DEVELOPMENT OF BIOMASS DERIVED CATALYST FOR THE PRODUCTION OF BIODIESEL USING NEEM, WASTE COOKING OIL AND JATHROPHA OIL BLEND

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CERTIFICATION

This is to certify that this project work was carried out by Jamgbadi Godspromise Ohilebo, of the Department of Chemical engineering, University of Benin, Benin City, Edo State, Nigeria.

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DEDICATION

This work is dedicated to God Almighty for his loving kindness and tender mercies, for keeping me throughout my stay in school and bringing me this far. And to my parents Mr and Mrs Jamgbadi for their unwavering support before and during the completion of this work

ACKNOWLEDGEMENT

My sincere gratitude's goes to God almighty for his loving kindness and tender mercy towards me all through my stay in the university and for seeing me through to this very day.

Special thanks goes to my project supervisor Prof. Mrs. E.A. Oyedoh for her advice, constructive criticism and corrections in the course of completing this project work. I also want to specially acknowledge my HOD, Prof. Mrs. E.T. Akhiero, and other lecturers of the department of chemical engineering, university of Benin that have impacted in my life, I say may God bless you. I also want to thank Engr. Fred who guided me on my project work, I say may God bless you abundantly in Jesus name.

I also wish to acknowledge my parents Mr. and Mrs. Jamgbadi, my siblings, jamgbadi Precious, Jamgbadi Goodness and Jamgbadi Ebenezar. You all have been a source of blessing to my life.

I will not fail to mention some's of my friends micheal Ikechukwu, victor Ikechukwu, Daniel Itoya, Abba Ihonde and so many who have made my stay here in Uniben a memorable one.

I pray that God in his infinite mercies will continue to enrich your lives with good things and we will all meet at the top.

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ABSTRACT

The increasing demand for sustainable energy sources has driven the exploration of biodiesel as a viable alternative to fossil fuels. This study focuses on the development of biomass-derived catalysts for the production of biodiesel using a blend of non-edible oils: neem, shea, and jatropha. The research aims to address the challenges associated with conventional catalytic processes, such as high costs, environmental impact, and inefficiencies, by utilizing agricultural waste products to create sustainable and cost-effective catalysts.

The project involved the synthesis of biomass-derived catalysts from plantain peels and coconut husks, which were characterized for their physical and chemical properties. The transesterification process was optimized using the Box-Behnken Design (BBD) to determine the effects of reaction temperature, reaction time, catalyst load, and methanol-to-oil mole ratio on biodiesel yield. The results indicated that the optimal conditions for biodiesel production were a reaction temperature of 60°C, a reaction time of 90 minutes, a catalyst load of 5.5 wt%, and a methanol-to-oil mole ratio of 6.5:1, yielding a maximum biodiesel yield of 92.37%.

The biodiesel produced from the oil blend was characterized according to ASTM standards, and the results showed that the physical and chemical properties, including density, viscosity, flash point, and acid value, were within acceptable limits for biodiesel. The study demonstrated that biomass-derived catalysts are effective in producing high-quality biodiesel from non-edible oil blends, offering a sustainable and economically viable alternative to conventional catalysts. This research contributes to the advancement of renewable energy technologies by providing a framework for the utilization of locally sourced agricultural waste in biodiesel production. The findings highlight the potential of biomass-derived catalysts to enhance biodiesel yield and quality while reducing environmental impact, thereby supporting the transition to sustainable energy solution.

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background of the Study

The pressing need to lessen dependency on fossil fuels and slow down climate change is driving up demand for sustainable energy sources. Made from sustainable biological resources like vegetable and animal fats, biodiesel has become a viable substitute. It is a desirable alternative for energy production in both developed and developing countries due to its environmental advantages, which include lower greenhouse gas emissions and better air quality. With a focus on the sustainability and efficiency of traditional catalytic methods, this project, "Development of Biomass-Derived Catalyst for the Production of Biodiesel Using Shea, Waste cooking oil, and Jatropha Oil blend," aims to address the ongoing issues in the manufacturing of biodiesel. Although several feedstocks can be used to produce biodiesel, this study focusses on non-edible oils derived from shea, neem and jatropha oil plant which are abundant in regions such as Africa and Asia. These oils present unique opportunities for biodiesel production due to their high oil content and minimal competition with food sources.

