PRODUCTION OF BIODIESEL FROM COCONUT OIL USING BIFUNCTIONAL CATALYST FROM CARB SHELLS AND COCONUT SHELL FOLLOWED BY ITS OPTIMIZATION

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A PROJECT SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF BACHELOR OF ENGINEERING (B.Eng) DEGREE. IN THE DEPARTMENT OF CHEMICAL ENGINEERING, FACULTY OF ENGINEERING, UNIVERSITY OF BENIN, BENIN CITY, NIGERIA.

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CERTIFICATION

This is to certify that this project research work was carried out by AGBONIGIARHUOYI OSASUMWEN of the Department of Chemical Engineering, University of Benin, Benin City, Edo State, Nigeria.

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ENGR. DR. O.U. OSAZUWA **PROJECT COORDINATOR**	**DATE**
ENGR. DR. MRS. E. A. OYEDOH **HEAD OF DEPARTMENT**	**DATE**
EXTERNAL EXAMINER	**DATE**

DEDICATION

This work is dedicated to God and to my parents. This work is also dedicated to those with an interest and passion to discover new things and also to impact the academic field. iv

ACKNOWLEDGEMENT

We thank God for giving us the ability and grace to complete this job.I want to express my gratitude to everyone who helped this project be completed successfully. I would like to thank my research supervisor, Engr. Prof. K. O. Obahiagbon, for his invaluable guidance, patience, and advice throughout the research's development.

I also want to thank my devoted parents and siblings for their understanding and patience in allowing me the time and space I needed to do my research as well as for being there for me when I needed them the most.

In addition, I'd want to thank my friends and classmates for helping to make this research project pleasurable by not just providing me with the support and motivation I needed to finish the study, but also participate in brainstorming sessions and help make this study project enjoyable.

ABSTRACT

In this study, coconut oil (CO), which had been characterised and shown to have an Acid Value of 5.8 mg KOH/g, was utilised to optimise the synthesis of biodiesel, which was catalysed by a bio-based bi-functional catalyst, by esterifying and trans-esterifying the CO simultaneously. The carbonised and sulfurized coconut shell was used to create the acid precursor for the bio-based bi-functional catalyst, while the calcined and KOH-treated crab shell was used to create the base precursor. Both precursors were then impregnated. The synthesised bio-based catalyst was characterised using SEM, XRD, FTIR, and BET/BJH techniques. The experimental design was optimised using Box Behnken Design (BBD) for the simultaneous esterification and transesterification of CO. The measured response was the production of coconut oil methyl ester (COME). The methanol-to-oil molar ratio, reaction temperature, and catalyst loading had the biggest effects on COME yield among the factors. The study's ideal conditions were determined to be a 16.75:1 methanol to oil ratio, 1% catalyst loading, 58.19 °C, and a reaction duration of 78.76 min, yielding a COME yield of 85.73 wt% and an AV of 0.28 mg KOH/g. RSM and ANFIS models were compared using statistical data, and the results showed that the ANFIS model was more accurate than the RSM model. After characterising the COME obtained in the best possible condition, it was determined that its physicochemical parameters satisfied the ASTM D6751 and EN 14214 standards for biodiesel.

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CHAPTER ONE

INTRODUCTION

1.1 RESEARCH BACKGROUND

The search for green energy is of utmost importance everywhere; hence numerous production methods have been considered.

The tradable currency of technology is energy. Without energy, society as we know it would fall apart from the ground up. A city's electricity supply demonstrates how dependent we are on this particularly useful form of energy. Hospitals drop to a level of care and maintenance, computers and elevators stop working, and the lights go off. Population growth often exceeds the average growth rate of 2%, which inincreasing energy needealthy industrialized economies, which are home to 25% of the world's population and have rising energy demands, use 75% of the world's energy supply (Dincer, 2000).

In addition to global warming, issues with energy supply and use also affect other environmental issues such air pollution, acid rain, ozone depletion, forest loss, and radioactive material emissions. If mankind is to have a successful transition to an energy future with minimal negative environmental effects, these factors must be taken into account simultaneously. There is a lot of evidence to show that if humans continue to damage the environment, the future will suffer.

The public and the energy sector are beginning to pay more attention to other environmental factors. More people are accepting the idea that customers share responsibility for pollution and its costs. Over the past one to two decades, the cost of the environment has contributed to price increases for numerous energy resources in several countries. By the middle of the twenty-first

century, the population of the world is projected to double, and economic growth will almost probably continue. By 2050, the demand for energy services might rise by an order of magnitude while the need for primary energy could rise by 1.5 to 3 times (Dincer, 2000). Concern over energy-related environmental issues such acid precipitation, stratospheric ozone depletion, and global climate change is projected to grow concurrently. Making greater use of renewable energy sources and technology is one way to combat the coming energy crisis. Sometimes, the enthusiasm with which this cause is promoted gives rise to excessive and implausible statements. Engineering viability, dependability, adaptability, economics, lack of supply, and public acceptance should all be taken into consideration.(Dincer, 2000).

1.2 STATEMENT OF THE PROBLEM

The urge to provide clean, reliable energy sources arose from the negative environmental effects of fossil fuels and their non-renewable nature; this led to the development of biofuels. Biofuels (such as biodiesel) are a class of fuels made from natural sources that have been shown to be less harmful to the environment and ecology than fossil fuels. Biodiesel is one of the many types of biofuels that may be produced utilizing catalyst from oils and alcohol.Non-edible, edible, inexpensive, and waste biomass are the main feedstock utilized to synthesis biodiesel in order to address the problems of fuel competing with food and high cost of biodiesel.

The focus is now on synthesizing a strong solid catalyst that would simultaneously catalyze esterification and transesterification reactions using wastes from natural sources in order to produce biodiesel. This is necessary for the economic viability of biodiesel production as well as in the effort to reduce production costs.

1.3 AIM

The aim of this study is to produce biodiesel through simultaneous transesterification and esterification of coconut oil using a heterogeneous bifunctional catalyst synthesized from crab shell and coconut shell

1.4 OBJECTIVE

1. Characterization of coconut oil gotten from a local source
2. Preparation of bio-based bifunctional catalyst for the simultaneous esterification and transesterification of coconut oil
3. Optimization of biodiesel production from coconut oil using RSM design software (Box Behnken)
4. Investigating the effects of process variables (Reaction time, catalyst loading, reaction temperature and methanol- oil ratio) on desired response, that is, the biodiesel yield.
5. Characterization of biobased catalyst.
6. Comparative study on the optimization of biodiesel production using RSM and ANFIS models.
7. Characterization of coconut oil Methyl Esters (COME)

1.5 SCOPE

I. Collection of crab shell from local sources.

II. Collection of coconut shells.

III. Purchase of the coconut oil from local source.

IV. Characterization of coconut oil purchased.

V. Preparation of bi-functional catalyst from collected crab shell and cocnut shell.

VI. Characterization of the prepared catalyst from collected shells.

VII. Transesterification of the coconut oil using the prepared heterogeneous catalyst for biodiesel production

VIII. Determination of biodiesel yield using RSM and ANFIS model

IX. Comparative study on the optimization of biodiesel production RSM and ANFIS model.

X. Determination of optimum reaction conditions and biodiesel yield using Response Surface Methodology (RSM)

1.6 SIGNIFICANCE OF STUDY

With growing understanding of the need to pursue sustainable development for the sake of the environment, biodiesel manufacturing has always been ongoing. Technology has continuously aided in the search for safer and more affordable production techniques. These approaches, which use biocatalysts, acid-base catalysts, and transition-metal assisted catalysts, each have advantages and limitations, and the search for a more workable solution is never-ending. A heterogeneous catalyst is used in the study to produce biodiesel from coconut oil and common natural fruit (coconut shells) as activated carbon.

CHAPTER 2

LITERATURE REVIEW

2.1 ENERGY

This is the quantitative attribute that needs to be imparted to a physical system or a body in order to heat or perform work on it.

Generally speaking, the term "energy" refers to the capacity to perform work; it cannot be created nor destroyed but can change its shape, according to the fundamental laws of thermodynamics.

2.1.1 TYPES OF ENERGY

Energy in the universe can be categorized in a variety of ways. Some of these criteria are based on the energy's nature or sources, while others may be on how it has been used or how clean it is. The earth can be defined by its physical characteristics, which include substances known as matter, and the fact that these substances can change from one form to another as a result of man's daily activities or other physical activities occurring nearby. In essence, energy can be in the form of a solid, liquid, or gas. Non-renewable energy comes from sources that are running out of fuel, although there are also recently developed sustainable energy sources, available naturally and are called renewable because they can be replenished.

2.1.2 FORMS OF ENERGY

All five of our senses cannot perceive energy in its pure state; instead, energy can take on a variety of shapes. Energy can take on various forms, including heat, which is the shape it takes when it is transferred as a result of a change in temperature, kinetic motion, potential, and atomic energy, which is the form it takes in the atomic realm.Other forms include: electrical energy-which causes the motion of electrons often involving a magnetic interaction. Etc.

2.2 APPLICATION AND USES OF ENERGY

This physical quantity has uses in every aspect of existence, from outer space to the kitchen in our own homes. According to one school of thought, the universe formed according to the Big Bang theory through the release of a tremendous amount of energy that provided the necessary energy for the universe's gradual creation some billions of years ago. This creation, it is claimed, is still ongoing today, with new stars being born every day and some dying somewhere in the universe.On earth, energy is employed to power transportation over long distances; what would have naturally taken months to cover using our legs would now just take days or even hours. This energy is used by sectors of the economy that create secondary goods and services to drive production and support daily operations. Approximately 90% of motorized transportation relies on oil, according to (Prentice et al., 2009) and accounts for almost half of the worlds use of oil.

2.3 RENEWABLE ENERGY

2.3.1 THE NEED FOR RENEWABLE ENERGY

Since the start of the industrial revolution, access to clean, affordable, and reliable energy has been the cornerstone of the world's rising affluence and economic growth (Chu and Majumdar, 2012). Energy sources that are renewable may be replenished, making them sustainable. They are sources of energy that are continuously replenished by natural processes including the sun, wind, water, heat from the Earth, and plants. Technologies that transform renewable energy into forms that are used to accomplish work in our daily activities, such as electricity, heat, chemicals, mechanical power, etc., are continuously being employed by the human race.Because there is little to no pollution released during consumption, energy from renewable sources is commonly referred to as "clean" or "green." However, certain energy sources have an adverse impact on the environment. By releasing greenhouse gases that destroy the ozone layer and trap solar radiation, burning fossil fuels contributes to climate change and global warming. This gradual increase in atmospheric

temperature is brought on by the release of these gasesScientists have predicted that if the atmospheric average temperature is allowed to continue to rise gradually, there will be flooding due to the sea level rising gradually, as well as heat waves, droughts, and other naturally occurring disasters. In 2001, the National Renewable Energy Laboratory. Additionally, if the supply of conventional oil declines due to rising demand, lesser grades of oil that are more expensive and less clean will start to be a feasible option, which will increase costs, environmental damage, and competition with the cosmetic and pharmaceutical industries for raw materials (Prentice et al., 2009).

2.3.2 ADVANTAGES OF RENEWABLE ENERGY

"Renewable energy is energy generated for direct end use or the production of electricity partially or totally from non-depleting energy sources. The majority of policy makers typically only have access to the costs of energy measures and are unaware of all of their advantages. For instance, calculating the entire energy cost savings of buying a new solar-powered electrical system without accounting for the advantages to the environment and human health brought about by lower emissions. Additionally, States can learn that the advantages of renewable energy policies extend to many different economic sectors, including power, the environment, and health, and they can help government organizations with integrated planning and management to maximize the advantages of having fewer policies and programs to achieve objectives.(Environmental Protection Agency (EPA), 2018, pp. 4-5).

2.3.2.1 Category of Renewable Energy Benefits

Energy efficiency and renewable energy benefits to power systems, emissions and public health, and to the economy were categorized by the Environmental Protection Agency in the United States (2018, pp. 6-7) based on energy and renewable energy savings generation as follows.

A. Electric System Benefits: Electricity distribution bottlenecks will be lessened by an energy-efficient, renewable system, bring stability, reliability and security to the system and reduce cost of increasing capacity.

B. Emissions and Health Benefits:When fossil fuels are used as energy sources, greenhouse gases, fine particles, and occasionally ground level ozone are released into the atmosphere, polluting it and the environment as a whole. These chemicals or particles are hazardous to health and can cause illness. The use of efficient renewable energy sources reduces these risks and is sustainable.

C. Economic Benefits: Companies that use efficient and renewable energy can benefit economically by using more accessible energy sources, paying less for operations and business operations in general, creating jobs, increasing profits, and increasing revenue. These companies can be in the manufacturing, construction, service, or even government-owned sectors.There are also economic benefits that culminates from sustainable energy systems, health and environmental benefits such as increase in productivity of the workers and people in general.

2.4 TYPES OF RENEWABLE ENERGY

Research has gone into the use of affordable and sustainable sources of energy such as solar, wind, tidal, ocean energy and hydropower etc.

2.4.1 HYDRPOWER

One of the most utilized and abundant natural energy sources, hydropower is now a crucial component of the energy roadmaps of the world's main economies, including the United States, China, and other nations. The hydropower plant, which uses a dam to store a lot of water on the river and turbines to act as outlets and generate power simultaneously, is the technology used to

convert hydropower energy into electricity. Although hydropower is a cleaner source of energy, technology used to transform it into useable forms, like the hydropower plant, nonetheless have an adverse impact on the river and the aquatic species that inhabits it.Hydropower systems use certain advances to mitigate this, reducing riverbank obstruction of natural water flow and decreasing the loss of aquatic life. (National Renewable Energy Laboratory, 2001)

2.4.2 GEOTHERMAL ENERGY

About 4,000 miles below the surface of the globe, geothermal energy is extracted from the Earth's core. It has been determined that the temperature of its core can reach 9000°F. The heat from the core is stored in underground reservoirs and used for a variety of things, like heating houses or producing power. The constant temperature from the ground can be channeled for home needs using equipment like geothermal heat pumps. In terms of energy potential, geothermal energy can be said to be incomparable to fossil fuels. The majority of nations have sizable geothermal reservoirs that can be used to generate electricity using geothermal heat pumps. (National Renewable Energy Laboratory, 2001).

Some practical uses for geothermal energy include the following: direct use of geothermal energy, such as in hot springs; production of electricity using geothermal energy in power facilities where steam turbines powered by hot water or stream from geothermal wells power electric generators; Heat pumps are used by geothermal heat pumps to extract heat from the earth for heating and cooling purposes through underground piping.

2.4.3 SOLAR ENERGY

Solar radiation, commonly known as solar energy, is the energy that comes from the sun. Using solar technology, this energy that enters the earth's atmosphere directly through waves is captured and stored until it can be converted into one or more other forms and used for labor. Conventional

solar technologies are made to directly access the sun's radiant energy and use it to create various types of energy, such as;heat, light, and power. (National Renewable Energy Laboratory, 2001).

Table 2.1 Advantages and Disadvantages of Solar Energy generation.

Advantages Disadvantages	**Advantages Disadvantages**
Resources (if well maintained) are infinite and will not extend regardless of the amount of consumption	Expensive for household
Climate friendly	Produces sufficient energy, but storage problematic (sometimes need for public energy supply)
Profitable investment	Cost of maintenance
No toxic gas production	Weather dependent
Every area has access to sunlight even if not Regularly	
No noise pollution	

(Adra, 2014, p.6)

2.4.3 WIND ENERGY

Wind energy has been utilized since navigators first used it to power boats on lakes and rivers. 2018 (Khillar). One of the most prevalent energy sources on the globe is wind energy. In recent years, wind mills have become a popular technique for capturing wind energy. Wind turbines are currently the most efficient way to convert huge amounts of wind energy.

