

A Dissertation thesis entitled

“Catalytic conversion of cost effective feedstocks to fuel grade esters using novel solid acid catalyst”

Submitted in partial fulfillment of the requirements for the award of the degree of

Master of Science

IN

INDUSTRIAL CHEMISTRY

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ATMIYA UNIVERSITY

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CERTIFICATE

This is to certify that the dissertation thesis entitled **“Catalytic conversion of cost effective feedstocks to fuel grade esters using novel solid acid catalyst”** is submitted by **MR. BADI MOHAMADAKIL MANJURHUSHEN (Enrolment No: 200722001)**, a Post-Graduate student of the Semester-IV, Department of Industrial Chemistry, Faculty of Science, Atmiya University, Rajkot.

He has undertaken and conducted this dissertation work as a part of the curriculum to earn credits for obtaining the degree of Master of Science (M. Sc.) in Industrial Chemistry during academic year 2022-2023.

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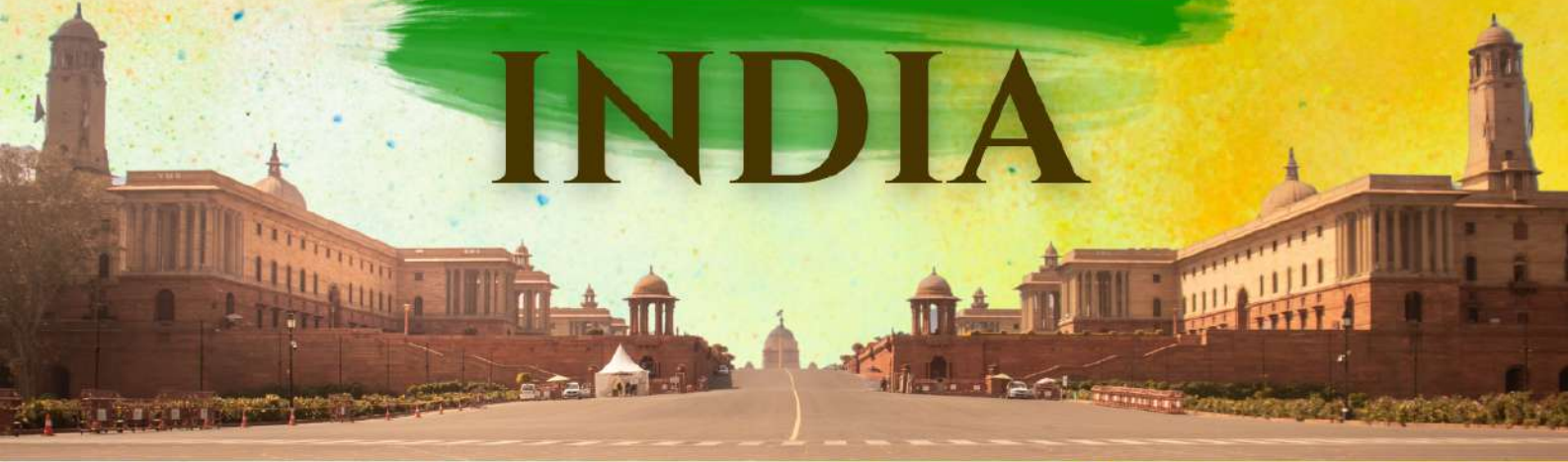
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DEDICATED TO MY NATION



INDIA



**WITHOUT THEIR LOVE, SUPPORT AND
CONSTANT ENCOURAGEMENT
THIS WOULD NOT HAVE BEEN POSSIBLE**



DECLARATION

We undersigned, hereby declare that the work assimilated in the dissertation thesis entitled **“Catalytic conversion of cost effective feedstock to fuel grade esters using novel solid acid catalyst”** has been carried out by us at Faculty of Science, Department of Industrial Chemistry, Atmiya University, Rajkot, Gujarat, India, under the supervision and Guidance of **Dr. Mehul L. Savaliya, Assistant Professor, Faculty of Science, Department of Industrial Chemistry, Atmiya University, Rajkot, Gujarat, India.**

To the best of our knowledge and belief, the work included in this thesis is quite original and has not submitted to any other Institution or University for the award of any degree either in this or any other form.

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ABSTRACT

Biodiesel is a renewable alternative fuel that can be produced from various feedstocks, including vegetable oils and animal fats. Heterogeneous acid catalysts have emerged as a promising alternative to traditional homogeneous catalysts for the synthesis of biodiesel due to their easy separation, reusability, and environmental friendliness. This paper presents an abstract for the synthesis of biodiesel from a heterogeneous acid catalyst. (1) The process involves the transesterification of the feedstock with an alcohol in the presence of a heterogeneous acid catalyst, which results in the production of biodiesel and glycerol as a by-product. The performance of the catalyst is influenced by various factors such as the type of acid, the type of support, and the reaction conditions. The use of heterogeneous acid catalysts for biodiesel synthesis is a promising approach that can lead to a more sustainable and environmentally friendly production process.

Keywords: Biodiesel, Groundnut oil, MNAS-Si catalyst, Transesterification.

1.0 INTRODUCTION

The use of solid acid catalysts in biodiesel production from groundnut oil has several advantages over traditional liquid catalysts. Solid acid catalysts are more efficient, cost-effective, and environmentally friendly than their liquid counterparts. Solid acid catalysts are also easier to separate from the product, can be reused multiple times without losing activity, and do not generate any toxic or corrosive waste. (1)(2)

The performance of solid acid catalysts in biodiesel production depends on several factors, including the type and concentration of the catalyst, reaction temperature, and reaction time. Researchers have studied various types of solid acid catalysts, including zeolites, sulfated metal oxides, and ion-exchange resins. The efficiency and selectivity of the catalyst can be improved by modifying its structure, adding dopants or modifiers, or using a combination of different catalysts.

Several studies have reported high yields of biodiesel using solid acid catalysts. For example, researchers have reported maximum yields of up to 98.5% for biodiesel production from groundnut oil using modified zeolite catalysts. These results demonstrate the potential of solid acid catalysts for the production of biodiesel from groundnut oil.(3)

The use of groundnut oil as a feedstock for biodiesel production is also advantageous. Groundnut oil is readily available and inexpensive, making it a cost-effective feedstock for biodiesel production. (2) Additionally, the use of groundnut oil for biodiesel production reduces the demand for petroleum-based fuels, which are finite and contribute to environmental pollution and climate change.

Overall, the use of solid acid catalysts in biodiesel production from groundnut oil is a promising approach to meet the increasing demand for renewable and sustainable energy sources while reducing our dependence on fossil fuels. The development of new and improved solid acid catalysts will further enhance the performance and efficiency of biodiesel production from groundnut oil. The use of groundnut oil as a feedstock for biodiesel production is also advantageous, as it is readily available and cost-effective. (2)(4)

2.0 LITERATURE REVIEW

2.1 BIODIESEL

Biofuels are alcohols, esters and different natural compounds created from biomass which includes herbaceous and woody plants, residues from agriculture and forestry, and a few agro- industrial wastes which includes waste from the meals industry.(1)

The term biomass refers to any substance which may be received via way of means of photosynthesis. Most plant life make use of sun strength to create sugars, beginning from easy materials along with water and carbon dioxide. This strength is saved in molecules of glucose, starch, oil, etc. Biofuels may want to consist of ethanol, biodiesel, and biomethanol among others. Most advanced and used are bioethanol and biodiesel (Stratta, 2003). The term biomass refers to any substance which may be received via way of means of photosynthesis. Most plant life make use of sun strength to create sugars, beginning from easy materials along with water and carbon dioxide.(4)

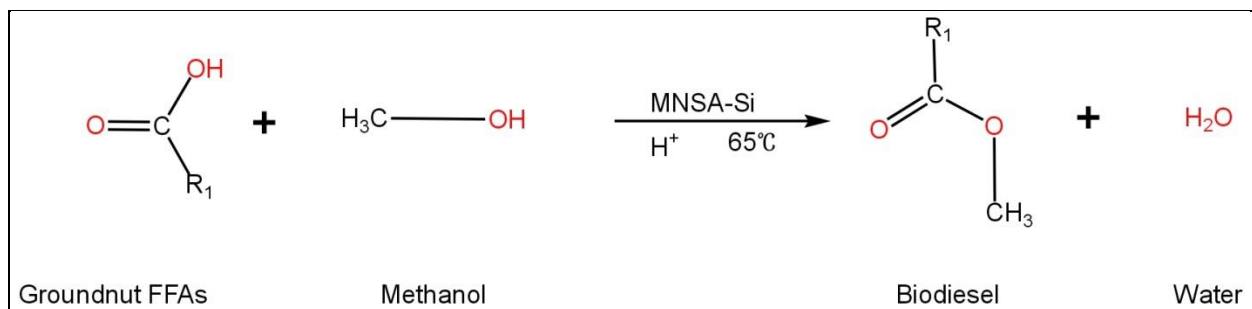


Figure.1 Reaction scheme for the synthesis of Biodiesel

This strength is saved in molecules of glucose, starch, oil, etc. Biofuels may want to consist of ethanol, biodiesel, and biomethanol among others. Most advanced and used are bioethanol and biodiesel (Stratta, 2003).(5)