Conventional biodiesel manufacturing techniques frequently include chemical catalysts, which are non-renewable and have high environmental costs both during production and use. Furthermore, these procedures can be costly, which raises the price of biodiesel relative to diesel made from petroleum. In order to produce biodiesel in a more sustainable manner, this study suggests using biomass-derived catalysts, which are made from agricultural waste products. This will take advantage of the renewable nature of these materials.

In addition to improving the output and purity of biodiesel, this project seeks to create a more sustainable and profitable production technique by developing novel catalysts and optimising the transesterification process. If this research is effective, it might have a big impact on the biodiesel sector by encouraging the use of sustainable, locally sourced materials.

1.2 Statement of the Problem

Biodiesel production must overcome several critical challenges to achieve widespread adoption and compete effectively with fossil fuels. Key issues include:

1. Reliance on Conventional Catalysts: Current catalytic processes predominantly rely on non-renewable resources for catalyst production. These catalysts can contribute to high production costs and environmental degradation from their manufacturing processes.

2. Low Efficiency and Yields: Many existing methods yield biodiesel at suboptimal rates, leading to inefficiencies in the overall production process. Factors such as improper reaction conditions, inadequate catalyst performance, and high energy requirements can severely limit yields.

3. Economic Viability: The initial investment and operational costs associated with biodiesel production using conventional catalysts can hinder its competitiveness against petroleum diesel, making it less attractive to consumers and producers.

4. Environmental Concerns: Conventional production methods often have a greater environmental impact due to higher emissions and waste generation compared to potential biomass-derived approaches.

By addressing these issues, this project aims to develop a more sustainable catalyst for biodiesel production that mitigates the environmental and economic barriers, facilitating the transition to renewable energy sources.

1.3 Aim and Objectives

The aim of this project is to develop effective biomass-derived catalysts from plantain peels, coconut husks ash for the production of biodiesel from non-edible oils such as waste cooking oil, neem oil, and jatropha oil. The objectives of the study are;

i. To determine the physical and chemical properties of the oil blends
ii. To synthesize Biomass-Derived Catalysts from plantain peels and coconut husk
iii. To optimize the production of biodiesel from oil blend using box behnken design of experiment.
iv. To determine the physical and chemical properties of optimized biodiesel synthesized from oil blend

1.4 Scope of the Study

The study is designed to focus on specific areas that align with the overall objectives of enhancing biodiesel production through innovative catalytic processes. The scope includes:

1. Catalyst Development: This project will specifically involve synthesizing biomass-derived catalysts using the by-products of neem, and jatropha plants, which are sourced from regions where these plants flourish.

2. Biodiesel Production: The primary objective centers on optimizing the transesterification process for these unique non-edible oils to improve overall biodiesel yield and quality, making it more commercially viable.

3. Sustainability Assessment: Emphasizing the environmental benefits, the study will conduct a comparative analysis of biomass-derived catalysts against conventional chemical catalysts, quantifying their sustainability advantages.

4. Technical Evaluation: Various reaction conditions (including temperature, pressure, and catalyst loading) will be experimentally evaluated to identify the optimal parameters for efficient biodiesel production.

5. Broader Implications: The findings may have implications for the larger biodiesel industry, contributing to advances in renewable energy technology and reinforcing the feasibility of employing local agricultural resources in energy production.

1.5 Relevance of the Study

The relevance of this study extends not only to academic discourse surrounding sustainable energy but also toward concrete applications in the biodiesel industry:

1. Supporting Sustainable Energy Solutions: The creation of catalysts produced from biomass is in line with international goals for attaining sustainability and energy independence. This study can offer a framework that encourages the use of locally produced agricultural goods and cleaner fuel substitutes.

2. Economic and Social Impact: By generating jobs in agriculture, production, and distribution, the successful implementation of biodiesel production from non-edible oils can support regional economies. Additionally, by making local fuel and feedstocks available, it can support rural development.

3. Environmental Benefits: By focusing on reducing carbon footprints and limiting the waste associated with traditional biodiesel production, this research will demonstrate a viable method

that supports renewable energy initiatives and places importance on environmental conservation practices.

