precision. It employs a focused electron beam at the particles, It reveals the unique surface morphology and composition of particles, which operates by magnifying the images of particles in nanoscale.

Its features is that it zooms deep into the surface of particles and elaborates minute details about the particle, in this case my catalyst. It shows in detail about the surface morphology of the catalyst, its composition and its performance. It also shows the particle size and the arrangement of the particles.

Vacuum chamber evacuation, equipment setup, and sample preparation are all steps for scanning electron microscopy (SEM). After being mounted on a conductive substrate, the sample is put inside the SEM chamber. The voltage and current of an electron beam are set. By using the electron beam to scan the sample surface and find secondary or backscattered electrons, images were obtained. After that elemental analysis was done through energy-dispersive X-ray spectroscopy (EDX). Surface shape, elemental composition, and other features were examined in the data analysis process. Reports were created from the results. High-resolution imaging and elemental analysis of materials are made possible by SEM, which helps with a variety of scientific and industrial applications.



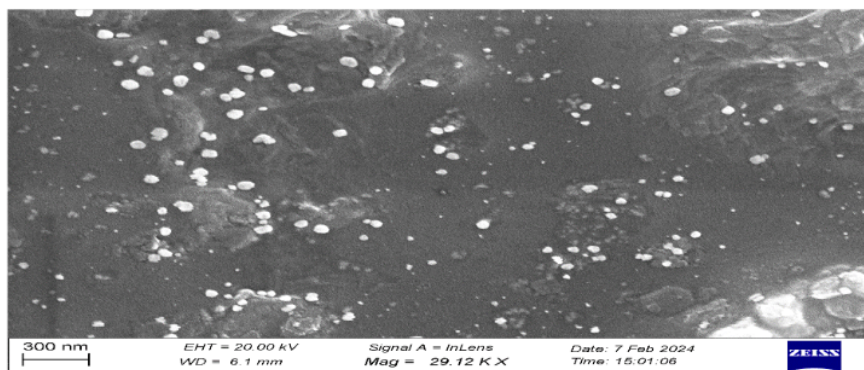
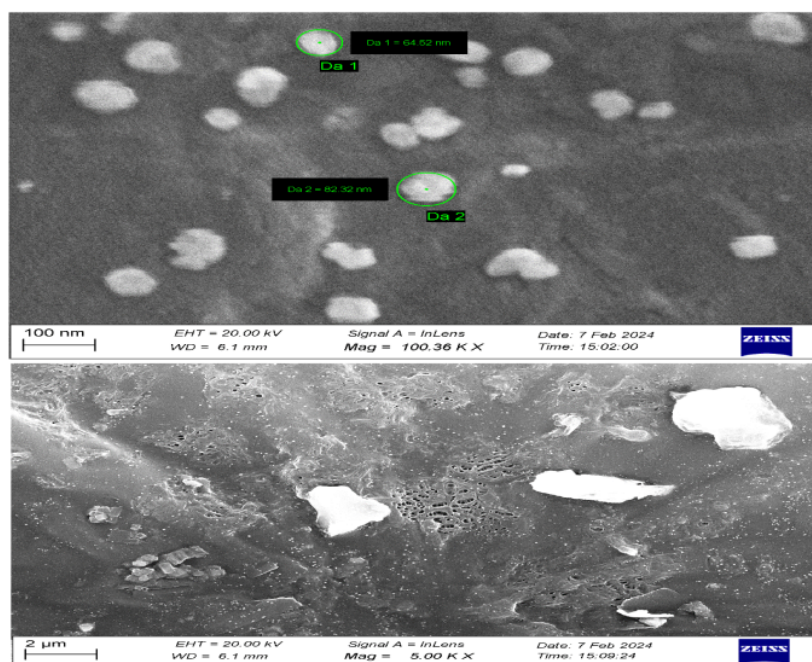


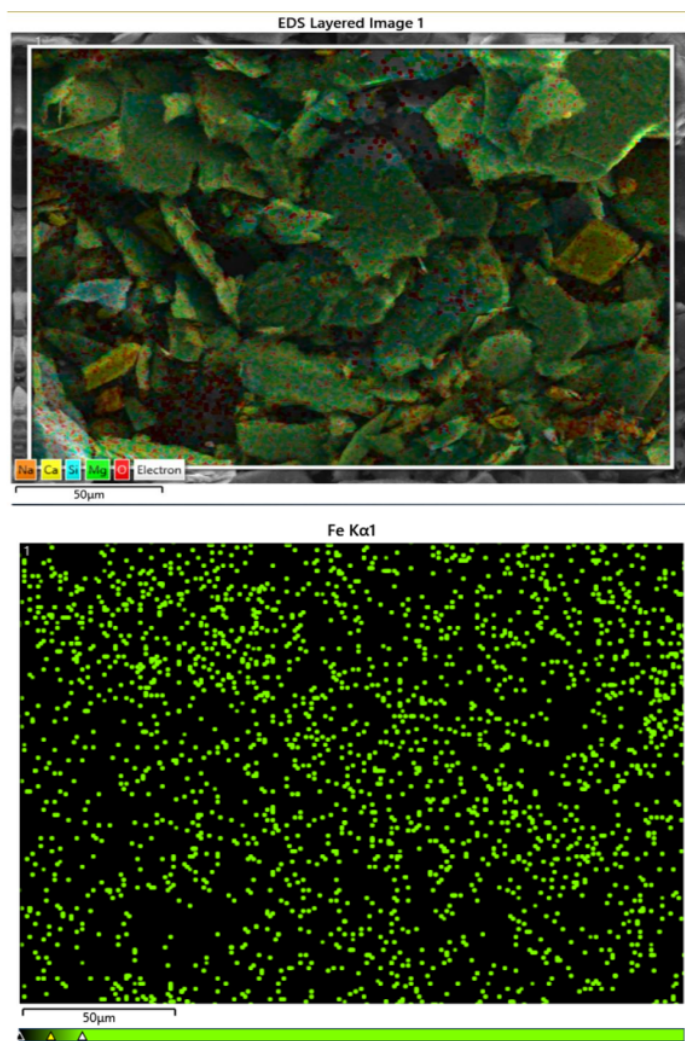
Fig 5.2 SEM for catalyst

Upon analyzing our catalyst sample of Fuller's Earth there were quite good revelations we had a good no of pores and particle diameters were ranging from 64.4 nm to 84.2 nm which indicated its potential efficiency in biodiesel production. The characterization denoted that the catalyst had a high surface area and optimum particle size distribution, which is viable for enhancing catalytic activities and providing efficient reactant diffusion while performing the transesterification process.