2.2 ADVANTAGES & DISADVANTAGES IN THE USE OF BIODIESEL FUEL

Advantages:

1. **Renewable and Sustainable:** Biodiesel is made from renewable resources, such as plant oils, animal fats, and waste cooking oil, and can be produced sustainably without depleting non-renewable resources.
2. **Lower Emissions:** Biodiesel emits significantly lower levels of greenhouse gases, particulate matter, and other pollutants compared to traditional diesel fuel, resulting in improved air quality and reduced negative impact on human health and the environment.
3. **Biodegradable:** Biodiesel is biodegradable, meaning it breaks down naturally and quickly in the environment without harming aquatic or terrestrial ecosystems.
4. **Better Lubricity:** Biodiesel has better lubricity than traditional diesel fuel, which can reduce engine wear and prolong the lifespan of engines and equipment.
5. **Economic Benefits:** Biodiesel production can create jobs and support local economies, particularly in rural areas where feedstocks are grown and processed.
6. **Reduced Dependence on Foreign Oil:** Biodiesel production can reduce dependence on foreign oil and improve energy security by using domestically produced feedstocks.
7. **Compatibility with Existing Infrastructure:** Biodiesel can be used in existing diesel engines and infrastructure without requiring significant modifications, making it a convenient and cost-effective alternative fuel option.(6)(7)

Disadvantages:

1. Land Use and Food Security Concerns: Biodiesel production can compete with food crops for land use, potentially leading to higher food prices and food insecurity, particularly in developing countries.
2. Cold Weather Performance Issues: Biodiesel has poor cold weather performance, which can cause fuel gelling, clogging of fuel filters, and other engine issues in cold climates.
3. Variability in Quality: The quality of biodiesel can vary depending on the feedstock source, production process, and other factors, which can affect engine performance and reliability.(6)(7)

2.3 TRANSESTERIFICATION

Biodiesel is commonly received through transesterification of oils or fat through reacting a short-chain alcohol, like methanol, with inside the presence of a heterogeneous acid- catalyst. The maximum common raw substances for manufacturing of biodiesel are vegetable oils from sunflower, soybean, palm oil, groundnut oil and other (Knothe, 2005; Dias, 2008).(8)

The transesterification reaction specifically depends on the character of the feedstock, catalyst concentration, the molar ratio alcohol-oil, temperature, agitationrate, pressure and reaction time in addition to moisture content material and quantity of unfastened fatty acids (Mittelbach, 2004; Canakci, 2001; Dorado, 2002;Ma, 1999).(9)

In the transesterification reaction, one mole of triglyceride is reacted with 3 moles of alcohol to shape one mole of glycerol and 3 moles of the respective fatty acid alkyl ester in a chain of 3 reversible reactions in which the triglycerides are transformed to diglycerides after which to monoglycerides. Glycerol is produced asbyproduct. After the reaction the combination forms two phases: the glycerin- rich-segment and ethyl esters-rich-segment. The phases may be separated via wayof means of decantation or centrifuging.(8)

There are numerous kinds of catalysts for transesterification. The maximum normally used catalyst for manufacturing of biodiesel is the homogeneous catalysis. The homogeneous catalysis used for biodiesel manufacturing may be divided particularly in alkaline and acidic catalysts. Heterogeneous catalysts (solid phase) is likewise used. Another exciting alternative can be to use lipases as enzymatic catalysts (Haas, 2005).(10)

2.4 ACID CATALYSIS

Acid catalysts consist of sulphuric, phosphoric and hydrochloric acid (Helwani, 2009). The acid catalysts are encouraged for oils with a excessive content material of free fatty acids which includes groundnut oil or waste oil (May 2004). The acid catalysis is also used to pre-deal with a few oils as a step previous to the fundamental transesterification. In this example the free fatty acids are esterified till the quantity of FFA is decrease than 0.5 % (Pinzi, 2009). Then, extra methanol and 26 base catalyst are brought to complete the response. However, the response is slow and requires higher temperatures and pressures, and also needs higher quantities of alcohol. (11)(12)

2.5 HETEROGENEOUS CATALYSIS

Heterogeneous catalysts act in a unique phase than the reactants. They do not no longer dissolve in alcohol, oil or biodiesel. They are in solid state and are without difficulty recoverable through decantation or filtration at the stop of the reaction (Helwani, 2009). Transesterification with homogeneous catalysis has offers issues such as formation of soaps and want for purification and disposal of byproducts of the reaction, the remedy of waste effluents and with inside the case of acid catalysts issues associated with corrosion (Meng, 2008). (10)

Using heterogeneous catalysis most effective the evaporation of methanol is needed and no waste effluents are produced, further it's also received natural glycerol (Melero, 2009). However their high price because of the need of increased temperature and pressure conditions as well as massive volumes of

alcohol isn't always still permitting a industrial manufacturing of biodiesel the use of heterogeneous catalysts (Mittelbach, 2004). (12)(13)

2.6 PRESENCE OF FREE FATTY ACIDS (FFAs)

A fatty acid is an natural molecule such as an extended aliphatic chain of an even range of carbon atoms. At the end of the chain there's a carboxyl group. A fatty acid is a carboxylic acid of excessive molecular weight. (14)

The content material of free fatty acids and moisture are key parameters that decidethe feasibility of the system of transesterfication. The content material of free fatty acids determines the catalytic path to be applied for the transesterfication. For a fundamental catalyst the quantity of FFA withinside the oil must be much less than 3% due to the fact the high acidity of the oil reasons a low biodiesel yield (Gold, 2002).(15) When oil has greater than 5% FFA, it can't be transesterified through alkaline catalysis, because of the formation of soaps which inhibit the separation of glycerol and biodiesel. The presence of FFA causes formation of cleaning soap and hampers the separation of the biodiesel and glycerol. Besides, a high quantity of catalyst is consumed and the catalytic performance is significantly reduced. (16)

One trouble of this technique is the presence of water. The accumulation of water produced for the duration of the esterification can stop the reaction earlier than it's far complete.

Using the acid catalysis biodiesel from groundnut oil containing 1-2% FFA has been acquired with an efficient conversion of 99% after 26 hours (Goff, 2004). For the transesterfication response at 100°C of dewaxed/degummed rice brain oil containing 24.5% and 49.5% FFA with methanol the reached conversion turned into 62% and 73% respectively (Zullaiker et al., 2005). An increase in the quantity of methanol or catalyst has a negligible impact on conversion. The water generated for the duration of the method forms any other phase which absorbs methanol and sulphuric acid. This water phase possibly prevents the

transesterification of triglycerides and similarly esterification of residual FFA (Goff, 2004). (14)(17)

The reaction of the transesterification of FFA, acid catalysis may be used. The transesterification method can be performed in two stages (Zullaikah, 2005). In step one FFA is transformed to alkyl esters through acid catalysis and within the 2nd step the transesterification method is finished through basic catalysis. (17)

2.7 ALCOHOLS

The alcohols which can be historically used in the transesterification are of short-chain, especially methanol (Rashid, 2008) and ethanol (Alamu, 2008). Other alcohols may be used consisting of propanol, isopropanol, butanol and pentanol (Encinar, 2002; Ma, 1999; Georgogianni, 2008), (18) (19) however those alcohols are a lot greater sensitive to water contamination. They also are greater high-priced and large molar ratios and higher temperatures are required. Methanol has a lower price and provides some chemical and physical benefits over different alcohols, because the high charge of response with triglycerides. (18) It is important to say the high toxicity of methanol, flammability, and the reality that methanol is specially constructed from nonrenewable fossil fuels such as methane gas. (20)

Ethanol, however, comes from renewable raw materials: sugar cane, sugar beet, greens and different cellulosic (Girard, 2006). (21) So ethanol is of specific interest particularly due to the fact it's far much less high-priced than methanol in a few regions (Moser, 2009). (19) It is also feasible to obtain through dry distillation of wood. However this method isn't relevant to huge scale. However, the ethyl ester formation is relatively tougher than that of methyl ester. The formation of a stable emulsion for the duration of the ethanolysis is a problem (Zhou, 2006). (22)

Ethanol and methanol do now no longer dissolve with the triglycerides at room temperature and the mixture have to be agitated automatically to allow

diffusion. During the reaction, typically an emulsion is formed. In the methanolysis this emulsion breaks down rapidly forming a decrease glycerol-rich layer and an higher layer rich in methyl ester. However in ethanolysis this emulsion is extra stable and complicates the separation and purification of the ethyl esters. The emulsion is precipitated partially through the formation of monoglycerides and diglycerides intermediates containing each polar hydroxyl groups and non-polar hydrocarbon chains.(21) (22) (23)

The stoichiometric ratio for transesterification requires three moles of alcohol and one mole of triglyceride to supply three moles of esters and one mole of glycerol. The transesterification is an equilibrium reaction requiring an extra of alcohol to drive the reaction to the right. The extra of methanol can be recovered after the reaction (Enweremadu, 2009). For maximum conversion it's far recommended to apply a molar ratio of 6:1. However this ratio will rely on the kind of feedstock used. An excessive molar ratio alcohol/oil impacts the separation of glycerol because of accelerated solubility. When the glycerol is maintained in the solution the opposite reaction takes place. The glycerol can react returned into a monoglycerides and diglycerides. The product will include monoglycerides and diglycerides, which crystallize very easily withinside the biodiesel and may cause clogging of filters and different engine problems (Agarwal, 2007; Knothe, 2005, Van Gerpen, 2005; Dorado, 2002).(24)

2.8 TEMPERATURE AND PRESSURE

The transesterification may be carried out at atmospheric pressure and at distinct temperatures depending on the used feedstock used. The temperature impacts the yield of received biodiesel (Ma, 1999; Meher, 2006). Often the transesterification is finished at a temperature close to the boiling point of alcohol (at 60 ° C while the usage of methanol) (Fukuda, 2001).