In order to generate power, wind turbines are "basic" machines. They are made up of turbine blades attached to a central hub and a shaft that power a generator's motor to generate electricity.Recent improvements to wind energy technology designs include the use of air foil blades, contemporary mechanical shaft systems, and improved generators, among others. Due to the rapid development of wind technologies worldwide, wind energy is one of the energy sources with the quickest growth rates. Currently, the United States is using up to 2500 MW of wind energy, which has the potential to be one of the nation's primary energy sources. In 2001, the National Renewable Energy Laboratory. Windmills would operate in complete opposition to electric fans (Adra, 2014, P.7). Windmills are a type of wind turbine that differ from them in that they are powered machinery that uses vanes to transform wind energy into mechanical energy (Khillar, 2018). Windmills transform wind energy into mechanical energy that can be utilized directly to power mechanical processes like grinding, pumping water, and operating electric motors, among others (Ecavo, 2021). They are typically positioned distant from residential areas because of the noise that the power-driven gadgets produce. (Adra, 2014 p.7).

Table 2.2 Advantages and Disadvantages of wind energy

Advantages	**Disadvantages**
Creates many job opportunities (e.g. installation, maintenance etc.)	Expensive
Climate friendly	Storage of energy
Low maintenance cost	Difficult maintenance
No air, ground or water pollution	Infrastructure to transport energy
Reduces CO2 emission	Weather dependent

Can be installed off-shore and on land	Surrounding wild life may be disturbed
Resource is infinite	Noise pollution in the installed areas
	Land disputes

(Adra, 2014)

2.4.4 HYDROGEN

Given that it only creates water as a byproduct of burning rather than other gases like carbon dioxide CO2 or carbon monoxide CO, hydrogen is a particularly pure source of energy. It is also the simplest fuel on earth and incredibly abundant; it just has one proton and one electron. Because it cannot be found in nature in its purest form, hydrogen is produced in industry and has a very high energy content. The most popular method of producing hydrogen is steam methane reforming (SMR), an industrial process that involves the application of heat to separate hydrogen from carbon. Methane from natural gas is reacted with steam to produce hydrogen gas as the end product. Proton Exchange Membrane fuel cells are an unique innovation for the reforming process. To generate, researchers are creating very effective, sophisticated reformers. One of the top renewable energy sources in the world, hydrogen fuel cells also have the ability to act as an energy delivery, storing, and transporting medium. The direct synthesis of hydrogen from water, one of the most common chemical species on earth, would open up new possibilities for the development of hydrogen fuel technology. (National Renewable Energy Laboratory, 2001).

2.4.5 OCEAN ENERGY

In terms of maritime technology, research, and development, Europe is a global leader. Ocean energy is generated mechanically by tides and waves or thermally by redistributing solar heat. The majority of ways that energy from the oceans is used involves its conversion to electrical energy, which is then used in electrical systems that use the warm surface water from the ocean's surface

or boil the water there to power turbines that are connected to electrical generators. The "Adam" gadget, in particular, uses a turbine system to transform tidal energy into electricity, whilst wave energy is employed to either directly or indirectly activate a generator via a hydraulic system. (National Renewable Energy Laboratory, 2001)

2.4.6 BIOMASS

Biomass is another form of renewable energy that has a strong potential to meet the energy needs of modern societies, whether they are developing or industrialized. The term "biomass" refers to non-fossilized, biodegradable organic material derived from plants, animals, and microbes. Agricultural, forestry, and associated industry products, byproducts, residues, and garbage are also included in this, as are the organic non-fossil and biodegradable fractions of industrial and municipal wastes. Gases and liquids that are recovered from the breakdown of non-fossilized and biodegradable organic material are also referred to as biomass.These days, households and businesses use a lot of wood, farming waste, fruit bunches that have been emptied of their fruit, leaves, and other agricultural products as fuel (Ashwani et al., 2014). About 10–15% (45 exajoules, EJ) of the world's energy consumption comes from biomass. In the vast majority of Asian nations, it is one of the main energy sources. There are now 5 different forms of biofuels that use biomass as a source: pyrolysis oil, bio-ethanol, bio-methanol, bio-briquettes, and hydrogen gas.Currently, only biodiesel and bioethanol are produced on an industrial scale as fuels (Ashnani et al., 2014). Fuels like biodiesel and bioethanol are at the forefront of alternative technologies to meet the growing energy demand and replace diminishing petroleum supplies. Consequently, biodiesel is a practical substitute for compression-ignition engines.(Demirbas, 2007).

2.4.7 BIO ENERGY

Energy obtained from organic materials, such as plants, is known as bioenergy or energy derived from living things. Because they can be renewed, these energy sources are also renewable energy sources. In daily life, bio energy is used for things like cooking edible items made from plants and animals for nourishment or burning wood in a fireplace or campfire. However, not all of the biomass we use comes from plants or trees. Large amounts of unwanted or leftover biomass can be produced by a variety of sectors in industrialized nations, including those engaged in building or the processing of agricultural products, which can then be used as a source of bioenergy. In 2001, the National Renewable Energy Laboratory.

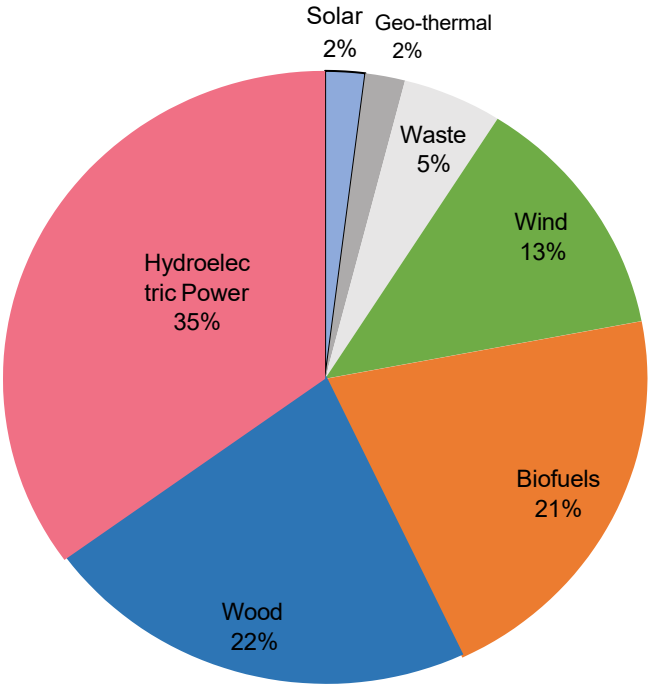


Fig 2.1 Global energy consumption rate as at 2011.

SOURCES OF BIO-RENEWABLE ENERGY

2.4.8 BIO POWER

Bioenergy, or energy derived from living things, is energy produced from organic resources like plants. These energy sources are called renewable energy sources because they can be replenished. Bio energy is utilized in everyday activities like preparing food made from plants and animals for sustenance or burning wood over a campfire or fireplace. Although humans use a lot of biomass from plants and trees, not all of it is. In industrialized countries, a range of industries, such as those involved in construction or the processing of agricultural products, can produce large volumes of unwanted or leftover biomass, which can then be used as a source of bioenergy.The National Renewable Energy Laboratory was founded in 2001. Additionally, biomass can be gasified to create biogas, which can be utilized in gas turbines to produce energy. The use of methane concentrated gas derived from landfills, which is burned in boilers to produce steam for use in electricity generation and as a utility in industrial operations, is another method for producing electricity from biomass. Another byproduct of exposing biomass to high temperatures is pyrolysis oil, which is similarly utilized for power generation or as a feedstock for chemical synthesis. (National Renewable Energy Laboratory, 2001).

2.4.9 BIOFUELS

Biofuels are straightforward fuels created by chemically converting biomass into liquid fuels, frequently after some sorting and purification processes. Ethanol and biodiesel are two popular types of biofuels. Crops that are high in carbohydrates, like corn and cassava, can be converted to ethanol through fermentation in the presence of biological catalysts. Fuel quality can be improved by adding ethanol as a fuel additive. By transesterifying lipids into less viscous liquids that can be

used in diesel engines or combined with regular diesel for use in gasoline-powered diesel engines, biodiesel is created. Transporting biofuels is simpler than moving feedstock.

According to reports, when refueling a car, biofuels have a higher energy density and a higher rate of energy transfer (Chu and Majumdar, 2012). Therefore, it is better to convert biomass into biofuels for use in transportation and energy production.

For example, ethanol from sugar cane, diesel-like fuel from soybean oil, dimethyl ether (DME), or Fischer-Tropsch liquids (FTL) from lignocellulosic biomass are all examples of biofuels that can be used as alternatives to petroleum-derived fuels.2008 United Nations, p. 1. Biofuels can be solid (bio char), liquid (ethanol and biodiesel), or gaseous (biogas, biohydrogen, and biosynthetic gas), depending on their state of matter (Thanh et al., 2012, p.192). Based on its origins, biofuel has been categorized as generational across time. Second generation fuels are made from non-edible lignocellulosic biomass, such as non-edible whole-plant biomass, such as grasses, or non-edible crop production residues, such as corn stalks and rice husks. First generation fuels are made from sugar, grains, and seeds. In a number of applications, biofuels are utilized as an alternative to fuels sourced from petroleum.Fuel produced by the Fischer-Tropsch process is also used in compression ignition engines, as well as alcohol fuels in spark-ignition engines, biodiesel, and DME. About 3 billion people in poor nations now utilize solid fuels, which can be replaced by biofuel, which can also be used for cooking. Since biofuel emits fewer pollutants than solid cooking fuels, it is safer to use and better for the environment. Even though these fuels are produced at a far larger scale for transportation needs than for cooking needs, many industrialized nations like the United States, France, England, and other European nations are paying greater attention to the development of biofuel for transportationThe current stage of biofuel production from biomass is labor-intensive by nature and ideal for rural communities, giving locals work and

bridging gaps in municipal power supply to such places. Despite the enormous advantages of using biofuels, there are concerns about land being diverted from agricultural and other ecological uses and strain on the water resources that are used to grow the feedstock for biodiesel. However, rising economic policies, actions taken to combat climate change, the current high price of oil, worries about energy security, etc. are projected to increase the market for biofuels.2008 United Nations. With already-existing automobiles and filling stations, biofuels are simply adaptable. They are made from readily available, easily biodegradable biomass. Additionally, compared to fossil fuels, biofuels are more inexpensive and environmentally responsible.(Thanh *et al.*, 2012, p. 192).

2.5 BIOFUEL CLASSIFICATION AND USES

2.5.1 FIRST GENERATION BIOFUELS

First generational biofuels are biofuels obtained from oil-bearing crops (((((Elias *et al.*, 2020 p.1). Due to the fact that plants store energy as carbohydrates in their seeds, grains, stems etc. The category of fuels can be used as Petroleum-gasoline substitutes such as Ethanol or Butanol by fermentation of starches (corn, wheat, potato) or sugars (sugar beets, sugar cane) obtained from the plants. They are also used as Petroleum diesel substitutes which include Biodiesel by transesterification of plant oils, also called Fatty Acid Methyl Ester (FAME) and Fatty Acid Ethyl Ester (FAEE), Pure plant oils (straight coconut oil)2008 United Nations. Despite their high utility, there are worries about food-fuel competitions if these oil-bearing crops become the primary resource for the manufacturing of biodiesel.(Elias *et al.*, 2020 p. 2).

2.5.2 SECOND GENERATIONAL BIOFUELS

Because lignin, the substance responsible for binding activities in plant cell walls, is present in plants, they are lignocellulosic in nature. This biomass has the ability to produce energy, which is tapped through various chemical and technological processes to produce bio-based fuels.

Examples of lignocellulosic sources of biofuels from plants include crop residues, woody crops, and energy grasses. These sources can be used thermochemically to produce petroleum-gasoline substitutes like methanol and mixed alcohols or biochemically to produce ethanol or butanol by enzymatic hydrolysis. Alternatively, petroleum-diesel substitutes such fischer-tropsch, diesel fuel, and dimethyl ether

(also a replacement for propane) and Green Diesel Source (United Nations, 2008).

They are still regarded as expensive to develop in big scale production even though they are more widely available and less expensive than first generations biofuels. (Elias *et al.*, 2020 p.2).

2.6 BIODIESEL

According to numerous biodiesel studies, biodiesel has a calorific value that is almost identical to that of fossil fuels. It also has a high flash point, a high cetane number, better lubrication, and lower environmental emissions. In general, biodiesel and regular diesel fuel have relatively similar physical and chemical properties. It can be used in conventional diesel engines with little to no modification, or it can be utilized exclusively in biodiesel engines by blending it with petrol diesel fuels at a component percentage of about 5% to 20%. There are numerous ways to make biodiesel, some of which include:

1. 1.Dilution
2. Preheating
3. Micro-emulsion
4. Pyrolysis
5. Transesterification

Transesterification has established itself as the most effective method for producing biodiesel out of all these methods. Coconut oil and animal fats can be transformed into biodiesel through the esterification and transesterification of an alcohol. A appropriate homogeneous or heterogeneous catalyst is used to speed up the reaction. Depending on the technology to be used, producing biodiesel through a transesterification reaction can be done in five steps. The stages are the following: pretreatment, reaction, washing, alcohol recovery, and glycerin purification. The removal of excess free fatty acids (FFA), water, and other undesirable elements is aided by pretreatment processes. Prior to adding the oil during the reaction stage, the alcohol and catalyst are mixed in a well-stirred reactor. To get rid of unreacted glycerol, soap that generated during the reaction, and other impurities, the product is washed—especially when a homogeneous catalyst is employed. The use of water during washing is an option, but it may come at an additional cost and raise questions concerning the solvent's availability, water treatment, and emulsion issues. While dry cleaning is accomplished via the use of chemical processes and iron exchange techniques.

Now that the product has dried, byproducts or unreacted parts are recovered (Hassan and Ayodeji, 2019).

Biodiesel is made from an oil or fat source that mostly contains triglyceride molecules. Production and processing of biodiesel (methyl ester) from agricultural feedstocks like coconut oil and animal fat is developing into a significant business in nations like the United States and Canada. (Hassan

and Ayodeji, 2019).

Fig 2.2 Renewable energy sources and application.

2.6.1 BIODIESEL PRODUCTION

A catalyst that would carry out simultaneous esterification and transesterification activities in the oil is required because it has previously been established through literature and calculations that the amount of free fatty acids contained in coconut oil is considerable. A quantity of methanol was added to the created bifunctional catalyst, and it was agitated for 6 to 8 minutes. The oil was placed in a 1000 ml round bottom flask (reactor) attached to a magnetic stirrer. The reflux condenser was used to condense methanol that had escaped in a gaseous state back into the reactor. The magnetic stirrer was used to slightly warm the oil. Using a magnetic stirrer, the catalyst and methanol combination was introduced to the oil and swirled continuously for varying amounts of time at 40, 50, and 60 °C. The resulting mixture was allowed to cool and then transferred to a separation funnel where, due to differences in density, the biodiesel and glycerol separated into two distinct

phases by gravity. After the tap was opened, the glycerol was separated into a separate container, leaving the biodiesel.

2.6.2 BIODIESEL PURIFICATION

In order to eliminate any dissolved glycerol or excess methanol, the produced biodiesel was rinsed with water, separated from the waste water in a funnel, and allowed to dry. To determine whether the purified biodiesel fulfilled required standards, it was taken for analysis.

2.6.3 FEEDSTOCK

Biodiesel is produced from coconut oil or animal fat through transesterification. The feedstock is primarily passed through this process to reduce the viscosity of the oil. The type of oil utilized is important for a good product yield and to obtain an alkyl ester with the desired chemical and physical properties. The quality of a feedstock for biodiesel production entails the feedstock is free from impurities. As noted by in his research, the feedstock should have a low free fatty acid content (< 1 FFA wt. %) and moisture (< 0.5 wt.%) (Elias *et al.*, 2020 p.2). when the free fatty acid (FFA) content of the oil is high or when the oil has high water content such as in jetropha oil, rubber seed oil, tobacco oil, waste cooking oil, it could result in problems during the conversion process if a homogeneous catalyst is used (Thanh *et al.*, 2012, p.194). The feedstock may be edible, non-edible or waste cooking oil. Some edible-oil sources include: Soya bean which is a major feedstock used in the United States and has a global feedstock percentage(GFP) use of 25%, Rapeseed and Sunflower are the feedstock mainly used in the European countries with Rapeseed providing 59% of global need and sunflower 5%, Palm oil produces the most yield per unit land compared with other feed stocks. Palm oil and Coconut oil are the main sources of biodiesel in Malaysia and Indonesia. While non-edible oil sources are produced from crops such as: *Jetropha curcas, Calophylluminophyllum, Nicotianatabacum, Ceibapentandra, Calophylluminophyllum*, and

Herveabrasiliensis etc. Non-edible oil is preferred economically as feedstock for the biodiesel industry since the use of edible oil may compete with food for consumption and eventually lead to scarcity (Hassan and Ayodeji, 2019, p.622).

