

BIODIESEL PRODUCTION FROM RUBBER SEED OIL USING CARBON NANOTUBES-TiO₂ CATALYST

Submitted in partial fulfilment of the requirement for the award of
Bachelor of Technology Degree in Chemical Engineering

by

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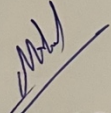
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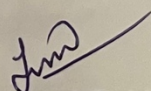
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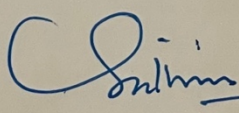
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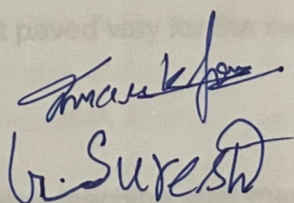

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DECLARATION

We MARK JOEVIN VINCENT (39190017) and G.SURESH (39190033) hereby declare that the Project Report entitled "**BIODIESEL PRODUCTION FROM RUBBER SEED OIL USING CARBON NANOTUBES-TiO₂ CATALYST**" done by us under the guidance of Mr. Micheal Rahul Soosai M.Tech and Dr.J.Arun, Ph.D. SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY, CHENNAI at is submitted in partial fulfilment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering.

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ABSTRACT

Research on biodiesel has increased as a sustainable and environmentally friendly alternative fuel to traditional fossil fuels. Vegetable oils, yellow grease, used cooking oils, or animal fats are used to make biodiesel. Transesterification, a process that turns fats and oils into glycerine and biodiesel, which is used to create the fuel, with the use of low molecular weight alcohol, in different reaction conditions and with different types of catalysts. However, compared to diesel fuel made from petroleum, production costs are still quite high. The use of solid heterogeneous catalyst in the production of biodiesel could lower its cost and make it economically competitive with diesel. However, compared to diesel fuel made from petroleum, production costs are still quite high. The use of a solid heterogeneous catalyst in the production of biodiesel could lower its cost and make it economically competitive with diesel. Carbon nanotubes-titanium dioxide composite as a catalyst has shown a great potential as heterogeneous catalyst in assisting the transesterification of rubber seed oil to biodiesel by producing a yield of 93.33%, under optimum conditions (0.5 wt% catalyst loading, 1:6 oil to methanol ratio, 60°C reaction temperature and 1 h 30 min reaction time), due to high surface area, strong metal support interaction, chemical stability, and acid–base property. The catalyst was characterized by XRD & FT-IR measurements to understand and evaluate their physical and structural properties. The biodiesel sample was characterized by gas chromatography-FID. Furthermore, its physiochemical properties met ASTM D6751 standards. Moreover, upon regeneration using n-hexane washing followed by calcination at 650°C for 3 h, the CNT-TiO₂ catalyst was used for transesterification reaction for five times.

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LIST OF ABBREVIATIONS

CNT	Carbon nanotube
GC-FID	Gas Chromatography-flame ionization detection
FFA	Free fatty acid
FAME	Fatty acid methyl esters
FTIR	Fourier transform infrared spectroscopy
RPM	Rotation per minute
XRD	X-Ray diffraction
RFS	Renewable fuel standards
MWCNT	Multi walled carbon standards
ASTM	American standard for testing material

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CHAPTER-1

INTRODUCTION

1.1 BIODIESEL

Due to its potential to displace conventional fossil fuels, biodiesel is an environmentally friendly replacement fuel that has attracted growing attention in recent years. It is a kind of diesel fuel produced using renewable materials like recycled grease, animal fats, and vegetable oils. This essay will give a thorough introduction to biodiesel, covering its background, production processes, benefits, drawbacks, environmental impact, governmental regulations, and prospects for the future (Ayoola et al., 2021).

1.1.1 History of biodiesel

Biodiesel has a lengthy history going back more than a century. The idea of utilizing vegetable oils and other naturally occurring substances as fuel dates return to the late 1800s, when Rudolf Diesel designed the first engine powered by diesel. Instead of using fossil fuels at first, Diesel built his engine to run on vegetable oils like peanut oil. He thought that using these fuels would give the engine a cleaner, longer-lasting source of energy.

In the early 1900s, the idea of utilizing vegetable oils as energy became more and more popular, particularly in rural areas where fuels made from petroleum were harder to come by. Different vegetable oils, including soybean oil and cottonseed oil, were tested by farmers and other rural residents as potential sources of fuel (Mythili et al., 2014).

Vegetable oil was initially used as a fuel, but its production and processing were expensive, which prevented its widespread use. Most consumers preferred petroleum-based fuels because they were more affordable and easily accessible.

The oil embargo and ensuing energy crisis in the 1970s gave rise to the idea of biodiesel as a sustainable and environmentally friendly fuel. Biodiesel was considered to be a potential solution as investigators and scientists started looking for other sources of energy.

James J. Beckman, who had invented a method for transforming vegetable oils into a fuel suitable for diesel engines, received the first official biodiesel patent in 1977. The vegetable oil was combined with methanol and sodium hydroxide in Beckman's procedure to produce methyl esters, which are the main constituent of biodiesel.

Researchers worked to improve the biodiesel manufacturing procedure throughout the 1980s and 1990s, and as a result, commercial production accelerated. The first commercial biodiesel plant opened in Austria in 1989, marking Europe's introduction of the fuel on a large scale (Khan et al., 2017).

Throughout the course of the 1990s as well as the beginning of the 2000s, the consumption of biodiesel grew in Europe, in part due to government initiatives that promoted the use of environmentally friendly fuels. After the Renewable Fuel Standard was passed in 2005 and made it mandatory to use sustainable fuels in transportation fuel, the biodiesel industry in America started to boom in the early 2000s.

As more people search for more sustainable and environmentally friendly energy sources, biodiesel is currently generated and utilized all over the world. Although there are still obstacles to be overcome, like the cost for generation and the scarcity of feedstocks, biodiesel has advanced significantly from its early days as a specialised fuel used by farmers in the countryside (Mohiddin et al., 2021).

1.1.2 Advantages of biodiesel

An environmentally friendly, non-toxic, and sustainable alternative to diesel fuel made from petroleum is biodiesel. Compared to conventional diesel fuel, it has a number of benefits, including (Datta et al., 2019):

1. Renewable: Vegetable oils and animal fats, which are both readily accessible and replenishable, are used to make biodiesel.
2. Reduced greenhouse gas emissions: When compared to conventional diesel fuel, biodiesel can cut greenhouse gas emissions by up to 80%. This is due to the fact that biodiesel is burned, it produces less carbon dioxide and other contaminants because it is made from organic materials that absorb carbon dioxide during their growth.

3. Enhanced air quality: Compared to conventional gasoline and diesel, biodiesel burns more cleanly, emitting less sulphur dioxide and other pollutants. This enhances the quality of the air and lowers respiratory issues in localised areas.
4. Biodegradable: Because biodiesel decomposes rapidly in the environment, it won't harm water or soil resources over the long term.
5. Better engine performance: Because biodiesel has a larger cetane rating than regular petroleum fuel, it explodes more effectively and ignites quicker. This enhances the engine's capabilities by increasing power, improving fuel efficiency, and lowering maintenance costs.
6. Energy security is improved because biodiesel can be produced locally, reducing reliance on imported oil. Additionally, this promotes local economies and jobs in the biodiesel sector.
7. Versatility: Biodiesel is a flexible and adaptable fuel source because it can be utilized in any engine without any modification.
8. Reduced reliance on fossil fuels: Biodiesel contributes to energy mix diversification and reduces reliance on fossil fuels. By doing this, the dangers of using only fossil fuels for energy are lessened.

Comparing to traditional fuel, biodiesel has a number of benefits, including lowered greenhouse gas emissions, better air quality, improved engine performance, increased energy security, and a decreased reliance on fossil fuels. As a result, it plays a crucial role in the shift to a future with more sustainable and renewable energy.

1.2 GOVERNMENT POLICIES

An important part of encouraging the production and use of biodiesel is played by government policies. A lot of nations have put programs and incentives in place to promote the utilization of biodiesel as a raw material for clean and renewable energy. The following are a few of the most prevalent government biodiesel policies:

1. Many nations have adopted renewable fuel standards (RFS), which call for a specific proportion of renewable fuels, like biodiesel, to be blended with conventional fossil fuels. For instance, the Renewable Fuel Standard in the

United States mandates that a specific amount of renewable fuel be blended with petrol and diesel fuel each year.

2. Tax breaks: A lot of governments provide tax breaks to promote the production and use of biodiesel. For manufacturers, processors, and consumers of biodiesel, such incentives may take the form of tax credits, exemptions, and deductions (Galadima & Muraza, 2020; Janda et al., 2012).
3. Governments may also provide grant programmes to aid in the study and advancement of biodiesel manufacturing methods and technologies. The development of new biodiesel production facilities as well as the research, development, and demonstration of new technologies can all be funded by these programmes.
4. Governments occasionally provide fuel subsidies to make biodiesel more accessible and competitive with conventional diesel fuel. The higher production costs of biodiesel can be partially offset by these subsidies, which will also increase consumer access to it.
5. Mandates and targets: Some governments have established biodiesel use mandates and targets. These regulations mandate the use of a specific volume of biodiesel in heating oil, transportation fuels, and other uses. For instance, by 2020, the European Union wants to use 10% renewable energy in transportation.
6. Research and development initiatives: To support the development of novel biodiesel technologies and manufacturing processes, many governments also fund research and development initiatives. These initiatives could advance the sector and increase biodiesel's parity with conventional diesel fuel (Williams et al., 2006).
7. Environmental laws: Environmental laws that promote the use of biodiesel may also be implemented by governments. For instance, vehicle and other equipment emissions regulations may favour the use of biodiesel over conventional diesel

In general, government regulations have an important impact on encouraging production and the utilization of biodiesel. Governments can contribute to the development of the biodiesel industry by offering mandates, incentives, and other forms of support. These regulations can aid in lowering greenhouse gas emissions,

enhancing air quality, and advancing the utilization of recyclable and sustainable power sources.

1.3 PRODUCTION METHODS OF BIODIESEL

Biodiesel can be made from a variety of raw materials, including cooking oils, fats from animals. There are several methods for producing biodiesel, each having advantages and disadvantages. The following are some of the popular techniques for producing biodiesel (Liu et al., 2009):

1. Base catalysed transesterification :The most popular technique for producing biodiesel is base catalysed transesterification, which entails reacting a triglyceride with an alcoholic content and a basic activator. The triglyceride is converted in this reaction into glycerin and fatty acid methyl esters (FAME), which are then separated. The FAME can be used as biodiesel.
2. Acid catalyzed transesterification: Similar to base transesterification, acidic catalysed transesterification uses an acid catalyst (such as sulfuric acid) rather than a base as the catalyst. Because it can tolerate higher levels of contaminants, the acid catalyst is usually employed with feedstocks that are of lower quality or are contaminated (Gholami et al., 2020; ji, 201 C.E.).
3. Enzymatic transesterification: Enzymes rather than chemical catalysts are used in this method. The cost of this method may be higher than that of chemical catalysis, but it can yield greater quantities of biodiesel and work with a wider variety of feedstocks.
4. Supercritical methanol: In this process, methanol is heated with vegetable or animal-derived fats at high temperatures and pressures, causing the triglycerides to disintegrate into methyl esters and glycerol.
5. Pyrolysis is a process that involves heating biomass feedstocks, like algae or plant matter, without the presence of oxygen in order to create a liquid bio-oil that can be further processed into biodiesel. This process produces high yields of biodiesel from a variety of feedstocks, but it needs specialised equipment and can be pricey.
6. Hydrogenation: Through the process of hydrogenation, triglycerides are changed into saturated fatty acids by reacting vegetable or animal fat with hydrogen gas in the presence of a catalyst. Following that, biodiesel can be

created using conventional chemical catalysis to transform the saturated fatty acids. The production of high-quality biodiesel using this method is possible from a variety of feedstocks, though it is less common than other biodiesel production methods (Janda et al., 2012).

Overall, the availability of feedstock, the price of fabrication, and desired end product will all influence the type of biodiesel production chosen. Regardless of the method of production, biodiesel has the potential to be a key player in the shift to a future with more renewable and sustainable energy sources.

1.4 FACTORS AFFECTING BIODIESEL

Several factors can influence the grade and amount of biofuel generated during production. The following are some of the crucial elements influencing the production of biodiesel (Mathiyazhagan & Ganapathi, 2011):

1. Feedstock quality: The grade of the raw materials used to create biodiesel can have an important influence on the quality of the end result.. The best feedstocks for producing biodiesel are those with low impurity and water content levels.
2. Feedstock accessibility: The generation of biodiesel may be impacted by the accessibility of feedstocks. To minimise the cost of transportation and lower the carbon footprint of the production of biodiesel, high and locally accessible raw materials are preferred (Chozhavendhan et al., 2020; Mathiyazhagan & Ganapathi, 2011).
3. Kind and amount of catalyst: The rate of reaction and yield of biodiesel can be impacted by the kind and amount of catalyst used in the process. The final product's quality can vary depending on the characteristics of the catalyst used.
4. Type and concentration of alcohol: the kind and amount of alcohol used in the production of biodiesel can have an impact on the yield and rate of reaction. The most widely used alcohol is methanol, though ethanol and other alcohols can also be used.
5. Reaction time and temperature: The yield and quality of biodiesel can be impacted by the reaction time and temperature used in the production

process. Higher temperatures and longer reaction times can produce more biodiesel, but they can also produce biodiesel of lower quality.

6. Methods for separation and purification: The methods used to separate the biodiesel from glycerol and other by-products can have an impact on the overall quality of the end product. Effective separation and purification techniques can reduce impurities and raise the biodiesel's quality (Chozhavendhan et al., 2020; Mathiyazhagan & Ganapathi, 2011).
7. Utilization of by-products: The use of by-products, such as glycerol, can have an effect on the production of biodiesel. Utilizing by-products effectively can reduce waste and increase the biodiesel industry's overall sustainability.

Overall, a number of variables that can affect the type and amount of biodiesel produced have an impact on its production. Paying close attention to these elements can aid in maximising the generation of biodiesel and enhancing the sustainability of this significant renewable energy source.

1.5 CHALLENGES FACING BY BIODIESEL

Despite the reality that biofuel is a sustainable and ecologically harmless energy source, there are several barriers to its widespread adoption. The following are some of the major challenges facing biodiesel (Chen et al., 2021):

1. Infrastructure and distribution: There may be issues with the infrastructure and distribution systems for biodiesel. Compared to petroleum-based diesel, biodiesel has different properties that necessitate specialised handling and storage amenities as well as engine and automobile modifications. Additionally, biodiesel's distribution networks are less established than those for petroleum-based diesel, which can restrict its accessibility and availability.
2. Energy balance and carbon footprint: Concerns can also be raised about the energy balance and carbon impact of biodiesel. While inputs of energy such as fuel for machinery and electricity for processing are needed during the production process of biodiesel, despite being an energy source that is renewable. The carbon impact of biodiesel is also influenced by the feedstock's origin and the manufacturing processes. In some cases, the carbon footprint of biodiesel production and transportation may be higher than that of diesel derived from petroleum.

3. Barriers to policy and regulation: These obstacles can make it difficult to produce and use biodiesel. While other nations have not, some have put policies in place to encourage the creation and use of biodiesel. Regulations may occasionally place restrictions on the use of biodiesel, such as prohibitions on biodiesel blends or demands for pricey certification and testing.
4. Technical restrictions: The use and production of biodiesel are also subject to some technical restrictions. For instance, biodiesel may contain less energy than gasoline derived from petroleum, which may result in decreased fuel economy and range for automobiles. In addition, biodiesel may be more prone to oxidation and degradation, which may affect storage and shelf life (Balan, 2014).
5. Cost and availability of feedstocks: The price and accessibility of feedstocks can be a significant barrier to the production of biodiesel. Competition for feedstocks may increase as demand for biodiesel rises, pushing up costs and possibly creating shortages. Additionally, some feedstocks might not be accessible in specific areas, which would restrict the possibility of producing local biodiesel there.

Overall, even though biodiesel has many benefits as a renewable and sustainable energy source, the project faces plenty of challenges that must be addressed before its full potential may be fulfilled. It will take a combination of technological advancement, policy support, and market expansion to address these challenges.

1.6 TRANSESTERIFICATION PROCESS

The main method for turning waste cooking oils, animal fats, or vegetable oils into biodiesel is transesterification. In this process, lipids and alcohol react with the help of a catalyst to produce fatty acid esters and glycerin..

The catalysts used in this can either be acidic or basic. Acidic catalysts like sulfuric acid or hydrochloric acid are less frequently used because of how dangerous and corrosive they are. Due to their high activity and low cost, basic catalysts like sodium hydroxide or potassium hydroxide are most frequently used in the production of commercial biodiesel.

Esterification, glycerolysis, trans-esterification are the three steps in the transesterification reaction. When triglycerides are allowed to react with alcohol with the help of an acidic catalyst, free fatty acids and glycerin are produced in the first stage. In the subsequent step, glycerol and the leftover triglycerides along with free fatty acids combine to create mono-, di-, and triglycerides. In the last step, glycerol and fatty acid esters are created when the rest of the triglycerides and glycerides react with alcohol with the help of a basic activator (Leung et al., 2010a).

The process of transesterification can be carried out in both batch & continuous operations. In a batch process, the catalyst and reacting substances are combined in a reactor and given time to react. When the desired level of conversion is reached, the reaction is stopped, and the resulting mixture is then split into biodiesel and glycerol phases. In a continuous process, reactants and catalyst are continually introduced to a reactor, and the resulting mixture is continuously withdrawn and segregated into glycerol and biofuel.

Transesterification requires the use of catalysts to achieve high yields and shorten reaction times. The catalysts serve as a mediator, supplying a different reaction pathway with lower activation energy and speeding up the reaction rate. Sodium hydroxide and potassium hydroxide are two common alkali metal hydroxides that are used as fundamental catalysts in transesterification. These catalysts are very reactive and have been demonstrated to yield large amounts of biodiesel quickly. When the desired level of conversion is reached, the reaction is stopped, and the resulting mixture is then split into biodiesel and glycerol phases. The reactants and catalyst are continuously added to a reactor in a continuous process, and the reaction mixture is continuously removed and separated into glycerol and biodiesel (Leung et al., 2010b; Veljković et al., 2015).

To attain significant yields and low reaction times, this technique involves the addition of catalysts. The catalysts serve as mediators, supplying a different reaction pathway with lower activation energy and speeding up the reaction rate. Two popular alkaline metallic hydroxides utilised as fundamental catalysts in transesterification are sodium hydroxide and potassium hydroxide, respectively. These catalysts are very reactive and have been demonstrated to yield large amounts of biodiesel quickly (Veljković et al., 2014).

1.6.1 Steps involved in transesterification

In this procedure, an alcohol, usually methanol or ethanol, is reacted with the feedstock with the help of a catalyst, like sodium hydroxide or potassium hydroxide. There are several phases to the transesterification process, such as:

1. Feedstock preparation: The preparation of the feedstock is the first step in the transesterification process. To do this, the raw material is typically filtered to get rid of any impurities like water and solid particles. It might be necessary to melt the feedstock if it is a solid, such as animal fat, in order to use it.
2. Alcohol preparation: The making of the alcohol is the next action. The alcohol, which is typically methanol or ethanol, must be anhydrous in nature (i.e., devoid of water) in order to avoid unintended reactions. Although alcohol is typically bought commercially, it can also be made using biomass and other sustainable resources (Suehara et al., 2005).
3. Preparation of the catalyst: The catalyst, which is typically either potassium or sodium hydroxide, is generally made by disintegrating it in methanol or ethanol. The catalyst plays an important part in this process because it speeds up the production of biodiesel and aids in the disintegration of the feedstock.
4. Mixing: After being ready, the feedstock, alcohol, and catalyst are combined in a reaction vessel. To facilitate the chemical process, the mixture of materials is exposed to a specified temperature, generally around 50°C and 70°C.
5. Reaction: After combining the feedstock, alcohol, and catalyst, the transesterification reaction starts. Alcohol breaks down the ester molecules in the feedstock during the reaction, creating biodiesel and molecules of glycerol in the process. Usually, it takes a long time for the reaction to finish (Leung et al., 2010a; Najafpour et al., 2008).
6. Separation : After the process is finished, the resultant solution is allowed to settle, at which point the layers of glycerol and biodiesel are separated. The biodiesel layer is typically lighter and floats on top, while the glycerol layer is typically heavier and sinks to the bottom.
7. Washing: To remove any remaining catalyst and impurities, the biodiesel is then washed with water. This is typically accomplished by combining

biodiesel and water, letting the mixture settle, and then separating the layers of water and biodiesel.

8. Drying: The biodiesel must be dried as the last step. Usually, this is accomplished by heating biodiesel in order to evaporate any lingering water.

In summary, this process is a crucial step in the preparation of biodiesel, and the use of catalysts is required to produce high yields and shorten reaction times. Due to their high reactivity and low cost, basic catalysts like NaOH or KOH are frequently used in chemical reactions.

1.7 HETEROGENOUS CATALYST FOR BIODIESEL SYNTHESIS

Heterogeneous catalysts, a kind of catalyst used in the manufacturing of biodiesel, exist as a solid form rather than being dispersed in a reaction mixture. They are frequently employed in the transesterification reaction, where they aid in the conversion of triglycerides into the main ingredients of biodiesel, fatty acid methyl esters. In comparison to homogeneous catalysts, heterogeneous catalysts have several advantages, including ease of segregation, reusability, and low waste creation.

Solid acid catalysts are one of the most prevalent kinds of heterogeneous catalysts employed in biodiesel manufacturing.. The most common foundations for these catalysts are zeolites, clays, or sulfonated resins. In comparison to homogeneous acid catalysts, they are more stable, less prone to corrosion, and capable of operating at lower temperatures and pressures. The need for longer reaction times and lower conversion rates when compared to homogeneous catalysts are some of their drawbacks (Veljković et al., 2015).