5.4 Energy Dispersive X-Ray(EDX) for Fuller's earth catalyst.

An effective method for analysing and for figuring out a material's elementary composition is Energy Dispersive X-ray Spectroscopy , which is used in junction with Scanning Electron Microscopy. With SEM, a sample's surface is bombarded with a concentrated electron beam, producing different X-rays. To determine which elements are present in the sample and how much of each is present relative to the others i.e the amount of it present,It examines these X-rays and gives a detailed information on elements and it's compositions.



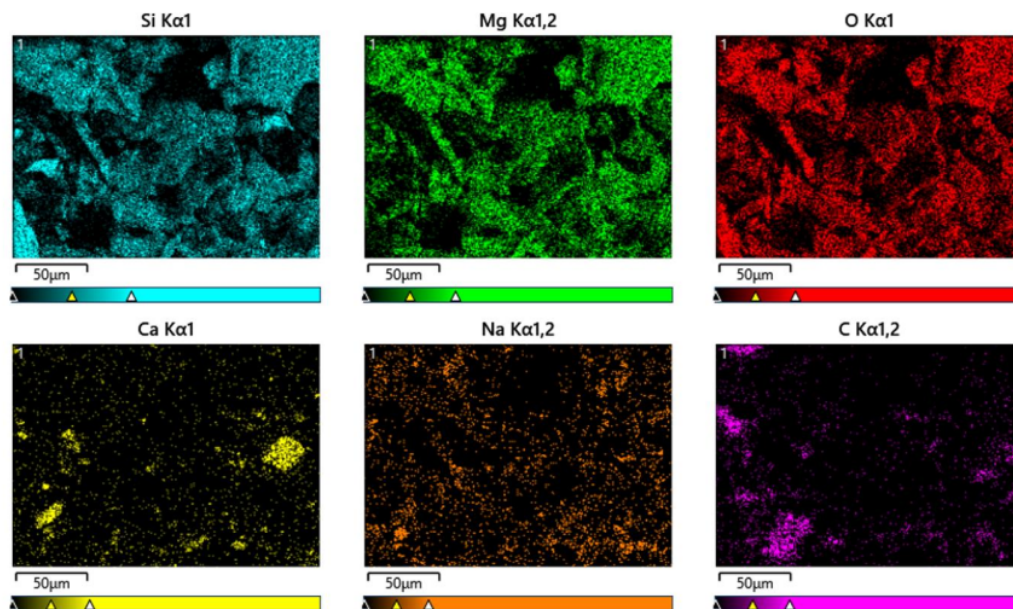


Fig 5.3 EDX for catalyst

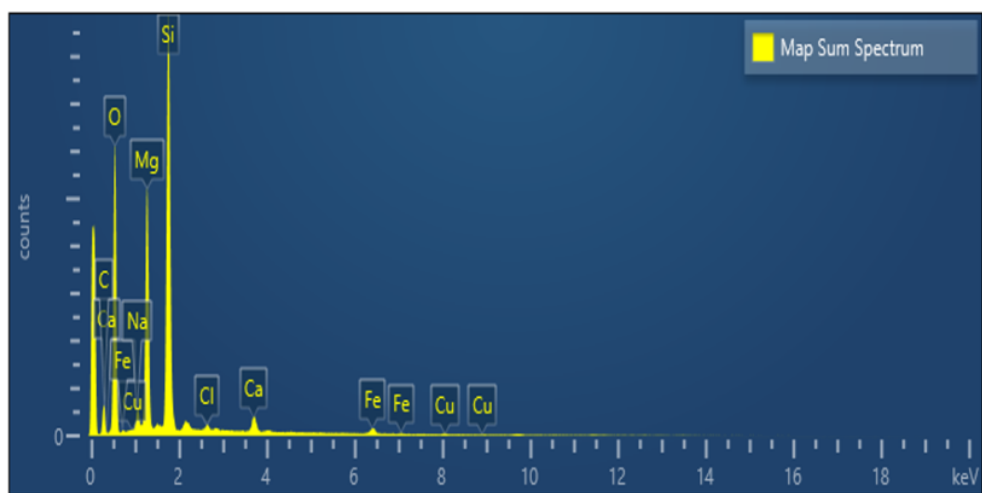


Fig 5.4 Elemental composition of catalyst

Table 5.1 :Elemental composition of catalyst

Map Sum Spectrum Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	22.61	0.58	31.91
O	K series	45.34	0.39	48.03
Na	K series	1.28	0.06	0.95
Mg	K series	11.27	0.12	7.86
Si	K series	17.40	0.17	10.50
Ca	K series	1.07	0.04	0.45
Fe	K series	1.03	0.06	0.31
Total		100.00		100.00

The EDX analysis of the sample indicated the existence of carbon (C), oxygen (O), sodium (Na), magnesium (Mg), silicon (Si), calcium (Ca), and iron (Fe). Among these elements that we found, oxygen has the highest weight percentage at 45.34%, second is carbon at 22.61%. The findings of these elements suggests the sample is mainly made up of organic compounds, along with materials such as polymers or organic coatings.

Furthermore, silicates or other inorganic compounds present due to the significant concentrations of silicon (17.40%) and magnesium (11.27%). The point that these elements can be found in glasses and minerals suggests the fact that composition of our catalyst is heterogeneous, meaning it contains both organic and inorganic elements.

We also found that iron, calcium, and salt together make up less than 2% of the sample's weight, it can be more likely that these elements are mostly additions or contaminants than main components. However, their existence might still affect the catalyst characteristics and uses, especially when it comes to reactivity or structural stability.

Therefore the EDX analysis provided an overview on the elements and its concentration by weight percentage also highlighting the organic and the inorganic compounds. The analysis and information provided through the analysis is very crucial if we want to understand the catalyst properties its application etc.

5.5 Brunauer-Emmett-Teller(BET) for Fuller's earth catalyst.

BET is a popular technique for figuring out the specific surface area of materials with pores, like catalysts, adsorbents, and nanoparticles. It gives information about surface area, porosity of materials and the methodology is based on measurements of gas adsorption, usually nitrogen adsorption at low temperatures.

BET is used along with BJH in order to calculate pore size distribution of the catalyst particle. The BET method gives information on the catalyst material's total surface area, but it does not tell us specific information about distribution of pore sizes. By offering data on the distribution of pore sizes which is a crucial component for calculating the porosity and functionality of the catalyst material the BJH approach enhances the BET analysis.