2.6.4 COCONUT OIL

The wick, meat, and milk of the coconut palm fruit are used to make coconut oil, an edible oil. Below about 25 °C (77 °F), coconut oil is a white solid fat; in warmer temperatures, it is a transparent, thin liquid oil. The unrefined varieties smell strongly like coconut. It is used as a culinary oil as well as in manufacturing detergents and cosmetics. Numerous health organizations advise restricting its usage as a food due to its high levels of saturated fat.

Production.

Production of coconut oil depends on the growth of palm trees. You can extract coconut oil either wetly or dryly.

Wet process

Philippines-style traditional extraction straight from the milk. Additionally, latik (curds), a garnish for Filipino sweets, are produced by the method.

Instead of dried copra, the all-wet process employs coconut milk that is derived from raw coconut. The coconut milk's proteins produce an emulsion of water and oil. The process of dissolving the emulsion to extract the oil presents the biggest challenge. Long-term boiling was once employed to do this, but it results in discolored oil and is not cost-effective. Centrifugal devices and pre-treatments with cold, heat, acids, salts, enzymes, electrolysis, shock waves, steam distillation, or a combination of these are used in modern procedures. Even after accounting for dry processing's losses from spoilage and pests, wet processing is less profitable than dry processing despite its

many variations and technological advancements because of a 10-15% lower yield. Wet processes typically demand a significant investment in energy and equipment, resulting in expensive capital and running expenses.

Dry process

To produce copra through dry processing, the flesh must first be removed from the shell and dried over fire, in the sun, or in kilns. The coconut oil and a high-protein, high-fiber mash are produced by pressing the copra or dissolving it in solvents. There is no method to extract protein from the mash; instead, it is fed to ruminants because it is of poor quality for human consumption. The efficiency of the oil-making process greatly depends on how the coconuts are harvested (they can be between 2 and 20 months old when plucked). Immature nut copra is harder to deal with, yields less, and provides a lower-quality product.

Hexane is a solvent used by traditional coconut oil processors to extract up to 10% more oil than is possible with just rotary mills and expellers. The oil is subsequently refined to eliminate certain free fatty acids to lessen rancidification susceptibility. Use of copra with a moisture content below 6%, maintaining the oil's moisture level below 2%, heating the oil to 130–150 °C (26–302 °F), and adding salt or citric acid are further methods to extend shelf life.

Fig 2.3 Coconut oil.

2.6.5 COCONUT SHELL

The hardest part of the coconut fruit is the coconut shell. The coconut husk and flesh are separated by the coconut shell. Typically, this shell is made to enclose the coconut's inside. Shell is a material that is used to create a variety of crafts and other products. Because of their resilience, coconut shells are used to make a lot of handcrafted decorations. The charcoal made from coconut shells can also be used as food and is significantly more durable than other charcoals.

Active carbon is frequently produced using coconut shell charcoal. Typically, activated carbon is referred to as oxygen-treated carbon dioxide. Commonly, active carbon is employed to remove contaminants. Coconut shell charcoal is frequently utilized in the active carbon-using industries of purification and other fields.

Fig 2.4 Coconut shell

2.6.6 CRAB SHELLS

A crab is a small aquatic animal with eight legs, claws, and a flat body. On the beach, you could occasionally discover little crabs in the sand.

Crabs can be found in both fresh and saltwater, and they have a thick, shell-like covering that is known as a "exoskeleton." Some varieties of crab are prized for its delicate, sweet flesh, which is frequently referred to as "crab meat." Many seashelled animals with the word "crab" in their names,

such as horseshoe crabs and hermit crabs, aren't actually crabs. Catalysts for the generation of biodiesel can be made from crab shells.

Fig 2.5 Crab

2.7 PROPERTIES OF BIODIESEL

(Chhetri et al., 2008) performed an experiment to produce biodiesel in order to conduct research on the economic viability of biodiesel in comparison to petro-based fuel. Waste cooking oil, ethyl alcohol, and sodium hydroxide were employed as the reaction's reactants. Gas chromatography was used to assess the ethyl ester's fatty acid content. According to ASTM standards, the physical and fuel characteristics of the resulting biodiesel were characterized, and some of the findings were as follows: At 40, the viscosity was 5.03 mm/sec, the flashpoint was 164 °C, the phosphorus level was 2 ppm, and the combined calcium and magnesium content was 1 ppm. There was no sediment or water present. The cetane index was 61, the cetane content was 2ppm, the total acid number was 0.29mgKOH/g, and the cloud point and pour point were each -1°C and -16°C.

2.7.1 FLASH POINT

The flash point is the lowest temperature at which a fuel may generate enough vapor to ignite, resulting in the creation of flames. The flash point of biodiesel is higher than that of regular diesel. Additionally, biodiesel has a higher flash point specification than diesel regulations. While diesel fuel's flash point ranges from 55°C to 66°C, biodiesel has an average flash point of 150°C. The elements found in each fuel account for this variation.

2.7.2 CLOUD POINT

The lowest temperature at which crystal formation in biodiesel can be seen as a cloudy suspension is known as the cloud point (CP) (Sanford et al., 2009).

It is the temperature below which the first crystals begin to form. The temperature at which the cloud originates is described. After the cloud point, the temperature is lowered in 1°C steps until it reaches the point when the fuel stops moving or loses its flow characteristics.

2.7.3 POUR POINT

The pour point is the lowest temperature at which a liquid loses its ability to flow. The temperature is reduced by 3°C increments in a freezer that can cool to below zero degrees until a fog, haze, or wax crystal forms at the test tube's bottom.

When using the fuel in cold climates, the cloud and pour point properties are important.

2.7.4 VISCOSITY

The viscosity of biodiesel is one of its most crucial characteristics since it has an impact on the fuel injection system, particularly in cold climates where viscosity rises with temperature. The viscosity of biodiesel is often higher than that of diesel, and this higher viscosity level results in slower liquid atomization and longer liquid penetration durations than in the case of diesel fuel.

2.7.5 ACID VALUE

The amount of potassium hydroxide (KOH), measured in milligrams, needed to neutralize one gram of a chemical compound is known as the acid value (also known as the neutralization number, acid number, or acidity). A chemical substance, such as a fatty acid, or a combination of chemical compounds can have one or more carboxylic acid groups, which is measured by the acid number. An organic solvent (typically isopropanol) is used to dissolve a given quantity of the sample in a typical process, and phenolphthalein is used as a color indicator to titrate the solution of alcoholic potassium hydroxide (KOH) at the known concentration. The acid number is used to measure a substance's acidity, such as biodiesel. The amount of base needed to neutralize the acidic components in 1 g of sample, measured in milligrams of potassium hydroxide. For mineral oils and biodiesel, there are standard methods for calculating the acid number, such as ASTM D 974 and DIN 51558. For biodiesel, the European Standard EN 14104 and ASTM D664 are both widely used globally. The acid number (mg KOH/g oil) for biodiesel in both EN 14214 and ASTM D6751 standard fuels should be less than 0.50 mg KOH/g.

2.7.6 ADVANTAGES AND DISADVANTAGES OF BIODIESEL

In terms of being a cleaner source of energy, biodiesel has a number of advantages over fossil fuels. Particularly, biodiesel has lower emissions of greenhouse gases such as carbon dioxide, carbon (IV) oxide, sulphur dioxide, and others, and is more biodegradable than fossil fuels. Agriculture is the primary source of the feedstock used to make biodiesel, which is a renewable fuel. In addition to being an economical fuel for farmers and other rural residents, biodiesel also offers a means of recycling agricultural waste. It also needs a simple production method.

Biodiesel is also free of sulfur and aromatic chemicals (Hassan and Ayodeji, 2019). (Sahani, Roy and Sharma, 2019, p.190).

In addition to the additional advantages of biodiesel over petroleum-based diesThere are still several production issues with diesel that, if resolved, would give biodiesel a comparative edge over non-renewable energy sources. One of these difficulties is that using edible oils as a feedstock for the production of biodiesel can endanger the availability of food and increase food scarcity. In addition, the cost of producing biodiesel is currently high, and its freezing point is a cause for concern for use in cold regions, though this can be fixed by using petro-biodiesel blends like B10, B5, etc.(Hassan and Ayodeji, 2019). (Hassan and Ayodeji, 2019). According to studies by Mahila et al. (2020, cited in Amenaghawon, Evbarunegbe, and Obahiagbon, 2021), approximately 70% of the production cost of biodiesel is attributable to the cost of the feedstock. This finding is consistent with the high cost of producing biodiesel.

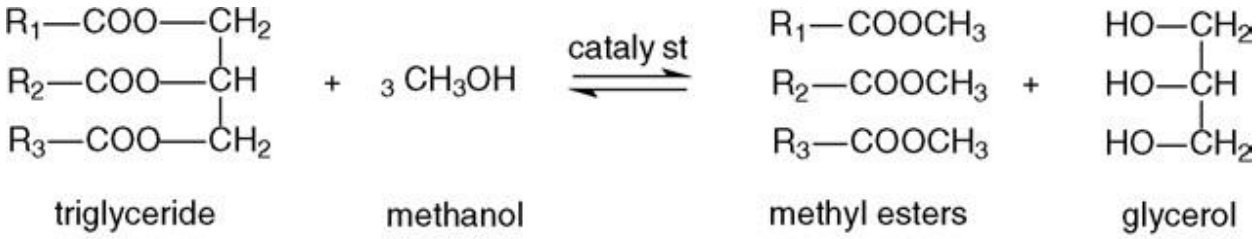
2.8.1 TRANSESTERIFICATION

Using a strong acid or base as a catalyst, transesterification of coconut oils involves the reaction of a triglyceride and an alcohol to create fatty acids alkyl esters (crude biodiesel) and glycerol. Diglycerides and monoglycerides, which are reaction intermediates, are created during the reaction from the processes involved. The type of alcohol utilized, the alcohol to oil ratio, the catalyst used, the reacting circumstances, the purity of the reactants, and the amount of the reacting oil are all variables that can affect the outcome and yield of the reaction, just like they might with any other chemical reaction. Ma, Hanna, and others (1999 as cited in Khurshid, 2014).

2.8.2 ALCOHOLYSIS

During the transesterification reaction, the alcohol used separates into an alkoxide and a hydrogen ion. These ions then interact with the molecules of the catalyst and the oil to produce the transesterification reaction's end products through a chemical process known as methanolysis of

oil. In the chemical reaction seen in figure 2.1, the dipole-dipole interaction brought about by the solid catalyst's inherent electronic configuration causes the hydroxyl bond (O-H) in the methanol to break into the methoxide ion (CH3O) and hydrogen ion (H+) at the surface of the solid catalyst.



with R1, R2, R3 = hydrocarbon chain from 15 to 21 carbon atoms

Figure 2.1 Methanolysis of Coconut oil

(Sahani, Roy and Sharma, 2019, p.190).

One aspect of the ionic interaction between the alcohol and triglyceride molecules is the availability of methoxide negatively charged ions for bonding; however, during the acid catalyzed reaction steps, the carbonyl group is protonated (an activity supported by the acid catalyst), increasing its electron affinity and making it more susceptible to a methoxide ion (CH3O) (Serio et al., 2008).The characteristics of the fatty acid alkyl ester formed are influenced by the physical and chemical characteristics of the alcohol that will be employed in the reaction. The performance (chemical activity) and cost of the alcohol are taken into consideration when choosing it for the reaction. Methanol, ethanol, and butanol are examples of short chain alcohols frequently employed in transesterification. Due to oil's solubility in ethanol, it may be desirable to use it as an extractive solvent; nevertheless, stable emulsions between the oil and ethanol form during the transesterification reaction, making it difficult to separate and purify the reaction's desired result, ethyl esters. While using methanol can result in emulsions that easily break down into methyl

ester-rich layers on top of glycerol-rich layers, this can also increase production costs. In addition, ethanol can be made from agricultural resources. Due to the addition of a second carbon molecule to its chain, ethanol is more ecologically friendly than methanol and has a higher cetane number and heat value. To favor the forward reaction, increase the product yield, and aid phase separation from the formed glycerol, the alcohol is used in excess of the stoichiometric amount of the triglycerides, which is typically used at a stoichiometric ratio of 3:1 or more practically 6:1 (depending on reaction conditions).

2.8.3 KINETICS OF TRANSESTERIFICATION

From the literature, it is clear that the kinetics of the reaction should be taken into account in order to facilitate and accelerate the biodiesel synthesis process. The various reaction stages that take place when the catalyst and alcohol combine are one of these factors. According to Dossin et al. (2006, cited in Rafaat 2011, p.205), the rate-determining step is the surface reaction step when the catalyst is MgO or La2O3, and the rate-determining step is the methanol adsorption onto the catalyst's surface when the catalyst is BaO, CaO, or SrO, which has higher basicity. According to Dhanasekaran krishnan and Dass (2012, p. 9798), there are three steps that must be completed in order to derive the kinetic model of transesterified biodiesel. These steps are the conversion of the final concentration of triglycerides to the final concentration of diglycerides and methyl esters, the conversion of the final concentration of diglycerides to the final concentration of monoglycerides and methyl esters, Any oil's rate of transesterification is influenced by its initial composition as well as operational factors including reaction temperature, catalyst concentration, and the methanol to oil molar ratio. Their study also revealed that the conversion of monoglycerides at their final concentration into methyl esters has a lower activation energy than the other conversion events.

2.8.4 EFFECT OF WATER MOLECULE IN TRANSESTERIFICATION

Depending on the quantity of water molecules available in the oil feedstock, the presence of water molecules in the transesterification reaction combining methanol and glycerides in the presence of a catalyst can be both beneficial to the reaction process and a barrier to it. When using solid metal oxides like CaO, small amounts of water help the catalytic activity of CaO because when the CaO molecule ionizes, it dissociates into O2 which are electron donors therefore exhibiting other characteristics of a Bronsted base. O2 behaves as a Lewis acid or electron acceptor and Ca+ ions which behave as a Lewis acid or electron acceptorThe O2 creates a dipole-dipole bond with the H+ ion from a water molecule when there isn't much water present in the reaction, freeing up the OH to form bonds with nearby positively charged cations. In order for the transesterification reaction with the triglyceride to proceed, the OH ion locates an alcohol molecule and exerts an electrochemical force of attraction on its hydroxyl group. This causes the methanol molecule to dissociate into a methoxide anion CH3O and hydrogen ions H+ (Refaat, 2011, p. 205).According to Chorkendorff and Niemantverdriet (2003, cited in Rafaat, 2011, p. 205), the hydrolysis of esters contained in the oil into fatty acids, which can then react with a basic catalyst present—in this case, CaO—to make soap, lowers the yield of the biodiesel produced.

2.8.5 CATALYSIS IN TRANSESTERIFICATION

The rate of most organic reactions must be sped up using a catalyst because they often occur at considerably slower rates that are unsuitable for use in industry. The reaction has been accelerated using catalysts, which has increased the process's economic viability. Serio et al. (2008) list the following as desirable characteristics for a catalyst: basic catalysis of the transesterification and esterification reactions; catalyst stability; ability to remain active in water and at low temperatures; high selectivity for the desired product; little to no susceptibility to leaching and poisoning.

According to Lotero et al., the catalyst should favor triglycerides for absorption over glycerol and water because of their polar nature and ability to render the catalyst inactive (2005 cited in Refaat, 2011, p.2).