4. Encouraging Technological Innovation: The innovative approaches proposed here in catalyst development may inspire further research and investment into advanced biodiesel production techniques, potentially leading to new discoveries and solutions within the renewable energy landscape.

In conclusion, this initiative is in line with more general environmental and social objectives and has the potential to significantly advance the domains of sustainable agriculture and renewable energy technology. It is anticipated that the results will offer theoretical as well as practical insights that can promote improvements in biodiesel production and foster a more sustainable future.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Biodiesel Production Processes

As a renewable substitute for fossil fuels, biodiesel has drawn a lot of interest since it can lessen greenhouse gas emissions and dependency on non-renewable resources. Modern manufacturing techniques emphasize how crucial it is to optimize them in order to improve productivity, sustainability, and financial sustainability.

An overview of the design, simulation, and sustainability assessment of the methods used to produce biodiesel is given by Pasha et al. (2021). They stress how crucial it is to optimize the process, taking into account variables like reaction conditions, catalyst selection, and feedstock selection that impact yield. In order to predict results and optimize designs, the study emphasizes the use of simulation tools, which eventually leads to more sustainable production methods. Through the incorporation of life cycle assessments, they offer a comprehensive method for assessing the environmental effects of biodiesel production.

Bashir et al. (2022) investigate new developments in biodiesel manufacturing processing methods. They emphasize the advantages and disadvantages of several techniques, including enzymatic processes, transesterification, and supercritical fluid processing, in their comprehensive evaluation. The necessity for novel technologies that can improve response rates and lower energy consumption in order to increase economic viability is highlighted by this study. The creation of sophisticated catalysts is emphasized by the authors as being essential to maximizing the production efficiency of biodiesel and reducing waste.

Role of Catalysts:

The choice of catalyst significantly influences the biodiesel production process. Zhang et al. (2022) provide insights into the application of heterogeneous nanocatalysts, detailing their operational mechanisms and performance characteristics. The review explains the advantages of using heterogeneous catalysts, including ease of separation and recycling, which can enhance the overall process efficiency. The authors discuss various types of nanocatalysts, their preparation methods, and their transition from laboratory-scale applications to industrial processes. This comprehensive examination of nanocatalysts suggests that their utilization can significantly improve biodiesel yield and quality.

Synthesis of Findings:

Collectively, these studies underscore the dynamic landscape of biodiesel production processes. Pasha et al. (2021) highlight the importance of sustainability assessments, while Bashir et al. (2022) focus on the adoption of advanced processing technologies to improve production efficiency. Zhang et al. (2022) further offer valuable insights into catalyst development, showcasing the potential of nanotechnology in enhancing biodiesel production.

Overall, the literature indicates that ongoing research and innovation are essential for overcoming current challenges in biodiesel production. By focusing on process optimization, advanced technologies, and effective catalyst solutions, the biodiesel industry can pursue a more sustainable and economically viable future. These reviews collectively provide a solid foundation for further exploration into biomass-derived catalysts, such as those derived from shea, waste cooking oil, and jatropha oil, as potential solutions for biodiesel production challenges.

2.1.1 Transesterification

A crucial chemical process in the creation of biodiesel is transesterification, which transforms the triglycerides found in vegetable oils and fats into glycerol and fatty acid methyl esters (FAME). In order to improve yield and efficiency, recent research has concentrated on optimizing this process through the use of different catalysts.

Developments in Transesterification Catalysts:

Wang et al. (2023) explore the developments in catalysts that make it possible to produce biodiesel through transesterification. A variety of catalyst types, such as enzymatic, heterogeneous, and homogenous catalysts, are covered in their study. They highlight the importance of catalyst selection in determining the reaction kinetics, overall yield, and sustainability of the biodiesel synthesis process. The authors also highlight new advances in catalyst design, emphasizing materials that can operate at lower pressures and temperatures, which lowers operating costs and energy consumption. In order to maximize production efficiency, the study also investigates the possibilities of bifunctional catalysts, which can improve transesterification and following reactions.