The total analysis of a porous material takes place in three simple steps.

- **Adsorption Isotherm Calculation:** Adsorption isotherms are produced by gas adsorption studies, usually across a range of relative pressures (P/P_0). Amount of gas adsorbed onto the material as a function of relative pressure is described by the isotherm. For this step mainly nitrogen gas is used.
- **BET Analysis:** After the adsorption isotherm calculation BET analysis is performed on this isotherm data. This analysis is done on BET equation which is mainly a plot of $P/V(P_0-P)$ v/s P/P_0 where P_0 is saturation pressure
- **BJH Desorption Analysis:** After BET analysis, the desorption of the adsorption isotherm takes place. It analyses the release of gas from pores as pressure decreases. This method is then used to calculate pore size distribution data.

Adsorption Isotherm Calculation.

Parameters for Data Reduction:-

- Temperature 77.350K
- Molec. Wt.: 28.013
- Cross Section: 16.200 Å²
- Liquid Density: 0.808

Adsorbate :-Nitrogen.

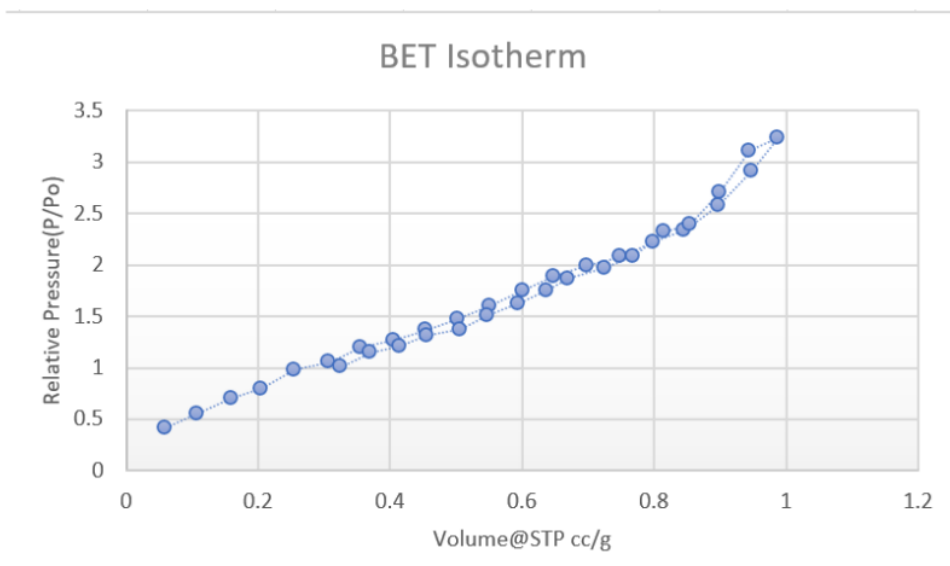


Fig 5.5 BET isotherm Graph.

- ❖ The above graph of relative pressure v/s pore volume represents the adsorption and the desorption curve, it helps in determination of surface area and pore diameter.
- ❖ The top line is the desorption line and the bottom one is adsorption.

BET summary

- Slope = 863.102 1/g
- Intercept = 8.019×10^1 1/g
- Correlation coefficient, $r = 0.999564$
- C constant = 11.763
- Surface Area = 3.692 m²/g

Surface Area: The catalyst surface area was calculated to be at 3.692 m²/g. It indicates a significant active surface area for catalytic reactions. A higher surface area generally correlates with more available active sites for reactions, potentially leading to enhanced catalytic activity.

Slope: The slope value comes within a certain range which depends upon the adsorption characteristics of the catalyst material and the experimental conditions at which experiments were performed. Generally, higher slopes indicate more favorable adsorption behavior. The slope value of 863.102 1/g suggests a reasonable indication of a material with viable adsorption capacity.

Intercept: The intercept value tells us the amount of adsorbate adsorbed at low relative pressures (approx close to zero). It has to be a positive value, but mainly its magnitude depends on conditions such as the surface properties of the material and the adsorbate we have used. The calculated intercept value of 8.019×10^1 1/g comes to be within an acceptable range.

Correlation Coefficient (r): A significantly high correlation coefficient value which was found to be 0.999564 tells us that there is a strong linear relationship between the relative pressure and the quantity of adsorbate. This shows that the data fits well to the BET model.

Constant C: The role of constant C in the BET equation is to transform the experimental data into surface area values. Its results depend on the specific adsorption procedure and experimental conditions. The calculated value of 11.763 comes under a typical range for this constant.

7 BJH Pore Size Distribution Desorption Summary

Surface Area = 7.176 m²/g

Pore Volume = 0.008 cc/g

Pore Diameter Dv(d) = 2.120 nm

5.6 Catalyst Reusability

Fuller's Earth catalysts can be reused several times depending on the reaction type, catalyst stability, and catalyst recovery method, among other variables. Depending upon microwave assisted reaction and filtration we were able to recover a significant amount of catalyst for approx 2 grams. The reusability of a catalyst can be cost-effective and sustainable solution. Fuller's Earth catalysts are very promising for producing biodiesel because they can be reused, which offers aid to financial and environment factors. Fuller's earth can be very useful for bulk production because it can maximize production efficiency and reduce costs by utilizing Fuller's Earth's long-lasting and reuseability catalytic characteristics by adhering to strict regeneration procedures and keeping performance in check. The long use of Fuller's Earth catalysts can be extended by regular optimization efforts and following maintenance procedures, guaranteeing its continuous contribution to environmentally friendly biodiesel production methods.