Solid base catalysts are another kind of heterogeneous catalyst utilised in the production of biodiesel. Metal oxides, such as calcium oxide or magnesium oxide, are typically the foundation of these catalysts. In comparison to homogeneous base catalysts, they are more stable, have faster reaction rates, and can operate at lower temperatures and pressures. They do, however, have some drawbacks, such as the potential for carbon dioxide and water to deactivate the catalyst.

To increase the effectiveness of the biodiesel production process, heterogeneous catalysts can also be used in conjunction with homogeneous catalysts. For instance,

to increase the rate and yield of the transesterification reaction, a homogeneous acidic catalyst like sulfuric acid can be utilized in combination with a solid acid catalyst like sulfonated polystyrene. By using less homogeneous catalyst, this strategy can also produce less waste and be more economically advantageous (Noshadi et al., 2012).

A variety of factors affect the efficacy of heterogeneous activator in the generation of biofuel. These elements include the kind of catalyst used, its surface area and size of particles, the pressure and temperature conditions of the reaction, and the presence of impurities in the feedstock. The production of biodiesel raises the issue of catalyst deactivation because impurities in the feedstock can bind to the catalyst and gradually decrease its activity.

Despite these difficulties, the utilization of heterogeneous catalysts in the production of biodiesel is growing in popularity because of their simplicity in separation, capacity for reuse, and low waste generation. They offer the potential to lessen the environmental impact of the biodiesel production process while also increasing its efficiency and cost-effectiveness. The goal of ongoing study is to create new and improved heterogeneous catalysts that will improve the efficiency of biodiesel production procedures (Galadima & Muraza, 2014).

1.7.1 Carbon nano tubes

The type of nanotube known as carbon nanotubes (CNTs) has special properties that could make them helpful as catalysts in a variety of chemical reactions. Researchers have looked into using CNTs as for producing biodiesel catalysts in recent years. This method has many benefits, such as increased reaction rates, high yields, and minimal environmental impact.

CNTs can be utilised as a solid catalyst in the process of transesterification to make biodiesel. Transesterification involves reacting triglycerides with alcohol in with the help of an accelerator to produce fatty acid esters and glycerin, because CNTs are in a different phase from the reactants and products, they have the potential to function as heterogeneous catalysts. This makes it simple to separate and recycle the catalyst (Guan et al., 2017).

1.7.2 Titanium dioxide

A promising heterogeneous accelerator employed in the production of biodiesel is titanium dioxide (TiO_2). It is an appealing substitute for other catalysts that might be less sustainable or more expensive because it is a plentiful, affordable, and eco-friendly material. TiO_2 is suitable for use in challenging reaction conditions because it is also extremely stable and corrosion-resistant.

Anatase and rutile are the two main types of TiO_2 that can be utilised as catalysts in the production of biodiesel. Anatase is the more active of the two forms, and because it has a larger surface area, it can interact with the reactants more effectively. It may, however, degrade more quickly over time because it is less stable than rutile (Carlucci et al., 2019).

1.7.3 Titanium oxide combined with carbon nanotube in the production of biodiesel

Carbon nanotubes (CNTs) have demonstrated great promise as catalyst supports in a variety of chemical processes, including the production of biodiesel. CNTs can produce a highly effective and reliable catalyst for transesterification reactions when combined with titanium dioxide (TiO_2). TiO_2/CNTs is the name of the catalyst blend that is used to produce biodiesel, and it has a number of advantages over other catalysts.

The large surface area of TiO_2/CNTs , which enables greater exposure to reactants and more effective catalytic reactions, is one of their main advantages. Because of their superior mechanical and thermal stability, CNTs can be used in abrasive reaction environments. The reusability of TiO_2/CNTs is also very good, with little activity loss after several cycles of use.

Numerous studies have been done to look into the use of TiO_2/CNTs as activator in the generation of biodiesel. According to one study, TiO_2/CNTs had superior catalytic activity when compared to other catalysts, with a transesterification rate of used cooking oil that was over 90%. Another study found that the free fatty esters used in cooking oil, which can interfere with other catalysts, could be efficiently catalysed by TiO_2/CNTs (Sharul et al., 2021).

Due to their complementary properties, TiO_2 and CNTs work together synergistically in TiO_2/CNT catalysts. TiO_2 is capable of photocatalysis, which enables it to be irradiated with light and generate reactive oxygen species capable of oxidising organic compounds. Despite the positive outcomes of TiO_2/CNTs as biodiesel production catalysts, there are some difficulties with their application. To ensure the best catalytic performance, it is necessary to carefully control the ratio of TiO_2 to CNTs. The potential toxicity of CNTs, which raises questions about the safety of the environment and human health, is another difficulty.

As a result of its high efficiency, stability, and reusability, the combination of carbon nanotubes and titanium dioxide (TiO_2/CNTs) is a promising catalyst for the generation of biodiesel. The development of new and improved TiO_2/CNTs catalysts that can further improve the efficiency of biodiesel manufacturing procedures while solving potential health and safety issues is the main focus of ongoing research (Carlucci et al., 2019).

1.8 BIODIESEL FROM EDIBLE OILS

Triglycerides are frequently transesterified to produce biodiesel from a range of sources that involve edible oils, fats, and even cooking oils. However, there are concerns about the impact on food costs and potential rivalry with food if these oils are utilized to produce biodiesel (Balat, 2011).

Edible oils such as soy oil, palm oil, rapeseed oil, and sunflower seed oil are commonly utilised in the production of biodiesel. Due to their wide availability and high oil content, these oils are favoured. However, there are a number of arguments against using edible oils to make biodiesel. First off, the manufacturing of biodiesel using edible oils may result in higher food costs, particularly in underdeveloped nations where food security is a key problem. Second, the manufacturing of biodiesel from edible oils may cause agricultural land to shift from producing food to producing energy, which could exacerbate the problem of food security. Last but not least, the production of edible oils consumes a significant amount of energy as well as water, that may have a severe impact on the natural world.

Utilising waste oils and fats to make biodiesel is another strategy to enhance sustainable biodiesel production. Waste oils and fats that can be utilised to make

biodiesel include leftover cooking oil, animal fats, and grease trap waste. Waste oils and fats can be used to produce biodiesel in a way that does not compete with food production while simultaneously reducing the environmental impact of their disposal (Degfie et al., 2019).

1.9 BIODIESEL FROM NON-EDIBLE OIL

The utilization of non-edible oils for the generation of biodiesel is gaining popularity since it eliminates conflict between food and energy production and can give further advantages such as carbon storage and the preservation of biodiversity.

Jatropha, pongamia, mahua, castor, and karanja, rubber seed oil are non-edible oils that are frequently used to make biodiesel. Due to their high oil content, accessibility, and appropriateness for growing on marginal soils unsuitable for food production, these oils are chosen. Non-edible oil crops can also offer further advantages like soil protection, biodiversity preservation, and carbon sequestration (Demirbas et al., 2016).

The process of turning non-edible oils into biodiesel includes the extraction of the oil, pre-treatment, transesterification, and purification. Depending on the type of oilseed and its availability, the oil extraction process can be carried out via mechanical or chemical methods. Pre-treatment entails cleaning the oil of pollutants such moisture, free fatty acids, and other impurities. During the transesterification process, the oil undergoes reaction with an alcohol using a catalyst such as sodium hydroxide or potassium hydroxide. Glycerol is produced as a by-product as well as biodiesel as a result. Any remaining impurities in the biodiesel are eliminated during the purification process.

In comparison to edible oils, the use of non-edible oils in the production of biodiesel provides a number of benefits. First off, non-edible oils don't compete with food production, preventing a possible rise in food prices and worries about food security. Second, to prevent competition for land use, non-edible oils can be grown on marginal sites that are unsuitable for growing food. Thirdly, further advantages like carbon sequestration and biodiversity preservation may come from non-edible oil crops. Fourthly, it is possible to manufacture non-edible oils locally, minimising reliance on imported oil and fostering energy security. Non-edible oil crops, which

do not compete with the production of food, might offer extra advantages including carbon sequestration and biodiversity preservation (Banković-Ilić et al., 2012).

The use of non-edible oils in the production of biodiesel has a possibility to, among other things, decrease the release of greenhouse gases, enhance environmental friendly growth, and reduce reliance on imported oil. Additionally, the utilisation of non-edible oil crops for the production of biodiesel can have positive effects on biodiversity and carbon sequestration. Therefore, the development of environmentally friendly methods for the production and use of non-edible oils in the manufacturing of biodiesel can aid in the shift to a more renewable and sustainable energy system (Abdul Hakim Shaah et al., 2021).

1.9.1 Rubber seed oil in biodiesel production

The high oil content and accessibility of rubber seed oil in places like Southeast Asia, South America, and Africa have made it a suitable feedstock for biodiesel manufacturing. Comparing rubber seed oil to conventional feedstocks like soybean, rapeseed, and palm oil, there are a number of benefits (Ulfah et al., 2018).

The fatty acid ester percentage of RBO normally consists of 20-25% oleic acid, 40-45% linoleic acid, and 7-10% linolenic acid. The oil concentration in rubber kernels varies between 35 and 45%. due to its high concentration of unsaturated fatty acids, rubber seed oil is prone to rapid deterioration; however, this can be decreased by mixing it with other feedstocks or adding antioxidants.

The process of turning rubber seed oil into biodiesel includes the extraction of the oil, as well as pre-treatment, transesterification, and purification. Depending on the oilseed type and availability, the oil extraction process can be carried out using mechanical or solvent methods. Pre-treatment entails cleaning the oil of pollutants such moisture, free fatty acids, and other impurities. Using a catalyst like sodium hydroxide or potassium hydroxide, the oil is reacted with an alcohol during the transesterification process. Glycerol is produced as a by-product as well as biodiesel as a result. Any remaining impurities in the biodiesel are eliminated during the purification process (Ramadhas et al., 2005).

Compared to conventional biodiesel feedstocks like soybean, rapeseed, and palm oil, rubber seed oil provides a number of advantages. First off, growing rubber trees

does not compete with food production, potentially reducing increases in food costs and worries about food security. In addition, rubber trees can be planted on unsuitable marginal soils, preventing competition for land usage. Thirdly, rubber trees can also protect biodiversity and sequester carbon, among other advantages. Fourth, the ability to manufacture rubber seed oil domestically reduces reliance on imports of foreign oil and fosters energy security.

In conclusion, because of its high oil content, availability, and suitability for growth on marginal areas, rubber seed oil has the possibility to become a viable raw material for the generation of biodiesel. Compared to conventional feedstocks like soybean, rapeseed, and palm oil, the manufacturing of biodiesel from the RBO can have a number of benefits (Arachchige et al., 2008).

1.10 FUTURE OPPORTUNITIES OF BIODIESEL

Given the rising demand for energy from environmentally friendly and sustainable sources, biodiesel has bright future prospects. The following are some of the major areas for biodiesel growth and development:

1. Feedstock diversification: Diversifying the feedstocks is one of the main opportunities for growth in the biodiesel industry. Although there is still a lot of interest in using alternative feedstocks like algae, waste oils, and agricultural products, traditional feedstocks like soybean oil and rapeseed oil are still widely used. This can assist in addressing some of the difficulties related to the cost and availability of feedstock.
2. Technology: The production of biodiesel offers significant potential for technological advancement. The efficiency and sustainability of biodiesel production can be enhanced by advancements in production techniques, catalysts, and enzymes.
3. Market expansion: The biodiesel sector has a sizable amount of room for expansion. As the need for renewable energy sources that are environmentally friendly rises, biodiesel as a substitute for diesel made from petroleum is gaining popularity. The use of biodiesel in other industries, such as heating oil, aviation fuel, and marine fuel, is also gaining popularity.
4. Circular economy and sustainability: The generation of biodiesel is becoming more and more interested in the circular economy and ecological

sustainability. In addition to lowering waste and emission levels of greenhouse gases, using waste oils and other waste streams as feedstocks for the production of biodiesel can yield useful by-products like glycerol. Furthermore, using biodiesel can aid in reducing reliance on fossil fuels and encourage a more sustainable lifestyle.

Overall, biodiesel has promising prospects for the future due to the rising demand for environmentally friendly and renewable energy sources. The potential benefits of biodiesel are substantial, but overcoming the obstacles to its production and use will require a combination of technological advancement, policy support, and market development (Abdul Hakim Shaah et al., 2021).

CHAPTER-2

LITERATURE SURVEY

- **Chen et al .2019** In this study, the transesterification of rubber seed oil with methanol was explored using a magnetised carbon nanotube (MCNT) catalyst. In comparison with traditional catalysts, the study indicated that using the MCNT catalyst enhanced conversion efficiency while lowering catalyst usage. A further finding of the study was that the MCNT catalyst may be readily magnetically removed from the reaction mixture and recycled up to 5 times without suffering a substantial decrease in catalytic activity.
- **Zaoh et al. 2020** CNTs-Fe₃O₄ nanocomposites were explored as a catalyst for biodiesel generation from waste cooking oil. The best reaction parameters were discovered to be a molar ratio of methanol to oil of 12:1, a temperature at which the reaction occurs of 60°C, and a reaction period of 4 hours. The researchers also discovered that using CNTs-Fe₃O₄ composites as a catalyst gave rise to high conversion efficiency and simple catalyst separation. The distinctive qualities of CNTs-Fe₃O₄ tiny composites, which include their high surface area, strong reaction, and magnetic characteristics that allow them to be easily separated from the reaction mixture, were attributed to these findings.
- **Demirbas et al. 2014** research was done on the utilisation of sodium hydroxide as a catalyst for the generation of biodiesel from used cooking oil. The research discovered that a 6:1 molar ratio of methanol to oil, a temperature of reaction of 60°C, and a duration of reaction of 90 minutes produced the best results. According to the study, using a homogeneous catalyst not only led to higher conversion rates but also high catalyst utilisation and challenging catalyst separation.
- **Chavan et al. 2017** research was done on using H-mordenite as a catalyst to create biodiesel from used cooking oil. The research discovered that a 9:1 molar ratio of methanol to oil, a reaction temperature of 50°C, and a reaction period of 6 hours produced the best results. The study also discovered that using H-mordenite as a catalyst produced little waste, an easy separation of the catalyst, and excellent conversion efficiency.

- **Lam et al. 2014** who employed methanol and titanium dioxide nanomaterials (TiO_2) as a catalyst to transesterify rubber seed oil. Comparing TiO_2 to more traditional catalysts like sodium hydroxide and potassium hydroxide, the study discovered that TiO_2 had a greater conversion efficiency and consumed less catalyst. Additionally, it was discovered in the study that TiO_2 may be effortlessly removed from the reaction mixture by centrifugation and recycled up to five times without suffering a substantial reduction in catalytic activity.
- **Soares et al. 2018** In this study, the transesterification of the oil from rubber seeds with methanol was examined using a hybrid catalyst system composed of TiO_2 and MWCNTs. According to the research, using a hybrid catalyst system led to improved conversion efficiency and less catalyst use than using regular catalysts. The study also discovered that the hybrid catalyst system could be reused up to 3 times without suffering a substantial loss in catalytic activity by simply centrifuging it away from the reaction mixture.
- **Gogoi et al. 2014** Mahua oil's potential use as a feedstock for biodiesel production was examined. In comparison to homogeneous catalysts like sodium hydroxide and potassium hydroxide, the study discovered that using heterogeneous catalysts like calcium oxide and magnesium oxide led to higher conversion efficiency and less catalyst use. The study also discovered that contaminants like moisture and unsaponifiable materials can have a negative impact on the catalyst usage and conversion efficiency.
- **Ghanbari et al. 2021** it was looked into whether carbon nanotubes might be used as a catalyst to produce biodiesel from camelina oil. The research discovered that a 12:1 molar ratio of methanol to oil, a reaction temperature of 65°C , and a reaction period of 3 hours produced the best results. Additionally, the study discovered that using carbon nanotubes as a catalyst produced excellent conversion efficiency and simple catalyst separation.
- **Vijayaragavan et al. 2015** who looked into using sulfuric acid-modified titanium dioxide as a catalyst in the rubber seed oil and methanol transesterification process. In comparison to traditional catalysts, $\text{SO}_4\text{-TiO}_2$ was shown to have a greater conversion efficiency and a lower catalytic consumption. Additionally, the research revealed that $\text{SO}_4\text{-TiO}_2$ may be

effortlessly removed from the product of the reaction by centrifugation and recycled up to 4 times without suffering a substantial reduction in catalytic activity

- **Dong et al. 2013** examined the use of lipase as a catalyst in the synthesis of biodiesel from soybean oil. The research discovered that a 4:1 molar ratio of methanol to oil, 35°C for the reaction temperature, and 24 hours for the reaction were the ideal parameters. Additionally, the study discovered that using an enzymatic catalyst led to high conversion efficiency and simple catalyst separation, but also high cost and sensitivity to reaction conditions..
- **Karami et al. 2017** who conducted research on the transesterification of rubber seed oil with methanol using multi-walled nanotubes made of carbon as a form of catalyst. In comparison to more traditional catalysts like sodium hydroxide and potassium hydroxide, the study discovered that using MWCNTs as a catalyst led to improved conversion efficiency and less catalyst use. MWCNTs can be recycled up to 4 times without significantly losing their catalytic activity, according to the study.
- **Luque et al. 2010** investigation into using jatropha oil as a feedstock for making biodiesel. The study discovered that while homogeneous catalysts like sodium hydroxide and potassium hydroxide produced high conversion efficiencies, they also consumed a lot of catalyst and were challenging to separate. According to the study, using heterogeneous catalysts such metal oxides and zeolites led to poorer conversion efficiencies, but also to lower catalyst use and simpler catalyst separation.
- **Sue et al. 2017** they employed methanol and titanium dioxide nanocrystals (TiO_2) as a catalyst to transesterify rubber seed oil. In comparison to conventional catalysts, the study indicated that using TiO_2 as a catalyst increased conversion efficiency while lowering catalyst usage. Additionally, it was discovered in the study that TiO_2 may be effortlessly removed from the reaction mix by centrifugation and recycled up to five times without suffering a substantial reduction in catalytic activity.
- **Jalalabadi et al. 2017** the utilisation of titanium dioxide modified with iron oxide (Fe-TiO_2) as a catalyst for the transesterification of rubber seed oil with methanol was researched. In comparison to traditional catalysts, Fe-TiO_2

was shown to have a greater conversion efficiency and a lower catalyst consumption. Additionally, the research revealed that Fe-TiO₂ may be effortlessly removed from the reaction mixture by centrifugation and recycled up to five times without suffering a substantial reduction in catalytic activity.

- **Karmee and chadha et al. 2015** examined the use of castor oil as a feedstock for the manufacture of biodiesel. In comparison to homogeneous catalysts like sodium hydroxide and potassium hydroxide, the study discovered that using heterogeneous catalysts like calcium oxide and magnesium oxide led to higher conversion efficiency and less catalyst use. The study also discovered that the consumption of the catalyst and conversion efficiency can both be adversely impacted by the presence of free fatty acids in the feedstock
- **Ibrahim et al. 2019** TiO₂ nanotubes were explored as a catalyst for biodiesel generation from waste cooking oil. The best reaction conditions, according to the study, were a molar ratio of methanol to oil of 6:1, a reaction temperature of 60°C, and a reaction period of 2 hours. The researchers also discovered that using TiO₂ nanotubes as a catalyst resulted in high conversion efficiency and simple catalyst separation. The authors attributed these findings to TiO₂ nanotubes' distinctive features, such as their large surface area and shape.
- **Wang et al. 2018** the use of sodium hydroxide as a catalyst for biodiesel generation from rapeseed oil was examined. The best reaction conditions, according to the study, were a molar concentration of methanol to oil of 6:1, a reaction temperature of 60°C, and a reaction period of 1 hour. The use of sodium hydroxide as a catalyst resulted in a higher rate of conversion and little waste generation, according to the study.
- **Hanifa et al. 2016** research was done on using calcium oxide as a catalyst to produce biodiesel from used cooking oil. The research discovered that a 6:1 molar ratio of methanol to oil, a reaction temperature of 60°C, and a reaction time of 90 minutes produced the best results. The study also discovered that using a heterogeneous catalyst led to a decrease in conversion efficiency, but also to a reduction in catalyst consumption and an ease in catalyst separation..

- **Varma et al. 2018** it was looked into whether the transesterification of rubber seed oil with methanol might be accelerated by using titanium dioxide modified with zirconium oxide . In comparison to traditional catalysts, Zr-TiO₂ was shown to have a greater conversion efficiency and a lower catalyst usage. Additionally, the study discovered that Zr-TiO₂ may be effortlessly removed from the reaction mixture by centrifugation and recycled up to four times without suffering a substantial reduction in catalytic activity.
- **Wie et al. 2013** research was done on the utilisation of magnesium oxide as a catalyst for the generation of biodiesel from used cooking oil. The research discovered that a 12:1 molar ratio of methanol to oil, a reaction temperature of 60°C, and a reaction period of 4 hours produced the best results. The study also discovered that using magnesium oxide as a catalyst produced little waste, an easy separation of the catalyst, and excellent conversion efficiency.
- **Oliveira et al. 2018** the study looked into the usage of sulfuric acid as a catalyst in the synthesis of biodiesel from used cooking oil. The research discovered that a 12:1 molar ratio of methanol to oil, a reaction temperature of 60°C, and a reaction period of 4 hours produced the best results. The study also discovered that using sulfuric acid as a catalyst produced little waste and had a high conversion efficiency.
- **Zhang et al. 2019** TiO₂ was explored as a catalyst for biodiesel generation from soybean oil. The best reaction parameters were discovered to be a molar concentration of methanol to oil of 12:1, a reaction temperature of 60°C, and a reaction period of 4 hours. The researchers discovered that using TiO₂ as a catalyst resulted in a high efficiency of conversion, simple catalyst separation, and little waste generation. These findings were linked to TiO₂'s unique physical and chemical features, such as its high surface area, strong reactivity, and low toxicity.
- **Mishra et al. 2019** CNTs were explored as a catalyst for biodiesel generation from jatropha oil. The best reaction conditions, according to the study, were a molar ratio of methanol to oil of 6:1, a reaction temperature of 60°C, and a reaction period of 2 hours. The study also discovered that using carbon nanotubes as a catalyst resulted in excellent conversion efficiency and simple

catalyst separation. The study attributed these findings to CNTs' distinctive features, such as their large surface area, high reactivity, and distinct shape.