Biodiesel also can be produced through methods at high pressures (eg 50- a

hundred bar) and high temperatures (200-250 ° C) the use of huge quantities of methanol and without pretreatment of the raw material. Methods the usage of methanol at supercritical situations at 350 ° C and 43 MPa had been also used. However, the prices of these methods are high (Pinzi, 2009, Mittelbach, 2004). (23) (24)

2.9 REACTION TIME

Freedman et al (1986) found the increase in fatty acid esters conversion when there's an increase in reaction time. The reaction is slow at the start due to mixing and dispersion of alcohol and oil. After that the reaction proceeds very fast. However the most ester conversion became completed within < 8hr similarly increase in reaction time does now no longer increase the yield product i.e. biodiesel/mono alkyl ester (Leung and Guo, 2006; Alamuet al., 2007). (19) Besides, longer reaction time results in the reduction of end product (biodiesel) because of the reversible reaction of transesterification resulting in lack of esters in addition to cleaning soap formation (Eeveraetal., 2009; Ma et al., 1998).(20)

2.10 PHASE SEPARATION

After the transesterification reaction, the reaction mixture (combination) is separated in two phases. The lower phase, the glycerol phase, may be separated through decantation or centrifugation.

Some ways to promote phase separation, mentioned by Mittelbach, are (Mittelbach, 2004):

- 1) The addition of water, glycerol more or hexane.
- 2) Cool down the mixture.
- 3) Application of excessive voltage to facilitate separation of the polar phase (glycerol and residues of alcohol and catalyst) from the non-polar phase (esters and alcohol).(12)

2.11 PURIFICATION OF BIODIESEL

After separation of phases, each the biodiesel and glycerol phase must be washed and purified. In the industry (enterprise) the methanol is recovered through heating the ester phase. Glycerol and traces of catalyst may be eliminated from the biodiesel segment through washing with acidified water with a purpose to neutralize the traces of catalyst. Free fatty acids may be recovered from the ester phase through distillation, the usage of the reality that FAME generally distilled at 30-50 °C lower than FFA. However this method will increase considerably the costs (Mittelbach, 2004; Van Gerpen, 2005; Enweremadu, 2009). (13)

Solid adsorbents have proven success with inside the elimination of impurities from biodiesel. Some used adsorbents are magnesium silicate and silica gel (Predojevic, 2008). Ion exchange resins had been also examined for purification of biodiesel. (12)

3.0 MATERIALS AND METHOD

3.1 MATERIALS

Groundnut oil was purchased from Giriraj Agency, Pushkardham chowk, Rajkot, Gujarat, India. Anhydrous dichloromethane (99% purity), were supplied by Pallav chemical Pvt. Ltd, Tarapur, Boisar, India. Methanol (AR grade) was purchased from a vantor performance material india limited. Chlorosulfonic acid, dichloromethane and silica gel (99% purity) were supplied by Qualikems Fine Chem Pvt. Ltd., Nandesari, Vadodara. Methanol less crude glycerol was produced using current biodiesel synthesis procedure.



Figure.2 Ground nut oil, DCM & Silica Gel

3.2 ANALYTICAL INSTRUMENTAL METHODS

The FT-IR analyses of fresh and regenerated MNAS-Si had been done on a FT-IR spectrophotometer. However, the FT-IR analysis of biodiesel was achieved on a FT-IR spectrophotometer. ¹H-NMR analysis was achieved on a FT- NMR spectrometer. Surface and structural morphologies of the fresh and regenerated MNAS-Si catalysts were predicted through scanning electron microscope. The overall acidity of the MNAS-Si was measured through neutralization titration. The biodiesel (%) yield was predicted through gas chromatography analysis. element FAME mixture was employed as a standard for recognition and determination of the peaks retrieved with inside the WF based biodiesel in gas chromatography analysis.

4.0 EXPERIMENTAL PROCEDURES

4.1 PREPARATION OF MNAS-Si CATALYST

A 250 ml flask (FBF) was charged with m-Nitroaniline and sulfurochloridic acid (13.56 ml) was charged drop wise. The m-Nitroaniline (26.28 g) was charged in little fractions over a t1 lime slot of 45 min at ambient conditions under nitrogen atmosphere (g) and constant stirring. As a result, the hydrochloric acid (g) was elaborated from the reaction flask immediately. The reaction mixture was rattled for 45 min and the remaining side product hydrochloric acid (g) was removed by vacuum. The mixture was triturated with dichloromethane (15 ml) and then filtered. The powdery residue was again treated with dichloromethane (15 ml) and dried under vacuum oven. The m-nitroaniline sulfonic acid (MNAS 40 g, 90%) was obtained as an off-white powder. Then after, 30 g silica gel (200-400mesh) was mixed with off-white MNAS and stirred for 30 min for the formation of an intermolecular hydrogen bond to support the MNAS-Si. At last, a dried and white powder material composed of MNAS-Si was obtained (75 g). The scheme for synthesis of MNAS-Si is given in fig. 3.

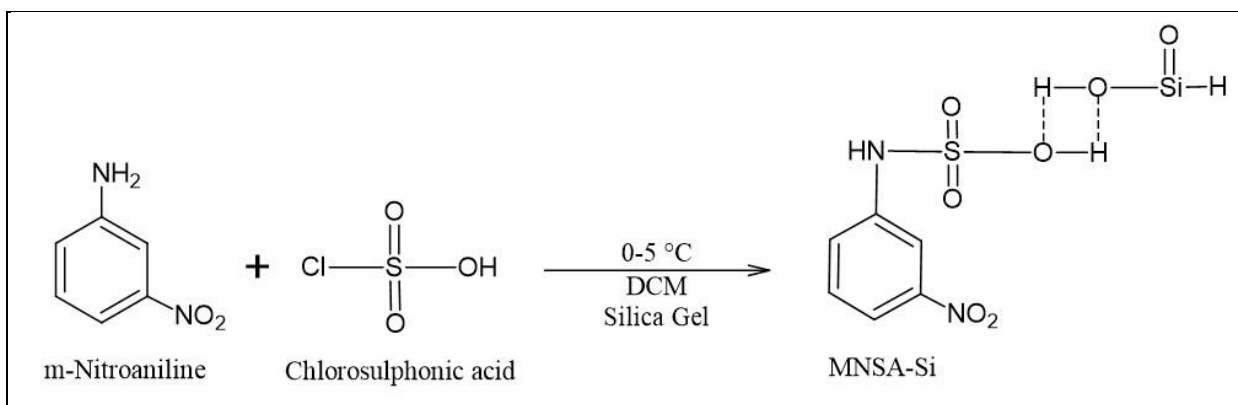


Figure.3 Reaction scheme for the synthesis of MNAS-Si



Figure.4 Preparation of MNAS-Si catalyst

4.2 PREPARATION OF BIODIESEL

The experimental part of the work includes the production of biodiesel from Groundnut oil. Methanol has been used in the transesterification. MNAS-Si has been selected as catalyst. The reaction takes place in an flat bottom flask on magnetic stirrer in a continuous process. The flask is placed on magnetic stirrer maintained at a desired temperature by one electrical heater.

The alcohol (methanol or ethanol) and catalyst MNAS-Si are mixed preferably at the temperature close to the main reaction temperature to avoid significant temperature drop in reactor. Groundnut oil (which also is maintained at the constant temperature) is added to the mixture of alcohol-catalyst. After reaction finished the product is poured into a funnel to obtain a separated two phases. Upper phase is biodiesel and downer is glycerol.