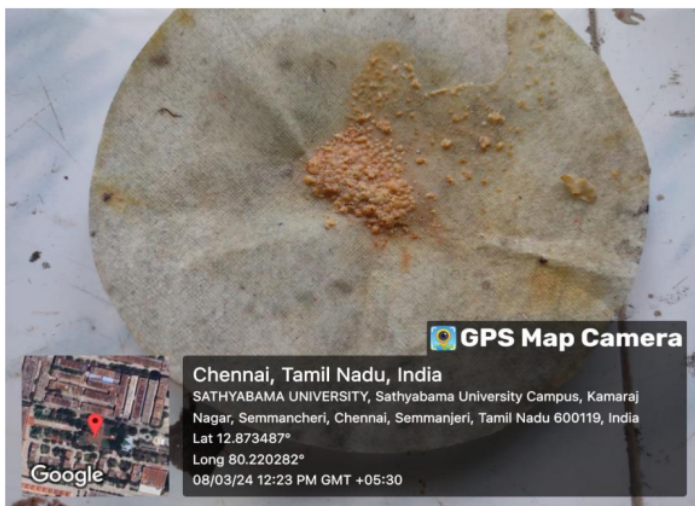


Fig 5.6 Recovered Catalyst

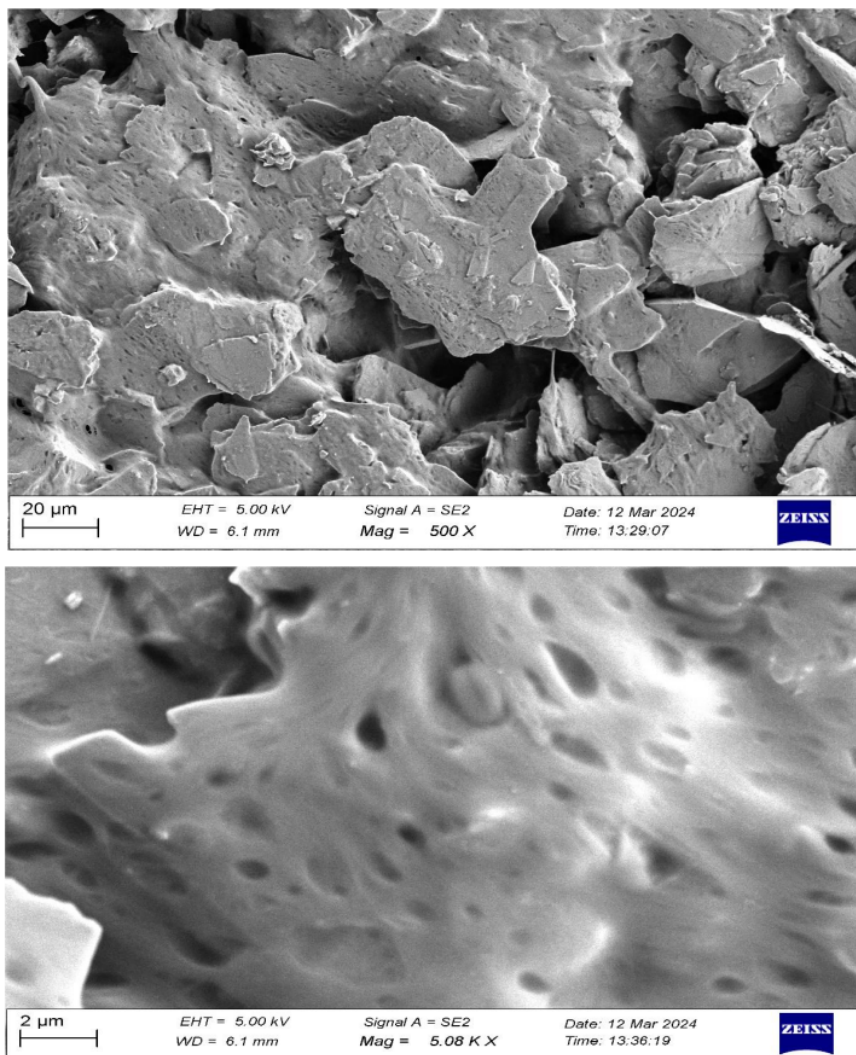


Fig 5.7 SEM images for recovered catalyst

The catalyst's surface morphology, structural integrity, and deposition of impurities or reaction byproducts can all be examined using after-use SEM analysis. After using a catalyst, SEM pictures have shown changes to the surface topography of the catalyst, such as the existence of pores or surface roughness due to catalytic reactions and regeneration processes. SEM analysis can also help assist in finding contaminants or organic residues on the catalyst's surface, which will affect its catalytic activity and require renewal.

5.7 Gas Chromatography Flame Ionization Detection(GCFID)for biodiesel

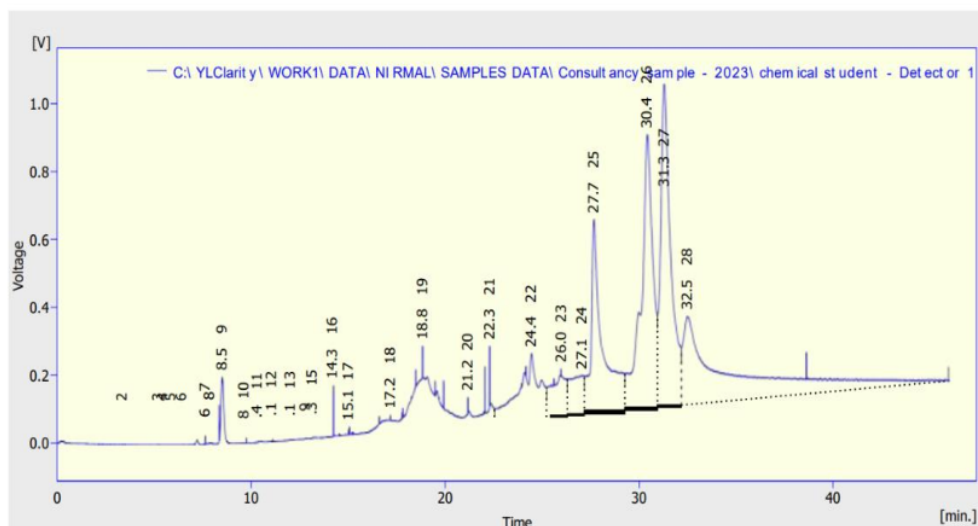


Fig 5.8 GCFID graph for Biodiesel

GC-FID is a technology that works well for assessing biodiesel samples since it can be used to extract and quantify specific components in a complicated mixture: The different elements contained in the biodiesel sample are separated and quantified using GC-FID. The presence of methyl esters, which are the main ingredients of biodiesel, is indicated in this instance. It allows the production and purity of biodiesel to be determined by quantifying the amount of methyl esters. This data is essential for evaluating the biodiesel's quality and appropriateness as a fuel. In order to make sure the biodiesel satisfies quality requirements, GC-FID analysis may also identify any contaminants or undesirable molecules that may have developed during the transesterification procedure.