2.8.6 CATALYSTS

Since the fundamental need for being called a catalyst is that it may interact with reactants in a reaction and still return to its original form, a catalyst can exist as a solid object, a liquid, or even in a gaseous state. The catalyst's actual physical form could affect a process's overall cost. The shape of the catalyst, which depends on the characteristics of the oil utilized, might influence the economics of the entire process in the transesterification of triglycerides into biodiesel to some amount.Solid catalysts have some advantages over liquid catalysts, according to Singh et al. (2020, referenced in Amenaghawon, Evbarunegbe, and Obahiagbon, 2021), since they are more thermally stable, reusable, and do not pose concerns with separation, purification, or environmental issues. The majority of catalysts are either acidic or basic in their chemical composition.While acidic catalysts, such as sulfuric acids, require longer reaction times (3 to 4 hours) to complete the reaction even at higher temperatures of about 100 degrees Celsius, basic catalysts, such as hydroxides like potassium and sodium hydroxides, alkoxides like methoxides, ethoxides, propoxides, and butoxides, or carbonates, require less time to complete the reaction even at room temperature. Catalysts can also be categorized according to their constituent materials, which can be either organic or inorganic. Based on the catalyst's function, a catalyst may also be referred to as acid-base, enzymes, photocatalytic, or electrocatalytic depending on how it interacts with the reactants and products in the process.Homogeneous catalysis and heterogeneous catalysis are the two categories of catalyzed reactions based on how the catalyst interacts with the chemical species present in the reaction mixture. When the catalyst, reactants, and products are all in the same phase

during a reaction, this is referred to as homogeneous catalysis. For the traditional manufacture of biodiesel, homogeneous alkaline catalysts like sodium hydroxyl (NaOH), potassium hydroxyl (KOH), sodium methoxide (NaOCH3), and potassium hydroxyl (KOCH3) are frequently utilized. Due to their efficiency, effectiveness, and affordability, these homogeneous catalyst are frequently used (Elias et al., 2020, p.2)

2.9 HOMOGENEOUS CATALYST

Homogeneous catalysts are catalysts used in homogeneous catalysis. When the catalyst, reactants, and products are all in the same phase, the reaction is said to be homogeneous. Because of the solubility of the components in the solution and other chemical characteristics of the chemical species in the mixture, compounds can chemically dissociate into ions when they are in solutionHomogeneous catalysts work as Bronsted-Lowry acids or bases in reactions by first dissociating into conjugate pairs of ions. For the synthesis of biodiesel, homogeneous alkaline catalysts are frequently utilized, including sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium methoxide (NaOCH3), and potassium methoxide (KOCH3) (Gaanty and Maniam, 2011, p.248).Despite being readily soluble in methanol, alkoxides and carbonates of sodium and potassium are less appropriate as homogenous catalysts (Elias et al., 2020, p 2). The homogeneous base catalyst's hydroxide group has the capacity to hydrolyze ester molecules and break their molecular bonds, producing methanol and a carboxylic acid molecule (free fatty acids- FFA). Another disadvantage of utilizing a homogeneous catalyst for the transesterification reaction is this side reaction. The following issues also arise while using homogeneous catalyst:Large amounts of waste water are produced when the catalyst is neutralized, and the catalyst is no longer useable or recoverable after the transesterification process. (P. 284, Gaanty and Maniam, 2011) Due to energy requirements, equipment corrosion, catalyst separation, and the need to purify Fatty

Acid Methyl Ester, homogeneous catalyzed transesterification reactions are typically expensive (Elias et al., 2020 p. 2).Due to the availability of basic and acidic sites, which are simple to separate from the product, require less water for manufacture, and solve the problem of soap formation, this has increased interest in the use of heterogeneous bi-functional catalysts.

2.9.1 HOMOGENEOUS ACID CATALYST

Acidic catalysts are preferred over basic homogeneous catalyst for the transesterification of coconut oil to biodiesel from low-quality feedstock (Elias et al., 2020 p.2). While water percentages as high as 5 wt% will fully stop an acid catalyst-catalyzed reaction, acid catalysts are sensitive to water concentrations as low as 0.1 weight percent (wt%), which can lower the yield of fatty acid methyl ester (FAME).Acid catalyzed transesterification reactions necessitate temperatures between 60 and 100 °C and reaction durations between 2 and 10 h. (Thanh et al., 2012 p.194). Homogeneous acid catalyst requires a high methanol to oil ratio of at least 12:1 in order to function (Elias et al., 2020, p.2) The acidic nature of the catalysts causes equipment used in the process to corrode as well.The FAME output, however, may be increased while the reaction time is sped up by utilizing an acid catalyst in a pretreatment phase to convert FFA into esters, followed by an alkaline catalyst for the transesterification step. Alkaline catalysts are essentially preferred for use alone if the oil feed has low FFA and water content due to greater process economics. (2012) p. 194 in Thanh et al.

2.9.2 HOMOGENEOUS BASE CATALYST

The alcohol to oil molar ratio is exceeded by alkaline alkoxides, such as sodium alkoxide, alkaline hydroxides, such as sodium hydroxide (NaOH), and potassium hydroxide (KOH), which are homogeneous base catalysts that give good yields quickly and within the temperature range of 60 to 50 °C. This is usually the case when a high-quality oil is used (FFA 1 wt% and moisture 0.5

wt%), according to the literature review. Although alkoxides are more reactive catalysts and create more methyl esters, hydroxides have the advantage of being less expensive. When utilizing alkoxide, increasing the oil's hydroxide content will increase the yield of the process.(2012), p. 198 (Thanh et al). (Thanh et al.). However, as was already noted, when a certain quantity of free fatty acid is present in the oil feedstock, the hydroxide group of the homogeneous catalyst is a disadvantage in the transesterification conversion of the current glycerides into fatty acid alkyl eaters. The presence of some water exacerbates this situation since it may interact with the esters to produce hydrolysis, which could result in the formation of more free fatty acids throughout the reaction.

2.9.3 HOMOGENEOUS SOLID CATALYST

was used in earlier studies on the generation of biodiesel and even in some recent studies. Because they allow for a simpler and more efficient purification process/step, heterogeneous solid-catalysts might be viewed as an improvement over homogeneous base or acid catalysts. Heterogeneous solid catalysts can be used again and again.Alkali or alkaline oxides supported on materials with a wide surface area make up the majority of heterogeneous solid catalysts. Metal oxides, zeolites, hydrotalcites, Y-alumina, and other heterogeneous solid catalysts are a few examples (Thanh et al., 2012, p.199). Basically, the solid heterogeneous catalyst offers enough surface area for the alcohol to bind to it and speeds up the reaction by allowing methanol and the outer molecules on the solid catalysts' surfaces to interact.Despite these advantages that using a heterogeneous catalyst over a homogeneous catalyst has over the former, heterogeneous catalyst has some disadvantages. One issue with employing heterogeneous catalyst is that it deactivates with time, most likely as a result of poisoning, according to (Sivasamy et al., 2009).coking, sintering or leaching. Poisoning has been noticed to occur more when using used oils (Lam et al., 2010).

2.9.4 COMPARISON BETWEEN HOMOGENEOUS AND HETEROGENEOUS CATALYSTS

Homogeneous base catalyst has the following characteristics

- Do not form water during transesterification reaction.
- About 4000 times faster reaction rate than acid catalyzed transesterification.
- Two -step alkaline-catalyzed transesterification from used coconut oil is an economic method for biodiesel production.
- Reaction can occur at mild reaction condition and thus less energy required.
- NaOH and KOH are economically feasible and widely available.

Heterogeneous base catalyst has the following characteristics.

- Reusable. And easy to separate from product.
- Relatively faster reaction rate than acid catalyzed transesterification.
- Reaction can occur at mild reaction condition and relatively lower energy.
- Long catalyst life time.

Homogeneous acid catalyst has the following characteristics

- Insensitive to FFA and water content in the oil.
- Preferred-method if low-grade oil is used.
- Esterification and transesterification occur simultaneously.
- Saponification can be avoided.
- Produce high yield of biodiesel.

Heterogeneous acid catalyst has the following characteristics

- Insensitive to FFA and water content in the oil.
- Preferred-method if low-grade oil is used.
- Esterification and transesterification occur simultaneously
- Easy separation of catalyst from product.
- High possibility to reuse and regenerate the catalyst.
- Recyclable.

2.10 COMPOSITE CATALYSTS FROM WASTE MATERIALS

Researchers are seeking for readily available and reasonably priced feedstock for biodiesel production as a result of the search for a more cost and efficient biodiesel manufacturing technique (Mahmood Khan et al., cited in Amenaghawon, Evbarunegbe and Obahiagbon, 2021). As a result, effective catalyst feedstock made from waste materials that contain high concentrations of metal oxides and other chemically active compounds is used in the production of biodiesel, either as catalyst support or as active sites when more expensive catalyst materials are dispersed on more affordable solid support.Using two different catalyst sources—in this example, crab and cockle shells—separately in the transesterification reaction and combining the sources had no discernible impact on the methyl esters generated, according to the experiment carried out by Gaanty and Maniam (2011). raising the prospect of combining two or more waste CaO sources as a catalyst for the conversion of coconut oils into biodiesel. Gaanty and Maniam (2011) transesterified waste chicken fat to create methyl esters using activated waste crab and Cockle shells as a catalyst. When the shells were heated to high temperatures in order to activate them, X-ray diffraction (XRD) and

Electron Dispersive X-ray (EDX) were used to watch how both shells changed into CaO. Additionally, a Central Composite Design (CCD) was employed to examine the effects of the catalyst loading, the mass ratio of methanol to oil, and the reaction time on the reaction. When used separately or together in different ratios, both shells had identical effects.The best reaction conditions were 4.9 weight percent catalyst and a methanol to oil mass ratio of 0.55: 1, which produced a 98% conversion of methyl esters in 3 hours.

2.11 CATALYST SELECTION

A good catalyst should, in general, not be deactivated by water, be stable, not deactivate at low temperatures, not leach while in use, and have excellent selectivity (Serio et al., 2008). When choosing a base catalyst for a particular reaction, the catalyst's catalytic activity and acid-basic strength have an impact on how the catalyst interacts with the oil and alcohol that will be used in the process.As water can bind to acid species (H+) more effectively than alcohols, it is crucial that the catalyst surface be hydrophobic in order to prevent the hydrophobic triglycerides from coming into contact with the catalyst. Since the transesterification reaction is more severe due to the larger hydrophobic triglyceride molecules, the catalyst should instead have preferential adsorption for the triglycerides (Lotero et al., 2005). When it comes to metal oxides, they exhibit other traits of a Bronsted base (O2 electron donor) due to the presence of negative oxygen ions (anions), which are electron donors, and positive metal ions (cations), which act as Lewis acids (M+ electron acceptors) when they are ionized (Refaat, 2011, p. 205). In addition to the catalyst's catalytic activity, suitability for a particular reaction, and physical parameters like surface area, pore size, pore volume, etc., other considerations for choosing a catalyst include its cost and availability. (2010) (Ejikeme et al.) stated that the distribution of pores across the inner and outer surfaces is a significant catalyst feature. CaO is more frequently used in the production of biodiesel because it

is inexpensive, less toxic, and easily accessible in natural formations. It is also an active basic catalyst. Lee and co. (2009, cited in Refaat 2011, p.205). Due to its limited solubility in methanol, it also possesses a high basic strength and is more environmentally friendly Zabeti et al (2009, cited in Refaat 2011, p. 205).

2.11.1 CATALYST CHARACTERIZATION

Analyses are conducted to determine whether the catalyst utilized or intended for use in the biodiesel manufacturing process possesses the desired qualities (physical and chemical) for the reaction. In techniques like X-ray fluorescence analysis (XRF), scanning electron microscopy-electron dispersive X-ray (SEM-EDX), gas chromatography (GC), flame ionization detection (FID), transmission electron microscopy (TEM), X-ray diffraction (XRD), etc., such analyses are typically based on fundamental principles of physics and chemistry such as photo-emission of particles, their magnetism, and electric properties of atom Catalyst Characterization can be done to determine a substance's qualities before it is used as a catalyst in a production process.process. These characteristics include the material's thermal stability, the chemical makeup of the bulk and surface solids, the bulk solid's structure, the size of the crystallites and the makeup of the phases, as well as the pore size, volume, surface area, and porosity. Based on the catalyst's property that is being studied, the characterisation analysis is generally categorized.

2.11.2 CHARACTERIZATION TECHNIQUES

I. The qualities and traits of a catalyst can be discovered in a variety of ways. These techniques can be divided into different categories depending on the type of property being studied, including surface morphology, surface chemical properties (such as the location and oxidation state of active ingredients, acid-base properties, redox properties, etc.), aggregate properties (such as aggregate or particle size, density, mechanical strength,

attrition resistance, etc.), and catalytic properties (activity, selectivity, stability etc.). These are a few of these methods:

1 Structural Analysis

2 Microscopic technique

3 Spectroscopic technique

4 Thermal stability analysis

2.11.3 STRUCTURAL ANALYSIS

The structure of the catalyst is analyzed using structural analysis techniques, such as pore analysis, pore size, porosity, and surface area. The BET (Brumaire, Emmet, and Teller) method of analysis, which gauges particle surface area by examining multilayer physisorption isotherms of inert gases like nitrogen N2, is an illustration of these techniques.

Another illustration is the X-Ray Diffraction, which uses a lattice structural parameter to identify the crystalline phases in a catalyst. Even during a reaction, his approach can give information on the sizes of the particles, but it is unable to detect very small or amorphous particles.The Extended X-Ray Absorption Fine Structure (EXAFS), which provides information on the size, number, distance, and type of neighboring atoms to a marked atomic specie, is a similar technique to the X-ray Photoelectron Spectroscopy (XPS), which is used to determine the external composition and oxidation states of elements.

2.11.4 MICROSCOPIC TECHNIQUE

involves using electron microscopy to examine the size, shape, and surface structure of supported particles and catalysts. Examples that are frequently used are transmission electron microscopy and scanning electron microscopy (TEM).

2.11.5 SPECTROSCOPIC TECHNIQUE

Such analytic techniques include infrared spectroscopy used in identifying absorbed useful species on a catalyst's surface; Fourier Transform Infrared Spectroscopy is one example of this (FTIR). Raman Spectroscopy is another one of these methods.for studying of oxidation states and the interaction of metal oxides.

2.11.6 THERMAL ANALYSIS

It’s used to study the functional stability of catalyst as a function of temperature such that it can relate information about the rate of thermal desorption of absorbed particles, loss of weight under controlled gaseous atmosphere and acid-base properties of catalyst.

2.12 CATALYST STRUCTURE AND PREPARATION

The active sites, catalyst support, catalyst promoter, and catalyst support or carrier make up the majority of a catalyst's three component sections in terms of its physical composition. Due to the fact that it contains the primary functional group needed for the reaction, the active phase is typically regarded as the catalyst's primary component. The majority of active sites for catalysts are accompanied by metal oxides of common elements that are abundant in nature. However, their primary function is to provide enough surface area and rigid support for the dispersion of functional groups as active sites on them. Research has also advanced to the utilization of functionalized nano-catalysts, such as the creation of nanoparticles supported by Fe3O4 (iron oxide) (Sharma et al., 2016). The catalyst can be made in a variety of ways that may favor a specific catalyst design, including the impregnation approach, bulk catalyst preparation, and precipitation method. Etc.

2.13 RSM AND ANFIS

The yield of biodiesel synthesis is examined using RSM (Response Surface Methodology) and ANFIS (Adaptive neural fuzzy detection system) softwares, both of which operate by adjusting various parameters like temperature, time, and concentration until an ideal state is reached. The best situation discovered using the response surface methodology is contrasted with the ANFIS situation. A model that has a higher yield is significantly more suitable for use on a wide scale.

CHAPTER THREE

MATERIALS AND METHODS

3.1 MATERIALS

3.1.1 REAGENTS AND RAW MATERIALS

The reagents and raw materials used for this study are shown in Table 3.1 below:

Table 3.1: Reagents and Raw Materials

S/N	Reagents	Sources	Uses
1.	Distilled water	Obtained from a local vendor	Used for analytical medium for both qualitative and quantitative analysis at neutral pH
2	Coconut oil	Obtained from a local vendor	Used as a feedstock for the biodiesel production
3.	Methanol	BDH UK	Used to dissolve the catalyst in the biodiesel production
4.	Sulphuric acid (H_2SO_4)	Obtained from Pyrex Laboratory in Benin City Edo State	For the sulphonation of the acid precursor
5.	Acetic acid		For Peroxide value test
6.	Chlorofoam ($CHCL_3$)		For Iodine and Peroxide value test

7.	Phonolphthalein indicator		For titration
8.	Starch indicator		For iodine value test
5.	Lithium carbonate		Basic (Li_2CO_3) salt used as a doping medium
6.	Crab shells	Obtained from local market	Raw material for the catalyst synthesis
7.	Coconut shell	Obtained from a local vendor	Raw material for the catalyst synthesis
8.	Benzene	BDH UK	Used to calculate the acid value and FFA of both oil and biodiesel
9.	Ethanol	BDH UK	Used to calculate the acid value and FFA of both oil and biodiesel

3.1.2 EQUIPMENT

Table 3.2 below shows the various equipment used in this study.