The influence of ratio alcohol to oil, the amount of catalyst, the reaction time, the rate of mixing, the reaction temperature on the yield of produced biodiesel is studied.(23)(24)

The performance of the experiments includes the following steps:

- Measure quickly the catalysts MNAS-Si on an electronic scale.
- Mix the alcohol and the catalysts in a flat bottom flask under stirring to ensure complete solution.
- The groundnut oil is preheated to the reaction temperature in a electric heater. Then the oil is added to the alcohol/catalyst solution in the flask (FBF). The reaction is maintained for 6-10 hours under stirring at the set temperature (65 °C) to get complete conversion.
- The solution is poured into a separation funnel since the reaction is completed, i.e. two large phases are formed. The biodiesel product at the top and the by-product glycerol in the bottom of the funnel.
- Biodiesel and glycerol have different specific gravities and separated using gravity decanter. The biodiesel was washed with hot water till no more alkali was found. Recovered alkali could be recycled while glycerol was the byproduct of the process.(22)(25)(26)



Figure.7 Biodiesel reaction process and gravitational Separation of biodiesel, glycerol & other unreacted components

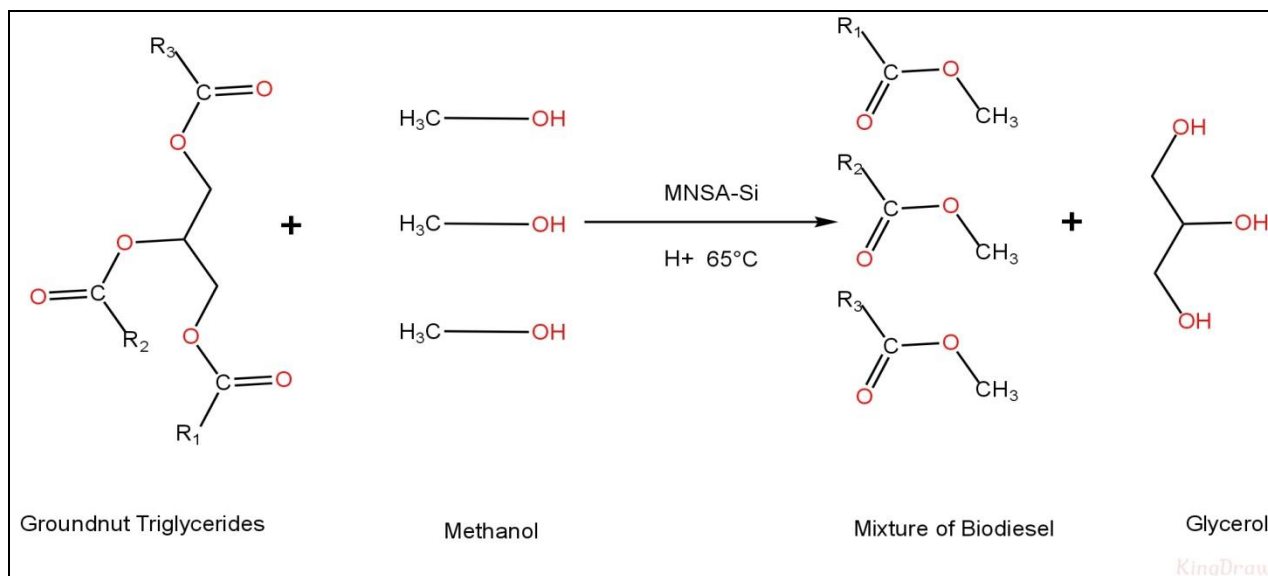


Figure.5 Reaction scheme via trans-esterification

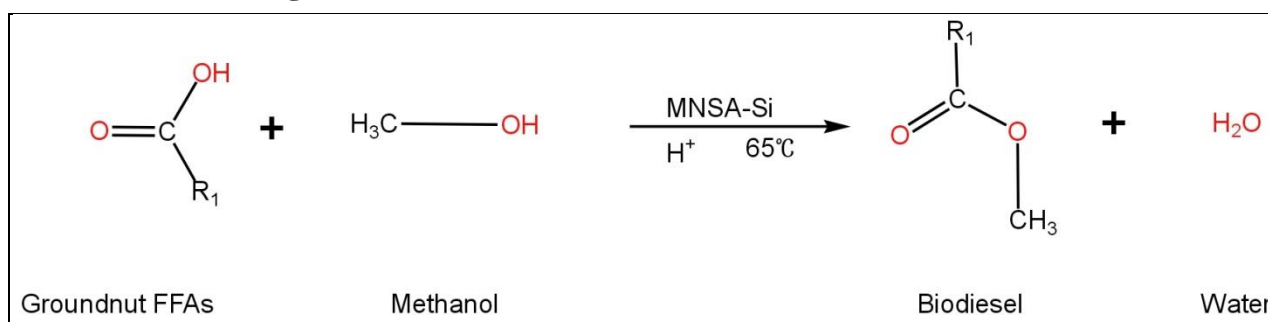


Figure.6 Reaction scheme via esterification

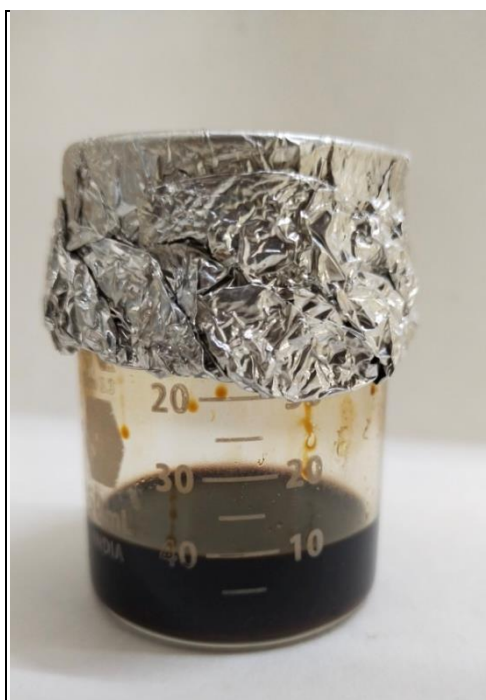


Figure.7 Biodiesel product

4.3 TRANSESTERIFICATION REACTION

Transesterification is the process where free fatty acids/TAG reacts with alcohol in the presence of a catalyst for methyl ester (biodiesel) production.

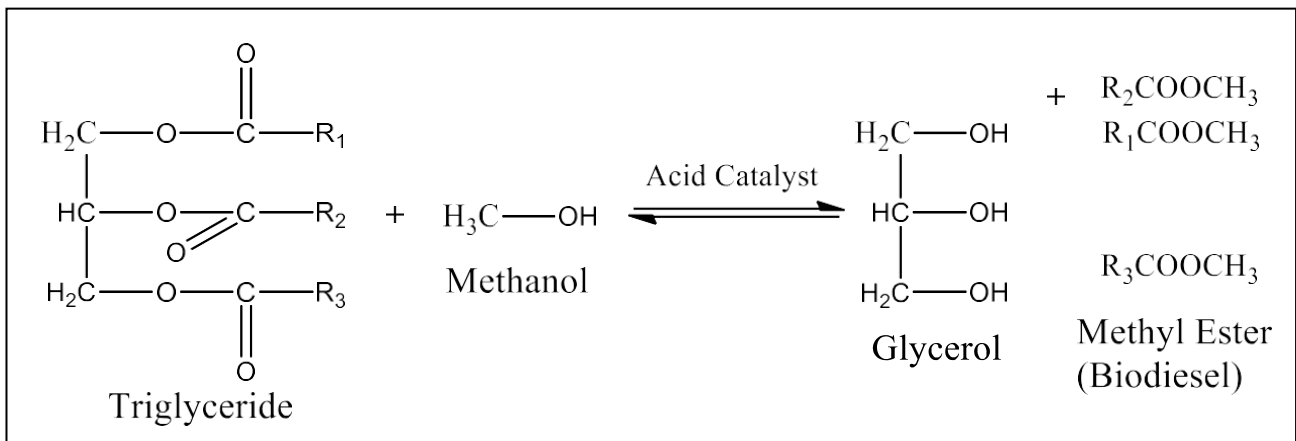
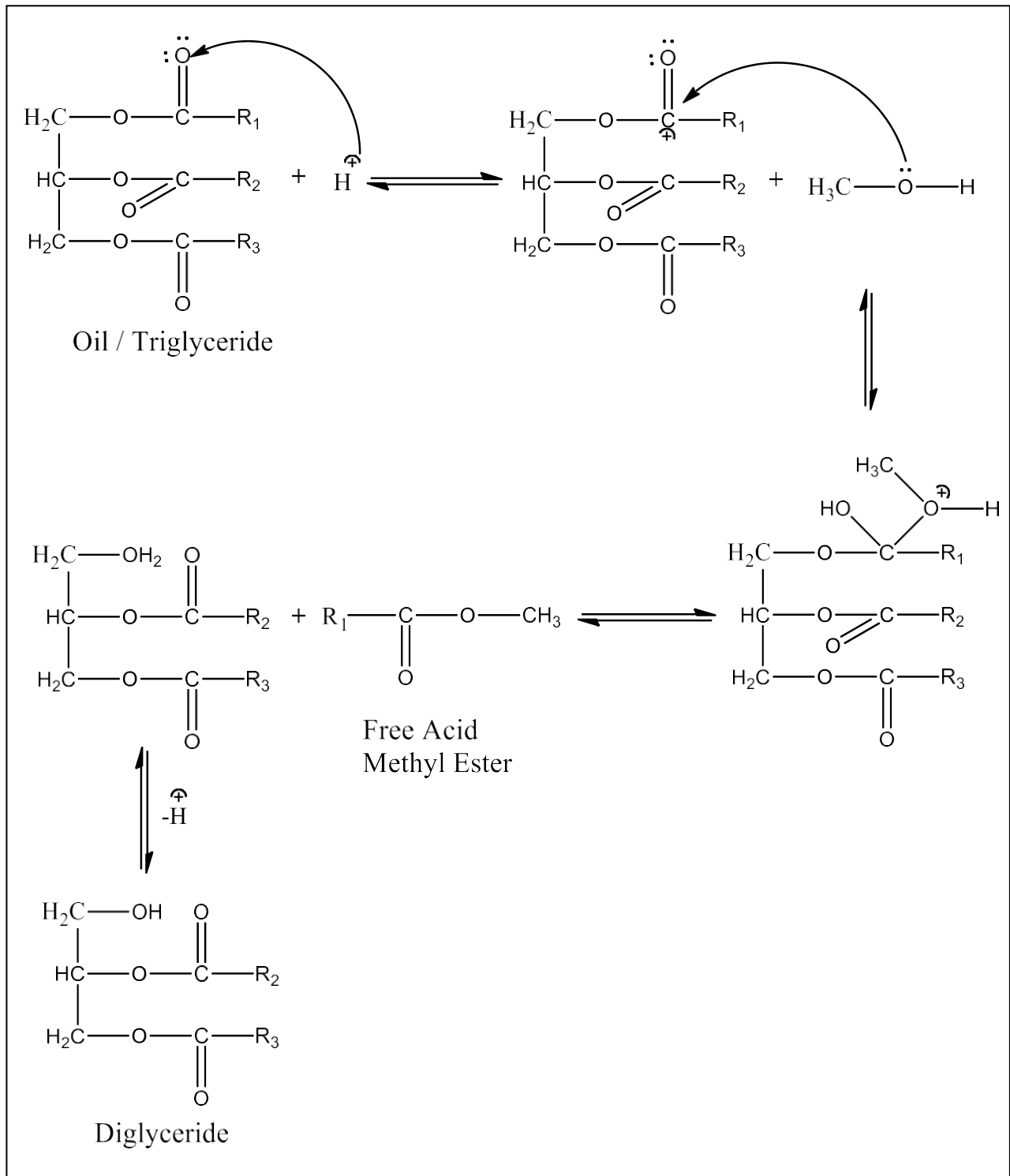