The steps that were involved in Gas Chromatography with Flame Ionization Detection (GC-FID) included sample preparation, instrument setup, and GC column installation. During chromatographic runs, samples were injected and sorted according to their affinity for the stationary phase. Organic chemicals that elute from the column were burned in flame ionization detection (FID), creating ions and a current proportional to their concentration. Compounds were then identified and quantified by acquiring and analyzing data. Quantification were aided by calibration curves that are derived from standards. Reports are created from the results,

analyzing the substances and concentrations found. Instrument performance is guaranteed by maintenance chores.

In this analysis, the results are presented in terms of retention time, area, and height for different compounds, along with their percentages. Here's an explanation of the observed results:-

- Heptadecanoic Acid, Methyl Ester (Compound 1): This compound is detected at a retention time of 10.427 minutes with an area of 33.185 mV.s. It makes up a small fraction of the total, with a percentage of 2.370%.
- Stearic Acid, Elaidic Acid, Methyl Ester (Compound 2): Detected at a retention time of 11.113 minutes with an area of 2.578 mV.s. It also constitutes a small portion of the sample, with a percentage of 6.333%.
- Linolenic Acid, Methyl Ester (Compounds 3 and 4): Two peaks at retention times 12.113 and 12.903 minutes correspond to this compound. They have relatively small areas and percentages (1.448% and 1.594%, respectively).
- Arachidic Acid, Methyl Ester (Compound 5): Detected at 13.260 minutes with an area of 43.124 mV.s. It represents a small portion of the sample, with a percentage of 2.620%.
- ⁵⁶cis-8,11,14-Eicosatrienoic Acid, Methyl Ester (Compound 6): This compound shows a significantly larger area of 72.479 mV.s, representing 150.462% of the total. This may be due to an anomaly in the analysis or the presence of an abundant compound.
- ³⁶cis-5,8,11,14,17-Eicosapentaenoic Acid, Methyl Ester (Compound 7): Detected at 15.077 minutes with an area of 113.770 mV.s. It constitutes a small fraction of the sample, with a percentage of 24.376%.
- Lignoceric Acid, Methyl Ester (Compound 8): This compound is detected at 17.187 minutes with a notably large area of 2407.856 mV.s, accounting for 48.227% of the total.
- ³¹cis-4,7,10,13,16,19-Docosahexaenoic Acid, Methyl Ester (Compound 9): Detected at 18.843 minutes with a substantial area of 15627.608 mV.s, representing 242.453% of the sample. This high percentage may indicate the presence of an abundant compound.

- Capric Acid, Methyl Ester (Compound 10): Detected at 21.183 minutes with an area of 1108.170 mV.s. It makes up a relatively small percentage of 79.701%.
- Undecanoic Acid, Methyl Ester (Compound 11): Detected at 22.283 minutes with an area of 2123.266 mV.s. It represents 224.992% of the total.
- Lauric Acid, Methyl Ester (Compound 12): Detected at 24.447 minutes with a substantial area of 13972.118 mV.s, accounting for 191.898% of the sample.
- Tridecanoic Acid, Methyl Ester (Compound 13): Detected at 25.973 minutes with an area of 6531.097 mV.s. It constitutes 138.992% of the total.
- Myristic Acid, Methyl Ester (Compound 14): Detected at 27.067 minutes with an area of 5735.785 mV.s, representing 113.638% of the sample.
- Myristoleic Acid, Methyl Ester (Compound 15): Detected at 27.663 minutes with a substantial area of 23536.503 mV.s, accounting for 570.606% of the total.
- Pentadecanoic Acid, Methyl Ester (Compounds 16 and 17): Two peaks at retention times 30.420 and 31.277 minutes correspond to this compound. They have significantly large areas and percentages (806.432% and 951.237%, respectively).
- cis-10-Pentadecenoic Acid, Methyl Ester (Compound 18): Detected at 32.477 minutes with an area of 43771.535 mV.s, representing 259.254% of the sample.

The total area for all compounds is 186004.728 mV.s, and the total height is 4064.804 mV. These results provide a detailed breakdown of the compounds present in the analyzed sample, with some compounds exhibiting notably high percentages.

5.8 Gas Chromatography Mass Spectroscopy for Biodiesel

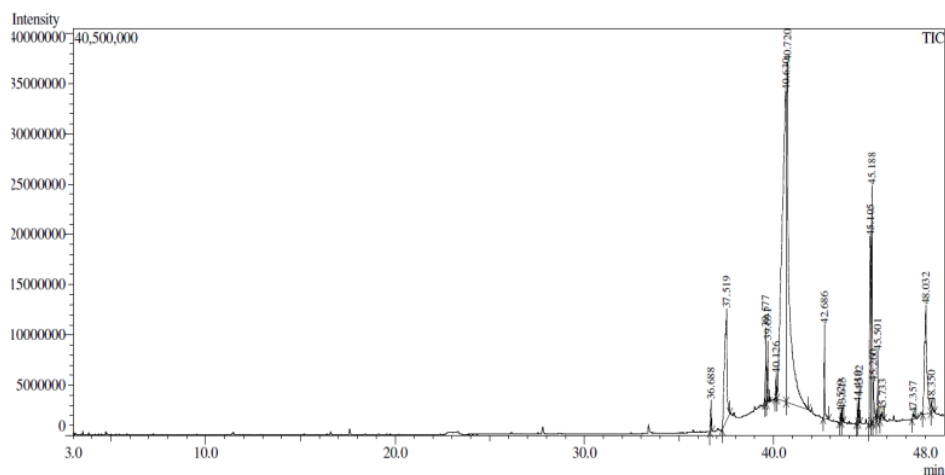


Fig 5.9 GCMS graph for biodiesel

Table 5.2: GCMS graph interpretation table

S.No	Compound Composition	Compound Name	Category
1	0.53	Palmitic acid,Methyl ester	Saturated
2	8.08	Linoleic acid,Methyl ester	Saturated
3	1.4	9,12-octadecadienoic acid,Methyl ester	poly-unsaturated
4	1.41	9-octadecenoic acid,Methyl ester	mono-unsaturated
5	0.43	Heptadecanoic acid,Methyl ester	Saturated
6	34.68	9,12-octadecadienoic acid,Methyl ester	poly-unsaturated
7	28.05	9-octadecenoic acid	mono-unsaturated
8	1.99	Hexadecanoic acid,2-Hydroxy,3 Propanediyl ester	Saturated
9	0.18	6,9-Octadecadienoic acid,Methyl ester	poly-unsaturated
10	0.24	9-octadecenamide	mono-unsaturated
11	0.38	6,9-Octadecadienoic acid,Methyl ester	poly-unsaturated
12	0.48	9-octadecenoic acid,1,2,3-propanetriyl ester	Saturated
13	5.09	9,12,15-octadecatrienoic acid, methyl acid	mono-unsaturated
14	5.85	9-octadecenoic acid,2,3-dihydroxypropyl ester	poly-unsaturated
15	1.42	14-methyl-8-Hexadecyn-1-ol	mono-unsaturated
16	1.41	Hexadecanoic acid,3 Propanediyl ester	Saturated
17	0.4	14-methyl-8-Hexadecyn-1-ol	Saturated
18	0.23	Linalol oxide,trimethylsiyl ester	-----
19	7.39	9-octadecenoic acid,9-Hexadecenyl ester	Saturated
20	0.36	Octadecanoic acid,2,3-dihydroxypropyl ester	Saturated