Table 3.2: Equipment Used

S/N	Equipment	Model	Uses
1.	Magnetic Stirrer	B.Bran HJ-3D	Used for liquid mixing

2.	Conical flask	Jinotech (250ml)	Used for storing and mixing chemicals
3.	Separating funnel	Jinotech (250ml)	Used for separating immiscible liquids like biodiesel, glycerol and methanol
4.	Droppers	3.0ml rubber droppers	Used to transfer small quantities of liquids
5.	Measuring Cylinder	Jinotech (100ml)	Used to measure accurately the volume of liquids
6.	Dessicator	Obtained from chemical engineering laboratory	Used to cool the catalyst to prevent it from reacting with moisture
7.	Beakers	Jinotech (250ml)	Used for mixing, measuring and heating chemicals
8.	Muffle furnace	Chemical engineering laboratory	Used to calcine the crab shells and plantain peels
9.	Oven	Chemical engineering laboratory	Used for sample drying
10.	Laboratory weighing scale	Chemical engineering laboratory	Used to measure precisely the mass of samples in grams
11.	Sieve	0.3mm mesh size (from Civil engineering laboratory, UNIBEN)	Used for particle sizing

12.	Volumetric flask	Brano (250ml)	Used to prepare and measure chemical solutions
13.	Funnel	Chemical engineering laboratory, UNIBEN	For guiding fluids or powder into a small opening
14.	Filter paper	Whatman No. 1	To separate fine solid substances from liquids
15.	Nose masks	Obtained from a local vendor	For protection against dusts, splashes or sprays of fluids and harmful odors during work
16.	Laboratory coat	Obtained from a local vendor	For protection against dusts, splashes, stains or sprays of fluids during work.
16.	Hand gloves	Obtained from a local vendor	For protection against hazardous chemicals and cuts
17.	SEM		To determine the surface morphology of the sample
18.	BET		To determine the surface area of the sample
19.	XRD		

20.	FTIR		To determine the catalytic activity of the conversion of the oil to fatty acid and methyl ester
21.	TGA		

3.2 METHODS

3.2.1 CATALYST PREPARATION

3.2.1.1 Preparation of precursor from crab shell

Warm distilled water was used to wash the crab shell in cycles, removing any leftover food particles or other contaminants. Then, a hammer was used to smash the crab shell into manageable chunks. Finally, the crab shell was given a last rinse in hot distilled water to get rid of any remaining oil.

After three days in the sun, the crab shell was baked at 110 degrees Celsius for two hours to finish the drying process. The powdered crab shell was then sieved through a 0.3mm screen, and the unsieved particles were thrown away. This process was accomplished using an engine grinder.

Purified CaO was extracted by calcining the finely sieved crab shell in a muffle furnace at 900°C for 4 hours. Crab shell was calcined, then promptly transported to a desiccator to stop any further reactions with air. Once it cooled, it was stored in an airtight container until it could be used or analyzed.

3.2.1.2 Preparation of precursor from coconut oil

The coconut shell was broken apart and cleaned in distilled water to get rid of any lingering debris or mud. In order to eliminate any remaining moisture, coconut shells were sun-dried for 14 days after being washed thoroughly. In order to remove any remaining moisture, the dried coconut shell was crushed and sieved through a 0.3mm screen.

Carbonizing the finely sieved coconut shell at 500°C for 3 hours in a muffle furnace turned it into ash. The carbonized coconut shell was then taken out and placed in a desiccator to stop any further reactions with the air. For later usage and examination, the material was stored in a sealed container.

3.2.1.3 Synthesis of the heterogeneous composite catalyst

Coconut shell and crab shell were prepared using the incipient wetness impregnation technique described by (Lani et al., 2016), Yusuf (2018), and (Yusuff et al., 2019), which was then used to the production of the biobased composite catalyst. The crab shell and coconut shell sample acquired during precursor preparation was basically blended to produce a slurry solution and agitated for approximately 30 minutes until full mixing had occurred. After forming a slurry, the solution was heated to 80 °C on a magnetic stirrer and swirled until all of the water had evaporated. It then took three hours in a 110°C oven to dry the resulting solid. After that, the freshly manufactured crab shell and coconut shell catalyst was calcined at 800°C for 3 hours to reactivate it.

3.2.1.4 Catalyst characterization of coconut shell and crab shell

A number of analytical methods were used to characterize and analyze the prepared catalyst to ensure that it met all of the required specifications. These methods included Fourier transform infrared spectroscopy (FTIR) for analyzing the functional group in the sample, scanning electron

microscopy (SEM) for analyzing the surface morphology, X-ray diffusion (XRD) for analyzing the crystalline compounds in the catalyst and precursors, and X-ray fluorescence (XRF) for determining the oxide composition.

3.3 CHARACTERIZATION OF THE COCUNUT OIL

In this experiment, coconut oil was purchased from a local store and refined specifically for use in biodiesel production. To establish whether the oil could be used to make biodiesel, the following tests were run on it:

3.3.1 DENSITY

An electronic weighing scale was used to find out how much a density bottle without its contents weighed. The density container was then filled to capacity with the sample, and their combined weight was calculated using Eqn. 3.3. (Najem and Khurshid, 2014)

$$\text{Density } (\rho) = \frac{W2-W1}{} \qquad \textit{Eqn.}\ 3.3$$

Where; W_1 = weight of empty density bottle,

W_2 = weight of sample + density bottle,

V = volume of sample.

3.3.2 ACID VALUE

The amount of potassium hydroxide, in milligrams, needed to neutralize the acid in 1 milligram of oil or fat. Triglyceride degradation in oil samples may be measured using this method. Triglyceride degradation can be caused by lipase activity or other processes. Titration, a technique previously reported by Yusuff et al., was used to calculate the WFO's acid value.(Yusuff et al., 2018)

A 250-milliliter conical flask was used to measure out 1g of WCO. The sample was prepared by adding 10 milliliters of benzene, 10 milliliters of ethanol, and 2 drops of phenolphthalein indicator. Next, it was titrated against 0.1M KOH while being vigorously shaken until a pink color was achieved that lasted for a few seconds. To calculate the acidity, we used Eqn. 3.4.

Acid value = $\frac{V \times 56.1}{}$ *Eqn.* 3.4

Where, W = the weight of oil,

V = volume of KOH required for titration.

3.3.3 FREE FATTY ACID (FFA)

The FFA value was determined using Eqn. 3.5;

FFA = $\frac{}{2}$ *Eqn.* 3.5

3.3.4 SAPONIFICATION VALUE

This is the average molecular weight of all fatty acids in a given sample, expressed as the number of milligrams of KOH required to saponify 1 milligram of fat. An alcoholic solution of KOH was made to determine the saponification value of 1 gram of the required seed oil sample. A solution of 5.51 milliliters of KOH was diluted with 200 milliliters of ethanol to create alcoholic KOH. After adding 50 ml of the alcoholic KOH to the weighed quantity of oil in a round bottom flask, refluxing it with a condenser and a heating mantle for an hour, cooling it, and then titrating it with a 0.5 M solution of HCL using phenolphthalein indicator, the desired concentration was reached.

$$= \frac{(\mathrm{B} - \mathrm{S}) * M * 56.1}{}$$

Where B represents the titre value for the blank solution

S represents the titre value for the oil solution

3.4 MODELLING AND OPTIMIZATION OF TRANSESTERIFICATION PROCESS

Design Expert 13 software modeled and optimized biodiesel production using Box Behnken Design (BBD) response surface approach. Four variables—reaction temperature (X1), catalyst loading (X2), reaction duration (X3), and methanol-oil ratio (X4)—affected biodiesel production. Table below shows Box-Behnken variable coded and real levels. Response surface regression using the second-order polynomial in Eqn. 3.2 analyzed the experimental data. (Amenaghawon, Evbarunegbe and Obahiagbon, 2021)

$$\mathbf{Y} = \boldsymbol{C_o} + \sum^{k}_{=} \boldsymbol{C_i X_i} + \sum^{k}_{=} \boldsymbol{C_{ii} X^2_i} + \sum^{k}_{<} \quad + \quad \textit{Eqn.}\ 3.2$$

Y represents the response variable (biodiesel yield), Co represents the intercept, Ci I = 1, 2...k) represents the coefficient of the first order, Cii represents the quadratic impact coefficient, and Cij represents the interaction effect coefficient. The experimental error term e is multiplied by the product of the independent response variables Xi and Xj that impact the biodiesel production.

Analysis of variance (ANOVA) and coefficient of determination were used to assess the goodness of model fit (R2). Using the quadratic equation found using the second order polynomial, response surface plots were made. This was accomplished by varying the values of two of the variables while keeping the values of the other two variables constant. Variables' optimal process values were calculated using numerical solutions. In addition, the software's numerical answers were examined to determine under what circumstances the highest possible biodiesel output might be achieved.

Table 3.1: Box Behnken Design for Transesterification Process

Variables (Coded factors)	**Levels**		
	-1	0	+1
Reaction temperature (X_1)	40	50	60
Catalyst loading (X_2)	0.5	1.0	1.5
Reaction time (X_3)	60	75	90
Methanol-oil ratio (X_4)	6	12	18

CHAPTER 4

RESULT AND DISCUSSION

4.1 PHYSIOCHEMICAL PROPERTIES OF COCONUT OIL

According to ASTM standards, the physiochemical characteristics of coconut oil were examined as in the prior chapter.

Table 4.1 Physiochemical Properties of COCONUT OIL (CO)

Properties	Values
Acid Value (mg KOH/g)	5.8
FFA (%)	2.9
Saponification Value	190
Molecular Weight (g)/mol	913.681
Peroxide Value	10
Viscosity at 31^{o}C (MPa.S)	26.53
Density (Kg/m^{3})	925
Moisture Content (%)	0.06

4.2 CHARACTERISATION OF PROCESSED CATALYST

The physicochemical properties of the synthesized catalyst and calcined crab shells were investigated. These samples' surface morphology were investigated using SEM, and their pores characteristics were examined using the Brunauer-Emmett-Teller (BET) and Barret-Joyner-

Halenda (BHJ) methods. Using an X-ray diffractometer, the crystalline phases discovered in these samples were determined. And lastly, the functional groups included in the treated catalyst were analysed using an FTIR spectrophotometer.

4.2.1. SURFACE MORPHOLOGY OF THE PROCESSED CATALYST

At different magnifications, the bi-functional catalyst (made from coconut and crab shells) and calcined crab shell surface shape were studied using scanning electron microscopy (SEM) images. The calcined crab shell contains some microscopic particles in mass form and others in agglomerated form, as seen in Fig. 4.1a. Figure 4.1a and Figure 4.1b both depict the presence of pores, which appear to be more common in the smaller aggregates than the bulk aggregates. Figure 4.1c depicts an uneven pore shape.

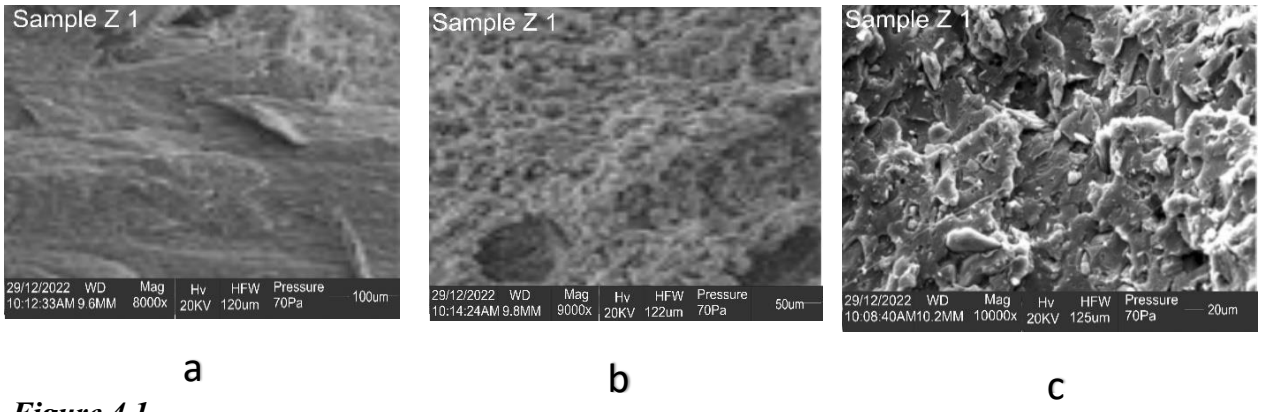


Figure 4.1

The bi-functional catalyst is depicted in Fig. 4.2 as having fine pore spaces and an irregularly distributed internal network of porous spaces that houses its active site. The bi-functional catalyst's ability to aggregate was constrained by the uneven distribution of porous spaces.

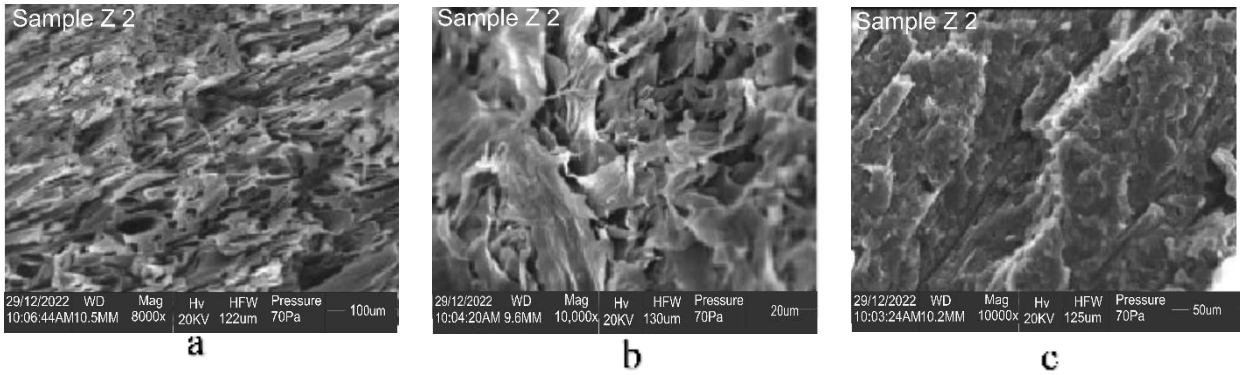


Figure 4.2

4.2.2 SURFACE AREA, PORE VOLUME AND PORE DIAMETER OF CATALYST

A catalyst's activity is directly impacted by its surface area. The concept of active sites, which refers to a single surface atom or group of such atoms with distinctive structure and properties where the catalysed transformation occurs, is central to discussions of the catalytic reaction processes. The synthesized catalyst's BET surface area was determined to be 268.330 m^2/g, while that of the calcined crab shell was found to be 265.720 m^2/g, this simply implies that the synthesized catalyst has a larger surface area than the calcined crab shell, Because the bio-based catalyst has a larger surface area, it likely has good catalytic activity. The summary of the BET analysis is shown below