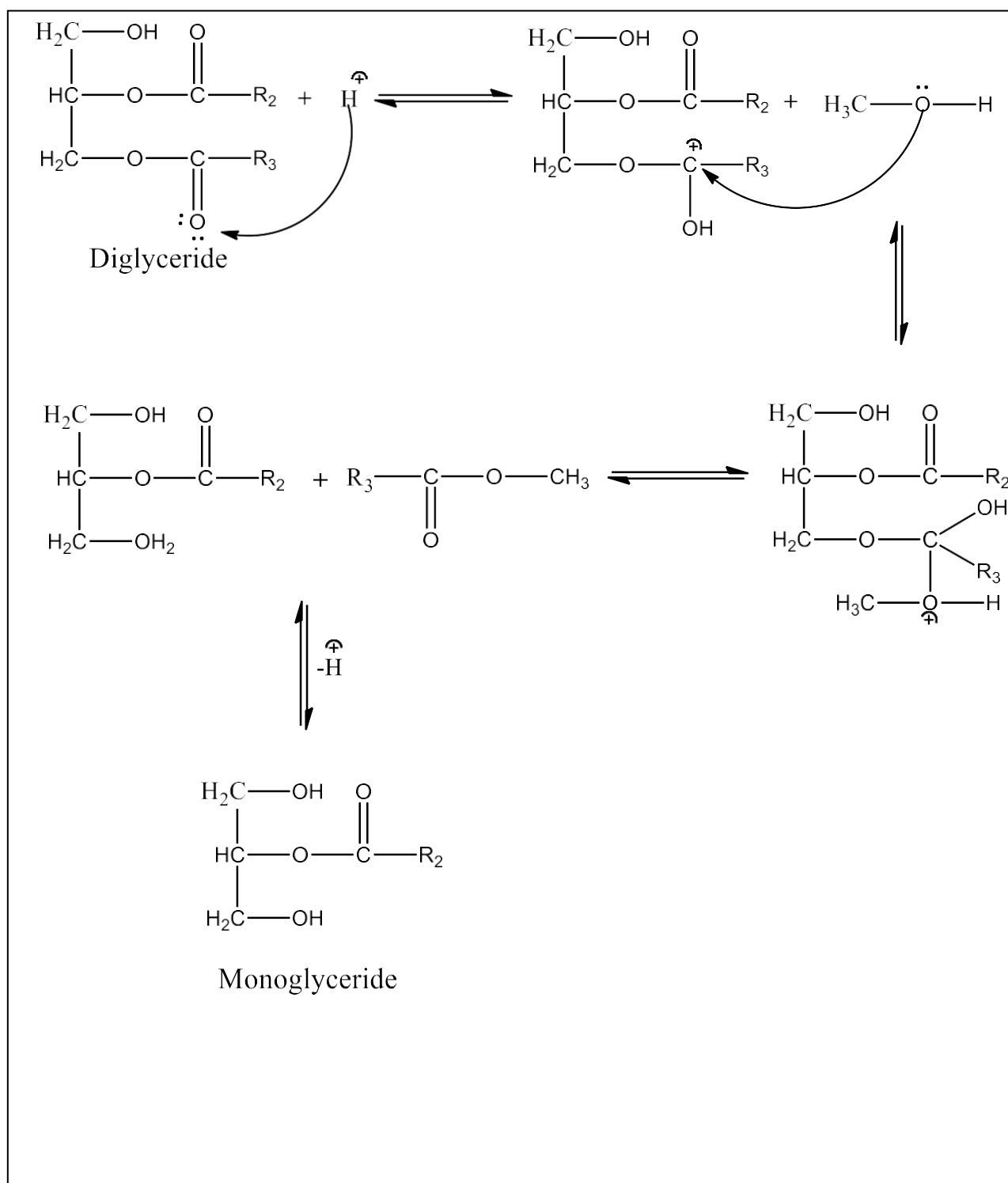
Figure.8 Transesterification Reaction

4.4 MECHANISM

STEP-1:



STEP-2:



STEP-3:

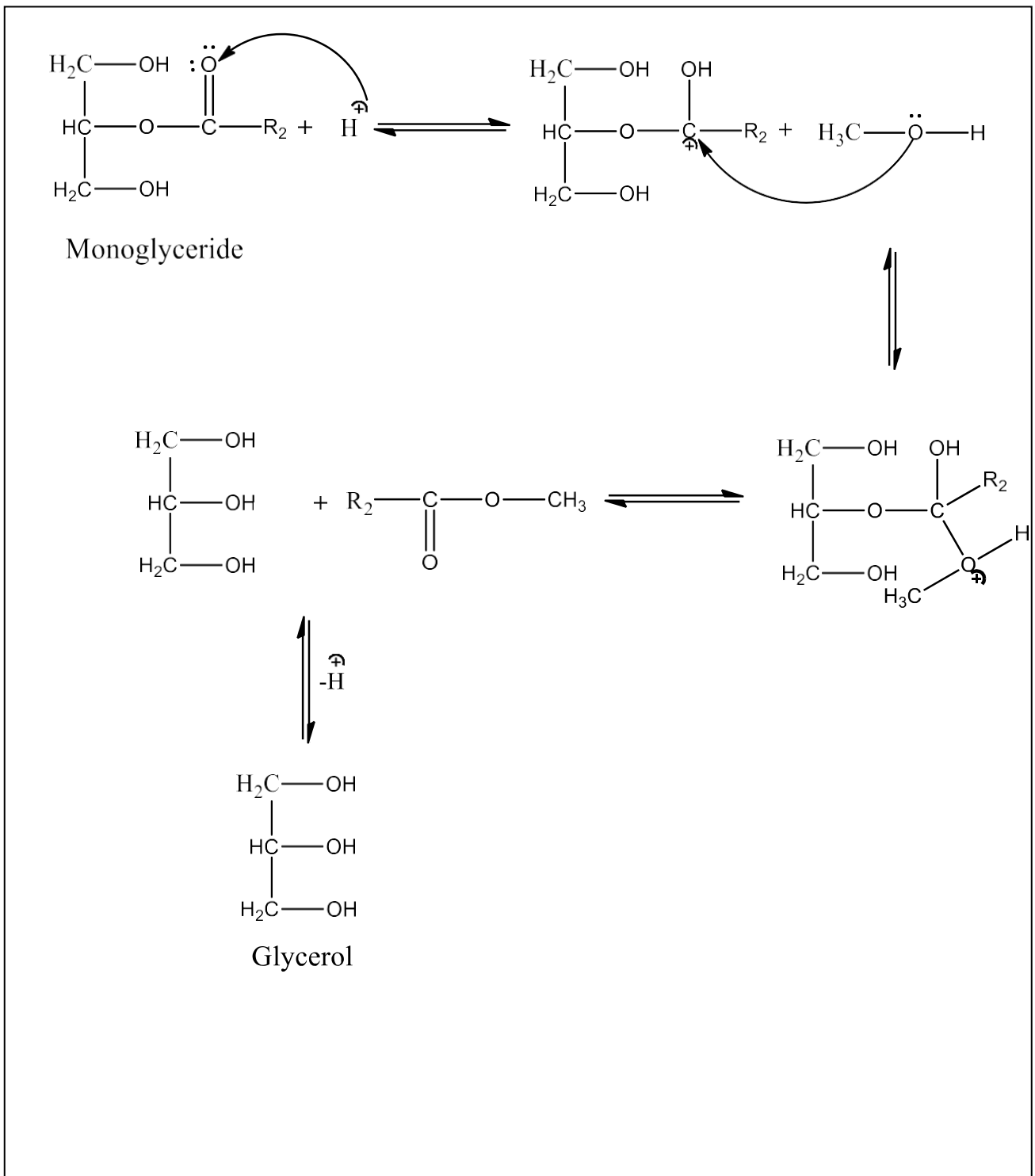
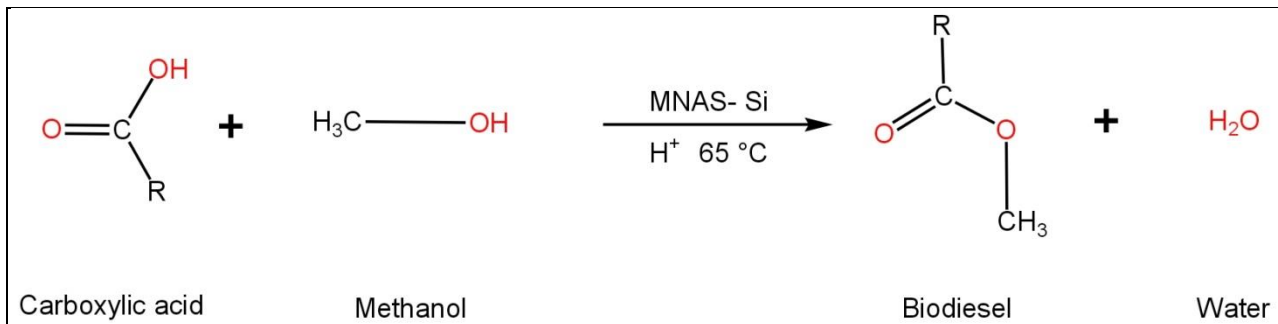


Figure.8 Mechanism of Transesterification Reaction

5.0 ESTERIFICATION

Esterification is reaction wherein reactant (alcohol and acid) form an ester as a product. Ester is common in organic materials.

5.1 ESTERIFICATION REACTION



5.2 MECHANISM

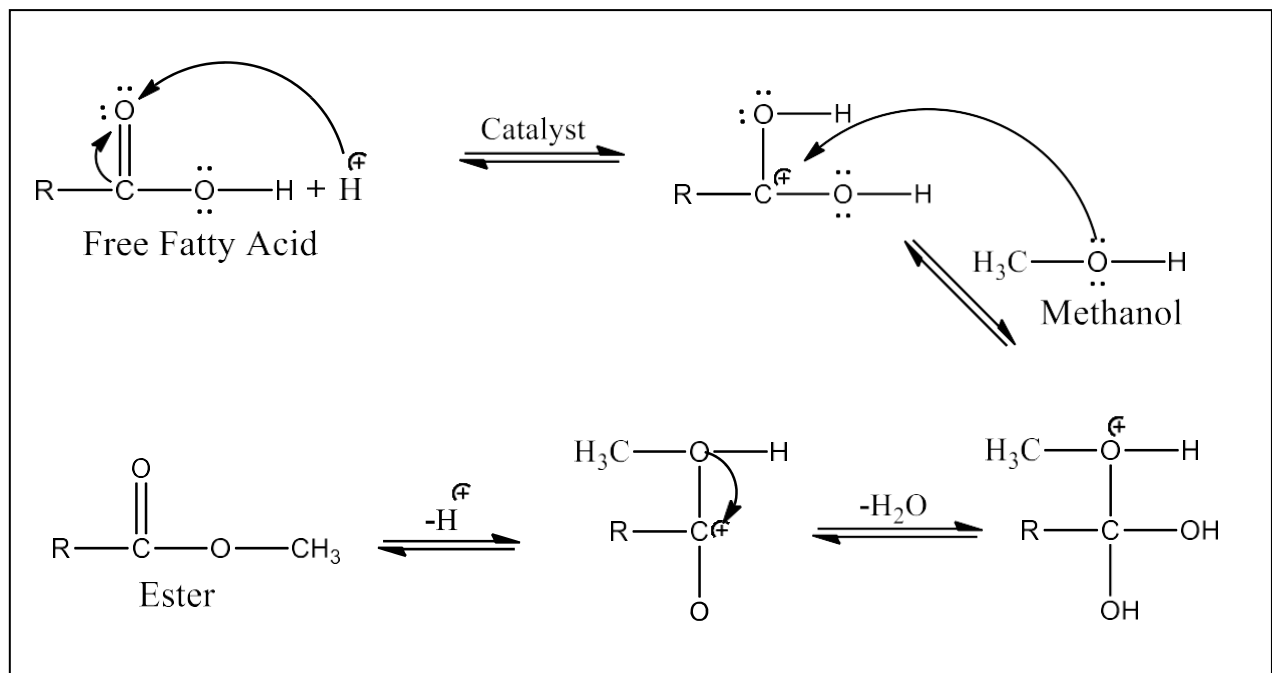


Figure.8 Esterification Reaction & Mechanism

6.0 BIODIESEL YIELD ESTIMATION (%)

The biodiesel yield (%) has been calculated using acid value estimation of groundnut acid oil and biodiesel as given in equation.

$$\% \text{ FFA Conversion (ppm)} = \frac{AV_i - AV_f}{AV_i} \times 100$$

Where: FFA = Free Fatty Acid

AV_i = Acid value initial

AV_f = Acid value final

7.0 RESULT DISCUSSION

Table-1: Result of % Yield of Biodiesel with varying reaction parameters

Sr. No.	O:M Molar ratio	MNAS-Si %(w/w)	Reaction Temp. (°C)	Reaction Time (t)	%Yield
1.	1:9	5	65	6	75.89%
2.	1:9	5	65	7	77.19%
3.	1:9	5	65	8	79.53%
4.	1:9	5	65	9	80.90%
5.	1:9	5	65	10	82.46%
6.	1:10	5	65	6	84.83%
7.	1:10	5	65	7	85.66%
8.	1:10	5	65	8	86.00%
9.	1:10	5	65	9	87.58%
10.	1:10	5	65	10	88.69%
11.	1:11	5	65	6	89.44%
12.	1:11	5	65	7	90.05%
13.	1:11	5	65	8	91.74%
14.	1:11	5	65	9	92.06%
15.	1:11	5	65	10	91.05%
16.	1:12	5	65	6	88.13%
17.	1:12	5	65	7	87.20%
18.	1:12	5	65	8	88.25%
19.	1:12	5	65	9	84.36%
20.	1:12	5	65	10	81.63%
21.	1:11	1	65	10	79.47%

22.	1:11	2	65	10	78.49%
23.	1:11	3	65	10	78.96%
24.	1:11	4	65	10	76.19%
25.	1:11	6	65	10	75.28%

It has been found from the experimental result; the 82.46% biodiesel yield has been obtained with 1:9 oil molar ratio, 5% (w/w) catalyst, 65°C reaction temperature and 10hr reaction time. However, it has also been obtained from the experimental result, the 88.69% biodiesel yield has been obtained with 1:10 oil molar ratio, 5% (w/w) catalyst, 65°C reaction temperature and 10hr reaction timer. However, It has been found from the experimental result; the 92.06% biodiesel yield has been obtained with 1:11 oil molar ratio, 5% (w/w) catalyst, 65°C reaction temperature and 9hr reaction time. It has been found from the experimental result; the 79.47% biodiesel yield has been obtained with 1:11 oil molar ratio, 1% (w/w) catalyst, 65°C reaction temperature and 10hr reaction time. It has been found from the experimental result; the 78.49% biodiesel yield has been obtained with 1:11 oil molar ratio, 2% (w/w) catalyst, 65°C reaction temperature and 10hr reaction time. It has been found from the experimental result; the 78.96% biodiesel yield has been obtained with 1:11 oil molar ratio, 3% (w/w) catalyst, 65°C reaction temperature and 10hr reaction time. It has been found from the experimental result; the 76.19% biodiesel yield has been obtained with 1:11 oil molar ratio, 4% (w/w) catalyst, 65°C reaction temperature and 10hr reaction time. It has been found from the experimental result; the 75.28% biodiesel yield has been obtained with 1:11 oil molar ratio, 6% (w/w) catalyst, 65°C reaction temperature and 10hr reaction time.