- **Liew et al. 2014** Carbon nanotubes (CNTs) were explored as a catalyst for palm oil biodiesel generation. The best reaction parameters were discovered to be a molar ratio of methanol to oil of 12:1, a temperature for the reaction is 60°C, and a reaction period of 4 hours. The study also discovered that using carbon nanotubes as a catalyst resulted in a high rate of conversion and lower waste creation. The study explained these findings to CNTs' distinctive features, such as their huge surface area, high degree of reactivity, and distinct shape.

CHAPTER-3

AIM AND SCOPE

3.1 AIM

The main goal of our project is to produce biodiesel from Rubber seed oil using Carbon nanotube Titanium Dioxide composite (CNT-TiO₂).

- Synthesis of CNT-TiO₂ catalyst.
- Characterization of CNT-TiO₂ catalyst to find its properties.
- To find properties of the biodiesel like Viscosity, Density, calorific value, Cloud point, FFA, etc., and also find the composition using GC.

3.2 SCOPE

Biodiesel acts like petroleum diesel, but produces less air pollution and it comes under renewable sources, is biodegradable and is safer for the environment. Producing biodiesel fuels can help create local environmental benefits. Replacing fossil fuels with biofuels has the potential to reduce some undesirable aspects of fossil fuels.

- Green-house gas(GHG) pollutant emissions.
- Exhaustible resources depletion.
- Dependence on unstable foreign suppliers.
- Increase in fossil fuel prices.
- Global warming and climatic change.

CHAPTER-4

MATERIALS AND METHODS

4.1 MATERIALS

Rubber seeds were purchased from farm near Marthandam, Kanyakumari, [8.3022 °N, 77.2231 °E]. Carbon-nanotubes, TiO₂, Sulfuric acid, Methanol, ethanol, Iso-Propyl alcohol, NaOH pellets, n-hexane, Phenolphthalein, Distilled Water, was used from laboratory in Centre for waste management, Sathyabama university, science and technology, Chennai-600119.

4.2 OIL EXTRACTION

Three methods of extraction were employed to extract oil from rubber seeds. The rubber seeds utilized for oil extraction weighed a total of 50 kg. The rubber seeds were cleaned to remove any dirt or other unwanted particles. The seeds were then dried to reduce their moisture content. In hydraulic pressing method, the seeds were placed in the hydraulic press machine and applied a pressure of 650-750 bar gradually to achieve high oil yield. The temperature during the extraction was kept around 65°C to avoid degradation of the oil. The oil extracted was collected in a container. Solid impurities were removed using a filter bag having a pore size of 5μ. In solvent extraction, 5 L of n-hexane was combined with 1 kg of powdered rubber seeds and stirred for 3 h. The solvent-oil mixture from solid residues was separated using a filter paper. The solvent-oil mixture was then separated by heating the mixture at 60°C for 30 min to evaporate the n-hexane. The remaining oil was weighed and stored in a container. In mechanical extraction, the seeds were heated to a temperature of 75°C before they were fed into the press. A pressure of 150 bar was applied to extract the oil. To separate the seed cake and oil mixture, centrifuge was used. The oil was then filtered at a temperature of about 80°C to prevent oil from becoming too viscous.

4.3 RUBBER SEED OIL ANALYSIS

The properties such as acid value, density, kinematic viscosity, acid value and FFA% were determined before utilizing this oil for the transesterification reaction. If

the values of the mentioned properties were not within the standards, then the oil was planned to be pre-treated in order to ensure effective synthesis of oil to quality biodiesel product.

4.4 PREPARATION OF CATALYST

0.47g of Carbon nanotubes and 50 mg of Titanium dioxide was taken. Titanium dioxide was mixed with distilled water in one beaker and Carbon nanotubes was mixed with ethanol in another beaker. Titanium dioxide was dissolved whereas Carbon nanotubes was still insoluble. Now both the solutions were combined in the single beaker. The beaker containing the solution was put in the sonification reactor for 30 min, after this process the solution became slightly soluble. The solution was kept on a magnetic stirrer at 500 rpm for 30 min, the solution became even more soluble than the last time. The solution was poured into a mini HTL which is a closed container tightened with bolts and nuts. The mini HTL was kept in an oven reactor at 130°C for 8 h. After 8 h, the solution was then poured from the mini-HTL into a 200 mL beaker. The solution was taken and it was water washed using ethanol for three times. The solution was sent to the centrifugation reactor operating at 25°C and 500 rpm for 50 min. Once the centrifugation reaction was complete the solution was then poured into a dry dish and after the drying process, catalyst was obtained in the form of minute powder.



Fig.4.1: a) Centrifugation of catalyst



b) Drying the catalyst

4.5 CHARACTERIZATION OF CATALYST

The catalyst was characterized by means of XRD (ARL EQUINOX 3000) and FT-IR (Shimadzu IRAffinity-1S). X-Ray Diffraction is a technique used to analyze the crystal structure of a material. By measuring the lattice parameters, crystal symmetry, and Miller indices of the diffraction peaks, XRD can provide details about a substance's crystal structure. The interplanar spacing (d) of the crystal lattice planes that diffract X-rays at this angle may be calculated using Bragg's Law ($n = 2d \sin \theta$), the sampling is scanned using 2θ , and the range is from 10° to 60° . The wavelength of Cu radiation is 1.54 Å.

FT-IR (Fourier Transform Infrared Spectroscopy) is a powerful analytical technique used to identify and quantify chemical compounds based on their vibrational modes. It works by measuring the absorption or transmission of infrared radiation through a sample, which is then used to determine the presence and concentration of specific functional groups in the sample.

4.6 ACID ESTERIFICATION

Due to the presence of high Fatty acid percentage in rubber seed oil esterification must be performed as a pre-treatment step to reduce the FFA in oil below 2% for effective conversion of oil to biodiesel, acid esterification is a chemical reaction in which triglycerides react with lower alcohol such as methanol in the presence of an acid catalyst and reduced the high amount of free fatty acids. In this work sulfuric acid (H_2SO_4) was used as the acid catalyst to reduce the FFA content. Before starting with the esterification process, moisture content in the oil was reduced by heating the oil to 110°C . 1 L of Crude rubber seed oil was taken into a 2000 mL three necked round bottom flask which was coupled with a condenser to recondense the evaporated methanol. The flask containing the oil was kept on a magnetic stirrer at 500 rpm and was preheated till 60°C . Methanol was taken in the ratio of 1:3 of oil to methanol, so for 1 L of oil 182 mL of methanol was taken, for every 1% of FFA 0.25 mL of concentrated sulfuric acid (H_2SO_4) was taken as an acid catalyst, so according to FFA present in the oil a total of 9.5 mL of concentrated sulfuric acid was taken and mixed with methanol in a conical flask. Once the oil reached the desired temperature, mixture of methanol and sulfuric acid was added to the oil in the round bottom flask, the mixture was maintained at 60°C at a stirring rate of 500

rpm for 2 h. Every 15 min a mercury thermometer was used to check the temperature. After 2 h the reaction mixture was allowed to cool down till room temperature and then poured into a separating funnel. The reaction mixture formed two separate layers, the upper layer constituted of the esterified oil, whereas the bottom layer constituted of methanol, sulfuric acid and other impurities. The bottom layer was separated and the esterified oil was transferred to a beaker and heated till 110°C in a mantle to vaporize methanol and to remove any moisture content present in the oil. The FFA for treated oil was checked and later collected for further transesterification process to convert the oil to biodiesel.

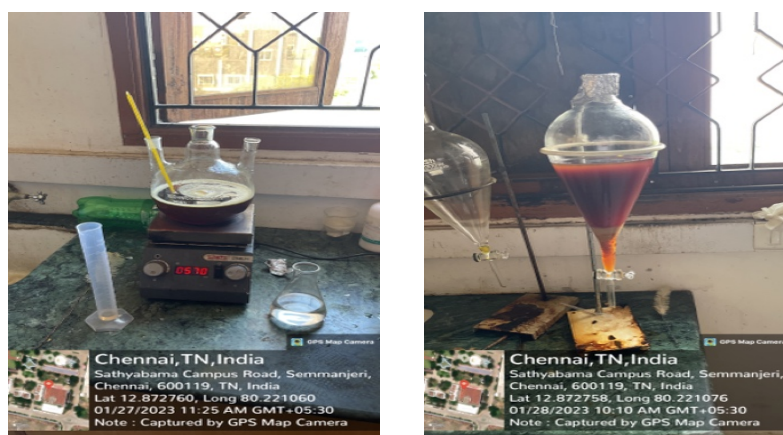


Fig. 4.2: a) Esterification of oil b) Separation of oil.

4.7 TRANSESTERIFICATION

After the FFA% in the oil was reduced below 2%, the oil was now ready to be sent for transesterification reaction. 3 samples of 100 mL oil was taken to produce 3 sample of biodiesel, where one sample of oil was taken and converted to biodiesel using NaOH which is conventional homogeneous catalyst, second sample of oil was converted to biodiesel using NaOH as well as CNT-TiO₂ which is a combination of both homogeneous and heterogeneous catalyst, and third sample was converted to biodiesel using CNT-TiO₂ alone which is a heterogeneous catalyst, these different combinations of catalyst were taken in order to compare the yield and biodiesel properties between the 3 samples and to understand the effect of heterogenous catalyst. The oil was poured into a 250 mL three necked round bottom flask and preheated till 60°C stirred at 500 rpm on a magnetic stirrer. Methanol was taken keeping oil to methanol ratio of 1:6, so for 100 mL of oil 18.2 mL of methanol was taken and mixed with 0.5g of the NaOH pellets in a conical flask. Once the oil

reached the desired temperature, the alcohol and catalyst mixture were poured into the flask containing the oil, the reaction was allowed to happen for 2 h maintaining the temperature at 60°C and at a stirring rate of 500 rpm, the temperature was checked every 15 min using mercury thermometer. Once the reaction was complete, the mixture was allowed to cool till room temperature and then sent to a separating funnel for separation process, there were two layers formed where the upper layer was biodiesel and the lower layer was glycerol, catalyst and other impurities which was removed.

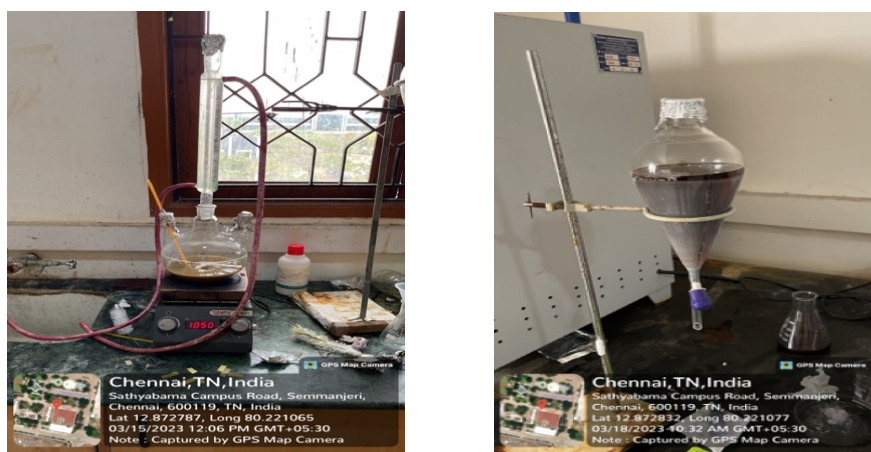


Fig. 4.3: a) Transesterification. b) Biodiesel separation.

4.8 WASHING TECHNIQUE

4.8.1 Wet Washing Technique

The biodiesel produced using the presence of homogenous catalyst was now sent for wet washing treatment to remove impurities such as methanol, catalyst, unreacted oil, etc. where the water was heated between 60°C and 79°C and then poured into a separator along with the biodiesel, and let it remain undisturbed for 15 min, the water collected at the bottom was removed and freshly heated water was again mixed with biodiesel in the separator. This process was repeated till the water was neutralized which was checked using litmus paper.

4.8.2 Membrane Washing technique

Biodiesel produced using heterogeneous catalyst was purified using membrane technique where a filter bag having a pore size of 5 μ was used to remove the catalyst and any other impurities present in the biodiesel.



Fig. 4.4: a) Membrane washing.

b) Wet washing.

4.9 BIODIESEL CHARACTERIZATION

The biodiesel was characterized by the means of GC-FID (Young In Chromass, YL6500GC), which was utilized to analyze the fatty acid methyl esters (FAMES) in our biodiesel samples in this research. Individual FAMES are separated and detected by the GC-FID based on their physical and chemical characteristics, such as boiling point and affinity for the stationary phase in the column. The FID detects fatty acid methyl esters (FAMES) by measuring their capacity to ionize in the flame and producing a current proportionate to the quantity of FAME in the sample.

4.10 DETERMINING THE PHYSIO-CHEMICAL PROPERTIES OF BIODIESEL

4.10.1 Moisture content (ASTM D2709)

The amount of moisture in biodiesel is measured using a heating process. A crucible is a substance that has been used in this procedure. 5 g of biodiesel sample was taken and then the total weight of the crucible with biodiesel sample was measured and recorded. The sample was then placed in a hot air oven set at 110°C for a period of 3 h. Once the sample was cooled. The total weight of the sample with crucible is then measured and recorded. Weight difference before and after heating was used to determine the loss of moisture content.



Fig. 4.5: a) Sample weighing.

b) Moisture removal in oven.

4.10.2 Density (ASTM D1298)

The density of biodiesel was determined using a pycnometer. A pycnometer is a stoppered glass container with a predetermined capacity. The mass of pycnometer and the mass of both pycnometer with the sample biodiesel is recorded. The difference is divided by the volume of the pycnometer and the density is arrived.

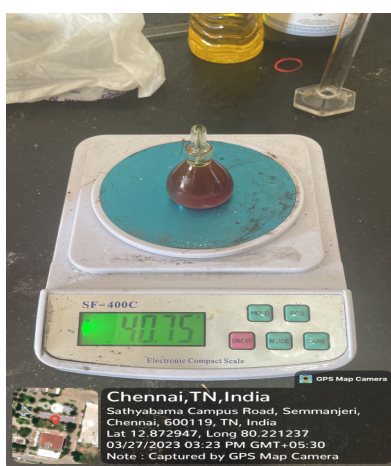


Fig. 4.6: Finding density using pycnometer.

4.10.3 Kinematic viscosity (ASTM D445)

It is an important property to determine as it affects the fuel's flow characteristics and lubricity. The fluids tendency to resist flow is called kinematic viscosity, which is measured in centistokes (cSt). According to ASTM standards the fluid viscosity should be within 1.9 centistokes (cSt) and 6.0 centistokes (cSt) at 40°C. The viscosity has been measured under the standard of ASTM D 445. First the U-tube

viscometer was ensured whether it was clean and dry. The biodiesel sample was poured into the U-tube viscometer and then kept in a temperature-controlled bath and was allowed to reach 40°C. Using a stopwatch, the time taken for a fixed volume of biodiesel sample to flow through the viscometer was recorded in seconds. The kinematic viscosity of the biodiesel was found using the following formula: time recorded (in secs) x 0.014 = kinematic viscosity (in centistokes)



Fig. 4.7: Viscosity

4.10.4 Free fatty acid and acid value (ASTM D664)

The FFA and acid value for the biodiesel was evaluated by performing titration procedure, for this process, a NaOH burette solution with a normality of 0.1 was first prepared. 5 g of biodiesel sample was taken in a conical flask. In addition, 25 mL of isopropyl alcohol was added to a conical flask containing the biodiesel sample. The combination was then given a total of four drops of burette solution. The solution was then heated until it reaches a temperature of 60°C. The sample was then allowed to cool to room temperature. Once the solution had reached room temperature, the phenolphthalein indicator was added. After that, the solution was titrated and the results were recorded and the acid value and FFA were calculated from the results using the Eq. (1) & Eq. (2) respectively.

$$\text{FFA (\%)} = \frac{28.2 \times (\text{burette reading}) \times 0.1}{5} \quad (1)$$

$$\text{Acid value} = \text{FFA (\%)} \times 1.99 \quad (2)$$



Fig. 4.8: Titration to find FFA%.

4.10.5 Cloud point (ASTM D2500) and Pour point (ASTM D97)

A sample of the petroleum product was placed in a glass test jar and progressively cooled to determine the cloud point. The cloud point temperature was determined by passing a light beam through the jar at the temperature at which the wax crystals begin to emerge and scatter the light. The pour point temperature was calculated by progressively chilling the sample in a test jar and monitoring the temperature at which the fluid stopped to flow when the jar was tilted. Typically, the pour point was determined by tilting the jar at regular intervals and watching the biodiesel sample until it no longer flowed. The temperatures were recorded for both cloud and pour point.



Fig. 4.9: Cloud point and Pour point

4.10.6 Calorific value (ASTM D6751)

The quantity of heat emitted when a unit of fuel is entirely burnt is referred to as its calorific value. It is often measured in J/g or kcal per kg of fuel. An instrument called bomb calorimeter was utilized to determine the calorific value of the biodiesel by determining the heat energy liberated during the process of combustion.

A little metal container called as “bomb” was filled with high-pressure oxygen and weighed. The bomb was put within a bigger container filled with water, which served as a heat sink, absorbing the heat emitted during burning. The metal container was filled with the biodiesel sample. The biodiesel was lit using a slight electrical discharge, which cause it to catch fire. The heat generated upon combustion was transmitted to the water, which heated it considerably. A thermometer was used to measure the increase in the temperature of the water. The biodiesel's calorific value was determined based on the rise in water temperature and the quantity of biodiesel utilized. The calorific value was calculated in terms of cal/g.



Fig. 4.10: a) Bomb calorimeter



b) Thermometer

4.10.7 Flash Point (ASTM D93)

The flash point of a liquid is the lowest point of temperature at which it can produce enough vapors to generate an ignitable combination in air near the liquid's surface. It is the temperature at which a liquid can ignite if it comes into contact with an ignition source, such as a flame or spark.

A laboratory apparatus called Pensky-Martens closed-cup tester was utilized to evaluate the flash point for the biodiesel sample. A small sample of biodiesel was poured in a cup, which was then heated at a regulated pace with a flame source. The flash point was defined as the temperature at which a flare of flame was visible over the outer layer of the liquid biodiesel sample.



Fig. 4.11: Flash Point

4.11 REUSABILITY OF CATALYST

The recovery of catalyst was studied for biodiesel produced using CNT-TiO₂ only. Once the transesterification was complete the catalyst was accumulated at the bottom of the separator which was easily separated and transferred to a separate beaker, the catalyst was mixed with some amount of oil and methanol in it, so the catalyst was then kept in oven at 110° C to evaporate the methanol. To remove the oil, 15 mL of n-hexane was added to the catalyst and oil mixture and it was stirred for few minutes to remove any oil and organic residues that may be present on the surface of the catalyst. The mixture was left to settle for several hours. The top layer of the mixture, which contains the n-hexane and organic residues, was then removed and by filtering the catalyst using filter paper. The washed catalyst was then dried to remove any residual n-hexane. This was done by heating the catalyst in an oven at a temperature of around 120°C-150°C for several hours. Once the catalyst was dried, it was then subjected to calcination, where the catalyst was heated in air or an oxygen-containing atmosphere at 650°C for 3 h. After calcination, the catalyst was cooled and was again used for transesterification.

CHAPTER-5

RESULTS AND DISCUSSION

5.1 OIL EXTRACTION

The extraction efficiency for hydraulic pressing, solvent extraction and mechanical pressing was about 39.47%, 31.65% and 27.78% respectively. The extraction efficiency of hydraulic press, showed the greatest oil yield, which was an appealing outcome. The hydraulic press produces a greater yield of oil with higher quality, has lower running costs, is ecologically friendly, and is adaptable. It is a dependable and efficient oil extraction technology that can be used on a wide range of plant materials, making it an affordable option for small-scale production. Rubber seed oil may be a feasible source for biodiesel or other industrial uses because of its high yield of oil that could be extracted from rubber seeds. In comparison with other sources of edible and non-edible oils, rubber seeds are a cheap supply of feedstock. A high extraction efficiency allows for the extraction of more oil per kilogram of seeds, which lowers the cost of the biodiesel synthesis process.

5.2 PROPERTIES OF RUBBER SEED OIL

Table 5.1 shows the properties of rubber seed oil utilized in this study and its FFA content. The basic oil analysis such as moisture content, density, kinematic viscosity, acid value, FFA% were performed on Crude Rubber seed oil. The properties are important to determine before transesterification reaction is employed ensuring product quality, reaction efficiency and safety. For oils having a high kinematic viscosity and density, may require longer reaction periods as well as higher reaction temperatures to complete the conversion to biodiesel. If Properties such as acid value and FFA% in the oil are high then it might cause undesired reactions and contaminants in the end biodiesel product leading to poor quality of biodiesel. Therefore, in order to reduce the acid value and FFA% of oil, acid esterification process was performed in order to reduce the FFA% of oil below the safer level of 2%. High moisture content in oil can also lead to various safety hazards during the transesterification reaction, as the water content in oil might cause the reaction to splatter, potentially resulting in flames or explosions. Therefore, in order

to reduce the moisture content, the oil was subjected to high temperature to evaporate the water molecules present in the oil.

Table 5.1: Properties of the Rubber seed oil.