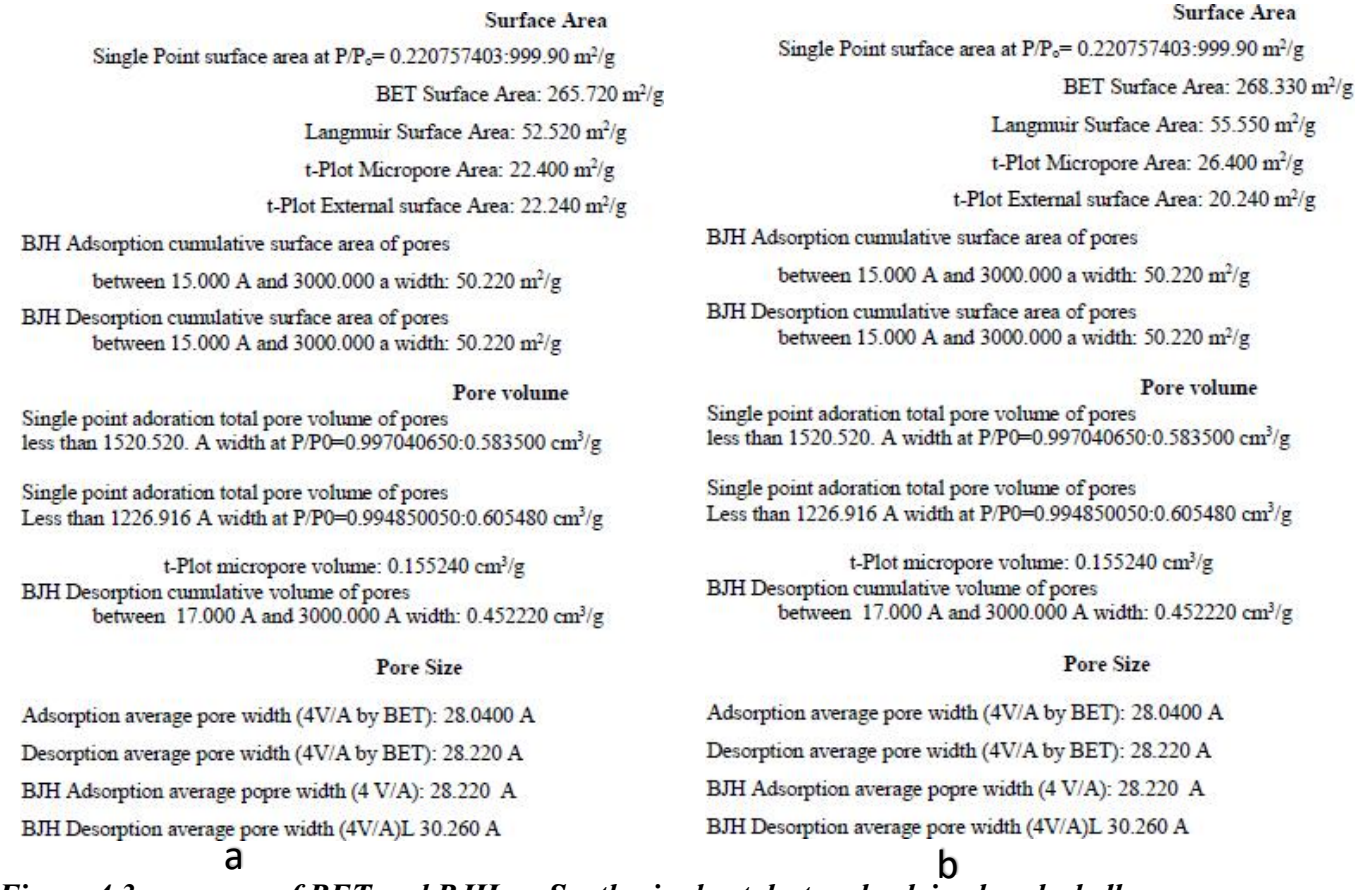


Figure 4.3 summary of BET and BJH on Synthesized catalyst and calcined crab shell

4.2.3 XRD ANALYSIS

The plots of the XRD analysis for the produced catalyst and calcine crab shell are shown in Figure 4.4. They also make the materials' crystalline structure visible. Figure 4.4a's plot, which depicts the crystals of crab shells, shows four different peaks for the minerals portlandite (at 2 Θ = 14.08°, 21°, 38°, and 42°), calcite (at 2Θ = 26°, 31°, 35°, 39.75°, and 43°), hydroxyapatite (at 2Θ = 16.78°, 23.24°, 23.87°, 28.45°, 34°, and 37.16° While figure 4.4b shows the plot for the synthesized catalyst crystals, there are four distinct peaks that correspond to portlandite, calcite, hydroxyapatite, and CaSIO. The portlandite peak is located at 2Θ = 12°, 21°, and 39°, the calcite peak is located at 2Θ = 35°, and the hydroxyapatite peak is located at 2Θ = 15°, 21.23°, 22°, 13°, 27°, 34°, and 37.16°.

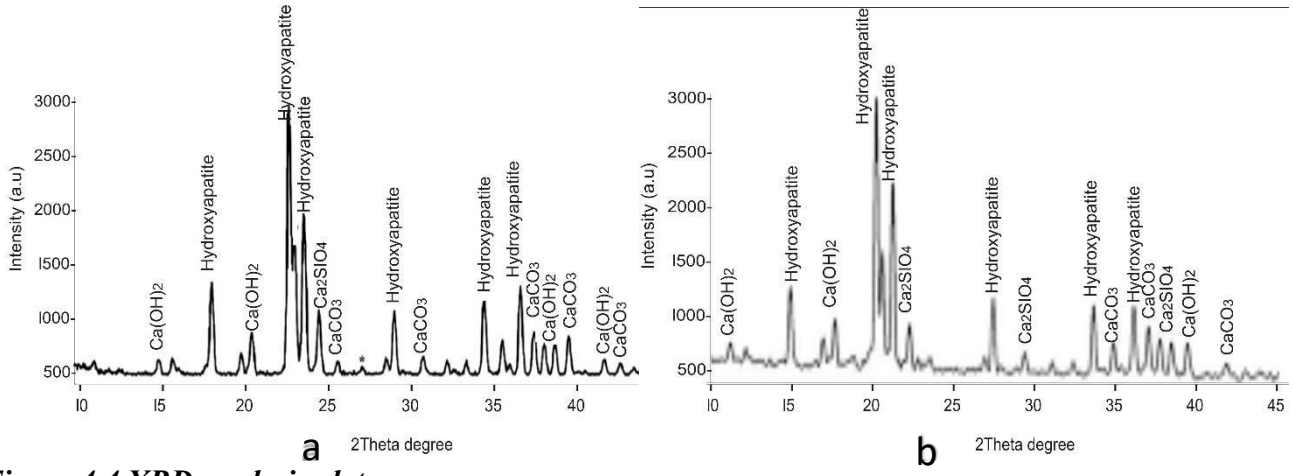


Figure 4.4 XRD analysis plot

4.2.4 FTIR

Figure 4.5 displays the Fourier transform infrared spectra used to determine the species' functional groups on the catalyst's surface. The CaO vibration caused the peaks (373.31-919 cm^{-1}) to appear in the catalyst that had not yet been employed. Other peaks (between 2966 and 4037.8 cm^{-1}) were

consistent with O-H stretching. The large signal at 2357.63 cm^{-1} may be caused by the carbonate C - O bonds, while the peak at 1686.39 cm^{-1} is attributable to the carbonyl functional group (C=O).

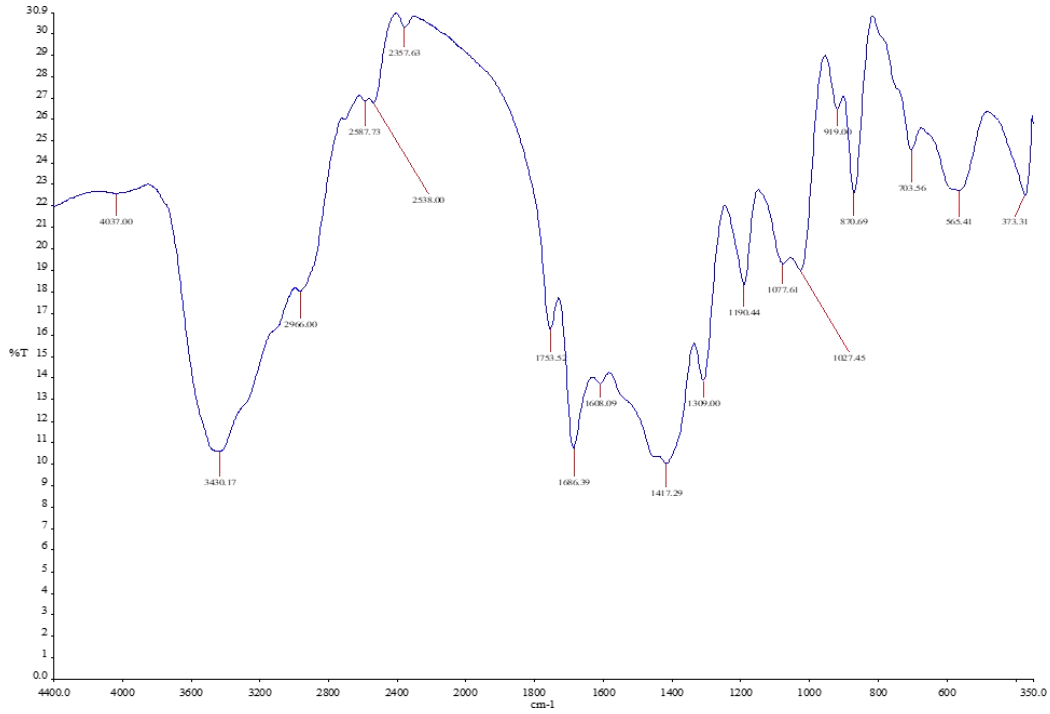


Figure 4.5 FTIR Spectrum

4.3 MODELING OF SIMULTANEOUS ESTERIFICATION AND TRANS-ESTERIFICATION OF COCONUT OIL (CO)

4.3.1. STATISTICAL ANALYSIS OF VARIANCE (RSM)

Response Surface Methodology (RSM) was used for the statistical analysis of the simultaneous esterification and trans-esterification of Coconut Oil (CO) utilising a bi-functional catalyst developed from Crab shells and Coconut shells. Using Design Expert 13.0, an experimental design utilising Box-Behnken Design (BBD) was carried out. An RSM model was created first in order to optimise the procedure. This was accomplished by using multiple regression analysis to fit the experimental data in Table 7 to the quadratic regression model (Equation (4.1)).

$$Y = \sum b_i X_i + \sum b_{ij} X_i X_j + \sum b_{ii} X_i^2 + e_i \quad \text{(Amenaghawon et al., 2021)(4.1).}$$

where X_i and X_j stand for the independent variables and Y is the dependent variable (biodiesel yield). B_i and B_{ij} are the single and interaction effect coefficients, respectively, and e_i is the experimental error term. B_o is the offset term. The set values and responses obtained are shown in Table 4.3.

Table 4.3. Experimental design developed by Box-Behnken Design (BBD)

Run No.	ACTUAL AND CODED VALUE (IN BRACKET) OF FACTORS				ACTUAL COME yield (wt.%)	PREDICTED COME yield (wt.%) (RSM)
	MeOH: Oil (mol/mol)	Temperature (^{0}C)	Cat. Loading (wt.%)	Time (min)		
1	6 (-1)	50 (0)	1 (0)	60 (-1)	75.73	74.73
2	18 (1)	50 (0)	0.5 (-1)	75 (0)	76.63	77.39
3	12 (0)	50 (0)	1 (0)	75 (0)	79.36	78.52
4	6 (-1)	50 (0)	1 (0)	90 (1)	78.36	77.21
5	18 (1)	40 (-1)	1 (0)	60 (-1)	80.37	81.11
6	6 (-1)	50 (0)	1 (0)	75 (0)	60.83	60.80
7	12 (0)	60 (1)	0.5 (-1)	60 (-1)	73.47	75.00
8	12 (0)	50 (0)	1 (0)	60 (-1)	84.2	82.61
9	18 (1)	40 (-1)	1.5 (1)	75 (0)	82.46	82.22
10	12 (0)	60 (1)	1 (0)	90 (1)	59.645	61.61
11	6 (-1)	50 (0)	1 (0)	75 (0)	81	82.94
12	12 (0)	50 (0)	1 (0)	75 (0)	80.46	78.52
13	12 (0)	50 (0)	1 (0)	75 (0)	77.6	78.52
14	12 (0)	50 (0)	1.5 (1)	60 (-1)	79.4	80.58
15	6 (-1)	40 (-1)	0.5 (-1)	75 (0)	74.6	75.22

16	12 (0)	60 (1)	0.5 (-1)	75 (0)	61	60.75
17	12 (0)	40 (-1)	1.5 (1)	75 (0)	85	84.85
18	12 (0)	40 (-1)	1.5 (1)	75 (0)	64.54	65.63
19	18 (1)	50 (0)	1 (0)	75 (0)	67.76	65.85
20	12 (0)	50 (0)	1 (0)	75 (0)	77.6	78.52
21	12 (0)	50 (0)	1.5 (1)	90 (1)	79.6	78.10
22	12 (0)	50 (0)	0.5 (-1)	90 (1)	78.6	77.45
23	6 (-1)	50 (0)	1.5 (1)	75 (0)	77	76.62
24	12 (0)	60 (1)	1 (0)	75 (0)	77.6	78.52
25	12 (0)	60 (1)	1 (0)	90 (1)	84.6	85.84
26	18 (1)	50 (0)	1 (0)	75 (0)	85.6	85.66
27	18 (1)	40 (-1)	1 (0)	90 (1)	78	78.60
28	12 (0)	60 (1)	1 (0)	60 (-1)	65.736	64.87
29	12 (0)	50 (0)	0.5 (-1)	75 (0)	85	83.50

After estimating the coefficients of the model terms (in terms of coded factors), the resulting equation is displayed in Equation (4.2). For comparison, the results of the predicted biodiesel yield using Equation (4.2) are also displayed in Table 4.3. The model's suitability is demonstrated by how well the experiment's findings match those expected. The predicted yield fell between 60.75 and 85.86%.

Y (COME Yield) =

$$78.524 + 1.94167A + 10.4907B + 1.55833C - 0.00841667D - 0.5825AB + 0.8575AC - 1.25AD - 0.885BC + 1.62275BD - 1.2325CD - 0.266625A^2 - 4.44525B^2 - 0.396625C^2 - 0.34525D^2 \quad (4.2)$$

Where: Y = COME Yield

A = MeOH: Oil (mol/mol)

B = Temperature (^{0}C)

C = Cat. Loading (wt.%)

D = Time (min)

From equation 4.2, it can be seen that A, B, C, AC and BD contributed positively in increasing the COME Yield, because they are positive, unlike D, AD, BC, CD, A^2, B^2, C^2 and D^2 that are negative.

One may forecast the reaction for certain levels of each element using Equation 4.2. By default, the factors' high levels are coded as +1 and their low levels as -1. By contrasting the factor coefficients, the coded equation can be used to determine the relative importance of the elements.

Equation 4.3 displays the COME to the process parameters (final equation) in terms of actual factors.

Y (COME Yield) =

$$-98.8044 + 1.74261A + 4.97645B + 24.0347C + 0.0196889D - 0.00970833AB + 0.285833AC - 0.0138889AD - 0.177BC + 0.0108183BD - 0.164333CD - 0.00740625A^2 - 0.0444525B^2 + -1.5865C^2 + -0.00153444D^2 \quad (4.3)$$

Where: Y = COME Yield

A = MeOH: Oil (mol/mol)

B = Temperature (^{0}C)

C = Cat. Loading (wt.%)

D = Time (min)

It is possible to anticipate the reaction for specific levels of each factor using the equation expressed in terms of the real factors. Here, the levels for each factor should be stated in their original units. Because the coefficients are scaled to account for the units of each element and the intercept is not at the centre of the design space, this equation should not be used to estimate the relative importance of each factor.

4.3.2.1 ANALYSIS OF VARIANCE (ANOVA) FOR RSM

Following the simultaneous esterification and transesterification of the Coconut Oil (CO), the suggested quadratic model has the highest R^2 value of 0.976. The results for the analysis of variance (ANOVA) are shown in Table 4.4 for COME yield.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1557.49	14	111.25	40.60	< 0.0001	significant
A-Methanol: oil ratio	45.24	1	45.24	16.51	0.0012	
B-Temperature	1320.67	1	1320.67	481.95	< 0.0001	
C-Catalyst loading	29.14	1	29.14	10.63	0.0057	
D-Time	0.0009	1	0.0009	0.0003	0.9862	
AB	1.36	1	1.36	0.4953	0.4931	
AC	2.94	1	2.94	1.07	0.3178	
AD	6.25	1	6.25	2.28	0.1532	
BC	3.13	1	3.13	1.14	0.3030	
BD	10.53	1	10.53	3.84	0.0701	
CD	6.08	1	6.08	2.22	0.1586	
A^2	0.4611	1	0.4611	0.1683	0.6879	
B^2	128.17	1	128.17	46.77	< 0.0001	
C^2	1.02	1	1.02	0.3724	0.5515	
D^2	0.7732	1	0.7732	0.2822	0.6036	
Residual	38.36	14	2.74			

Lack of Fit	31.36	10	3.14	1.79	0.3021	not significant
Pure Error	7.01	4	1.75			
Cor Total	1595.85	28				

Table 4.4(results for the analysis of variance (ANOVA) for COME yield

To describe the effect of any factor on the answer, one can use the sum of squares and the corresponding F-value. Indicators of how essential a component is to the process response include a bigger sum of squares and F-values. Additionally, the probability value was used to assess each component's relevance to the outcome (P-value). The model is apparently noteworthy because of its Model F-value of 40.60. An F-value this large could only happen owing to noise with a 0.01% chance. Model terms are considered significant when the P-value is less than 0.0500. A, B, C, and B^2 are important model terms in this instance. Model terms are not significant if the value is higher than 0.1000. Model reduction may enhance your model if it has a large number of unnecessary terms (excluding those necessary to maintain hierarchy).