8.0 CONFORMATIONAL METHOD

8.1 CLASSICAL METHOD

It is conformed on the basis of various properties like viscosity, density, boiling point, etc.

Table 2: Some properties of Groundnut Biodiesel

Properties	Biodiesel
Density (kg/m ³)	922
Specific gravity	0.89
Cloud point (°C)	7.0
Pour point (°C)	-2
Acid value (mg KOH/gm)	0.45
Flash point (°C)	167

8.2 FLAME TEST



Figure.11 Flame Test of Biodiesel

For the conformation of Biodiesel product synthesized or not, flame test is used. Transfer some portion of biodiesel into the aluminum foil/ petri dish and ignite it using matchstick. If it starts continuously burning and then we can say biodiesel are synthesized.

9.0 SPECTRAL CHARACTERIZATION

• FT-IR ANALYSIS OF GROUNDNUT OIL:

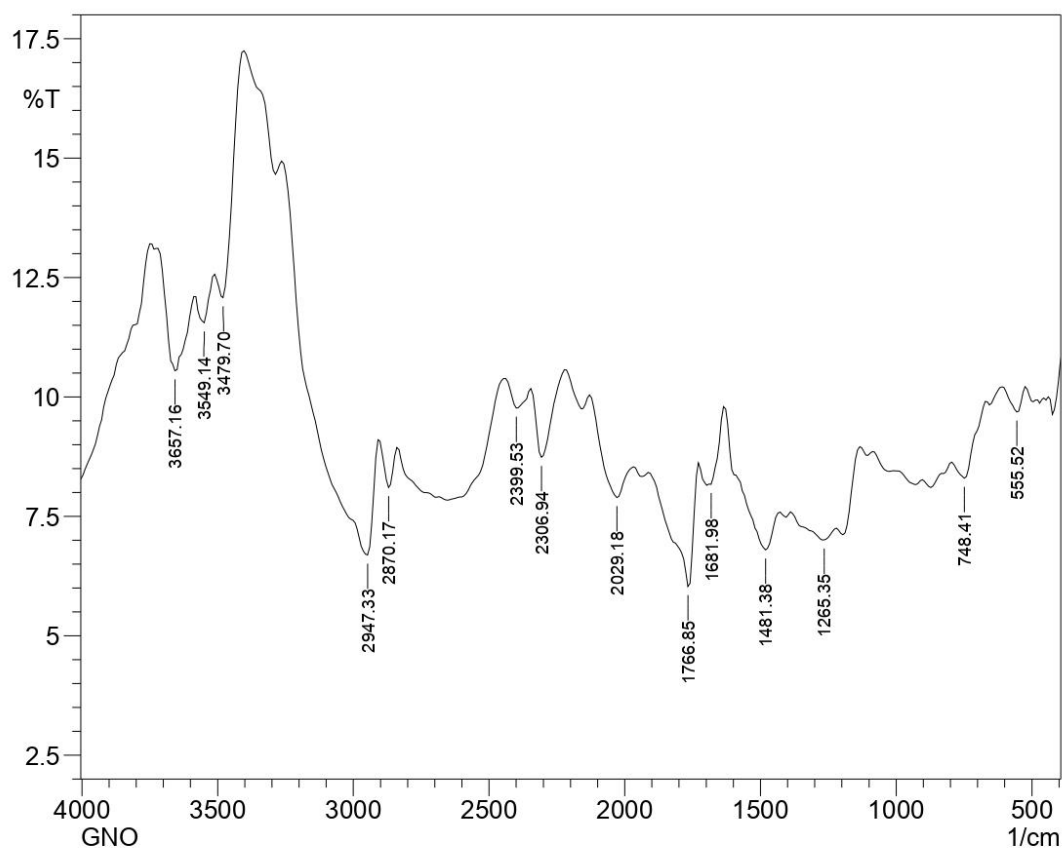


Figure.12 FT-IR of Groundnut acid oil

FT IR Spectrum of Groundnut oil is confirmed with the presence of 3657.16 cm⁻¹ (-OH stretching), 3549.14 cm⁻¹ (N-H stretching) 2947.33 cm⁻¹ (C-H stretching), 2870.17 cm⁻¹ (CH₂ stretching), 1766.86 cm⁻¹ (C=O) 1265.35 cm⁻¹ (CH bending) and 748.41 cm⁻¹ (CH rocking) respectively.

- **FT-IR ANALYSIS OF SYNTHESIZED GROUNDNUT BIODIESEL:**

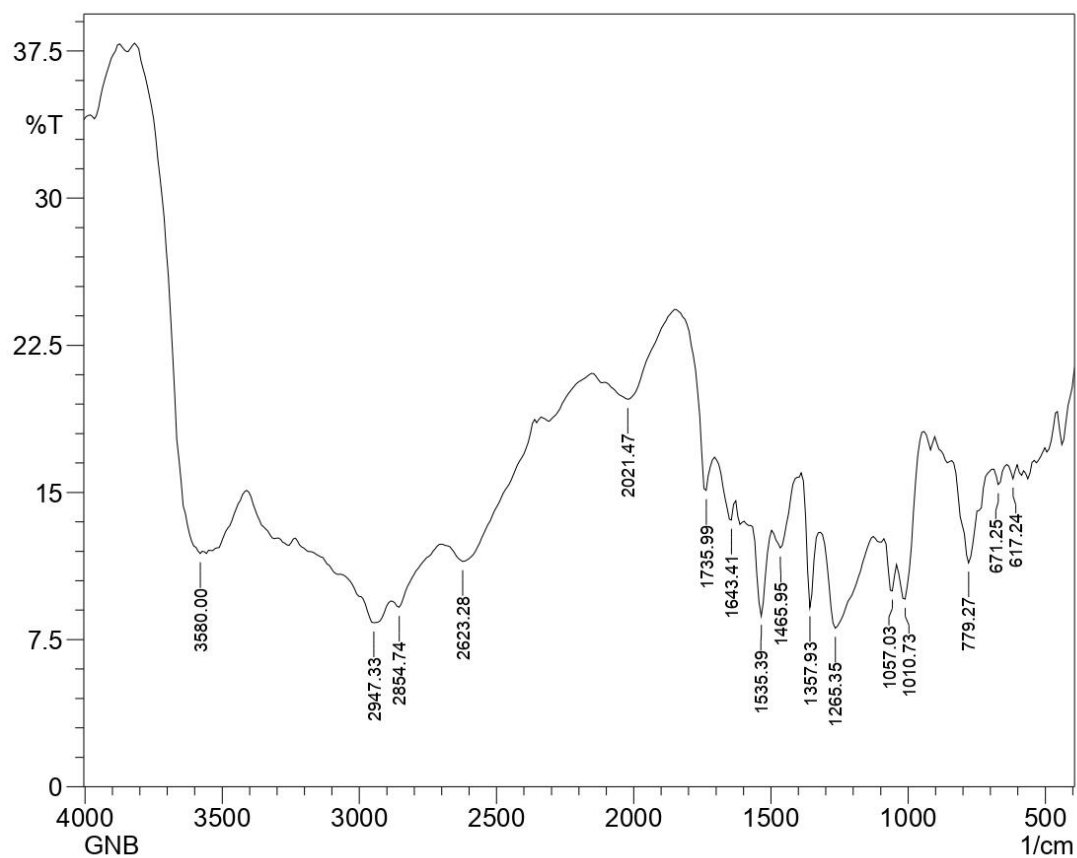


Figure.13 FT-IR of Groundnut biodiesel

FT-IR spectrum of synthesized Groundnut biodiesel is confirmed with the presence of 3580.00 cm⁻¹(O-H stretching) 2947.33 cm⁻¹ (CH₃ stretching), 2854.74 cm⁻¹ (CH₂ stretching), 1735.99 cm⁻¹ (C=O) ,1465.95 cm⁻¹ (CH bending), 1010.73 cm⁻¹ (C-O) & 779.27 cm⁻¹ (CH - rocking) respectively.