The performance of homogenous catalysts (like sodium hydroxide and potassium hydroxide), heterogeneous catalysts (like solid acid and base catalysts), and biocatalysts (enzymes) is evaluated in a review by Maleki et al. (2022). The authors discuss the benefits and drawbacks of each type of catalyst in terms of cost-effectiveness, reaction conditions, and reusability. Notably, they note that although homogenous catalysts are very effective, they frequently require rigorous separation processes, which can be costly. In contrast, heterogeneous catalysts are positioned as a more sustainable option because of their reusability and decreased waste generation.

Synthesis of Insights:

By highlighting the importance of catalyst design and optimization, this body of research lays the groundwork for continued innovation in biodiesel production processes. As the industry moves towards more sustainable practices, focusing on transesterification with advanced catalysts remains essential for improving biodiesel yield and economic viability. Wang et al. (2023) advocate for ongoing research into innovative catalysts that can improve reaction conditions and overall yield, while Maleki et al. (2022) effectively highlight the trade-offs between different catalyst types, offering invaluable insights for developing sustainable biodiesel production methods.

Together, these studies illustrate a trend toward integrating material science and engineering approaches to create more effective catalysts. By emphasizing the importance of catalyst design and optimization, this body of research lays the groundwork for continued innovation in biodiesel production processes. As the industry moves towards more sustainable practices, focusing on transesterification with advanced catalysts remains essential for improving biodiesel yield and economic viability.

2.1.2 Comparison of Catalytic Methods

The production of biodiesel through catalytic processes has garnered much attention due to the potential for improving yield, efficiency, and sustainability. A growing body of literature compares various catalytic methods, focusing on their effectiveness, cost, and environmental impact.

Comparative Analysis of Catalytic Activities:

Bedir and Doğan (2024) investigate the catalytic activities of calcium-based catalysts derived from waste materials in biodiesel production. Their research underscores the potential for using low-cost, sustainable catalysts sourced from agricultural waste and industrial by-products. The study provides a detailed comparison of different calcium-based catalysts, examining their performance in transesterification reactions. The authors present data demonstrating that these catalysts not only achieve substantial biodiesel yields but also offer advantages in terms of reusability and lower environmental impact compared to traditional catalysts. Their findings suggest that waste-derived catalysts can be a viable alternative in the biodiesel industry, promoting circular economy principles.

Bohlouli and Mahdavian (2021) offer a comprehensive review of various catalysts used in biodiesel production, emphasizing the distinctions between homogenous, heterogeneous, and biocatalysts. They classify catalysts based on their origins and describe their efficiencies in transesterification processes. The paper discusses the trade-offs associated with each type: while homogenous catalysts like sodium hydroxide are highly effective, they often require complex separation processes and generate more waste. Heterogeneous catalysts, though sometimes less efficient, facilitate easier separation and recycling, leading to lower operational costs. The review also highlights the growing interest in biocatalysts, which, while offering milder reaction conditions, may face challenges related to enzyme stability and cost-effectiveness.

Integration of Findings:

These studies present a nuanced comparison of various catalytic methods for biodiesel production. Bedir and Doğan (2024) specifically emphasize the advantages of using waste-derived calcium catalysts, while Bohlouli and Mahdavian (2021) provide an overarching review of multiple catalyst types and their respective benefits and limitations. Both studies advocate for further research into sustainable and economically viable catalytic solutions.

This indicates a clear trend towards exploring alternative catalysts that not only improve the efficiency of biodiesel production but also align with sustainability goals. As the industry faces challenges related to cost and environmental impact, the integration of waste-derived catalysts and an understanding of the comparative advantages of different catalytic methods will be essential for advancing biodiesel production technologies.

2.2 Biomass Derived Catalysts

In recent years, there has been a lot of interest in using biomass as a feedstock for the synthesis of catalysts, mainly because of its sustainability and capacity to improve the efficiency of different chemical reactions. Catalysts generated from biomass offer a possible substitute for conventional metal-based catalysts, frequently providing similar performance with a less environmental effect.

The conversion of waste biomass into catalysts for the production of biodiesel is one particularly promising area of research. In a thorough review, Hamza et al. (2021) address different types of waste biomass and their conversion processes, highlighting the potential of these materials in catalyzing transesterification reactions. They highlight the benefits of using catalysts derived from waste biomass, such as their low cost, availability, and ability to reduce harmful waste, which promotes a circular economy in the biodiesel sector. Their findings imply that improving the catalysts' properties could greatly increase the yield and quality of biodiesel.