The sum of squares for COME Yield in this study's ANOVA model was determined to be 1557.49, with an F-value of 40.60 and a P-value of 0.0001 (< 0.0001). According to the analysis of variance performed on the COME yield result, the temperature, methanol to oil ratio and the second order effect of temperature had the most significant impact on the COME yield, with the temperature having the greatest impact with a sum of squares value of 1320.67, F-value of 481.95, and P-value of 0.0001. The F-value for the lack of fit, which is 1.79, indicates that the lack of fit is not

significant in comparison to pure error. A significant Lack of Fit F-value has a 30.21% likelihood of being caused by noise. Since we want the model to fit, a non-significant lack of fit is desirable.

2.7.1.1 4.3.2. FIT STATISTICS

The term "goodness of fit" refers to how well a statistical model predicts a set of observations. In order to summarise the difference between actual values and those predicted by the in-question model, goodness of fit measures is typically used.

Table 4.5 Fit Statistics of this study

Parameters	COME yield Value
R^2	0.9760
Adjusted R^2	0.9519
Predicted R^2	0.8800
Adeq. Precision	21.0750
Std. Dev.	1.66
Mean	76.27
C.V. %	2.17
PRESS	191.56

A high R^2 value (around unity) shows that the fitted model predicts the COME yield with respectable accuracy. The discrepancy between the Predicted R^2 of 0.8800 and the Adjusted R^2 of 0.9519, which is reasonable agreement, is less than 0.2. A good reproductivity for the model is indicated by a comparatively low value for the coefficient of variation (CV), 2.17%. A

measurement of how well the model fits each part of the design is the sum 191.56 of PRESS. The signal-to-noise ratio is measured with 21.075's sufficient precision. The ideal ratio is greater than 4. Consequently, this approach can be applied to explore the design space.

Table 4.6 Sequential Model Sum of Squares

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Mean vs Total	1.687E+05	1	1.687E+05			
Linear vs Mean	1395.05	4	348.76	41.68	< 0.0001	
2FI vs Linear	30.29	6	5.05	0.5329	0.7762	
Quadratic vs 2FI	**132.15**	**4**	**33.04**	**12.06**	**0.0002**	**Suggested**
Cubic vs Quadratic	30.74	8	3.84	3.02	0.0973	Aliased
Residual	7.62	6	1.27			
Total	1.703E+05	29	5871.73			

4.3.3. EFFECT OF REACTION PARAMETERS ON COME YIELD

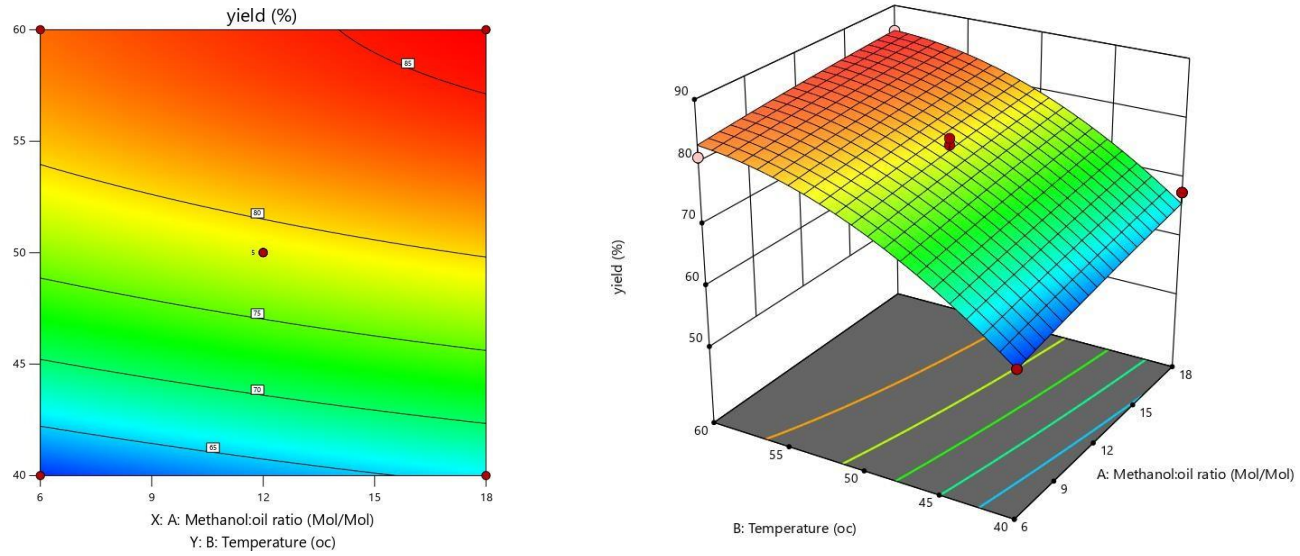


Figure 4.6 Response Surface plot for the interaction between Temperature and Methanol Ratio in

The effect of the temperature and methanol ratio relationship on COME Yield is depicted in Figure 4.6 Methanol ratio was adjusted between 6:1 and 18:1 while maintaining catalyst loading constant at 75 minutes and 1 weight percent, respectively, at 40 °C. When the temperature was at 60 °C and the methanol ratio was changed from 6:1 to 18:1, the COME Yield achieved was 60.83 wt% and 67.76 wt%, respectively. The COME Yields achieved were 81 weight percent and 85.60 weight percent, respectively.

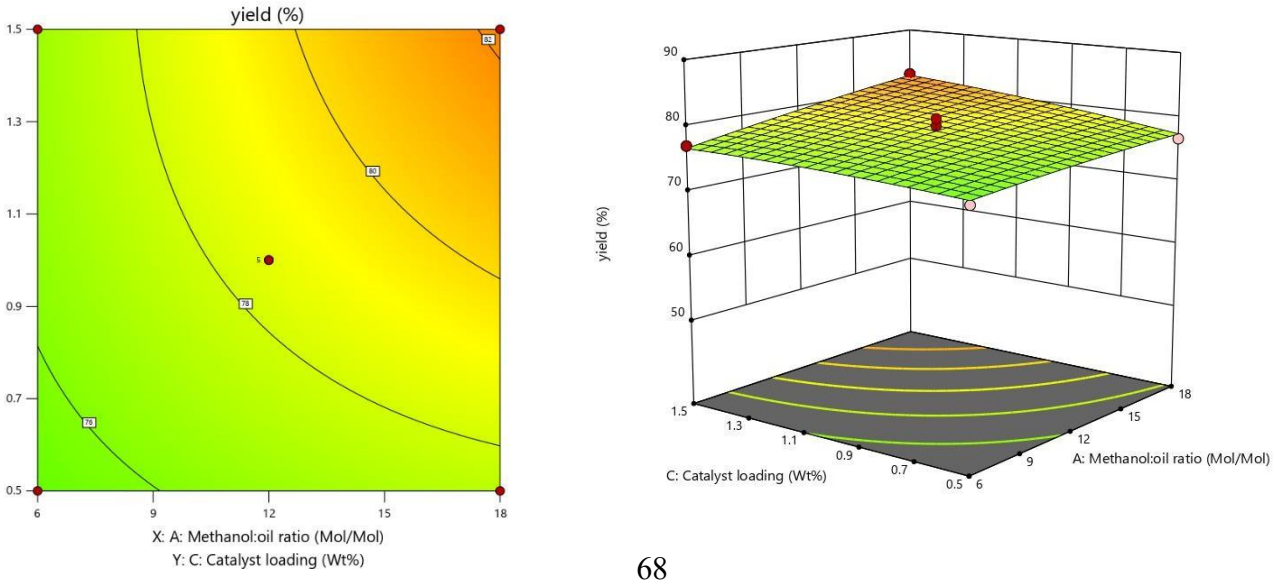


Figure 4.7 Response Surface plot for the interaction between Catalyst loading (wt. %) and Methanol

Figure 4.7 shows the impact of the methanol ratio and catalyst loading relationship on COME yield. While keeping the temperature and duration at 50 0C and 75 minutes, respectively, the methanol ratio was changed between 6:1 and 18:1 when the catalyst loading was at 0.5 weight percent. The COME yield was 74.6 weight percent and 76.63 weight percent, respectively, with 1.5 weight percent catalyst loading. A 6:1 to 18:1 methanol ratio adjustment was made. Both 77 and 82.46 weight percent of COME Yields were achieved.

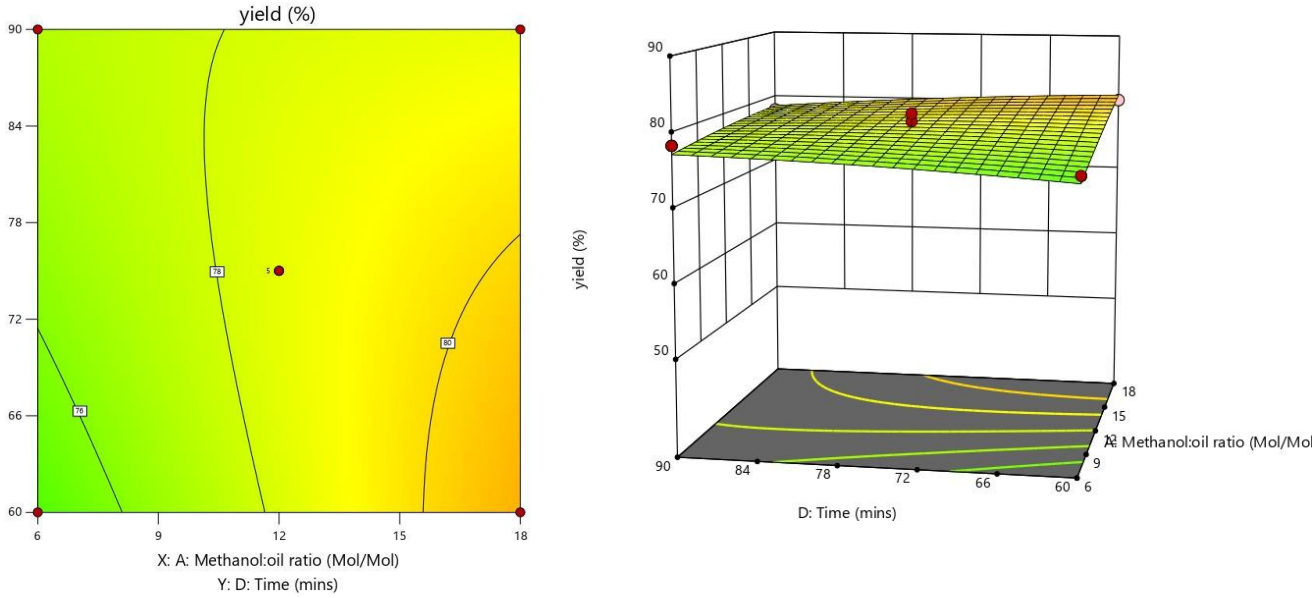


Figure 4.8 Response Surface plot for the interaction between Time and Methanol Ratio in COME

Figure 4.8 shows how time and methanol ratio interaction affect COME Yield. Over the course of the 60 minutes, the methanol ratio was varied between 6:1 and 18:1, while keeping the catalyst loading at 1% and the temperature at 50 0C, respectively. The COME Yield was obtained at 75.73 and 80.37 weight percent when the time was 90 minutes and the methanol ratio was changed between 6:1 and 18:1. COME Yields of 78.36 and 78.0% were attained, respectively.

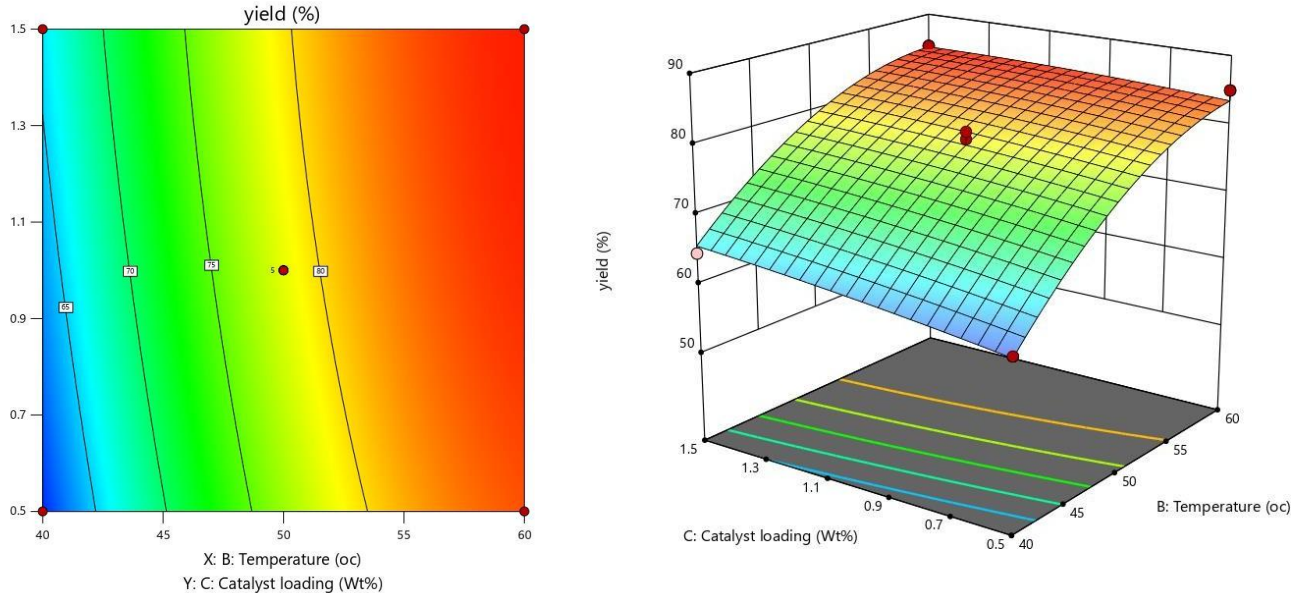


Figure 4.9 Response Surface plot for the interaction between Catalyst Loading and Temperature in

Figure 4.9 shows how the catalyst loading and temperature interact to affect COME Yield. While keeping time and methanol ratio constant at 75 minutes and 12:1, COME Yield was obtained at 61 weight percent and 85 weight percent, respectively, when catalyst loading was at 0.5 weight percent and temperature was varied between 40 °C and 60 °C, and when catalyst loading was at 1.5 weight percent and temperature was varied between 40 °C and 60 °C, respectively.

Figure 4.10 illustrates the influence of time and temperature interactions on COME Yield. The COME Yield was produced at 65.74 weight percent and 84.2 weight percent, respectively, when the time was 60 minutes and the temperature was altered between 40 and 60 degrees Celsius. The identical process was carried out again when the time had passed 90 minutes and the temperature had been changed from 40 to 60 degrees Celsius. COME Yields of 58.645 wt.% and 84.6 wt.%, respectively, were obtained.