Properties	Testing procedure	Rubber seed oil
Moisture content%	ASTM D2709	0.414
Density (g/mL)	ASTM D1298	0.904 g/mL
Kinematic Viscosity (cSt)	ASTM D445	34.86 cSt
Acid value	ASTM D664	74.625
FFA%	ASTM D664	37.5

5.3 CHARACTERIZATION OF THE CATALYST

5.3.1 Fourier Transform Infrared (FT-IR)

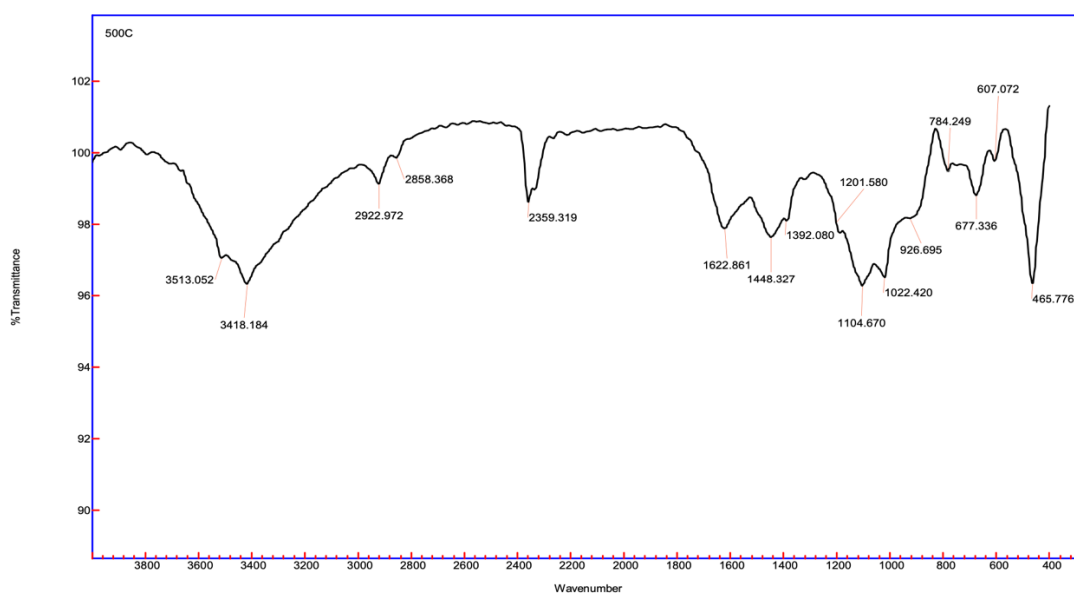


Fig. 5.1: FT-IR analysis image of CNT-TiO₂ catalyst

The FT-IR analysis of the CNT-TiO₂ revealed the presence of C-H stretching at peak values of 2922.972 and 2858.368 cm⁻¹. The presence of O-H stretching was observed at peak values of 3513.052, 3418.184, 1392.080 cm⁻¹. The existence of C=O stretching was suggested at the peak values of 1622.861, 784.249, 677.336

cm^{-1} . The presence of C-O stretching was observed at peak values of 1201.580 and 1104.670 cm^{-1} .

5.3.2 X-Ray Diffraction (XRD)

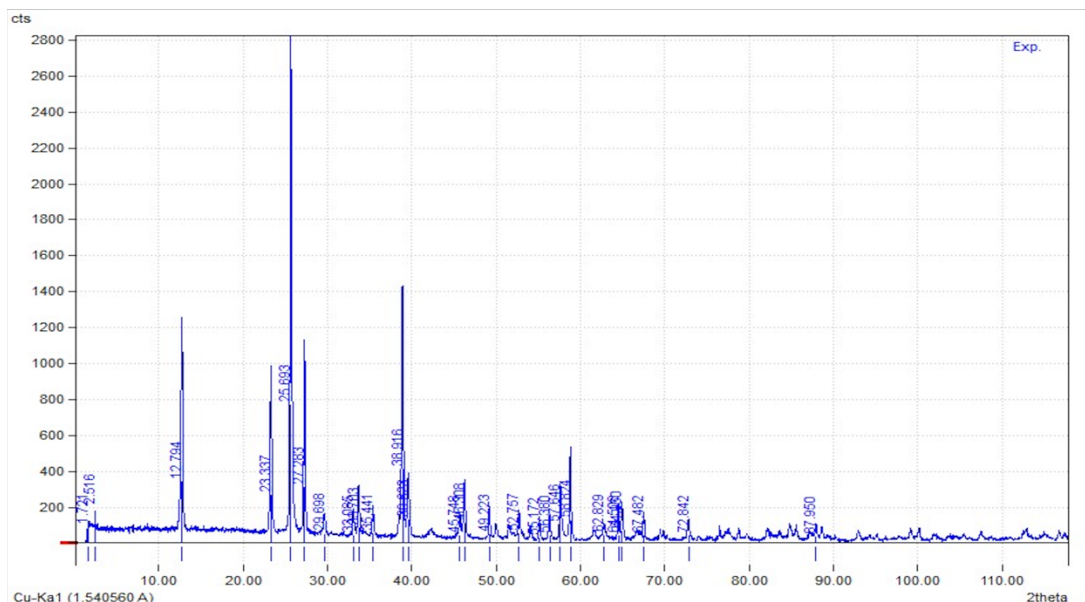


Fig. 5.2: XRD analysis image of the CNT-TiO₂ catalyst.

One of the key advantages of XRD analysis is its capacity to distinguish between different crystal structures of the exact same material and identify unknown compounds. XRD analysis may also provide quantitative data on the relative amounts of each phase present in a sample.

Finding H, K, L, the basic idea underlying X-ray diffractogram analysis (XRD) is Bragg's rule, which connects the wavelength of X-rays to the distance between crystal surfaces in a substance:

$$n\lambda = 2d \sin\theta, \text{ where } d = a / \sqrt{h^2 + k^2 + l^2}$$

$$n \lambda / 2 \sin \theta = a / \sqrt{h^2 + k^2 + l^2},$$

where d is the distance between crystal planes, n is an integer, θ is the angle of incidence of X-rays on the crystal and λ is the wavelength of X-rays.

The sampling is scanned using 2θ , and the range is from 10° to 60° . The wavelength of Cu radiation is $1.54 \text{ \AA}$, where $n=1$, a is lattice constant $=10 \text{ \AA}$.

The possible values of h , k , and l that satisfy this equation are: $h = 2, k = 1, l = 0$ or $h = 0, k = 2, l = 1$ or $h = 1, k = 0, l = 2$

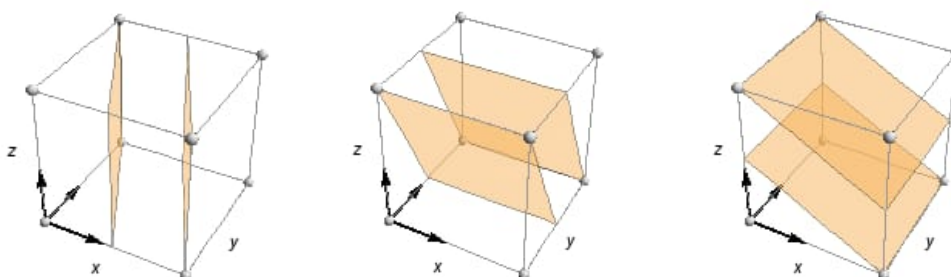


Fig. 5.3: Crystal structure from H,K,L

5.4 FFA REDUCTION OF RUBBER SEED OIL BY ESTERIFICATION

The results for FFA% reduction in crude rubber seed oil which was done by acid esterification technique with intermediate moisture removal technique is summarized in table 5.2.

Table 5.2: FFA% reduction results for rubber seed oil

Technique used	FFA% results
After 1 st moisture removal	33.72%
After 1 st esterification	8.17%
After 2 nd moisture removal	6.09%
After 2 nd esterification	1.12%

From the results it can be concluded that to reduce the FFA% for rubber seed oil below the safer level of 2% esterification has to be performed two times. Using the esterification procedure to reduce the FFA concentration of oil to less than 2% has various advantages. The oil's quality and stability are improved since excessive FFA levels can cause oxidation and degradation. The production of biodiesel reduces significantly when the FFA concentration of the oil exceeds 4%. The reason for this is because sometimes commercially used alkaline catalysts react with FFAs to

generate soap, which hinders the separation of ester and glycerin. In conclusion, lowering the FFA concentration of oil to less than 2% can result in enhanced quality and stability, lower processing costs, and higher yield during refining.

5.5 EFFECT OF TYPE OF CATALYST ON BIODIESEL YIELD

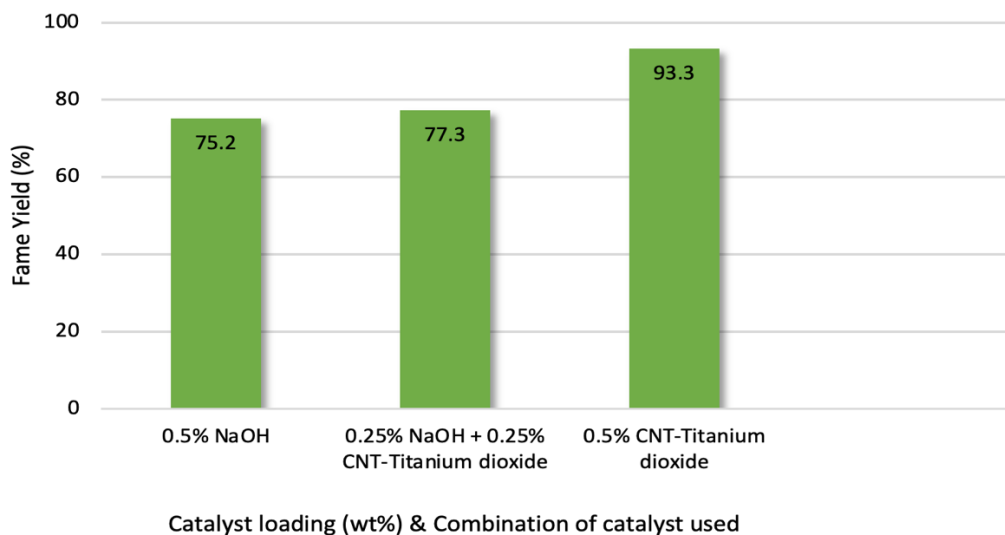


Fig. 5.4: Comparison of yield% for all the three samples

The yield percentage for all the three biodiesel samples which were produced using the two types of catalyst categories in the combination of NaOH only (homogenous catalyst), NaOH (homogeneous) + CNT-TiO₂ (heterogenous) and CNT-TiO₂ only (heterogeneous) were recorded and compared, which is represented in Fig 5.3. From the yield% results it can be concluded that when heterogenous catalyst was used biodiesel yield was significantly higher. The reason being is due to the type of washing technique used. When the biodiesel was prepared using homogeneous catalyst, wet washing technique had to be employed, which significantly reduced the biodiesel yield as during wet washing the process of separation was difficult, as when the water was drained out from the separating funnel, some amount of biodiesel also came along with water leading to reduction in overall biodiesel amount, resulting in reduced biodiesel yield. Since the wet washing technique was repeated 6 times for biodiesel produced using homogeneous catalyst and 4 times for biodiesel produced using combination of homogeneous and heterogeneous, each time the wet washing technique was employed the wastage of biodiesel during the separation process took place leading to a significant reduction in the yield of biodiesel. Whereas when membrane washing was used the separation of the

biodiesel from the catalyst and other impurities was easy and less time consuming. There was no need for water washing and the wastage of biodiesel during the process was significantly less, therefore leading to a better yield.

5.6 CHARACTERIZATION OF THE BIODIESEL

The chromatogram in Fig.5.4 obtained was analyzed and the types of fatty acid methyl esters were determined by interpreting the retention time, area time and height% of each FAME visibly present in the chromatograph with standard reference library and database which contains retention time index and spectra of relevant FAME.

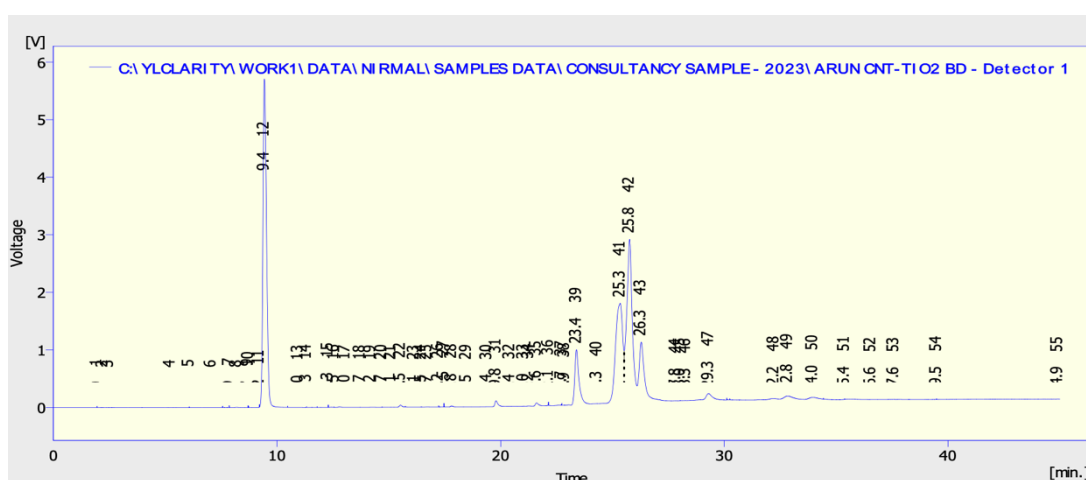


Fig. 5.5: GC-FID chromatograph for biodiesel produced using CNT-TiO₂ catalyst.

In the biodiesel sample, there was a total of 80% saturated fatty acid methyl ester content, where myristic acid, tricosanoic acid and lignoceric acid were present in the percentage of 26.4, 48.2, and 5.4. The presence of these saturated fatty acids help can increase the cetane number of the biodiesel, which is a measure of its ignition quality. Higher cetane numbers result in more efficient combustion and smoother engine operation. These saturated fatty acids methyl esters have a relatively high melting point and can improve the low-temperature properties of biodiesel. They can reduce the cloud point and pour point, which are the temperatures at which the fuel begins to solidify and lose its fluidity. By reducing these temperatures, saturated fatty acids can help to ensure that biodiesel remains liquid and flows freely even in cold weather conditions.

5.7 PHYSIO-CHEMICAL PROPERTIES OF THE BIODIESEL

The properties of the biodiesel produced using CNT-TiO₂ were recorded and compared with the ASTM6751 standards to analyze the fuel's characteristics which is summarized in table 5.3.

Table 5.3: Properties of Biodiesel synthesized using CNT-TiO₂ catalyst

Property type	Biodiesel using CNT-TiO ₂	ASTM D6751
Moisture content (%)	0.087	< 0.03
Density (g/mL)	0.887	0.85-0.9
Viscosity (cSt)	4.234	1.9-6.0
Cloud point (°C)	5	-3 to 12
Pour point (°C)	-22	-15 to 10
Acid value	0.5662	0.80 max
FFA (%)	0.28	0.42 max
Calorific Value (cal/g)	8763.88	8359.61 – 9553.84
Flash Point (°C)	165	130 min – 191max

5.7.1 Moisture content

The moisture content % of the biodiesel met the ASTM D6751 standard with the value of 0.087%, as the standard specifies the value to be less than <0.03%. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below:

1. Avoiding microbial growth: Microbial development may occur in biodiesel, which can lead to its degradation and spoiling. Microorganisms require moisture in order to thrive, therefore keeping the moisture level of biodiesel low can help limit microbe development.

2. Preventing hydrolysis: When biodiesel comes into contact with water, it can undergo hydrolysis, a chemical process that breaks up the bonds of ester in the fuel. This might result in the development of free fatty acids and other byproducts, which can harm the engine and degrade the fuel economy. Keeping the moisture level of biodiesel low can help avoid hydrolysis.

3. Improving fuel quality and stability: Moisture can also have an impact on biodiesel's overall quality and stability. Water in the fuel can cause emulsions to develop, making the gasoline challenging for separation from water and reducing its energy content. Furthermore, moisture can contribute to biodiesel oxidation, which can result in the creation of deposits and other byproducts that degrade the fuel's performance and longevity. Biodiesel can preserve its quality and stability over time by keeping the moisture content low.

5.7.2 Density

The density of the biodiesel determined was 0.887 g/mL, which lies between the acceptable range of 0.85 - 0.9 g/mL as specified by the ASTM D6751 standard. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below:

1. Quality control: One of the most significant indicators of biodiesel fuel quality is density. Evaluating the density of biodiesel aids in ensuring that it fulfills the necessary standards and regulations, as well as detecting any potential impurities or pollutants that may impair its efficiency and performance.

2. Performance and efficiency: The density of biodiesel fuel is critical to its performance and economy. It has an influence on the energy content, combustion characteristics, and also viscosity of the fuel, all of which can affect engine performance, fuel efficiency, and greenhouse gas emissions.

3. Blending and mixing: Biodiesel is frequently blended with other fuels to increase its qualities and performance, such as diesel, petrol or sustainable diesel. The density of biodiesel fuel is a crucial component in calculating the optimum blend ratio and ensuring correct fuel mixing and distribution, which can impact the overall quality and consistency of the blended fuel.

5.7.3 Kinematic viscosity

The kinematic viscosity of the biodiesel determined was 4.234 cSt at 40°C, which lies between the acceptable range of 1.9 – 6.0 cSt at 40°C specified by the ASTM D6751 standard. This result is significant as it indicates that the biodiesel synthesized in this study will have the following favorable characteristics.

1. Engine performance: Biodiesel's kinematic viscosity might impair the fuel's potential to flow correctly and effectively in an engine. If the viscosity is excessively high, it can lead to very poor fuel atomization, unfinished combustion, and increased engine wear. If the viscosity is excessively low, it might cause leaking and reduced lubrication. Finding the proper kinematic viscosity for biodiesel is critical for guaranteeing optimal engine efficiency.
2. Fuel transportation: Biodiesel's kinematic viscosity influences its ability to be transferred through pipes, tanks, and other means of transportation. When the viscosity is excessively high, it increases the likelihood of blockages and makes transportation more challenging and costly. If the viscosity is excessively low, however, it might cause spills and leaks during shipping. As a result, determining the proper kinematic viscosity is critical for guaranteeing safe fuel delivery.

5.7.4 Cloud point and pour point

The cloud and pour point of the biodiesel were determined to be 5°C and -22°C, which are within the acceptable range of -3 to 12°C for cloud point and -15 to 10°C for pour point as specified by the ASTM D6751 standard. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below;

1. Cold Weather Conditions: The cloud point is the temperature at which microscopic solid particles begin to form in biodiesel, giving it a foggy or hazy appearance. This can pose issues in cold weather because the particles can block gasoline filters and fuel lines, making it harder for the fuel to flow correctly.

The pour point is the temperature at which biodiesel thickens and becomes viscous, making it impossible to flow. In cold temperatures, the biodiesel may become too

thick to pump or atomize adequately, resulting in partial combustion and poor engine performance.

5.7.5 Free fatty acid and acid value

The FFA% and acid value of the biodiesel was determined to be 0.28% and 0.5662, respectively, which met the ASTM D6751 standards. The maximum FFA% and acid value allowed is 0.42% and 0.80 respectively, as specified by the standard. This result is significant as it indicates that the biodiesel synthesized in this study has low acid value which helps prevents the following undesired conditions;

1. Accelerated Oxidation: FFA may speed up the process of oxidation in biodiesel, leading to the development of hazardous chemicals such peroxides and acids. This can cause the gasoline to deteriorate over time, lowering its efficiency and potentially causing engine damage.
2. Corrosion: FFA in biodiesel can also cause corrosion in fuel system components such as tanks, pipes, and injectors. This can result in clogs, leaks, and other mechanical issues.
3. Formation of Deposits: FFA may react with metal ions to generate insoluble salts and other deposits that can clog fuel filters and other components of the fuel system.

5.7.6 Calorific value

The calorific value of the biodiesel was determined to be 8763.88 cal/g, which lies within the acceptable range of 8359.61 – 9553.84 cal/g as specified by the ASTM D6751 standards. A Biodiesel's calorific value is a significant component in assessing its energy content and appropriateness for usage in various applications such as combustion engines, electricity generating, and heating systems. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below;

1. Energy Content: The calorific value of biodiesel is critical in calculating its energy content, which influences engine efficiency. A greater calorific value indicates that more energy is accessible from the fuel, which results in improved engine performance. If the calorific value is lower than that of typical diesel fuel, the engine

may require more fuel to generate the same amount of energy, resulting in worse fuel efficiency.

2. Economic analysis: Knowing biodiesel's calorific value is critical in establishing its cost-effectiveness in comparison to other fuels. This data is used to assess the retail price of biodiesel as well as the potential savings for users.

Furthermore, measuring the calorific value of biodiesel enables for precise assessments of energy content and prices, allowing producers and customers to make better decisions.

5.7.7 Flash point

The flash point of the biodiesel was determined to be 165°C, which lies within the acceptable range of 130°C – 191°C as specified by the ASTM D6751 standards. The flash point of a liquid is the lowest point of temperature at which it can produce enough vapors to generate an ignitable combination in air near the liquid's surface. It is the temperature at which a liquid can ignite if it comes into contact with an ignition source, such as a flame or spark. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below;

1. Reduced explosion risk and fire: Biofuels with higher flash points are far less likely to catch flames and cause an explosion or fire. As a result, they are easier to handle during storage, shipping, and dispensing. Higher flash points assist to safeguard personnel, equipment, and facilities by lowering the danger of fire and explosion.

2. Easiest transportation and storage: Because they require fewer severe safety procedures, biodiesel with a higher flash point may be stored and transported more simply. For example, if a biodiesel flash point exceeds a specific level, it may not be pivotal to store it in a specially constructed, fire-rated storage facility. This can save both time and money on storage and transit charges.

5.8 REUSABILITY OF THE CATALYST

The yield of biodiesel decreased each time the catalyst was regenerated and used for transesterification reaction as depicted in the Fig 5.5. Under the specified optimal process parameters, the biodiesel output decreased from 93.33% to 67.8% for 5

cycles of catalyst reuse. The reason being the increase in the formation of glycerol and decrease in the formation of biodiesel. When the catalytic activity reduces, its selectivity towards the transesterification decreases while its selectivity towards the hydrolysis of unreacted triglycerides increases, leading to increase in glycerol formation. The reason being due to reduced catalytic activity, partial transesterification of the triglycerides takes place leading to the formation of intermediates such as monoacylglycerols (MAGs) and diacylglycerols (DAGs) which undergoes hydrolysis in order to form fatty acids and glycerol. All of the ester bonds from triglycerides may not break to react with alcohol to form desired esters due to reduced catalytic activity. The catalytic activity decreases after each reuse which might be owing to molecules of oil blocking the active sites on the catalyst surface despite thorough washing with n-hexane and other factors like poisoning, leaching, sintering. Nonetheless, the catalyst could be reused at least for 5 times before employing new catalyst. Since it permitted catalyst re-use, the total process can be described as affordable as well as environmentally friendly.