Catalysts made from biomass have been investigated for use in electrochemical processes in addition to the manufacture of biodiesel. A thorough analysis of nonprecious metal catalysts made from biomass for oxygen reduction processes is given by Du et al. (2020). Their research focusses on engineering structures and active sites to satisfy particular requirements in energy conversion applications. The authors show that biomass-derived materials can rival traditional precious metal catalysts by concentrating on the customization of these catalysts, providing a more economical and environmentally friendly option for energy applications.

Furthermore, nitrogen-doped carbon compounds derived from biomass and their function as electrocatalysts in diverse electrochemical energy applications are the main focus of Park et al. (2022). Their review emphasises the many advantages of doping carbons produced from biomass with nitrogen, which can greatly increase the materials' stability and catalytic performance. According to the authors, these changes not only boost efficiency but also advance environmentally friendly energy conversion technologies, opening the door for more advancements in the area.

In summary, studies on catalysts made from biomass show that the chemical and energy industries are becoming more dedicated to sustainable practices. Researchers are moving closer to more environmentally friendly production techniques by turning waste materials into useful catalysts. These results highlight the need for more research into biomass resources in order to promote catalyst development improvements, which have ramifications for electrochemical applications that go beyond biodiesel. This demonstrates the enormous potential of these materials in sustainable chemistry and energy solutions by synthesising the noteworthy accomplishments in the field of biomass-derived catalysts.

2.2.1 Types of Biomass Catalysts

The development and application of biomass catalysts have gained significant attention as researchers seek sustainable alternatives for the conversion of biomass into valuable chemicals and fuels. Biomass-derived catalysts can be classified into various categories based on their origin, composition, and specific applications in different conversion processes, such as pyrolysis, gasification, and hydrothermal liquefaction.

Norouzi et al. (2021) provide an essential overview of the different catalysts employed in biomass pyrolysis, discussing their efficiency and effectiveness. Their study emphasizes the role of both homogeneous and heterogeneous catalysts, with a focus on how different materials can enhance the yield and quality of bio-oil produced from biomass. The authors conclude that while there is no "one-size-fits-all" catalyst for biomass pyrolysis, the choice of catalyst significantly influences the products formed during the pyrolysis process, making it critical to tailor catalyst selection to specific biomass types and desired outputs.

In a related context, Hamza et al. (2021) explore waste biomass-derived catalysts specifically for biodiesel production. They discuss various catalysts that can be produced from waste materials, highlighting the benefits of using non-edible biomass sources. The review indicates that these catalysts offer not only economic advantages due to their low-cost characteristics but also environmental benefits by promoting waste reduction. The performance of catalysts is largely dictated by their chemical composition and structural properties, which can be optimized for better catalytic activity.

Further emphasizing the versatility of biomass catalysts, Lin et al. (2021) investigate emerging heterogeneous catalysts employed in biomass conversion. Their work provides insights into the mechanisms of reactions involving these catalysts, showcasing how advancements in catalyst design are leading to more efficient biomass conversion methods. Their review identifies recent trends in catalyst development, including the incorporation of nanomaterials and tailored active sites, which enhance the selectivity and efficiency of biomass transformation processes.

Wu et al. (2022) contribute to the discussion by critically reviewing the applications of various catalysts in thermochemical biomass conversion processes. Their research covers pyrolysis, hydrothermal liquefaction, and gasification, exploring how different catalysts impact the overall efficiency and product distribution of these processes. They highlight the necessity for ongoing research to better understand the interactions between biomass and catalysts, which can lead to improved process designs and higher yields of desired products.

The diversity of biomass catalysts reflects the complexity of biomass conversion processes. From the utilization of waste materials to the development of novel heterogeneous catalysts, the literature indicates a strong inclination toward enhancing the efficiency and sustainability of biomass utilization. Future research should continue to explore the intricacies of catalyst design and application, focusing on achieving a deeper understanding of reaction mechanisms and optimizing catalytic performance across various biomass conversion processes.

2.2.2 Advantages and Disadvantages

The potential of biomass-derived catalysts for environmentally friendly and sustainable energy production has made them a focus of research. This review of the literature examines the benefits and drawbacks of using catalysts produced from biomass, based on current research in the area.