This gas chromatography-mass spectrometry (GCMS) analysis of my biodiesel sample indicates multiple composition of Fatty Acid Methyl Esters (FAMES) and other compounds.

The compounds which were present in ample quantity were saturated fatty acids like Palmitic acid, methyl ester, Linoleic acid, Heptadecanoic acid, and Hexadecanoic acid. These compounds help in maintaining the stability and viscosity of the biodiesel.

The methyl esters that was found in the biodiesel sample are of great importance due to their crucial roles in biodiesel quality, it's performance, and environment impact it will cause. These esters make up the bulk of biodiesel composition, and impact crucial fuel properties such as viscosity, combustion characteristics, and engine compatibility. These methyl esters found in the sample also indicates that the given sample is biodiesel confirmed.

Besides from methyl esters, polyunsaturated fatty acids as 9,12-Octadecadienoic acid and 6,9-octadecadienoic acid were also found in the test, providing detailing into the oxidative stability potential and cold flow properties of the biodiesel.

Mono-unsaturated fatty acids, along with 9-octadecenoic and 9-octadecenamide, were found in considerable proportions, influencing the biodiesel's combustion characteristics and oxidative stability.

More importantly, minor components such as 9-octadecenoic acid, 2,3-dihydroxypropyl ester, and 14-methyl-8-hexadecyn-1-ol make up and form the overall composition, which also affects the biodiesel's lubricating potential and thermal stability.

Also, the compounds present like hexadecanoic acid, 2-hydroxy, 3-propanediyl ester, and linalol oxide, trimethylsilyl ester shows the occurrence of esterification reaction and oxidation reaction during the synthesis of biodiesel.

Therefore, the GCMS analysis provides a detailed information into the chemical constituents that make up the biodiesel, it also helps in understanding the performance and quality of biodiesel, moreover it shows the potential of biodiesel for use in energy sector as a green source of energy.

5.9 Process Analysis with RSM

A statistical method for improving processes such as the manufacture of biodiesel is called response surface methodology, or RSM. It involves developing tests, usually using a Central Composite Design (CCD), to collect information on the effects of input variables (such as temperature and catalyst concentration) on response variables (such as yield and quality). By fitting a model to this data, design expert software makes it possible to forecast responses for other component combinations. Optimization, which frequently makes use of numerical methods or response surface visualization, seeks to identify circumstances that maximize yield or quality. By methodically determining the ideal circumstances, RSM improves the production of biodiesel and eventually lowers costs by increasing yields, improving quality, and increasing resource efficiency.

5.9.1 Analysis of Variance

Response Surface Methodology (RSM)'s ANOVA (Analysis of Variance) assesses the importance of variables affecting biodiesel output or quality in the context of biodiesel production. ANOVA evaluates the effects of each component and their interactions on the generation of biodiesel by planning experiments and gathering data on variables such as temperature, catalyst concentration, and reaction time. It directs process optimization by assisting in the identification of important variables influencing yield or quality. ANOVA in RSM assesses how well regression models fit the experimental data to ascertain whether the model can account for variance in the response variable. In the end, ANOVA helps researchers identify crucial variables for increasing output, improving productivity, and lowering expenses in the procedures involved in producing biodiesel.

Table 5.3 RSM response value.

Response 1: Biodiesel Yield

¹⁰ Source	Sum of squares	df	Mean Square	F-value	P-value	
Model	323.77	¹⁴	23.13	3.35	0.0153	significant
A-Alcohol to oil molar ratio	0.333	1	0.333	0.0483	0.8292	
B-Catalyst wt%	3.00	1	3.00	0.4349	0.5203	
C-Microwave Power	0.3333	1	0.3333	0.0483	0.8292	
D-Time	5.33	1	5.33	0.7731	0.3941	
²⁹ AB	6.25	¹	6.25	0.9060	0.3573	
AC	9.00	¹	9.00	1.30	0.2725	
¹³ AD	0.2500	¹	0.2500	0.0362	0.8518	
BC	0.2500	¹	0.2500	0.0362	0.8518	
BD	1.00	¹	1.00	0.1450	0.7091	
CD	0.2500	¹	0.2500	0.0362	0.8518	
A ²	141.67	1	141.67	20.54	0.0005	
B ²	190.78	1	190.78	27.66	0.0001	
C ²	55.43	1	55.43	8.04	0.0132	
D ²	46.36	1	46.36	6.72	0.0213	
Residual	96.58	14	6.90			
Lack of Fit	96.33	10	9.63	155.38	<0.0001	¹ significant

Pure Error	0.2480	4	0.0620			
Cor Total	420.35	28				

Factor coding is **Coded**.

Sum of squares is **Type III - Partial**

The **Model F-value** of 3.35 implies the model is significant. There is only a 1.53% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A^2 , B^2 , C^2 , D^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 155.38 implies the Lack of Fit is significant. There is only a 0.01% chance that a Lack of Fit F-value this large could occur due to noise. Significant lack of fit is bad -- we want the model to fit.

Table 5.4 Fit Statistics

Std Dev.	2.63	R²	0.7702
Mean	85.69	Adjusted R²	0.5405
C.V.%	3.07	Predicted R²	0.3210
		Adeq Precision	6.1832

A negative **Predicted R²** implies that the overall mean may be a better predictor of your response than the current model. In some cases, a higher order model may also predict better.

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 6.183 indicates an adequate signal. This model can be used to navigate the design space.