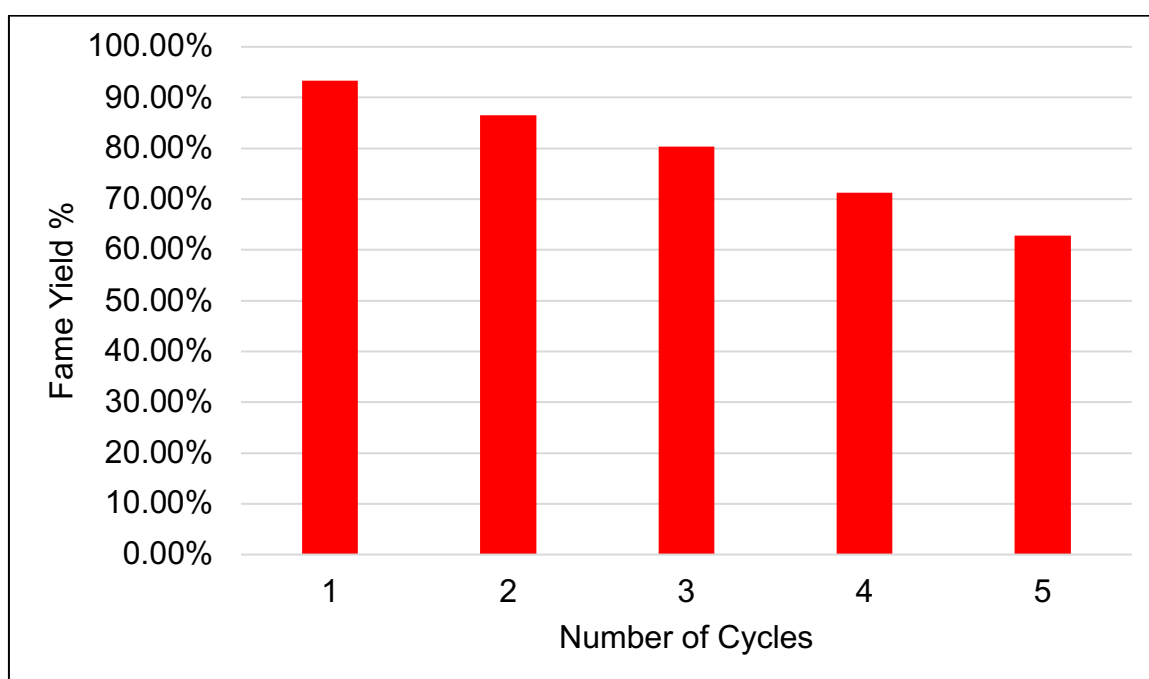


Fig. 5.6: Regeneration studies

CHAPTER-6

SUMMARY AND CONCLUSION

Biodiesel was successfully synthesized from rubber seed oil using a novel catalyst called CNT-TiO₂ composite which is categorized as a heterogeneous catalyst. Transesterification reaction using homogeneous catalyst proved to give less yield of biodiesel when compared with when transesterification was done using heterogeneous catalyst, the reason being less glycerol formation and easy separation of biodiesel from glycerol resulting in less wastage of biodiesel when heterogeneous catalyst was used. The easy separation was assisted by membrane washing technique which was less time consuming, and there was no waste water generation, the separation technique helped to prevent wastage of biodiesel during the process, thus improving the biodiesel yield as well as increasing the efficiency of the whole synthesis process. Regeneration studies showed that the catalyst's activity was reduced each time it was reused resulting in a decrease in biodiesel yield, but from the results it can be inferred that the catalyst could be reused at least 5 times before employing a new catalyst. This proves that the catalyst has a good life and is economical for the biodiesel synthesis. The properties of the biodiesel were compared with ASTM D6751, and all the values of the properties met the standards, thereby proving that the biodiesel produced is of good quality to be able to attract fuel buyers and suppliers and can meet regulatory benefit and other tax incentives.

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CHAPTER-1

INTRODUCTION

1.1 BIODIESEL

Due to its potential to displace conventional fossil fuels, biodiesel is an environmentally friendly replacement fuel that has attracted growing attention in recent years. It is a kind of diesel fuel produced using renewable materials like recycled grease, animal fats, and vegetable oils. This essay will give a thorough introduction to biodiesel, covering its background, production processes, benefits, drawbacks, environmental impact, governmental regulations, and prospects for the future (Ayoola et al., 2021).

1.1.1 History of biodiesel

Biodiesel has a lengthy history going back more than a century. The idea of utilizing vegetable oils and other naturally occurring substances as fuel dates return to the late 1800s, when Rudolf Diesel designed the first engine powered by diesel. Instead of using fossil fuels at first, Diesel built his engine to run on vegetable oils like peanut oil. He thought that using these fuels would give the engine a cleaner, longer-lasting source of energy.

In the early 1900s, the idea of utilizing vegetable oils as energy became more and more popular, particularly in rural areas where fuels made from petroleum were harder to come by. Different vegetable oils, including soybean oil and cottonseed oil, were tested by farmers and other rural residents as potential sources of fuel (Mythili et al., 2014).

Vegetable oil was initially used as a fuel, but its production and processing were expensive, which prevented its widespread use. Most consumers preferred petroleum-based fuels because they were more affordable and easily accessible.

The oil embargo and ensuing energy crisis in the 1970s gave rise to the idea of biodiesel as a sustainable and environmentally friendly fuel. Biodiesel was considered to be a potential solution as investigators and scientists started looking for other sources of energy.

James J. Beckman, who had invented a method for transforming vegetable oils into a fuel suitable for diesel engines, received the first official biodiesel patent in 1977. The vegetable oil was combined with methanol and sodium hydroxide in Beckman's procedure to produce methyl esters, which are the main constituent of biodiesel.

Researchers worked to improve the biodiesel manufacturing procedure throughout the 1980s and 1990s, and as a result, commercial production accelerated. The first commercial biodiesel plant opened in Austria in 1989, marking Europe's introduction of the fuel on a large scale (Khan et al., 2017).

Throughout the course of the 1990s as well as the beginning of the 2000s, the consumption of biodiesel grew in Europe, in part due to government initiatives that promoted the use of environmentally friendly fuels. After the Renewable Fuel Standard was passed in 2005 and made it mandatory to use sustainable fuels in transportation fuel, the biodiesel industry in America started to boom in the early 2000s.

As more people search for more sustainable and environmentally friendly energy sources, biodiesel is currently generated and utilized all over the world. Although there are still obstacles to be overcome, like the cost for generation and the scarcity of feedstocks, biodiesel has advanced significantly from its early days as a specialised fuel used by farmers in the countryside (Mohiddin et al., 2021).

1.1.2 Advantages of biodiesel

An environmentally friendly, non-toxic, and sustainable alternative to diesel fuel made from petroleum is biodiesel. Compared to conventional diesel fuel, it has a number of benefits, including (Datta et al., 2019):

1. Renewable: Vegetable oils and animal fats, which are both readily accessible and replenishable, are used to make biodiesel.
2. Reduced greenhouse gas emissions: When compared to conventional diesel fuel, biodiesel can cut greenhouse gas emissions by up to 80%. This is due to the fact that biodiesel is burned, it produces less carbon dioxide and other contaminants because it is made from organic materials that absorb carbon dioxide during their growth.

3. Enhanced air quality: Compared to conventional gasoline and diesel, biodiesel burns more cleanly, emitting less sulphur dioxide and other pollutants. This enhances the quality of the air and lowers respiratory issues in localised areas.
4. Biodegradable: Because biodiesel decomposes rapidly in the environment, it won't harm water or soil resources over the long term.
5. Better engine performance: Because biodiesel has a larger cetane rating than regular petroleum fuel, it explodes more effectively and ignites quicker. This enhances the engine's capabilities by increasing power, improving fuel efficiency, and lowering maintenance costs.
6. Energy security is improved because biodiesel can be produced locally, reducing reliance on imported oil. Additionally, this promotes local economies and jobs in the biodiesel sector.
7. Versatility: Biodiesel is a flexible and adaptable fuel source because it can be utilized in any engine without any modification.
8. Reduced reliance on fossil fuels: Biodiesel contributes to energy mix diversification and reduces reliance on fossil fuels. By doing this, the dangers of using only fossil fuels for energy are lessened.

Comparing to traditional fuel, biodiesel has a number of benefits, including lowered greenhouse gas emissions, better air quality, improved engine performance, increased energy security, and a decreased reliance on fossil fuels. As a result, it plays a crucial role in the shift to a future with more sustainable and renewable energy.

1.2 GOVERNMENT POLICIES

An important part of encouraging the production and use of biodiesel is played by government policies. A lot of nations have put programs and incentives in place to promote the utilization of biodiesel as a raw material for clean and renewable energy. The following are a few of the most prevalent government biodiesel policies:

1. Many nations have adopted renewable fuel standards (RFS), which call for a specific proportion of renewable fuels, like biodiesel, to be blended with conventional fossil fuels. For instance, the Renewable Fuel Standard in the

United States mandates that a specific amount of renewable fuel be blended with petrol and diesel fuel each year.

2. Tax breaks: A lot of governments provide tax breaks to promote the production and use of biodiesel. For manufacturers, processors, and consumers of biodiesel, such incentives may take the form of tax credits, exemptions, and deductions (Galadima & Muraza, 2020; Janda et al., 2012).
3. Governments may also provide grant programmes to aid in the study and advancement of biodiesel manufacturing methods and technologies. The development of new biodiesel production facilities as well as the research, development, and demonstration of new technologies can all be funded by these programmes.
4. Governments occasionally provide fuel subsidies to make biodiesel more accessible and competitive with conventional diesel fuel. The higher production costs of biodiesel can be partially offset by these subsidies, which will also increase consumer access to it.
5. Mandates and targets: Some governments have established biodiesel use mandates and targets. These regulations mandate the use of a specific volume of biodiesel in heating oil, transportation fuels, and other uses. For instance, by 2020, the European Union wants to use 10% renewable energy in transportation.
6. Research and development initiatives: To support the development of novel biodiesel technologies and manufacturing processes, many governments also fund research and development initiatives. These initiatives could advance the sector and increase biodiesel's parity with conventional diesel fuel (Williams et al., 2006).
7. Environmental laws: Environmental laws that promote the use of biodiesel may also be implemented by governments. For instance, vehicle and other equipment emissions regulations may favour the use of biodiesel over conventional diesel

In general, government regulations have an important impact on encouraging production and the utilization of biodiesel. Governments can contribute to the development of the biodiesel industry by offering mandates, incentives, and other forms of support. These regulations can aid in lowering greenhouse gas emissions,

enhancing air quality, and advancing the utilization of recyclable and sustainable power sources.

1.3 PRODUCTION METHODS OF BIODIESEL

Biodiesel can be made from a variety of raw materials, including cooking oils, fats from animals. There are several methods for producing biodiesel, each having advantages and disadvantages. The following are some of the popular techniques for producing biodiesel (Liu et al., 2009):

1. Base catalysed transesterification :The most popular technique for producing biodiesel is base catalysed transesterification, which entails reacting a triglyceride with an alcoholic content and a basic activator. The triglyceride is converted in this reaction into glycerin and fatty acid methyl esters (FAME), which are then separated. The FAME can be used as biodiesel.
2. Acid catalyzed transesterification: Similar to base transesterification, acidic catalysed transesterification uses an acid catalyst (such as sulfuric acid) rather than a base as the catalyst. Because it can tolerate higher levels of contaminants, the acid catalyst is usually employed with feedstocks that are of lower quality or are contaminated (Gholami et al., 2020; ji, 201 C.E.).
3. Enzymatic transesterification: Enzymes rather than chemical catalysts are used in this method. The cost of this method may be higher than that of chemical catalysis, but it can yield greater quantities of biodiesel and work with a wider variety of feedstocks.
4. Supercritical methanol: In this process, methanol is heated with vegetable or animal-derived fats at high temperatures and pressures, causing the triglycerides to disintegrate into methyl esters and glycerol.
5. Pyrolysis is a process that involves heating biomass feedstocks, like algae or plant matter, without the presence of oxygen in order to create a liquid bio-oil that can be further processed into biodiesel. This process produces high yields of biodiesel from a variety of feedstocks, but it needs specialised equipment and can be pricey.
6. Hydrogenation: Through the process of hydrogenation, triglycerides are changed into saturated fatty acids by reacting vegetable or animal fat with hydrogen gas in the presence of a catalyst. Following that, biodiesel can be

created using conventional chemical catalysis to transform the saturated fatty acids. The production of high-quality biodiesel using this method is possible from a variety of feedstocks, though it is less common than other biodiesel production methods (Janda et al., 2012).

Overall, the availability of feedstock, the price of fabrication, and desired end product will all influence the type of biodiesel production chosen. Regardless of the method of production, biodiesel has the potential to be a key player in the shift to a future with more renewable and sustainable energy sources.

1.4 FACTORS AFFECTING BIODIESEL

Several factors can influence the grade and amount of biofuel generated during production. The following are some of the crucial elements influencing the production of biodiesel (Mathiyazhagan & Ganapathi, 2011):

1. Feedstock quality: The grade of the raw materials used to create biodiesel can have an important influence on the quality of the end result.. The best feedstocks for producing biodiesel are those with low impurity and water content levels.
2. Feedstock accessibility: The generation of biodiesel may be impacted by the accessibility of feedstocks. To minimise the cost of transportation and lower the carbon footprint of the production of biodiesel, high and locally accessible raw materials are preferred (Chozhavendhan et al., 2020; Mathiyazhagan & Ganapathi, 2011).
3. Kind and amount of catalyst: The rate of reaction and yield of biodiesel can be impacted by the kind and amount of catalyst used in the process. The final product's quality can vary depending on the characteristics of the catalyst used.
4. Type and concentration of alcohol: the kind and amount of alcohol used in the production of biodiesel can have an impact on the yield and rate of reaction. The most widely used alcohol is methanol, though ethanol and other alcohols can also be used.
5. Reaction time and temperature: The yield and quality of biodiesel can be impacted by the reaction time and temperature used in the production

process. Higher temperatures and longer reaction times can produce more biodiesel, but they can also produce biodiesel of lower quality.

6. Methods for separation and purification: The methods used to separate the biodiesel from glycerol and other by-products can have an impact on the overall quality of the end product. Effective separation and purification techniques can reduce impurities and raise the biodiesel's quality (Chozhavendhan et al., 2020; Mathiyazhagan & Ganapathi, 2011).
7. Utilization of by-products: The use of by-products, such as glycerol, can have an effect on the production of biodiesel. Utilizing by-products effectively can reduce waste and increase the biodiesel industry's overall sustainability.

Overall, a number of variables that can affect the type and amount of biodiesel produced have an impact on its production. Paying close attention to these elements can aid in maximising the generation of biodiesel and enhancing the sustainability of this significant renewable energy source.

1.5 CHALLENGES FACING BY BIODIESEL

Despite the reality that biofuel is a sustainable and ecologically harmless energy source, there are several barriers to its widespread adoption. The following are some of the major challenges facing biodiesel (Chen et al., 2021):

1. Infrastructure and distribution: There may be issues with the infrastructure and distribution systems for biodiesel. Compared to petroleum-based diesel, biodiesel has different properties that necessitate specialised handling and storage amenities as well as engine and automobile modifications. Additionally, biodiesel's distribution networks are less established than those for petroleum-based diesel, which can restrict its accessibility and availability.
2. Energy balance and carbon footprint: Concerns can also be raised about the energy balance and carbon impact of biodiesel. While inputs of energy such as fuel for machinery and electricity for processing are needed during the production process of biodiesel, despite being an energy source that is renewable. The carbon impact of biodiesel is also influenced by the feedstock's origin and the manufacturing processes. In some cases, the carbon footprint of biodiesel production and transportation may be higher than that of diesel derived from petroleum.

3. Barriers to policy and regulation: These obstacles can make it difficult to produce and use biodiesel. While other nations have not, some have put policies in place to encourage the creation and use of biodiesel. Regulations may occasionally place restrictions on the use of biodiesel, such as prohibitions on biodiesel blends or demands for pricey certification and testing.
4. Technical restrictions: The use and production of biodiesel are also subject to some technical restrictions. For instance, biodiesel may contain less energy than gasoline derived from petroleum, which may result in decreased fuel economy and range for automobiles. In addition, biodiesel may be more prone to oxidation and degradation, which may affect storage and shelf life (Balan, 2014).
5. Cost and availability of feedstocks: The price and accessibility of feedstocks can be a significant barrier to the production of biodiesel. Competition for feedstocks may increase as demand for biodiesel rises, pushing up costs and possibly creating shortages. Additionally, some feedstocks might not be accessible in specific areas, which would restrict the possibility of producing local biodiesel there.

Overall, even though biodiesel has many benefits as a renewable and sustainable energy source, the project faces plenty of challenges that must be addressed before its full potential may be fulfilled. It will take a combination of technological advancement, policy support, and market expansion to address these challenges.

1.6 TRANSESTERIFICATION PROCESS

The main method for turning waste cooking oils, animal fats, or vegetable oils into biodiesel is transesterification. In this process, lipids and alcohol react with the help of a catalyst to produce fatty acid esters and glycerin..

The catalysts used in this can either be acidic or basic. Acidic catalysts like sulfuric acid or hydrochloric acid are less frequently used because of how dangerous and corrosive they are. Due to their high activity and low cost, basic catalysts like sodium hydroxide or potassium hydroxide are most frequently used in the production of commercial biodiesel.

Esterification, glycerolysis, trans-esterification are the three steps in the transesterification reaction. When triglycerides are allowed to react with alcohol with the help of an acidic catalyst, free fatty acids and glycerin are produced in the first stage. In the subsequent step, glycerol and the leftover triglycerides along with free fatty acids combine to create mono-, di-, and triglycerides. In the last step, glycerol and fatty acid esters are created when the rest of the triglycerides and glycerides react with alcohol with the help of a basic activator (Leung et al., 2010a).

The process of transesterification can be carried out in both batch & continuous operations. In a batch process, the catalyst and reacting substances are combined in a reactor and given time to react. When the desired level of conversion is reached, the reaction is stopped, and the resulting mixture is then split into biodiesel and glycerol phases. In a continuous process, reactants and catalyst are continually introduced to a reactor, and the resulting mixture is continuously withdrawn and segregated into glycerol and biofuel.

Transesterification requires the use of catalysts to achieve high yields and shorten reaction times. The catalysts serve as a mediator, supplying a different reaction pathway with lower activation energy and speeding up the reaction rate. Sodium hydroxide and potassium hydroxide are two common alkali metal hydroxides that are used as fundamental catalysts in transesterification. These catalysts are very reactive and have been demonstrated to yield large amounts of biodiesel quickly. When the desired level of conversion is reached, the reaction is stopped, and the resulting mixture is then split into biodiesel and glycerol phases. The reactants and catalyst are continuously added to a reactor in a continuous process, and the reaction mixture is continuously removed and separated into glycerol and biodiesel (Leung et al., 2010b; Veljković et al., 2015).

To attain significant yields and low reaction times, this technique involves the addition of catalysts. The catalysts serve as mediators, supplying a different reaction pathway with lower activation energy and speeding up the reaction rate. Two popular alkaline metallic hydroxides utilised as fundamental catalysts in transesterification are sodium hydroxide and potassium hydroxide, respectively. These catalysts are very reactive and have been demonstrated to yield large amounts of biodiesel quickly (Veljković et al., 2014).

1.6.1 Steps involved in transesterification

In this procedure, an alcohol, usually methanol or ethanol, is reacted with the feedstock with the help of a catalyst, like sodium hydroxide or potassium hydroxide.

There are several phases to the transesterification process, such as:

1. Feedstock preparation: The preparation of the feedstock is the first step in the transesterification process. To do this, the raw material is typically filtered to get rid of any impurities like water and solid particles. It might be necessary to melt the feedstock if it is a solid, such as animal fat, in order to use it.
2. Alcohol preparation: The making of the alcohol is the next action. The alcohol, which is typically methanol or ethanol, must be anhydrous in nature (i.e., devoid of water) in order to avoid unintended reactions. Although alcohol is typically bought commercially, it can also be made using biomass and other sustainable resources (Suehara et al., 2005).
3. Preparation of the catalyst: The catalyst, which is typically either potassium or sodium hydroxide, is generally made by disintegrating it in methanol or ethanol. The catalyst plays a important part in this process because it speeds up the production of biodiesel and aids in the disintegration of the feedstock.
4. Mixing: After being ready, the feedstock, alcohol, and catalyst are combined in a reaction vessel. To facilitate the chemical process, the mixture of materials is exposed to a specified temperature, generally around 50°C and 70°C.
5. Reaction: After combining the feedstock, alcohol, and catalyst, the transesterification reaction starts. Alcohol breaks down the ester molecules in the feedstock during the reaction, creating biodiesel and molecules of glycerol in the process. Usually, it takes a long time for the reaction to finish (Leung et al., 2010a; Najafpour et al., 2008).
6. Separation : After the process is finished, the resultant solution is allowed to settle, at which point the layers of glycerol and biodiesel are separated. The biodiesel layer is typically lighter and floats on top, while the glycerol layer is typically heavier and sinks to the bottom.
7. Washing: To remove any remaining catalyst and impurities, the biodiesel is then washed with water. This is typically accomplished by combining

biodiesel and water, letting the mixture settle, and then separating the layers of water and biodiesel.

8. Drying: The biodiesel must be dried as the last step. Usually, this is accomplished by heating biodiesel in order to evaporate any lingering water.

In summary, this process is a crucial step in the preparation of biodiesel, and the use of catalysts is required to produce high yields and shorten reaction times. Due to their high reactivity and low cost, basic catalysts like NaOH or KOH are frequently used in chemical reactions.