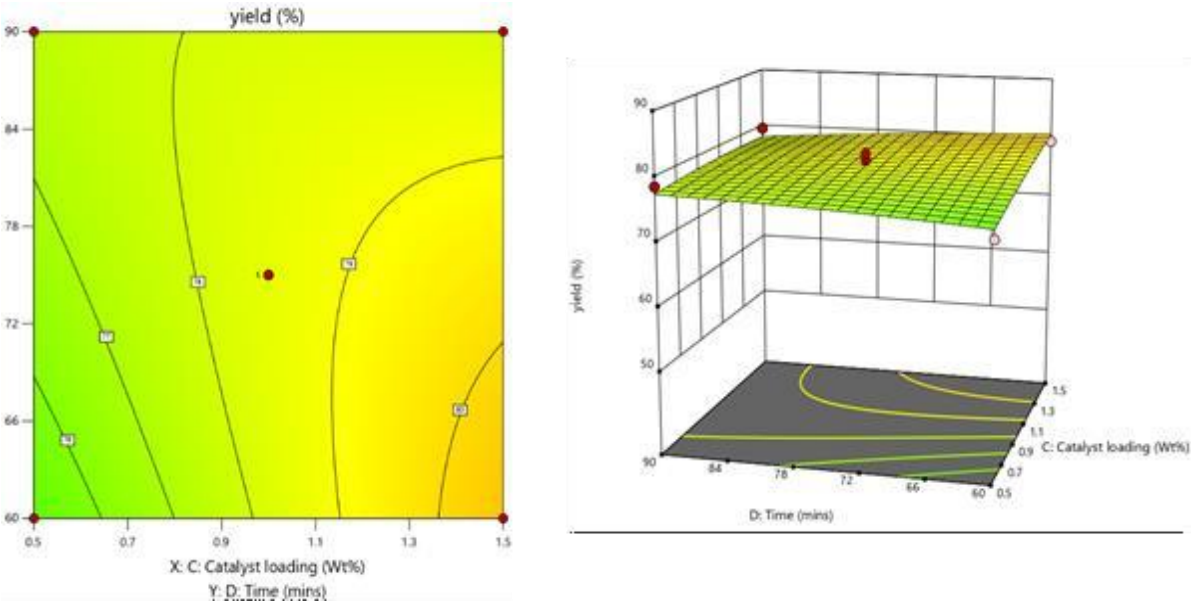


Figure 4.11 Response Surface plot for the interaction between Time and catalyst loading in COME Yield

On COME Yield, Figure 4.11 depicts the effects of time and catalyst loading. When the timer reached 60 minutes, the catalyst loading was changed from 0.5 weight percent to 1.5 weight percent while the temperature and methanol ratio were maintained at 75 minutes and 1 weight percent, respectively. The COME Yield obtained varied between 73.47 and 79.4 weight percent when the catalyst loading was altered between 0.5 and 1.5 weight percent. 78.6 and 79.6 weight percent were the respective COME Yields.

4.4 MODEL COMPARISON ON STATISTICAL ANALYSIS ON VARIANCE BETWEEN RSM AND ANFIS.

The right training data set was used to appropriately train the ANFIS model. This resulted in the creation of the parameters for the Sugeno-type fuzzy inference system (FIS), a hybrid learning technique that bases parameter creation on the gradient descent method and least squares estimate. Three membership functions were found to be the perfect number for each input when using a membership function with a trapezium structure. With the aid of the defuzzifier, the output values were calculated from the inputs. Fig. 4.12 shows the layout of the ANFIS model. The values predicted by the ANFIS model are shown in Table 4.7 and the high correlation between the experimental and predicted values was backed by the statistical parameters for ANFIS and RSM models ($R = 1$, $R^2 = 0.9673$, adjusted $R^2 = 0.96185$) (Table 4.8).

Run No.	ACTUAL AND CODED VALUE (IN BRACKET) OF FACTORS				ACTUAL COME yield (wt.%)	PREDICTED COME yield (wt.%) (RSM)	PREDICTED COME yield (wt.%) (ANFIS)
	MeOH: Oil (mol/mol)	Temperature (^{0}C)	Cat. Loading (wt.%)	Time (min)			
1	6 (-1)	50 (0)	1 (0)	60 (-1)	75.73	74.73	75.73
2	18 (1)	50 (0)	0.5 (-1)	75 (0)	76.63	77.39	76.63
3	12 (0)	50 (0)	1 (0)	75 (0)	79.36	78.52	78.52
4	6 (-1)	50 (0)	1 (0)	90 (1)	78.36	77.21	78.36
5	18 (1)	40 (-1)	1 (0)	60 (-1)	80.37	81.11	80.37
6	6 (-1)	50 (0)	1 (0)	75 (0)	60.83	60.80	60.83
7	12 (0)	60 (1)	0.5 (-1)	60 (-1)	73.47	75.00	73.47
8	12 (0)	50 (0)	1 (0)	60 (-1)	84.2	82.61	84.20
9	18 (1)	40 (-1)	1.5 (1)	75 (0)	82.46	82.22	82.46
10	12 (0)	60 (1)	1 (0)	90 (1)	59.645	61.61	59.64
11	6 (-1)	50 (0)	1 (0)	75 (0)	81	82.94	81.00
12	12 (0)	50 (0)	1 (0)	75 (0)	80.46	78.52	78.52
13	12 (0)	50 (0)	1 (0)	75 (0)	77.6	78.52	78.52
14	12 (0)	50 (0)	1.5 (1)	60 (-1)	79.4	80.58	79.40
15	6 (-1)	40 (-1)	0.5 (-1)	75 (0)	74.6	75.22	74.60
16	12 (0)	60 (1)	0.5 (-1)	75 (0)	61	60.75	61.00

17	12 (0)	40 (-1)	1.5 (1)	75 (0)	85	84.85	85.00
18	12 (0)	40 (-1)	1.5 (1)	75 (0)	64.54	65.63	64.54
19	18 (1)	50 (0)	1 (0)	75 (0)	67.76	65.85	67.76
20	12 (0)	50 (0)	1 (0)	75 (0)	77.6	78.52	78.52
21	12 (0)	50 (0)	1.5 (1)	90 (1)	79.6	78.10	79.60
22	12 (0)	50 (0)	0.5 (-1)	90 (1)	78.6	77.45	78.60
23	6 (-1)	50 (0)	1.5 (1)	75 (0)	77	76.62	70.00
24	12 (0)	60 (1)	1 (0)	75 (0)	77.6	78.52	78.52
25	12 (0)	60 (1)	1 (0)	90 (1)	84.6	85.84	84.60
26	18 (1)	50 (0)	1 (0)	75 (0)	85.6	85.66	84.60
27	18 (1)	40 (-1)	1 (0)	90 (1)	78	78.60	78.00
28	12 (0)	60 (1)	1 (0)	60 (-1)	65.736	64.87	65.74
29	12 (0)	50 (0)	0.5 (-1)	75 (0)	85	83.50	85.00

Table 4.7 Results from experiments and predictions for COME Yield

PARAMETER	RMS	ANFIS
R	0.98240606	0.9999
R^2	0.9603	0.9673
Adjusted R^2	0.95368333	0.96185
MSE	1.32153383	1.965808
RMSE	1.14957985	1.402073

SEP (%)	1.50730419	1.838367565
MAE	0.99934483	0.467206897
AAD (%)	1.32300107	0.596548483

Table 4.8 Evaluation of RSM and ANFIS performance

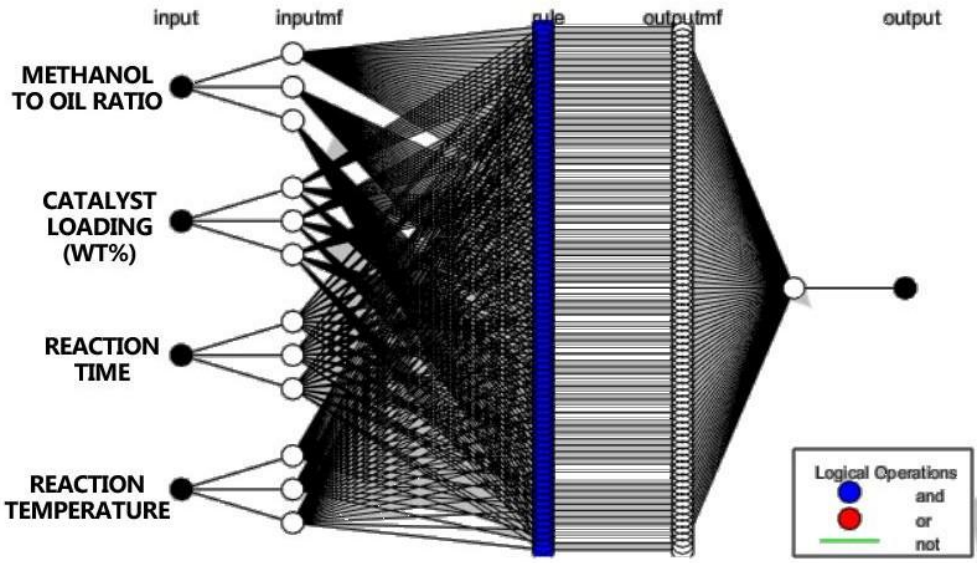


Figure 4.12 ANFIS architecture to forecast biodiesel yield

The various statistical metrics utilised to evaluate the effectiveness of the RSM and ANFIS models are summarised in Table 4.7. Generally speaking, both techniques demonstrated a strong ability to forecast the independent factors' effects on biodiesel yield. RSM was the least effective in terms of performance, with ANFIS outperforming RSM. These were shown by the extremely high R, R^2, and corrected R^2 values (0.9999, 0.9673, and 0.96185 respectively). The parity plots that compare experimental and anticipated biodiesel production provided more evidence that ANFIS was superior to RSM (Fig. 11). Fig. 11 makes it abundantly evident that the data points for the

ANFIS instance clustered more closely to the 45^0 line than the rest. This shows that the experimental data and the results predicted by the model fit each other quite well.

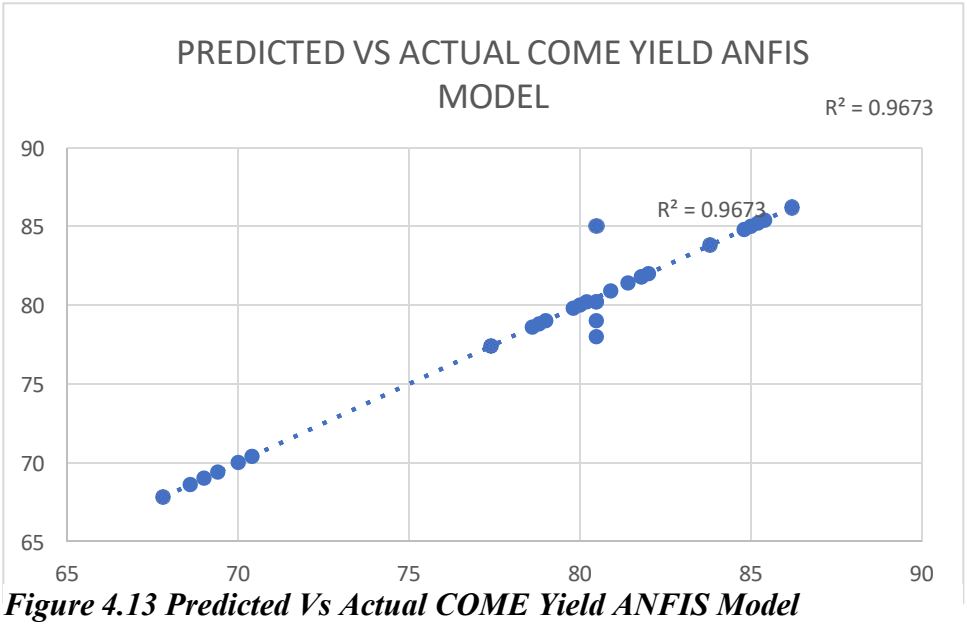


Figure 4.13 Predicted Vs Actual COME Yield ANFIS Model

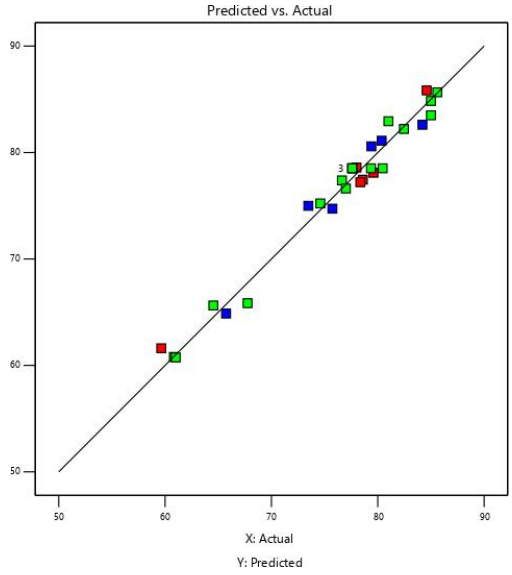


Figure 4.14 Predicted Vs Actual COME Yield RSM Model

4.5 PROCESS PARAMETER OPTIMIZATION AND MODEL VALIDATION

In order to maximise production efficiency while minimising costs, it is essential to optimise the process parameters involved in simultaneous esterification and transesterification.

According to the actual COME yield, the maximum yield was achieved with a catalyst loading of **1%**, a methanol to oil molar ratio of **18:1**, a reaction temperature of **60 °C**, and a reaction time of 7**5 minutes**, with a COME yield of **85.6 wt.%**. A **16.75:1** methanol to oil molar ratio, **1.28%** catalyst loading, **58.19 °C** reaction temperature, and **78.76 min**. of reaction duration were the parameters that Box Behnken Design indicated would produce the best results, with an **85.73 wt.%** COME. The similarity of these results demonstrates the accuracy of the assumptions.

4.6 CHARACTERISATION OF COME

The COME generated under the optimum reaction conditions was characterised to determine its fuel characteristics. Kinematic viscosity and density of the COME were found to be 3.76mm^2/s and 854.76kg/m^3, respectively, which were within the ASTM and EN biodiesel standard limits. The COME's AV, flash point, and calorific value were determined to be 0.28 mg KOH/g, 1280C, and 39.23MJ/kg. The values that were obtained for each characterization of COME are shown in Table 4.6.

Table 4.6 Physiochemical Attributes of Biodiesel obtained

Acid Value (mg KOH/g)	0.28
FFA (%)	0.14
Saponification Value	99.86
Viscosity at 31^0C (MPa.S)	3.76

Density (Kg/m^3)	854.76
Moisture Content (%)	0.006
Iodine Value	65.73
Pour Point (^0C)	-10.1
Cloud Point (^0C)	-2
Flash Point (^0C)	128
Cetane Number	68.52
HHV (Calorific Value)	39.23

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

The usage of biodiesel in the power sector has been claimed to assure both energy security and ecological balance. Commercial biodiesel production would need to use inexpensive and plentiful biomass as a raw source in order to be profitable.

This study used COCONUT OIL (CO), which was assessed and found to have an acid value of 5.8 mg KOH/g, to optimise the synthesis of biodiesel, catalysed by a bio-based bi-functional catalyst, by esterifying and trans-esterifying the COCONUT OIL (CO) at the same time. The crab shells were calcined and treated with KOH to form a base precursor, while the carbonised and sulfurized coconut shell was utilised to create an acid precursor. Both acid and base precursors were then impregnated to create the bio-based bi-functional catalyst. SEM, XRD, FTIR, and BET/BJH techniques were used to analyse the characteristics of the synthesised bio-based catalyst. At the optimum conditions of 18:1 methanol to oil molar ratio, 75 minutes reaction time, 60 ^{0}C reaction temperature, and a 1 weight percent catalyst loading, the catalysis produced a COME yield of 85.6 weight percent and an AV of 0.28 mg KOH/g, whereas the predicted COME Yield was discovered to be 85.73 weight percent, at 58.19^{0}C, 16.75:1 methanol ratio, 1.28 weight percent, and 78.76 minutes.

The ANOVA results revealed that the catalyst loading, reaction temperature, and methanol ratio were the key factors of the COME yield.

The parameters of the COME created in ideal conditions fell within the acceptable range of ASTM D6751 and EN 14214 criteria, and the synthesised catalyst was found to be an economical and environmentally friendly means of producing biodiesel.

5.2 RECOMMENDATIONS

The following topics should be looked at to gain a better understanding of this research, which involves the simultaneous esterification and transesterification of Coconut Oil (CO) to produce biodiesel utilising a bio-based bi-functional catalyst made from coconut shells and crab shells.:

1. Effect of temperature on the calcination and carbonization of crab and coconut shells.
2. Effect of impregnation ratios of the waste biomasses
3. Reaction kinetics of the reactions involved.
4. Effect of the synthetic catalyst's particle size on the simultaneous reaction.
5. Reusability study of the catalyst.

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