5.9.2 Final Equation In Terms of Actual Factors

Biodiesel Yield = $-430.50 + 27.90111A + 57.38667B + 0.062481C + 0.176370D - 0.416667AB + 0.005556AC + 0.000926AD - 0.002778BC - 0.005556BD - 0.000031CD - 0.519259A^2 - 5.42333B^2 - 0.000361C^2 - 0.000330D^2$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

5.9.3 Validation of Model

It is essential to distinguish between projected (anticipated) and actual responses when using Response Surface Methodology (RSM). Statistical models predict the response, which informs the best values for the input variables. As this is going on, real answers from trials help to develop the model and confirm its predictions. Confirmation of the model's predictive power is achieved by doing validation trials at software-proposed optimal parameters. When the findings of these trials match the predicted reactions, the model's predictions are likely accurate and resilient. By improving decision-making and problem-solving skills, this validation procedure guarantees dependability when using the model in real-world situations. Thus, in addition to streamlining procedures, RSM's dual emphasis on anticipated and actual reactions also strengthens the statistical rigor necessary for successful testing and analysis.

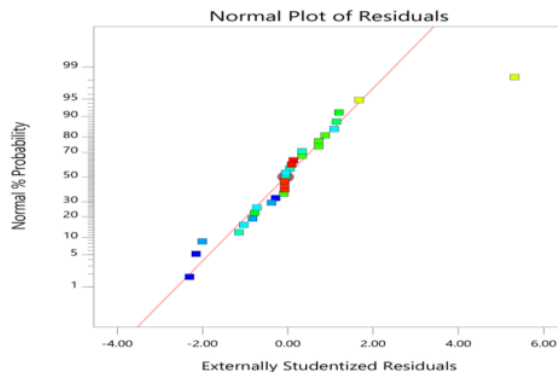


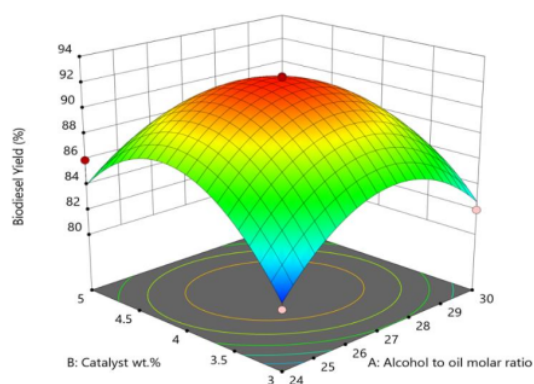
Fig 5.10 Validation of Model

Table 5.5 Report

Actual value	Predicted value	Standard order
80.00	83.25	13
92.00	92.18	28
82.00	85.08	10
86.00	87.33	6
84.00	84.08	11
82.00	82.67	2
83.00	83.08	19
92.00	92.18	29
85.00	83.08	18
87.00	86.42	17
89.00	86.33	7
83.00	84.25	12
92.00	92.18	25
87.00	85.50	8
83.00	81.17	4
83.00	84.75	14
86.00	84.00	3
87.00	85.75	20
92.50	92.18	26
84.00	83.42	23
82.00	83.42	15
89.00	83.75	21
84.00	85.92	9
80.00	83.42	24
92.40	92.18	27
87.00	87.17	5
87.00	85.75	22
80.00	80.50	1
84.00	83.92	16

5.9.4 3-D Surface Plots

When displaying the findings of Response Surface Methodology (RSM) experiments with several input variables, three-dimensional (3-D) graphs are essential. When the number of input variables exceeds two, 3-D graphs show the response variable (biodiesel yield, for instance) as a function of the first two input variables, with the vertical axis representing the third input variable. These graphs give researchers a thorough understanding of how the response variable varies with respect to various combinations of input factors, making it possible to pinpoint ideal circumstances. Researchers may better understand the intricate linkages and interactions among the input elements by visualizing the response surface in three dimensions. This helps to optimize the processes involved in producing biodiesel, resulting in increased output and efficiency.



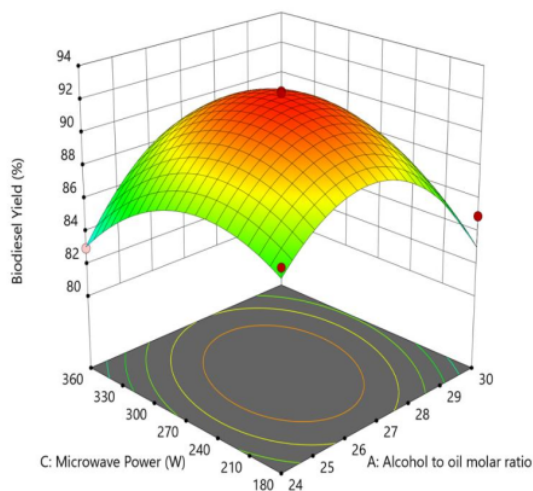
Biodiesel Yield (%)

- Design points above predicted value
- Design points below predicted value

80 92.5

X1 = A: Alcohol to oil molar ratio
X2 = B: Catalyst wt. %

Actual Factors
C: Microwave Power = 270
D: Time = 270



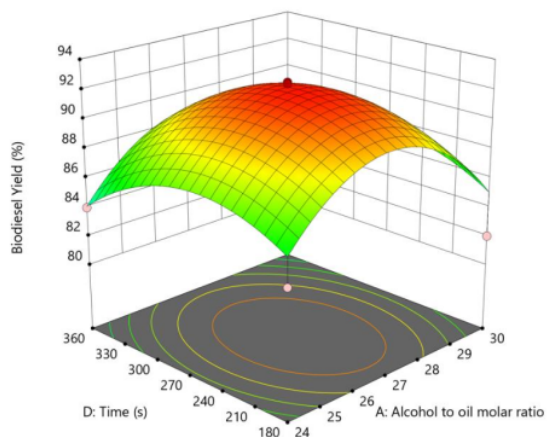
Biodiesel Yield (%)

- Design points above predicted value
- Design points below predicted value

80 92.5

X1 = A: Alcohol to oil molar ratio
X2 = C: Microwave Power

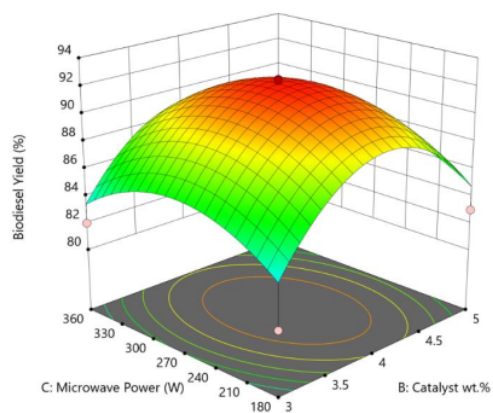
Actual Factors
B: Catalyst wt. % = 4
D: Time = 270