1.7 HETEROGENOUS CATALYST FOR BIODIESEL SYNTHESIS

Heterogeneous catalysts, a kind of catalyst used in the manufacturing of biodiesel, exist as a solid form rather than being dispersed in a reaction mixture. They are frequently employed in the transesterification reaction, where they aid in the conversion of triglycerides into the main ingredients of biodiesel, fatty acid methyl esters. In comparison to homogeneous catalysts, heterogeneous catalysts have several advantages, including ease of segregation, reusability, and low waste creation.

Solid acid catalysts are one of the most prevalent kinds of heterogeneous catalysts employed in biodiesel manufacturing.. The most common foundations for these catalysts are zeolites, clays, or sulfonated resins. In comparison to homogeneous acid catalysts, they are more stable, less prone to corrosion, and capable of operating at lower temperatures and pressures. The need for longer reaction times and lower conversion rates when compared to homogeneous catalysts are some of their drawbacks (Veljković et al., 2015).

Solid base catalysts are another kind of heterogeneous catalyst utilised in the production of biodiesel. Metal oxides, such as calcium oxide or magnesium oxide, are typically the foundation of these catalysts. In comparison to homogeneous base catalysts, they are more stable, have faster reaction rates, and can operate at lower temperatures and pressures. They do, however, have some drawbacks, such as the potential for carbon dioxide and water to deactivate the catalyst.

To increase the effectiveness of the biodiesel production process, heterogeneous catalysts can also be used in conjunction with homogeneous catalysts. For instance,

103 to increase the rate and yield of the transesterification reaction, a homogeneous acidic catalyst like sulfuric acid can be utilized in combination with a solid acid catalyst like sulfonated polystyrene. By using less homogeneous catalyst, this strategy can also produce less waste and be more economically advantageous (Noshadi et al., 2012).

A variety of factors affect the efficacy of heterogeneous activator in the generation of biofuel. These elements include the kind of catalyst used, its surface area and size of particles, the pressure and temperature conditions of the reaction, and the presence of impurities in the feedstock. The production of biodiesel raises the issue of catalyst deactivation because impurities in the feedstock can bind to the catalyst and gradually decrease its activity.

123 Despite these difficulties, the utilization of heterogeneous catalysts in the production of biodiesel is growing in popularity because of their simplicity in separation, capacity for reuse, and low waste generation. They offer the potential to lessen the environmental impact of the biodiesel production process while also increasing its efficiency and cost-effectiveness. The goal of ongoing study is to create new and improved heterogeneous catalysts that will improve the efficiency of biodiesel production procedures (Galadima & Muraza, 2014).

1.7.1 Carbon nano tubes

The type of nanotube known as carbon nanotubes (CNTs) has special properties that could make them helpful as catalysts in a variety of chemical reactions. Researchers have looked into using CNTs as for producing biodiesel catalysts in recent years. This method has many benefits, such as increased reaction rates, high yields, and minimal environmental impact.

30 CNTs can be utilised as a solid catalyst in the process of transesterification to make biodiesel. Transesterification involves reacting triglycerides with alcohol in with the help of an accelerator to produce fatty acid esters and glycerin, because CNTs are in a different phase from the reactants and products, they have the potential to function as heterogeneous catalysts. This makes it simple to separate and recycle the catalyst (Guan et al., 2017).

1.7.2 Titanium dioxide

A promising heterogeneous accelerator employed in the production of biodiesel is titanium dioxide (TiO_2). It is an appealing substitute for other catalysts that might be less sustainable or more expensive because it is a plentiful, affordable, and eco-friendly material. TiO_2 is suitable for use in challenging reaction conditions because it is also extremely stable and corrosion-resistant.

Anatase and rutile are the two main types of TiO_2 that can be utilised as catalysts in the production of biodiesel. Anatase is the more active of the two forms, and because it has a larger surface area, it can interact with the reactants more effectively. It may, however, degrade more quickly over time because it is less stable than rutile (Carlucci et al., 2019).

1.7.3 Titanium oxide combined with carbon nanotube in the production of biodiesel

Carbon nanotubes (CNTs) have demonstrated great promise as catalyst supports in a variety of chemical processes, including the production of biodiesel. CNTs can produce a highly effective and reliable catalyst for transesterification reactions when combined with titanium dioxide (TiO_2). TiO_2/CNTs is the name of the catalyst blend that is used to produce biodiesel, and it has a number of advantages over other catalysts.

The large surface area of TiO_2/CNTs , which enables greater exposure to reactants and more effective catalytic reactions, is one of their main advantages. Because of their superior mechanical and thermal stability, CNTs can be used in abrasive reaction environments. The reusability of TiO_2/CNTs is also very good, with little activity loss after several cycles of use.

Numerous studies have been done to look into the use of TiO_2/CNTs as activator in the generation of biodiesel. According to one study, TiO_2/CNTs had superior catalytic activity when compared to other catalysts, with a transesterification rate of used cooking oil that was over 90%. Another study found that the free fatty esters used in cooking oil, which can interfere with other catalysts, could be efficiently catalysed by TiO_2/CNTs (Sharul et al., 2021).

Due to their complementary properties, TiO_2 and CNTs work together synergistically in TiO_2/CNT catalysts. TiO_2 is capable of photocatalysis, which enables it to be irradiated with light and generate reactive oxygen species capable of oxidising organic compounds. Despite the positive outcomes of TiO_2/CNTs as biodiesel production catalysts, there are some difficulties with their application. To ensure the best catalytic performance, it is necessary to carefully control the ratio of TiO_2 to CNTs. The potential toxicity of CNTs, which raises questions about the safety of the environment and human health, is another difficulty.

As a result of its high efficiency, stability, and reusability, the combination of carbon nanotubes and titanium dioxide (TiO_2/CNTs) is a promising catalyst for the generation of biodiesel. The development of new and improved TiO_2/CNTs catalysts that can further improve the efficiency of biodiesel manufacturing procedures while solving potential health and safety issues is the main focus of ongoing research (Carlucci et al., 2019).

1.8 BIODIESEL FROM EDIBLE OILS

Triglycerides are frequently transesterified to produce biodiesel from a range of sources that involve edible oils, fats, and even cooking oils. However, there are concerns about the impact on food costs and potential rivalry with food if these oils are utilized to produce biodiesel (Balat, 2011).

Edible oils such as soy oil, palm oil, rapeseed oil, and sunflower seed oil are commonly utilised in the production of biodiesel. Due to their wide availability and high oil content, these oils are favoured. However, there are a number of arguments against using edible oils to make biodiesel. First off, the manufacturing of biodiesel using edible oils may result in higher food costs, particularly in underdeveloped nations where food security is a key problem. Second, the manufacturing of biodiesel from edible oils may cause agricultural land to shift from producing food to producing energy, which could exacerbate the problem of food security. Last but not least, the production of edible oils consumes a significant amount of energy as well as water, that may have a severe impact on the natural world.

Utilising waste oils and fats to make biodiesel is another strategy to enhance sustainable biodiesel production. Waste oils and fats that can be utilised to make biodiesel include leftover cooking oil, animal fats, and grease trap waste. Waste oils

and fats can be used to produce biodiesel in a way that does not compete with food production while simultaneously reducing the environmental impact of their disposal (Degfie et al., 2019).

1.9 BIODIESEL FROM NON-EDIBLE OIL

The utilization of non-edible oils for the generation of biodiesel is gaining popularity since it eliminates conflict between food and energy production and can give further advantages such as carbon storage and the preservation of biodiversity.

Jatropha, pongamia, mahua, castor, and karanja, rubber seed oil are non-edible oils that are frequently used to make biodiesel. Due to their high oil content, accessibility, and appropriateness for growing on marginal soils unsuitable for food production, these oils are chosen. Non-edible oil crops can also offer further advantages like soil protection, biodiversity preservation, and carbon sequestration (Demirbas et al., 2016).

The process of turning non-edible oils into biodiesel includes the extraction of the oil, pre-treatment, transesterification, and purification. Depending on the type of oilseed and its availability, the oil extraction process can be carried out via mechanical or chemical methods. Pre-treatment entails cleaning the oil of pollutants such moisture, free fatty acids, and other impurities. During the transesterification process, the oil undergoes reaction with an alcohol using a catalyst such as sodium hydroxide or potassium hydroxide. Glycerol is produced as a by-product as well as biodiesel as a result. Any remaining impurities in the biodiesel are eliminated during the purification process.

In comparison to edible oils, the use of non-edible oils in the production of biodiesel provides a number of benefits. First off, non-edible oils don't compete with food production, preventing a possible rise in food prices and worries about food security. Second, to prevent competition for land use, non-edible oils can be grown on marginal sites that are unsuitable for growing food. Thirdly, further advantages like carbon sequestration and biodiversity preservation may come from non-edible oil crops. Fourthly, it is possible to manufacture non-edible oils locally, minimising reliance on imported oil and fostering energy security. Non-edible oil crops, which do not compete with the production of food, might offer extra advantages including carbon sequestration and biodiversity preservation (Banković-Ilić et al., 2012).

The use of non-edible oils in the production of biodiesel has a possibility to, among other things, decrease the release of greenhouse gases, enhance environmental friendly growth, and reduce reliance on imported oil. Additionally, the utilisation of non-edible oil crops for the production of biodiesel can have positive effects on biodiversity and carbon sequestration. Therefore, the development of environmentally friendly methods for the production and use of non-edible oils in the manufacturing of biodiesel can aid in the shift to a more renewable and sustainable energy system (Abdul Hakim Shaah et al., 2021).

1.9.1 Rubber seed oil in biodiesel production

The high oil content and accessibility of rubber seed oil in places like Southeast Asia, South America, and Africa have made it a suitable feedstock for biodiesel manufacturing. Comparing rubber seed oil to conventional feedstocks like soybean, rapeseed, and palm oil, there are a number of benefits (Ulfah et al., 2018).

The fatty acid ester percentage of RBO normally consists of 20-25% oleic acid, 40-45% linoleic acid, and 7-10% linolenic acid. The oil concentration in rubber kernels varies between 35 and 45%. due to its high concentration of unsaturated fatty acids, rubber seed oil is prone to rapid deterioration; however, this can be decreased by mixing it with other feedstocks or adding antioxidants.

The process of turning rubber seed oil into biodiesel includes the extraction of the oil, as well as pre-treatment, transesterification, and purification. Depending on the oilseed type and availability, the oil extraction process can be carried out using mechanical or solvent methods. Pre-treatment entails cleaning the oil of pollutants such moisture, free fatty acids, and other impurities. Using a catalyst like sodium hydroxide or potassium hydroxide, the oil is reacted with an alcohol during the transesterification process. Glycerol is produced as a by-product as well as biodiesel as a result. Any remaining impurities in the biodiesel are eliminated during the purification process (Ramadhas et al., 2005).

Compared to conventional biodiesel feedstocks like soybean, rapeseed, and palm oil, rubber seed oil provides a number of advantages. First off, growing rubber trees does not compete with food production, potentially reducing increases in food costs and worries about food security. In addition, rubber trees can be planted on unsuitable marginal soils, preventing competition for land usage. Thirdly, rubber

trees can also protect biodiversity and sequester carbon, among other advantages. Fourth, the ability to manufacture rubber seed oil domestically reduces reliance on imports of foreign oil and fosters energy security.

In conclusion, because of its high oil content, availability, and suitability for growth on marginal areas, rubber seed oil has the possibility to become a viable raw material for the generation of biodiesel. Compared to conventional feedstocks like soybean, rapeseed, and palm oil, the manufacturing of biodiesel from the RBO can have a number of benefits (Arachchige et al., 2008).

1.10 FUTURE OPPORTUNITIES OF BIODIESEL

Given the rising demand for energy from environmentally friendly and sustainable sources, biodiesel has bright future prospects. The following are some of the major areas for biodiesel growth and development:

1. Feedstock diversification: Diversifying the feedstocks is one of the main opportunities for growth in the biodiesel industry. Although there is still a lot of interest in using alternative feedstocks like algae, waste oils, and agricultural products, traditional feedstocks like soybean oil and rapeseed oil are still widely used. This can assist in addressing some of the difficulties related to the cost and availability of feedstock.
2. Technology: The production of biodiesel offers significant potential for technological advancement. The efficiency and sustainability of biodiesel production can be enhanced by advancements in production techniques, catalysts, and enzymes.
3. Market expansion: The biodiesel sector has a sizable amount of room for expansion. As the need for renewable energy sources that are environmentally friendly rises, biodiesel as a substitute for diesel made from petroleum is gaining popularity. The use of biodiesel in other industries, such as heating oil, aviation fuel, and marine fuel, is also gaining popularity.
4. Circular economy and sustainability: The generation of biodiesel is becoming more and more interested in the circular economy and ecological sustainability. In addition to lowering waste and emission levels of greenhouse gases, using waste oils and other waste streams as feedstocks for the production of biodiesel can yield useful by-products like glycerol.

Furthermore, using biodiesel can aid in reducing reliance on fossil fuels and encourage a more sustainable lifestyle.

Overall, biodiesel has promising prospects for the future due to the rising demand for environmentally friendly and renewable energy sources. The potential benefits of biodiesel are substantial, but overcoming the obstacles to its production and use will require a combination of technological advancement, policy support, and market development (Abdul Hakim Shaah et al., 2021).

CHAPTER-2

LITERATURE SURVEY

- **Chen et al. 2019** In this study, the transesterification of rubber seed oil with methanol was explored using a magnetised carbon nanotube (MCNT) catalyst. In comparison with traditional catalysts, the study indicated that using the MCNT catalyst enhanced conversion efficiency while lowering catalyst usage. A further finding of the study was that the MCNT catalyst may be readily magnetically removed from the reaction mixture and recycled up to 5 times without suffering a substantial decrease in catalytic activity.
- **Zaoh et al. 2020** CNTs-Fe₃SO₄ nanocomposites were explored as a catalyst for biodiesel generation from waste cooking oil. The best reaction parameters were discovered to be a molar ratio of methanol to oil of 12:1, a temperature at which the reaction occurs of 60°C, and a reaction period of 4 hours. The researchers also discovered that using CNTs-Fe₃O₄ composites as a catalyst gave rise to high conversion efficiency and simple catalyst separation. The distinctive qualities of CNTs-Fe₃O₄ tiny composites, which include their high surface area, strong reaction, and magnetic characteristics that allow them to be easily separated from the reaction mixture, were attributed to these findings.
- **Demirbas et al. 2014** research was done on the utilisation of sodium hydroxide as a catalyst for the generation of biodiesel from used cooking oil. The research discovered that a 6:1 molar ratio of methanol to oil, a temperature of reaction of 60°C, and a duration of reaction of 90 minutes produced the best results. According to the study, using a homogeneous catalyst not only led to higher conversion rates but also high catalyst utilisation and challenging catalyst separation.
- **Chavan et al. 2017** research was done on using H-mordenite as a catalyst to create biodiesel from used cooking oil. The research discovered that a 9:1 molar ratio of methanol to oil, a reaction temperature of 50°C, and a reaction period of 6 hours produced the best results. The study also discovered that using H-mordenite as a catalyst produced little waste, an easy separation of the catalyst, and excellent conversion efficiency.

- **Lam et al. 2014** who employed methanol and titanium dioxide nanomaterials (TiO_2) as a catalyst to transesterify rubber seed oil. Comparing TiO_2 to more traditional catalysts like sodium hydroxide and potassium hydroxide, the study discovered that TiO_2 had a greater conversion efficiency and consumed less catalyst. Additionally, it was discovered in the study that TiO_2 may be effortlessly removed from the reaction mixture by centrifugation and recycled up to five times without suffering a substantial reduction in catalytic activity.
- **Soares et al. 2018** In this study, the transesterification of the oil from rubber seeds with methanol was examined using a hybrid catalyst system composed of TiO_2 and MWCNTs. According to the research, using a hybrid catalyst system led to improved conversion efficiency and less catalyst use than using regular catalysts. The study also discovered that the hybrid catalyst system could be reused up to 3 times without suffering a substantial loss in catalytic activity by simply centrifuging it away from the reaction mixture.
- **Gogoi et al. 2014** Mahua oil's potential use as a feedstock for biodiesel production was examined. In comparison to homogeneous catalysts like sodium hydroxide and potassium hydroxide, the study discovered that using heterogeneous catalysts like calcium oxide and magnesium oxide led to higher conversion efficiency and less catalyst use. The study also discovered that contaminants like moisture and unsaponifiable materials can have a negative impact on the catalyst usage and conversion efficiency.
- **Ghanbari et al. 2021** it was looked into whether carbon nanotubes might be used as a catalyst to produce biodiesel from camelina oil. The research discovered that a 12:1 molar ratio of methanol to oil, a reaction temperature of 65°C , and a reaction period of 3 hours produced the best results. Additionally, the study discovered that using carbon nanotubes as a catalyst produced excellent conversion efficiency and simple catalyst separation.
- **Vijayaragavan et al. 2015** who looked into using sulfuric acid-modified titanium dioxide as a catalyst in the rubber seed oil and methanol transesterification process. In comparison to traditional catalysts, $\text{SO}_4\text{-TiO}_2$ was shown to have a greater conversion efficiency and a lower catalytic consumption. Additionally, the research revealed that $\text{SO}_4\text{-TiO}_2$ may be

effortlessly removed from the product of the reaction by centrifugation and recycled up to 4 times without suffering a substantial reduction in catalytic activity

- **Dong et al. 2013** examined the use of lipase as a catalyst in the synthesis of biodiesel from soybean oil. The research discovered that a 4:1 molar ratio of methanol to oil, 35°C for the reaction temperature, and 24 hours for the reaction were the ideal parameters. Additionally, the study discovered that using an enzymatic catalyst led to high conversion efficiency and simple catalyst separation, but also high cost and sensitivity to reaction conditions..
- **Karami et al. 2017** who conducted research on the transesterification of rubber seed oil with methanol using multi-walled nanotubes made of carbon as a form of catalyst. In comparison to more traditional catalysts like sodium hydroxide and potassium hydroxide, the study discovered that using MWCNTs as a catalyst led to improved conversion efficiency and less catalyst use. MWCNTs can be recycled up to 4 times without significantly losing their catalytic activity, according to the study.
- **Luque et al. 2010** investigation into using jatropha oil as a feedstock for making biodiesel. The study discovered that while homogeneous catalysts like sodium hydroxide and potassium hydroxide produced high conversion efficiencies, they also consumed a lot of catalyst and were challenging to separate. According to the study, using heterogeneous catalysts such metal oxides and zeolites led to poorer conversion efficiencies, but also to lower catalyst use and simpler catalyst separation.
- **Sue et al. 2017** they employed methanol and titanium dioxide nanocrystals (TiO₂) as a catalyst to transesterify rubber seed oil. In comparison to conventional catalysts, the study indicated that using TiO₂ as a catalyst increased conversion efficiency while lowering catalyst usage. Additionally, it was discovered in the study that TiO₂ may be effortlessly removed from the reaction mix by centrifugation and recycled up to five times without suffering a substantial reduction in catalytic activity.
- **Jalalabadi et al. 2017** the utilisation of titanium dioxide modified with iron oxide (Fe-TiO₂) as a catalyst for the transesterification of rubber seed oil with methanol was researched. In comparison to traditional catalysts, Fe-TiO₂

was shown to have a greater conversion efficiency and a lower catalyst consumption. Additionally, the research revealed that Fe-TiO₂ may be effortlessly removed from the reaction mixture by centrifugation and recycled up to five times without suffering a substantial reduction in catalytic activity.

- **Karmee and chadha et al. 2015** examined the use of castor oil as a feedstock for the manufacture of biodiesel. In comparison to homogeneous catalysts like sodium hydroxide and potassium hydroxide, the study discovered that using heterogeneous catalysts like calcium oxide and magnesium oxide led to higher conversion efficiency and less catalyst use. The study also discovered that the consumption of the catalyst and conversion efficiency can both be adversely impacted by the presence of free fatty acids in the feedstock
- **Ibrahim et al. 2019** TiO₂ nanotubes were explored as a catalyst for biodiesel generation from waste cooking oil. The best reaction conditions, according to the study, were a molar ratio of methanol to oil of 6:1, a reaction temperature of 60°C, and a reaction period of 2 hours. The researchers also discovered that using TiO₂ nanotubes as a catalyst resulted in high conversion efficiency and simple catalyst separation. The authors attributed these findings to TiO₂ nanotubes' distinctive features, such as their large surface area and shape.
- **Wang et al. 2018** the use of sodium hydroxide as a catalyst for biodiesel generation from rapeseed oil was examined. The best reaction conditions, according to the study, were a molar concentration of methanol to oil of 6:1, a reaction temperature of 60°C, and a reaction period of 1 hour. The use of sodium hydroxide as a catalyst resulted in a higher rate of conversion and little waste generation, according to the study.
- **Hanifa et al. 2016** research was done on using calcium oxide as a catalyst to produce biodiesel from used cooking oil. The research discovered that a 6:1 molar ratio of methanol to oil, a reaction temperature of 60°C, and a reaction time of 90 minutes produced the best results. The study also discovered that using a heterogeneous catalyst led to a decrease in conversion efficiency, but also to a reduction in catalyst consumption and an ease in catalyst separation..