Benefits of Catalysts Derived from Biomass:

The sustainability and environmental friendliness of catalysts made from biomass are among their main benefits. According to Hamza et al. (2021), employing waste biomass as a catalyst encourages a circular economy in addition to reducing trash. Compared to traditional catalysts, which frequently use non-renewable resources, this use results in less carbon footprints. Furthermore, because biomass-derived catalysts can be made from easily accessible forestry and agricultural waste, they may be reasonably priced.

Zhang et al. (2022) further highlight the efficiency of carbon-based materials obtained from biomass in electrochemical energy devices by demonstrating their superior conductivity and large surface area. Because of these characteristics, biomass-based materials are a desirable alternative for green energy technologies since they improve the efficiency of energy storage and conversion devices like batteries and supercapacitors.

Furthermore, catalysts produced from biomass frequently show good selectivity and catalytic activity. In their review of different carbon-based and carbon-supported nanomaterials, Xia et al. (2022) describe how these catalysts can effectively support biomass conversion processes. These materials' distinct physicochemical characteristics allow them to function competitively with conventional catalysts, offering a sustainable substitute for chemical reactions.

The drawbacks of catalysts derived from biomass:

Although biomass-derived catalysts have many benefits, they also have several drawbacks. Their inconsistent quality and performance, which can be greatly influenced by the biomass's source and the catalyst preparation technique, is one major problem. According to Ren et al. (2022), differences in the content of biomass can result in inconsistent catalyst properties, which can affect how well catalysts work in catalytic processes. When procedures are scaled up for industrial applications, this discrepancy is very troublesome.

Another concern is the potential leaching of active components in biomass-derived catalysts. Hamza et al. (2021) indicate that certain biomass-derived catalysts may release undesirable substances during reactions, which can contaminate products and pose environmental risks. Ensuring stability and minimizing leaching are crucial aspects that need to be addressed in future catalyst design. Additionally, the commercial viability of biomass-derived catalysts is often hindered by the limited knowledge surrounding their long-term performance and reusability. Zhang et al. (2022) note that while research on these materials is progressing, further investigation is required to understand their behavior over extended operational periods. Establishing their long-term reliability is essential for widespread adoption in industrial applications.

In conclusion, catalysts made from biomass have many benefits, such as good performance in a range of catalytic processes, cost effectiveness, and sustainability. To reach their full potential, though, issues like quality variation, possible leaching, and concerns about long-term stability must be resolved. Overcoming these obstacles and encouraging the use of catalysts generated from biomass in green technology applications require ongoing research and development in this area.

2.3 Feedstocks for Biodiesel

Biomass-derived catalysts have emerged as a focal point for research due to their potential in sustainable energy production and environmental benefits. This literature review explores the advantages and disadvantages associated with the use of biomass-derived catalysts, drawing from recent studies in the field.

Advantages of Biomass-Derived Catalysts:

One of the primary advantages of biomass-derived catalysts is their sustainability and environmental friendliness. Hamza et al. (2021) highlight that using waste biomass as a catalyst not only reduces waste but also promotes a circular economy. This utilization leads to lower carbon footprints compared to conventional catalysts that often rely on non-renewable materials. Moreover, biomass-derived catalysts can be cost-effective, as they can be produced from readily available agricultural and forestry residues.

Zhang et al. (2022) further emphasize the effectiveness of biomass-derived carbon-based materials in electrochemical energy devices, showcasing their high surface area and excellent conductivity. These properties enhance the performance of energy storage and conversion systems, such as batteries and supercapacitors, making biomass-based materials an attractive option for green energy technologies.

Additionally, biomass-derived catalysts often exhibit good catalytic activity and selectivity. Xia et al. (2022) review various carbon-based and carbon-supported nanomaterials, explaining how these catalysts can facilitate biomass conversion processes efficiently. The unique physicochemical properties of these materials enable them to perform competitively against traditional catalysts, providing an eco-friendly alternative for chemical reactions.