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = A: Alcohol to oil molar ratio
 X2 = D: Time

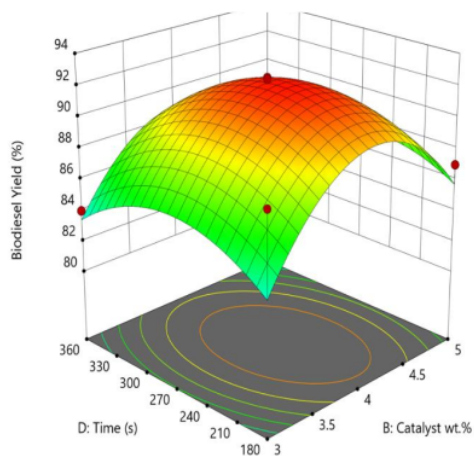
Actual Factors
 B: Catalyst wt.% = 4
 C: Microwave Power = 270



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = B: Catalyst wt.%
 X2 = C: Microwave Power

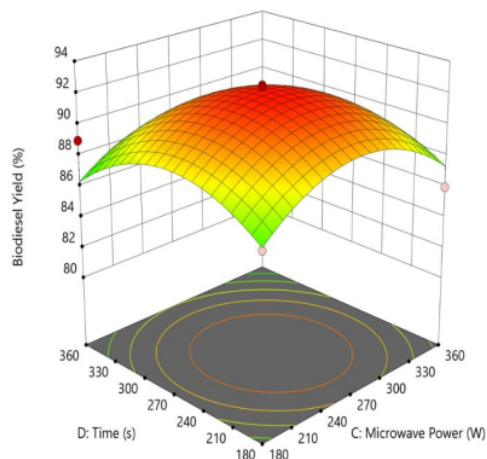
Actual Factors
 A: Alcohol to oil molar ratio = 27
 D: Time = 270



Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = B: Catalyst wt.%
 X2 = D: Time

Actual Factors
 A: Alcohol to oil molar ratio = 27
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Biodiesel Yield (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 80 92.5

X1 = C: Microwave Power
 X2 = D: Time

Actual Factors
 A: Alcohol to oil molar ratio = 27
 B: Catalyst wt.% = 4

Fig 5.11- 3-D Surface Plots

5.10 Properties of Biodiesel

5.10.1 Density

Mass of biodiesel per unit volume is expressed as density (g/cc). Biodiesel has a density of 0.863 g/cc, which is lower than that of water, which normally has a density of 1 g/cc. Reduced density suggests that biodiesel is simpler to handle and carry since it is lighter and less dense than water. Density can also impact engine emissions performance and combustion efficiency; for example, biodiesel with a lower density may burn and atomize fuel more effectively.

5.10.2 Viscosity

Viscosity evaluates a fluid's resistance to flowing. Biodiesel flows more readily than fluids with a greater viscosity, as seen by its comparatively low viscosity of 2.22 mm²/s. Biodiesel with a lower viscosity is preferred because it improves fuel atomization and mixing in engines, resulting in more effective combustion and less wear on the engines. Additionally, it guarantees simpler biodiesel handling and pumping, particularly in colder climates.

5.10.3 Calorific Value

Calorific value, often referred to as heating value or energy content, is the quantity of energy produced upon full combustion of one unit mass of biodiesel. With a high calorific value of 37.3 MJ/kg, burning biodiesel releases a sizable quantity of energy per unit mass. Biodiesel with a higher calorific value produces more energy, which boosts engine power and fuel economy.

5.10.4 Flash Point

The lowest temperature at which biodiesel releases sufficient vapor to catch fire when an ignition source is present is known as the flash point. Biodiesel with a flash point of 168°C is considered to have a moderately high flash point, which is advantageous for handling, storage, and transportation safety. Biodiesel with a higher flash point lowers the possibility of fire risks and unintentional ignition.

5.10.5 Fire Point

The temperature at which biodiesel may continue to burn after it is ignited is known as the "fire point." Biodiesel has a fire point of 188°C, meaning it can burn at

temperatures higher than its flash point. An extra safety margin offered by a higher fire point lowers the possibility of fire dangers during handling and use.

20 5.10.6 Pour Point

The lowest temperature at which biodiesel can still flow and remain fluid is known as the pour point. Biodiesel can still flow at relatively low temperatures, as indicated by its pour point of 3°C, which is advantageous for its use in cold climes. Lower pour point biodiesel ensures continued fuel flow and engine performance even in subfreezing temperatures since it is less prone to gel or freeze in cold weather.

Table 5.6 Properties of Biodiesel

Property	Biodiesel
Density (g/cc)	863
Viscosity (mm ² /s)	2.22
32 Calorific value (MJ/Kg)	37.3
Flash point(°C)	168
Fire point(°C)	188
Pour point(°C)	3

All in all, these attributes point to a high-quality biodiesel that has the qualities that are needed for safe operation of engines, good quality, and adaptability to different environmental settings.

6.CONCLUSION

The biodiesel synthesis from silk-cotton seed oil, methanol, and a Fuller's Earth catalyst represents an efficient and innovative process for producing a sustainable alternative fuel. The step-by-step procedure, which incorporates microwave irradiation and subsequent testing, demonstrates the viability of this method for biodiesel production.

The catalyst preparation involved meticulous steps, from mixing Fuller's Earth and NaOH, to sonication and drying. These steps ensure the catalyst's optimal performance in promoting the transesterification reaction. The resulting biodiesel product exhibited yield of 67% and the conditions were further optimized through RSM to get a higher yield, which meets key quality criteria.

The analysis of both the catalyst and biodiesel through modern testing techniques confirmed the presence of essential chemical groups and methyl esters in the biodiesel, validating its suitability as a fuel. These analytical techniques provide insights into the chemical composition and purity of the catalyst and biodiesel.

In summary, this biodiesel synthesis method, combining microwave irradiation, catalyst preparation, and rigorous testing, offers a rapid and efficient approach to biodiesel production. It underscores the importance of catalyst preparation, quality testing, and process optimization to ensure the viability and sustainability of biodiesel as a renewable fuel source. This research and methodology contribute to the ongoing pursuit of cleaner and more sustainable energy alternatives, decreasing our dependence on fossil fuels and mitigating environment impact.

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