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Varma et al. 2018 it was looked into whether the transesterification of rubber seed oil with methanol might be accelerated by using titanium dioxide modified with zirconium oxide. In comparison to traditional catalysts, Zr-TiO₂ was shown to have a greater conversion efficiency and a lower catalyst usage. Additionally, the study discovered that Zr-TiO₂ may be effortlessly removed from the reaction mixture by centrifugation and recycled up to four times without suffering a substantial reduction in catalytic activity.
- Wie et al. 2013** research was done on the utilisation of magnesium oxide as a catalyst for the generation of biodiesel from used cooking oil. The research discovered that a 12:1 molar ratio of methanol to oil, a reaction temperature of 60°C, and a reaction period of 4 hours produced the best results. The study also discovered that using magnesium oxide as a catalyst produced little waste, an easy separation of the catalyst, and excellent conversion efficiency.
- Oliveira et al. 2018** the study looked into the usage of sulfuric acid as a catalyst in the synthesis of biodiesel from used cooking oil. The research discovered that a 12:1 molar ratio of methanol to oil, a reaction temperature of 60°C, and a reaction period of 4 hours produced the best results. The study also discovered that using sulfuric acid as a catalyst produced little waste and had a high conversion efficiency.
- Zhang et al. 2019** TiO₂ was explored as a catalyst for biodiesel generation from soybean oil. The best reaction parameters were discovered to be a molar concentration of methanol to oil of 12:1, a reaction temperature of 60°C, and a reaction period of 4 hours. The researchers discovered that using TiO₂ as a catalyst resulted in a high efficiency of conversion, simple catalyst separation, and little waste generation. These findings were linked to TiO₂'s unique physical and chemical features, such as its high surface area, strong reactivity, and low toxicity.
- Mishra et al. 2019** CNTs were explored as a catalyst for biodiesel generation from jatropha oil. The best reaction conditions, according to the study, were a molar ratio of methanol to oil of 6:1, a reaction temperature of 60°C, and a reaction period of 2 hours. The study also discovered that using carbon nanotubes as a catalyst resulted in excellent conversion efficiency and simple

catalyst separation. The study attributed these findings to CNTs' distinctive features, such as their large surface area, high reactivity, and distinct shape.

- **Liew et al. 2014** Carbon nanotubes (CNTs) were explored as a catalyst for palm oil biodiesel generation. The best reaction parameters were discovered to be a molar ratio of methanol to oil of 12:1, a temperature for the reaction is 60°C, and a reaction period of 4 hours. The study also discovered that using carbon nanotubes as a catalyst resulted in a high rate of conversion and lower waste creation. The study explained these findings to CNTs' distinctive features, such as their huge surface area, high degree of reactivity, and distinct shape.

CHAPTER-3

AIM AND SCOPE

3.1 AIM

The main goal of our project is to produce biodiesel from Rubber seed oil using Carbon nanotube Titanium Dioxide composite (CNT-TiO₂).

- Synthesis of CNT-TiO₂ catalyst.
- Characterization of CNT-TiO₂ catalyst to find its properties.
- To find properties of the biodiesel like Viscosity, Density, calorific value, Cloud point, FFA, etc., and also find the composition using GC.

3.2 SCOPE

Biodiesel acts like petroleum diesel, but produces less air pollution and it comes under renewable sources, is biodegradable and is safer for the environment. Producing biodiesel fuels can help create local environmental benefits. Replacing fossil fuels with biofuels has the potential to reduce some undesirable aspects of fossil fuels.

- Green-house gas(GHG) pollutant emissions.
- Exhaustible resources depletion.
- Dependence on unstable foreign suppliers.
- Increase in fossil fuel prices.
- Global warming and climatic change.

CHAPTER-4

MATERIALS AND METHODS

4.1 MATERIALS

Rubber seeds were purchased from farm near Marthandam, Kanyakumari, [8.3022 °N, 77.2231 °E]. Carbon-nanotubes, TiO₂, Sulfuric acid, Methanol, ethanol, Iso-Propyl alcohol, NaOH pellets, n-hexane, Phenolphthalein, Distilled Water, was used from laboratory in Centre for waste management, Sathyabama university, science and technology, Chennai-600119.

4.2 OIL EXTRACTION

Three methods of extraction were employed to extract oil from rubber seeds. The rubber seeds utilized for oil extraction weighed a total of 50 kg. The rubber seeds were cleaned to remove any dirt or other unwanted particles. The seeds were then dried to reduce their moisture content. In hydraulic pressing method, the seeds were placed in the hydraulic press machine and applied a pressure of 650-750 bar gradually to achieve high oil yield. The temperature during the extraction was kept around 65°C to avoid degradation of the oil. The oil extracted was collected in a container. Solid impurities were removed using a filter bag having a pore size of 5μ. In solvent extraction, 5 L of n-hexane was combined with 1 kg of powdered rubber seeds and stirred for 3 h. The solvent-oil mixture from solid residues was separated using a filter paper. The solvent-oil mixture was then separated by heating the mixture at 60°C for 30 min to evaporate the n-hexane. The remaining oil was weighed and stored in a container. In mechanical extraction, the seeds were heated to a temperature of 75°C before they were fed into the press. A pressure of 150 bar was applied to extract the oil. To separate the seed cake and oil mixture, centrifuge was used. The oil was then filtered at a temperature of about 80°C to prevent oil from becoming too viscous.

4.3 RUBBER SEED OIL ANALYSIS

The properties such as acid value, density, kinematic viscosity, acid value and FFA% were determined before utilizing this oil for the transesterification reaction. If the values of the mentioned properties were not within the standards, then the oil

was planned to be pre-treated in order to ensure effective synthesis of oil to quality biodiesel product.

4.4 PREPARATION OF CATALYST

0.47g of Carbon nanotubes and 50 mg of Titanium dioxide was taken. Titanium dioxide was mixed with distilled water in one beaker and Carbon nanotubes was mixed with ethanol in another beaker. Titanium dioxide was dissolved whereas Carbon nanotubes was still insoluble. Now both the solutions were combined in the single beaker. The beaker containing the solution was put in the sonification reactor for 30 min, after this process the solution became slightly soluble. The solution was kept on a magnetic stirrer at 500 rpm for 30 min, the solution became even more soluble than the last time. The solution was poured into a mini HTL which is a closed container tightened with bolts and nuts. The mini HTL was kept in an oven reactor at 130°C for 8 h. After 8 h, the solution was then poured from the mini-HTL into a 200 mL beaker. The solution was taken and it was water washed using ethanol for three times. The solution was sent to the centrifugation reactor operating at 25°C and 500 rpm for 50 min. Once the centrifugation reaction was complete the solution was then poured into a dry dish and after the drying process, catalyst was obtained in the form of minute powder.



Fig.4.1: a) Centrifugation of catalyst



b) Drying the catalyst

4.5 CHARACTERIZATION OF CATALYST

The catalyst was characterized by means of XRD (ARL EQUINOX 3000) and FT-IR (Shimadzu IRAffinity-1S). X-Ray Diffraction is a technique used to analyze the crystal structure of a material. By measuring the lattice parameters, crystal symmetry, and Miller indices of the diffraction peaks, XRD can provide details about a substance's crystal structure. The interplanar spacing (d) of the crystal lattice planes that diffract X-rays at this angle may be calculated using Bragg's Law ($n = 2d \sin \theta$), the sampling is scanned using 2θ , and the range is from 10° to 60° . The wavelength of Cu radiation is 1.54 Å.

FT-IR (Fourier Transform Infrared Spectroscopy) is a powerful analytical technique used to identify and quantify chemical compounds based on their vibrational modes. It works by measuring the absorption or transmission of infrared radiation through a sample, which is then used to determine the presence and concentration of specific functional groups in the sample.

4.6 ACID ESTERIFICATION

Due to the presence of high Fatty acid percentage in rubber seed oil esterification must be performed as a pre-treatment step to reduce the FFA in oil below 2% for effective conversion of oil to biodiesel, acid esterification is a chemical reaction in which triglycerides react with lower alcohol such as methanol in the presence of an acid catalyst and reduced the high amount of free fatty acids. In this work sulfuric acid (H_2SO_4) was used as the acid catalyst to reduce the FFA content. Before starting with the esterification process, moisture content in the oil was reduced by heating the oil to $110^\circ C$. 1 L of Crude rubber seed oil was taken into a 2000 mL three necked round bottom flask which was coupled with a condenser to recondense the evaporated methanol. The flask containing the oil was kept on a magnetic stirrer at 500 rpm and was preheated till $60^\circ C$. Methanol was taken in the ratio of 1:3 of oil to methanol, so for 1 L of oil 182 mL of methanol was taken, for every 1% of FFA 0.25 mL of concentrated sulfuric acid (H_2SO_4) was taken as an acid catalyst, so according to FFA present in the oil a total of 9.5 mL of concentrated sulfuric acid was taken and mixed with methanol in a conical flask. Once the oil reached the desired temperature, mixture of methanol and sulfuric acid was added to the oil in the round bottom flask, the mixture was maintained at $60^\circ C$ at a stirring rate of 500 rpm for 2 h. Every 15 min a mercury thermometer was used to check the

temperature. After 2 h the reaction mixture was allowed to cool down till room temperature and then poured into a separating funnel. The reaction mixture formed two separate layers, the upper layer constituted of the esterified oil, whereas the bottom layer constituted of methanol, sulfuric acid and other impurities. The bottom layer was separated and the esterified oil was transferred to a beaker and heated till 110°C to in a mantle to vaporize methanol and to remove any moisture content present in the oil. The FFA for treated oil was checked and later collected for further transesterification process to convert the oil to biodiesel.



Fig. 4.2: a) Esterification of oil b) Separation of oil.

4.7 TRANSESTERIFICATION

After the FFA% in the oil was reduced below 2%, the oil was now ready to be sent for transesterification reaction. 3 samples of 100 mL oil was taken to produce 3 sample of biodiesel, where one sample of oil was taken and converted to biodiesel using NaOH which is conventional homogeneous catalyst, second sample of oil was converted to biodiesel using NaOH as well as CNT-TiO₂ which is a combination of both homogeneous and heterogeneous catalyst, and third sample was converted to biodiesel using CNT-TiO₂ alone which is a heterogeneous catalyst, these different combinations of catalyst were taken in order to compare the yield and biodiesel properties between the 3 samples and to understand the effect of heterogenous catalyst. The oil was poured into a 250 mL three necked round bottom flask and preheated till 60°C stirred at 500 rpm on a magnetic stirrer. Methanol was taken keeping oil to methanol ratio of 1:6, so for 100 mL of oil 18.2 mL of methanol was taken and mixed with 0.5g of the NaOH pellets in a conical flask. Once the oil reached the desired temperature, the alcohol and catalyst mixture were poured into

the flask containing the oil, the reaction was allowed to happen for 2 h maintaining the temperature at 60°C and at a stirring rate of 500 rpm, the temperature was checked every 15 min using mercury thermometer. Once the reaction was complete, the mixture was allowed to cool till room temperature and then sent to a separating funnel for separation process, there were two layers formed where the upper layer was biodiesel and the lower layer was glycerol, catalyst and other impurities which was removed.

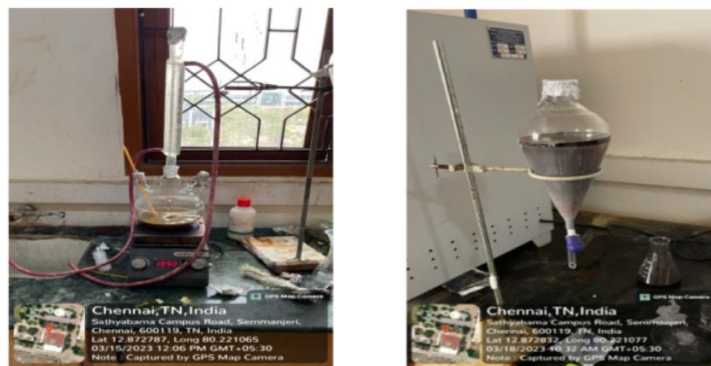


Fig. 4.3: a) Transesterification. b) Biodiesel separation.

4.8 WASHING TECHNIQUE

4.8.1 Wet Washing Technique

The biodiesel produced using the presence of homogenous catalyst was now sent for wet washing treatment to remove impurities such as methanol, catalyst, unreacted oil, etc. where the water was heated between 60°C and 79°C and then poured into a separator along with the biodiesel, and let it remain undisturbed for 15 min, the water collected at the bottom was removed and freshly heated water was again mixed with biodiesel in the separator. This process was repeated till the water was neutralized which was checked using litmus paper.

4.8.2 Membrane Washing technique

Biodiesel produced using heterogeneous catalyst was purified using membrane technique where a filter bag having a pore size of 5 μ was used to remove the catalyst and any other impurities present in the biodiesel.



Fig. 4.4: a) Membrane washing.

b) Wet washing.

4.9 BIODIESEL CHARACTERIZATION

The biodiesel was characterized by the means of GC-FID (Young In Chromass, YL6500GC), which was utilized to analyze the fatty acid methyl esters (FAMES) in our biodiesel samples in this research. Individual FAMES are separated and detected by the GC-FID based on their physical and chemical characteristics, such as boiling point and affinity for the stationary phase in the column. The FID detects fatty acid methyl esters (FAMES) by measuring their capacity to ionize in the flame and producing a current proportionate to the quantity of FAME in the sample.

4.10 DETERMINING THE PHYSIO-CHEMICAL PROPERTIES OF BIODIESEL

4.10.1 Moisture content (ASTM D2709)

The amount of moisture in biodiesel is measured using a heating process. A crucible is a substance that has been used in this procedure. 5 g of biodiesel sample was taken and then the total weight of the crucible with biodiesel sample was measured and recorded. The sample was then placed in a hot air oven set at 110°C for a period of 3 h. Once the sample was cooled. The total weight of the sample with crucible is then measured and recorded. Weight difference before and after heating was used to determine the loss of moisture content.



Fig. 4.5: a) Sample weighing.

b) Moisture removal in oven.

4.10.2 Density (ASTM D1298)

The density of biodiesel was determined using a pycnometer. A pycnometer is a stoppered glass container with a predetermined capacity. The mass of pycnometer and the mass of both pycnometer with the sample biodiesel is recorded. The difference is divided by the volume of the pycnometer and the density is arrived.



Fig. 4.6: Finding density using pycnometer.

4.10.3 Kinematic viscosity (ASTM D445)

It is an important property to determine as it affects the fuel's flow characteristics and lubricity. The fluids tendency to resist flow is called kinematic viscosity, which is measured in centistokes (cSt). According to ASTM standards the fluid viscosity should be within 1.9 centistokes (cSt) and 6.0 centistokes (cSt) at 40°C. The viscosity has been measured under the standard of ASTM D 445. First the U-tube viscometer was ensured whether it was clean and dry. The biodiesel sample was

poured into the U-tube viscometer and then kept in a temperature-controlled bath and was allowed to reach 40°C. Using a stopwatch, the time taken for a fixed volume of biodiesel sample to flow through the viscometer was recorded in seconds. The kinematic viscosity of the biodiesel was found using the following formula: time recorded (in secs) x 0.014 = kinematic viscosity (in centistokes)



Fig. 4.7: Viscosity

4.10.4 Free fatty acid and acid value (ASTM D664)

The FFA and acid value for the biodiesel was evaluated by performing titration procedure, for this process, a NaOH burette solution with a normality of 0.1 was first prepared. 5 g of biodiesel sample was taken in a conical flask. In addition, 25 mL of isopropyl alcohol was added to a conical flask containing the biodiesel sample. The combination was then given a total of four drops of burette solution. The solution was then heated until it reaches a temperature of 60°C. The sample was then allowed to cool to room temperature. Once the solution had reached room temperature, the phenolphthalein indicator was added. After that, the solution was titrated and the results were recorded and the acid value and FFA were calculated from the results using the Eq. (1) & Eq. (2) respectively.

$$\text{FFA (\%)} = \frac{28.2 \times (\text{burette reading}) \times 0.1}{5} \quad (1)$$

$$\text{Acid value} = \text{FFA (\%)} \times 1.99 \quad (2)$$



Fig. 4.8: Titration to find FFA%.

4.10.5 Cloud point (ASTM D2500) and Pour point (ASTM D97)

A sample of the petroleum product was placed in a glass test jar and progressively cooled to determine the cloud point. The cloud point temperature was determined by passing a light beam through the jar at the temperature at which the wax crystals begin to emerge and scatter the light. The pour point temperature was calculated by progressively chilling the sample in a test jar and monitoring the temperature at which the fluid stopped to flow when the jar was tilted. Typically, the pour point was determined by tilting the jar at regular intervals and watching the biodiesel sample until it no longer flowed. The temperatures were recorded for both cloud and pour point.

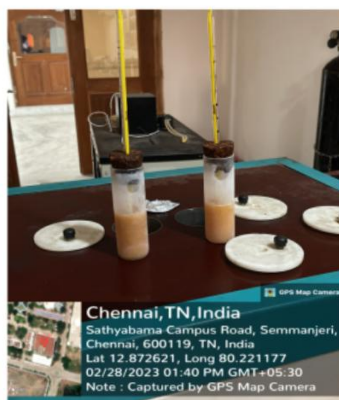


Fig. 4.9: Cloud point and Pour point

4.10.6 Calorific value (ASTM D6751)

The quantity of heat emitted when a unit of fuel is entirely burnt is referred to as its calorific value. It is often measured in J/g or kcal per kg of fuel. An instrument called bomb calorimeter was utilized to determine the calorific value of the biodiesel by determining the heat energy liberated during the process of combustion.

A little metal container called as “bomb” was filled with high-pressure oxygen and weighed. The bomb was put within a bigger container filled with water, which served as a heat sink, absorbing the heat emitted during burning. The metal container was filled with the biodiesel sample. The biodiesel was lit using a slight electrical discharge, which cause it to catch fire. The heat generated upon combustion was transmitted to the water, which heated it considerably. A thermometer was used to measure the increase in the temperature of the water. The biodiesel's calorific value was determined based on the rise in water temperature and the quantity of biodiesel utilized. The calorific value was calculated in terms of cal/g.



Fig. 4.10: a) Bomb calorimeter

b) Thermometer

4.10.7 Flash Point (ASTM D93)

The flash point of a liquid is the lowest point of temperature at which it can produce enough vapors to generate an ignitable combination in air near the liquid's surface. It is the temperature at which a liquid can ignite if it comes into contact with an ignition source, such as a flame or spark.

A laboratory apparatus called Pensky-Martens closed-cup tester was utilized to evaluate the flash point for the biodiesel sample. A small sample of biodiesel was poured in a cup, which was then heated at a regulated pace with a flame source. The flash point was defined as the temperature at which a flare of flame was visible over the outer layer of the liquid biodiesel sample.



Fig. 4.11: Flash Point

4.11 REUSABILITY OF CATALYST

The recovery of catalyst was studied for biodiesel produced using CNT-TiO₂ only. Once the transesterification was complete the catalyst was accumulated at the bottom of the separator which was easily separated and transferred to a separate beaker, the catalyst was mixed with some amount of oil and methanol in it, so the catalyst was then kept in oven at 110° C to evaporate the methanol. To remove the oil, 15 mL of n-hexane was added to the catalyst and oil mixture and it was stirred for few minutes to remove any oil and organic residues that may be present on the surface of the catalyst. The mixture was left to settle for several hours. The top layer of the mixture, which contains the n-hexane and organic residues, was then removed and by filtering the catalyst using filter paper. The washed catalyst was then dried to remove any residual n-hexane. This was done by heating the catalyst in an oven at a temperature of around 120°C-150°C for several hours. Once the catalyst was dried, it was then subjected to calcination, where the catalyst was heated in air or an oxygen-containing atmosphere at 650°C for 3 h. After calcination, the catalyst was cooled and was again used for transesterification.

CHAPTER-5

RESULTS AND DISCUSSION

5.1 OIL EXTRACTION

The extraction efficiency for hydraulic pressing, solvent extraction and mechanical pressing was about 39.47%, 31.65% and 27.78% respectively. The extraction efficiency of hydraulic press, showed the greatest oil yield, which was an appealing outcome. The hydraulic press produces a greater yield of oil with higher quality, has lower running costs, is ecologically friendly, and is adaptable. It is a dependable and efficient oil extraction technology that can be used on a wide range of plant materials, making it an affordable option for small-scale production. Rubber seed oil may be a feasible source for biodiesel or other industrial uses because of its high yield of oil that could be extracted from rubber seeds. In comparison with other sources of edible and non-edible oils, rubber seeds are a cheap supply of feedstock. A high extraction efficiency allows for the extraction of more oil per kilogram of seeds, which lowers the cost of the biodiesel synthesis process.

5.2 PROPERTIES OF RUBBER SEED OIL

Table 5.1 shows the properties of rubber seed oil utilized in this study and its FFA content. The basic oil analysis such as moisture content, density, kinematic viscosity, acid value, FFA% were performed on Crude Rubber seed oil. The properties are important to determine before transesterification reaction is employed ensuring product quality, reaction efficiency and safety. For oils having a high kinematic viscosity and density, may require longer reaction periods as well as higher reaction temperatures to complete the conversion to biodiesel. If Properties such as acid value and FFA% in the oil are high then it might cause undesired reactions and contaminants in the end biodiesel product leading to poor quality of biodiesel. Therefore, in order to reduce the acid value and FFA% of oil, acid esterification process was performed in order to reduce the FFA% of oil below the safer level of 2%. High moisture content in oil can also lead to various safety hazards during the transesterification reaction, as the water content in oil might cause the reaction to splatter, potentially resulting in flames or explosions. Therefore, in order to reduce the moisture content, the oil was subjected to high temperature to evaporate the water molecules present in the oil.

Table 5.1: Properties of the Rubber seed oil.

Properties	Testing procedure	Rubber seed oil
Moisture content%	ASTM D2709	0.414
Density (g/mL)	ASTM D1298	0.904 g/mL
Kinematic Viscosity (cSt)	ASTM D445	34.86 cSt
Acid value	ASTM D664	74.625
FFA%	ASTM D664	37.5

5.3 CHARACTERIZATION OF THE CATALYST

5.3.1 Fourier Transform Infrared (FT-IR)

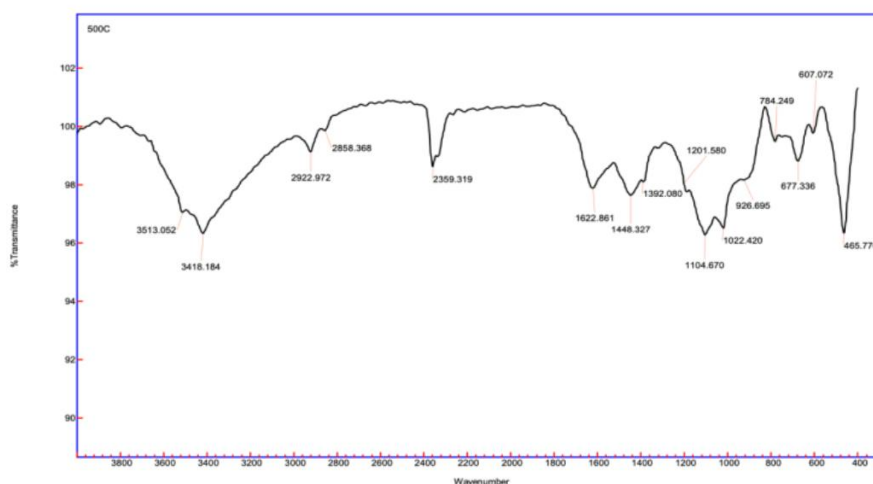


Fig. 5.1: FT-IR analysis image of CNT-TiO₂ catalyst

The FT-IR analysis of the CNT-TiO₂ revealed the presence of C-H stretching at peak values of 2922.972 and 2858.368 cm⁻¹. The presence of O-H stretching was observed at peak values of 3513.052, 3418.184, 1392.080 cm⁻¹. The existence of C=O stretching was suggested at the peak values of 1622.861, 784.249, 677.336 cm⁻¹. The presence of C-O stretching was observed at peak values of 1201.580 and 1104.670 cm⁻¹.