- **$^1\text{H-NMR}$ ANALYSIS OF GROUNDNUT OIL:**

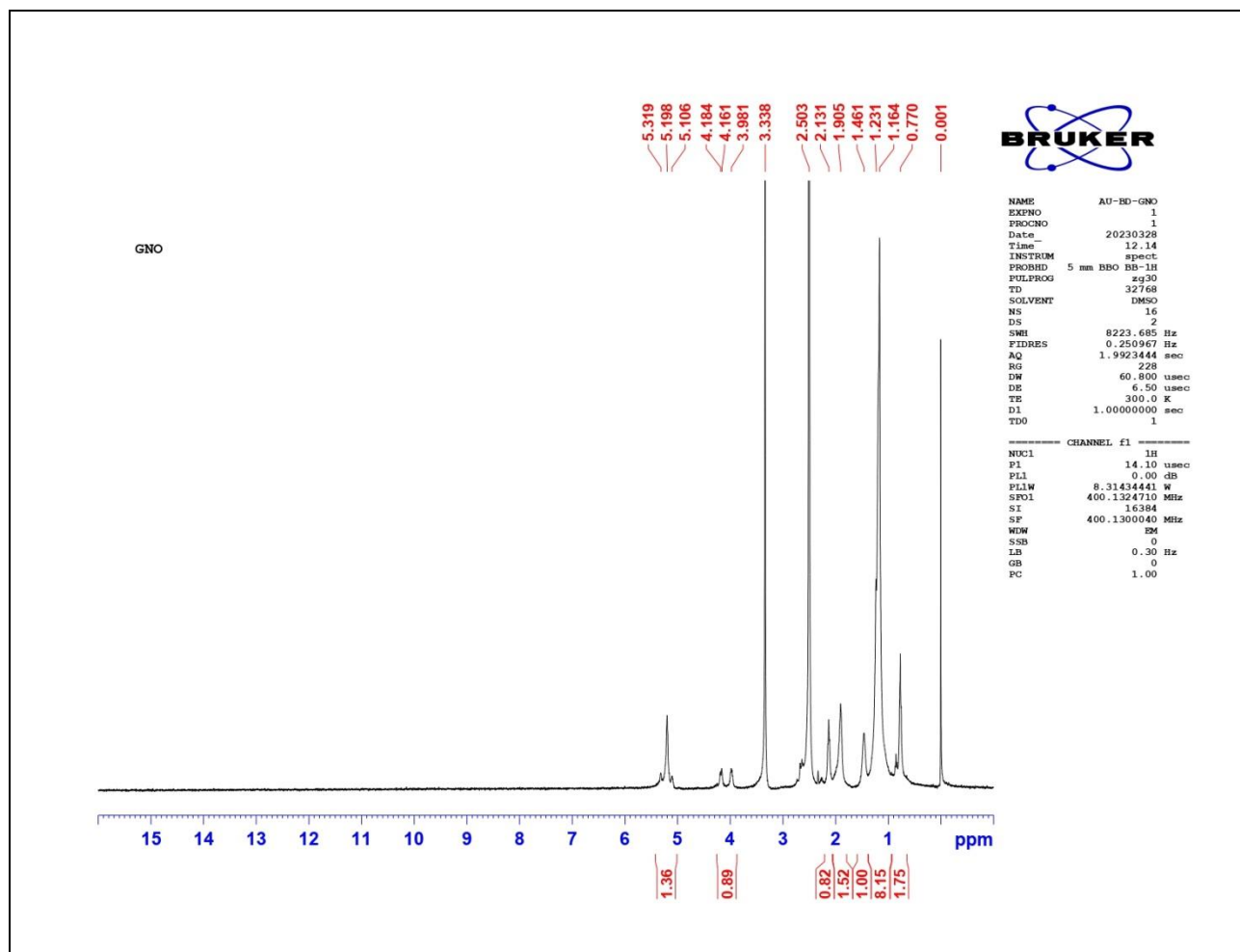


Figure.14 $^1\text{H-NMR}$ of Groundnut oil

The $^1\text{H-NMR}$ is a promising spectroscopic approach conventionally employed for structural assurance of organic molecules in synthetic organic chemistry. The structure of groundnut oil was accustomed by an essential peaks at 5.10-5.31 ppm ($-\text{CH}=\text{CH}$), 3.98-4.14 ppm ($\text{X}-\text{C}-\text{H}$), 3.33 ppm ($-\text{O}-\text{CH}_3$), 2.50 ppm ($\text{Ar}-\text{CH}_3$), 1.31-1.90 ppm (allylic compounds) and 1.16-1.46 ppm ($-\text{CH}_3$) commonly.

- **¹H-NMR ANALYSIS OF SYNTHESIZED GROUNDNUT BIODIESEL:**

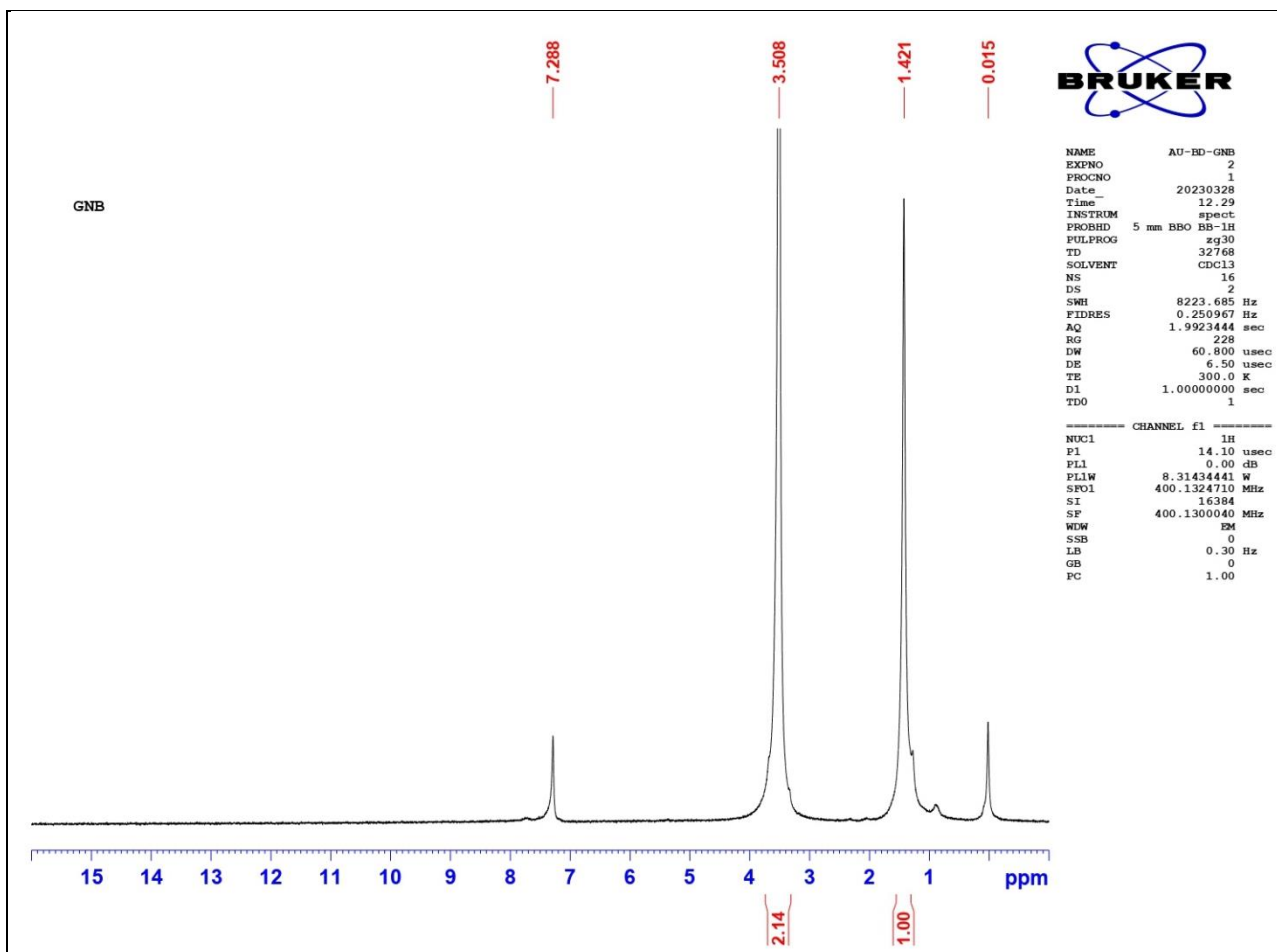


Figure.15 ¹H-NMR of Groundnut biodiesel

The structure of groundnut acid oil biodiesel was accustomed by the presence of an essential peaks at 7.28 ppm (CDCl₃), 3.50 ppm (CH₃O-) and 1.42 ppm (saturated alkane) commonly.

10.0 PHYSICAL PROPERTIES OF BIODIESEL

Table 2: Some properties of Groundnut oil Biodiesel

Properties	Biodiesel
Density (kg/m ³)	922
Specific gravity	0.89
Cloud point (°C)	7.0
Pour point (°C)	-2
Acid value (mg KOH/gm)	0.45
Flash point (°C)	167

11.0 CONCLUSION

Biodiesel is eco-friendly. Biodiesel is easy burning alternative gasoline. Biodiesel include no petroleum, however may be mixed with traditional diesel gasoline. This fuel may be used in any diesel engine without any modification. Biodiesel is degradable, non poisonous and free from sulphur and lead.

Hence, the primary goal is to provide biodiesel from groundnut acid oil and to apply esterification and transesterification with Ethanol and MNAS-Si Catalyst method. The most excellent parameters which gave most conversion of 92.06% the use of 5% w/w catalyst, 1:11 oil to alcohol molar ratio, at 65°C in 9 hours.

12.0 REFERENCE

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