5.3.2 X-Ray Diffraction (XRD)

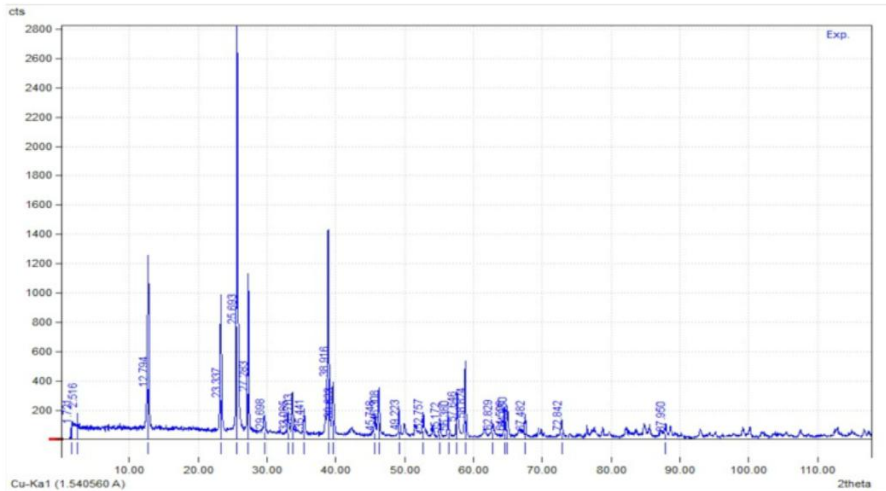


Fig. 5.2: XRD analysis image of the CNT-TiO₂ catalyst.

One of the key advantages of XRD analysis is its capacity to distinguish between different crystal structures of the exact same material and identify unknown compounds. XRD analysis may also provide quantitative data on the relative amounts of each phase present in a sample.

Finding H, K, L, the basic idea underlying X-ray diffractogram analysis (XRD) is Bragg's rule, which connects the wavelength of X-rays to the distance between crystal surfaces in a substance:

$$n\lambda = 2d \sin\theta, \text{ where } d = a / \sqrt{h^2 + k^2 + l^2}$$

$$n \lambda / 2 \sin \theta = a / \sqrt{h^2 + k^2 + l^2},$$

where d is the distance between crystal planes, n is an integer, is the angle of incidence of X-rays on the crystal and is the wavelength of X-rays.

The sampling is scanned using 2θ, and the range is from 10° to 60°. The wavelength of Cu radiation is 1.54 Å, where n=1, a is lattice constant = 10 Å.

The possible values of h, k, and l that satisfy this equation are: h = 2, k = 1, l = 0 or h = 0, k = 2, l = 1 or h = 1, k = 0, l = 2

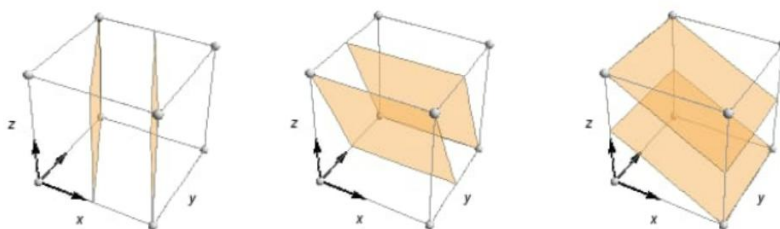


Fig. 5.3: Crystal structure from H, K, L

5.4 FFA REDUCTION OF RUBBER SEED OIL BY ESTERIFICATION

The results for FFA% reduction in crude rubber seed oil which was done by acid esterification technique with intermediate moisture removal technique is summarized in table 5.2.

Table 5.2: FFA% reduction results for rubber seed oil

Technique used	FFA% results
After 1 st moisture removal	33.72%
After 1 st esterification	8.17%
After 2 nd moisture removal	6.09%
After 2 nd esterification	1.12%

From the results it can be concluded that to reduce the FFA% for rubber seed oil below the safer level of 2% esterification has to be performed two times. Using the esterification procedure to reduce the FFA concentration of oil to less than 2% has various advantages. The oil's quality and stability are improved since excessive FFA levels can cause oxidation and degradation. The production of biodiesel reduces significantly when the FFA concentration of the oil exceeds 4%. The reason for this is because sometimes commercially used alkaline catalysts react with FFAs to generate soap, which hinders the separation of ester and glycerin. In conclusion, lowering the FFA concentration of oil to less than 2% can result in enhanced quality and stability, lower processing costs, and higher yield during refining.

5.5 EFFECT OF TYPE OF CATALYST ON BIODIESEL YIELD

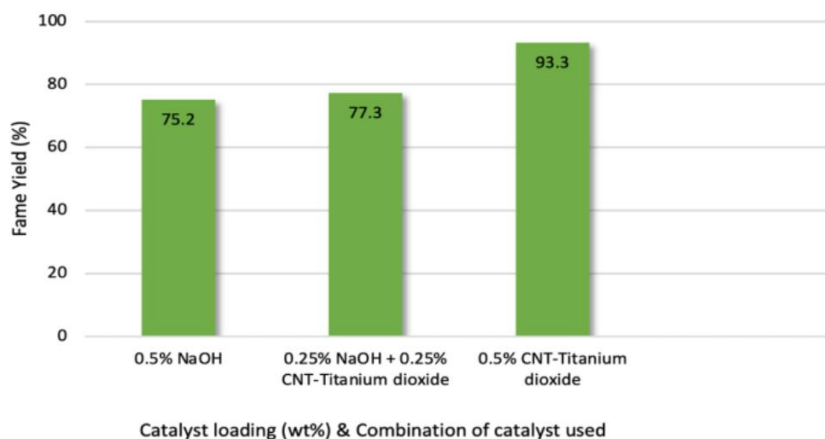


Fig. 5.4: Comparison of yield% for all the three samples

The yield percentage for all the three biodiesel samples which were produced using the two types of catalyst categories in the combination of NaOH only (homogenous catalyst), NaOH (homogeneous) + CNT-TiO₂ (heterogenous) and CNT-TiO₂ only (heterogeneous) were recorded and compared, which is represented in Fig 5.3. From the yield% results it can be concluded that when heterogenous catalyst was used biodiesel yield was significantly higher. The reason being is due to the type of washing technique used. When the biodiesel was prepared using homogeneous catalyst, wet washing technique had to be employed, which significantly reduced the biodiesel yield as during wet washing the process of separation was difficult, as when the water was drained out from the separating funnel, some amount of biodiesel also came along with water leading to reduction in overall biodiesel amount, resulting in reduced biodiesel yield. Since the wet washing technique was repeated 6 times for biodiesel produced using homogeneous catalyst and 4 times for biodiesel produced using combination of homogeneous and heterogeneous, each time the wet washing technique was employed the wastage of biodiesel during the separation process took place leading to a significant reduction in the yield of biodiesel. Whereas when membrane washing was used the separation of the biodiesel from the catalyst and other impurities was easy and less time consuming. There was no need for water washing and the wastage of biodiesel during the process was significantly less, therefore leading to a better yield.

5.6 CHARACTERIZATION OF THE BIODIESEL

The chromatogram in Fig.5.4 obtained was analyzed and the types of fatty acid methyl esters were determined by interpreting the retention time, area time and height% of each FAME visibly present in the chromatograph with standard reference library and database which contains retention time index and spectra of relevant FAME.

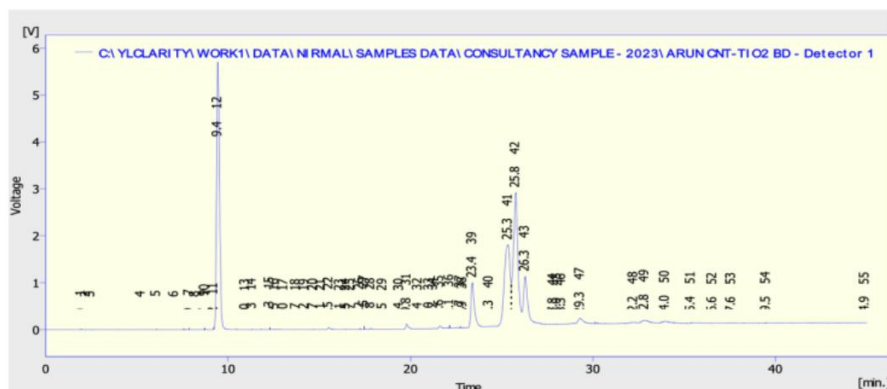


Fig. 5.5: GC-FID chromatograph for biodiesel produced using CNT-TiO₂ catalyst.

In the biodiesel sample, there was a total of 80% saturated fatty acid methyl ester content, where myristic acid, tricosanoic acid and lignoceric acid were present in the percentage of 26.4, 48.2, and 5.4. The presence of these saturated fatty acids help can increase the cetane number of the biodiesel, which is a measure of its ignition quality. Higher cetane numbers result in more efficient combustion and smoother engine operation. These saturated fatty acids methyl esters have a relatively high melting point and can improve the low-temperature properties of biodiesel. They can reduce the cloud point and pour point, which are the temperatures at which the fuel begins to solidify and lose its fluidity. By reducing these temperatures, saturated fatty acids can help to ensure that biodiesel remains liquid and flows freely even in cold weather conditions.

5.7 PHYSIO-CHEMICAL PROPERTIES OF THE BIODIESEL

The properties of the biodiesel produced using CNT-TiO₂ were recorded and compared with the ASTM6751 standards to analyze the fuel's characteristics which is summarized in table 5.3.

Table 5.3: Properties of Biodiesel synthesized using CNT-TiO₂ catalyst

Property type	Biodiesel using CNT-TiO ₂	ASTM D6751
Moisture content (%)	0.087	< 0.03
Density (g/mL)	0.887	0.85-0.9
Viscosity (cSt)	4.234	1.9-6.0
Cloud point (°C)	5	-3 to 12
Pour point (°C)	-22	-15 to 10
Acid value	0.5662	0.80 max
FFA (%)	0.28	0.42 max
Calorific Value (cal/g)	8763.88	8359.61 – 9553.84
Flash Point (°C)	165	130 min – 191max

5.7.1 Moisture content

The moisture content % of the biodiesel met the ASTM D6751 standard with the value of 0.087%, as the standard specifies the value to be less than <0.03%. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below:

1. Avoiding microbial growth: Microbial development may occur in biodiesel, which can lead to its degradation and spoiling. Microorganisms require moisture in order to thrive, therefore keeping the moisture level of biodiesel low can help limit microbe development.
2. Preventing hydrolysis: When biodiesel comes into contact with water, it can undergo hydrolysis, a chemical process that breaks up the bonds of ester in the fuel. This might result in the development of free fatty acids and other byproducts, which can harm the engine and degrade the fuel economy. Keeping the moisture level of biodiesel low can help avoid hydrolysis.

3. Improving fuel quality and stability: Moisture can also have an impact on biodiesel's overall quality and stability. Water in the fuel can cause emulsions to develop, making the gasoline challenging for separation from water and reducing its energy content. Furthermore, moisture can contribute to biodiesel oxidation, which can result in the creation of deposits and other byproducts that degrade the fuel's performance and longevity. Biodiesel can preserve its quality and stability over time by keeping the moisture content low.

5.7.2 Density

The density of the biodiesel determined was 0.887 g/mL, which lies between the acceptable range of 0.85 - 0.9 g/mL as specified by the ASTM D6751 standard. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below:

1. Quality control: One of the most significant indicators of biodiesel fuel quality is density. Evaluating the density of biodiesel aids in ensuring that it fulfills the necessary standards and regulations, as well as detecting any potential impurities or pollutants that may impair its efficiency and performance.
2. Performance and efficiency: The density of biodiesel fuel is critical to its performance and economy. It has an influence on the energy content, combustion characteristics, and also viscosity of the fuel, all of which can affect engine performance, fuel efficiency, and greenhouse gas emissions.
3. Blending and mixing: Biodiesel is frequently blended with other fuels to increase its qualities and performance, such as diesel, petrol or sustainable diesel. The density of biodiesel fuel is a crucial component in calculating the optimum blend ratio and ensuring correct fuel mixing and distribution, which can impact the overall quality and consistency of the blended fuel.

5.7.3 Kinematic viscosity

The kinematic viscosity of the biodiesel determined was 4.234 cSt at 40°C, which lies between the acceptable range of 1.9 – 6.0 cSt at 40°C specified by the ASTM D6751 standard. This result is significant as it indicates that the biodiesel synthesized in this study will have the following favorable characteristics.

1. Engine performance: Biodiesel's kinematic viscosity might impair the fuel's potential to flow correctly and effectively in an engine. If the viscosity is excessively high, it can lead to very poor fuel atomization, unfinished combustion, and increased engine wear. If the viscosity is excessively low, it might cause leaking and reduced lubrication. Finding the proper kinematic viscosity for biodiesel is critical for guaranteeing optimal engine efficiency.

2. Fuel transportation: Biodiesel's kinematic viscosity influences its ability to be transferred through pipes, tanks, and other means of transportation. When the viscosity is excessively high, it increases the likelihood of blockages and makes transportation more challenging and costly. If the viscosity is excessively low, however, it might cause spills and leaks during shipping. As a result, determining the proper kinematic viscosity is critical for guaranteeing safe fuel delivery.

5.7.4 Cloud point and pour point

The cloud and pour point of the biodiesel were determined to be 5°C and -22°C, which are within the acceptable range of -3 to 12°C for cloud point and -15 to 10°C for pour point as specified by the ASTM D6751 standard. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below;

1. Cold Weather Conditions: The cloud point is the temperature at which microscopic solid particles begin to form in biodiesel, giving it a foggy or hazy appearance. This can pose issues in cold weather because the particles can block gasoline filters and fuel lines, making it harder for the fuel to flow correctly.

The pour point is the temperature at which biodiesel thickens and becomes viscous, making it impossible to flow. In cold temperatures, the biodiesel may become too thick to pump or atomize adequately, resulting in partial combustion and poor engine performance.

5.7.5 Free fatty acid and acid value

The FFA% and acid value of the biodiesel was determined to be 0.28% and 0.5662, respectively, which met the ASTM D6751 standards. The maximum FFA% and acid value allowed is 0.42% and 0.80 respectively, as specified by the standard. This result

is significant as it indicates that the biodiesel synthesized in this study has low acid value which helps prevent the following undesired conditions;

1. Accelerated Oxidation: FFA may speed up the process of oxidation in biodiesel, leading to the development of hazardous chemicals such as peroxides and acids. This can cause the gasoline to deteriorate over time, lowering its efficiency and potentially causing engine damage.
2. Corrosion: FFA in biodiesel can also cause corrosion in fuel system components such as tanks, pipes, and injectors. This can result in clogs, leaks, and other mechanical issues.
3. Formation of Deposits: FFA may react with metal ions to generate insoluble salts and other deposits that can clog fuel filters and other components of the fuel system.

5.7.6 Calorific value

The calorific value of the biodiesel was determined to be 8763.88 cal/g, which lies within the acceptable range of 8359.61 – 9553.84 cal/g as specified by the ASTM D6751 standards. A Biodiesel's calorific value is a significant component in assessing its energy content and appropriateness for usage in various applications such as combustion engines, electricity generating, and heating systems. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below;

1. Energy Content: The calorific value of biodiesel is critical in calculating its energy content, which influences engine efficiency. A greater calorific value indicates that more energy is accessible from the fuel, which results in improved engine performance. If the calorific value is lower than that of typical diesel fuel, the engine may require more fuel to generate the same amount of energy, resulting in worse fuel efficiency.
2. Economic analysis: Knowing biodiesel's calorific value is critical in establishing its cost-effectiveness in comparison to other fuels. This data is used to assess the retail price of biodiesel as well as the potential savings for users.

Furthermore, measuring the calorific value of biodiesel enables for precise assessments of energy content and prices, allowing producers and customers to make better decisions.

5.7.7 Flash point

The flash point of the biodiesel was determined to be 165°C, which lies within the acceptable range of 130°C – 191°C as specified by the ASTM D6751 standards. The flash point of a liquid is the lowest point of temperature at which it can produce enough vapors to generate an ignitable combination in air near the liquid's surface. It is the temperature at which a liquid can ignite if it comes into contact with an ignition source, such as a flame or spark. This result is significant as it indicates that the biodiesel synthesized in this study will have various favorable characteristics as mentioned below;

1. Reduced explosion risk and fire: Biofuels with higher flash points are far less likely to catch flames and cause an explosion or fire. As a result, they are easier to handle during storage, shipping, and dispensing. Higher flash points assist to safeguard personnel, equipment, and facilities by lowering the danger of fire and explosion.
2. Easiest transportation and storage: Because they require fewer severe safety procedures, biodiesel with a higher flash point may be stored and transported more simply. For example, if a biodiesel flash point exceeds a specific level, it may not be pivotal to store it in a specially constructed, fire-rated storage facility. This can save both time and money on storage and transit charges.

5.8 REUSABILITY OF THE CATALYST

The yield of biodiesel decreased each time the catalyst was regenerated and used for transesterification reaction as depicted in the Fig 5.5. Under the specified optimal process parameters, the biodiesel output decreased from 93.33% to 67.8% for 5 cycles of catalyst reuse. The reason being the increase in the formation of glycerol and decrease in the formation of biodiesel. When the catalytic activity reduces, its selectivity towards the transesterification decreases while its selectivity towards the hydrolysis of unreacted triglycerides increases, leading to increase in glycerol formation. The reason being due to reduced catalytic activity, partial transesterification of the triglycerides takes place leading to the formation of intermediates such as

monoacylglycerols (MAGs) and diacylglycerols (DAGs) which undergoes hydrolysis in order to form fatty acids and glycerol. All of the ester bonds from triglycerides may not break to react with alcohol to form desired esters due to reduced catalytic activity. The catalytic activity decreases after each reuse which might be owing to molecules of oil blocking the active sites on the catalyst surface despite thorough washing with n-hexane and other factors like poisoning, leaching, sintering. Nonetheless, the catalyst could be reused at least for 5 times before employing new catalyst. Since it permitted catalyst re-use, the total process can be described as affordable as well as environmentally friendly.

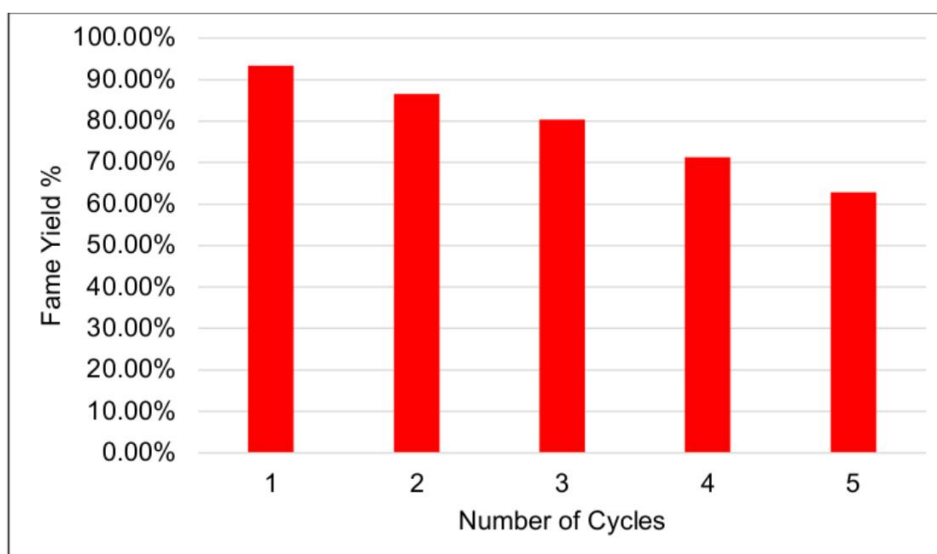


Fig. 5.6: Regeneration studies

CHAPTER-6

SUMMARY AND CONCLUSION

Biodiesel was successfully synthesized from rubber seed oil using a novel catalyst called CNT-TiO₂ composite which is categorized as a heterogeneous catalyst. Transesterification reaction using homogeneous catalyst proved to give less yield of biodiesel when compared with when transesterification was done using heterogeneous catalyst, the reason being less glycerol formation and easy separation of biodiesel from glycerol resulting in less wastage of biodiesel when heterogeneous catalyst was used. The easy separation was assisted by membrane washing technique which was less time consuming, and there was no waste water generation, the separation technique helped to prevent wastage of biodiesel during the process, thus improving the biodiesel yield as well as increasing the efficiency of the whole synthesis process. Regeneration studies showed that the catalyst's activity was reduced each time it was reused resulting in a decrease in biodiesel yield, but from the results it can be inferred that the catalyst could be reused at least 5 times before employing a new catalyst. This proves that the catalyst has a good life and is economical for the biodiesel synthesis. The properties of the biodiesel were compared with ASTM D6751, and all the values of the properties met the standards, thereby proving that the biodiesel produced is of good quality to be able to attract fuel buyers and suppliers and can meet regulatory benefit and other tax incentives.

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