Disadvantages of Biomass-Derived Catalysts:

Despite their numerous advantages, biomass-derived catalysts also present some disadvantages. One significant challenge is their variable quality and performance, which can depend heavily on the source of biomass and the method of catalyst preparation. Ren et al. (2022) discuss how variations in biomass composition can lead to inconsistencies in catalyst characteristics, impacting their effectiveness in catalytic processes. This inconsistency is particularly problematic when scaling up processes for industrial applications.

Another concern is the potential leaching of active components in biomass-derived catalysts. Hamza et al. (2021) indicate that certain biomass-derived catalysts may release undesirable substances during reactions, which can contaminate products and pose environmental risks. Ensuring stability and minimizing leaching are crucial aspects that need to be addressed in future catalyst design.

Additionally, the commercial viability of biomass-derived catalysts is often hindered by the limited knowledge surrounding their long-term performance and reusability. Zhang et al. (2022) note that while research on these materials is progressing, further investigation is required to understand their behavior over extended operational periods. Establishing their long-term reliability is essential for widespread adoption in industrial applications.

biomass-derived catalysts offer an array of advantages, including sustainability, cost-effectiveness, and high performance in various catalytic processes. However, challenges such as variability in quality, potential leaching, and questions regarding long-term stability must be addressed to fully realize their potential. Continued research and development in this field are critical to overcoming these limitations and promoting the adoption of biomass-derived catalysts in green technology applications.

This underscores the dual nature of biomass-derived catalysts, balancing their sustainable promise with the hurdles that must be overcome for their effective implementation in various applications.

Biodiesel is recognized as a promising alternative to conventional fossil fuels due to its renewable nature and lower environmental impact. The choice of feedstock is crucial for biodiesel production, as it influences the economics, sustainability, and quality of the final product. This literature review explores various feedstocks utilized in biodiesel production, highlighting their advantages and challenges.

1. Vegetable Oils

Vegetable oils are among the most common feedstocks for biodiesel production. The use of oils such as soybean, palm, and canola has been extensively studied. According to Ma and Hanna (1999), vegetable oils offer high lipid content, making them suitable for transesterification processes to produce biodiesel. However, the use of food crops raises concerns about food security and competition with food supply. Additionally, the seasonal availability of these crops can lead to supply fluctuations.

2. Animal Fats

Animal fats, including tallow and used cooking oils, represent another significant feedstock for biodiesel. Marchetti et al. (2007) note that animal fats have a higher cetane number compared to vegetable oils, which can result in better combustion properties. The recycling of used cooking oils also contributes to sustainability by reducing waste. However, the collection and processing of these fats can be cumbersome, and the presence of free fatty acids (FFAs) can complicate the transesterification process.

3. Algae

Algae are gaining attention as a future feedstock for biodiesel due to their rapid growth rates and high lipid content. According to Chisti (2007), microalgae can produce up to 60% of their biomass as lipids and are not confined to arable land, thereby eliminating competition with food crops. Additionally, algae can utilize wastewater and carbon dioxide, potentially leading to reduced environmental impacts. Despite these advantages, challenges such as high production costs and complex harvesting processes remain significant barriers to the commercial viability of algal biodiesel.

4. Non-Food Feedstocks

To address concerns over food security, researchers are exploring non-food feedstocks, such as jatropha and camelina. These plants can grow in arid environments and have lower input requirements compared to traditional crops. According to Dias et al. (2020), jatropha can yield significant oil content while requiring less water and fertilizers, making it a sustainable option for biodiesel. However, the commercial viability of non-food feedstocks is often hindered by agricultural challenges, market acceptance, and the need for extensive research and development.

The choice of feedstock for biodiesel production is critical in determining the sustainability, economic feasibility, and environmental impact of biodiesel as an alternative fuel. While conventional feedstocks such as vegetable oils and animal fats have been widely used, the exploration of algae and non-food feedstocks presents exciting opportunities for the future. Addressing challenges related to cost, consistency, and agricultural practices will be essential to enhance the viability of biodiesel feedstocks.

This literature review underscores the diversity of feedstocks available for biodiesel production and the importance of addressing their respective advantages and challenges to promote sustainable energy solutions.

5. Shea Oil

Shea oil is derived from the nuts of the shea tree (*Vitellaria paradoxa*) and has become popular in a number of industries, such as the manufacturing of biodiesel, food, and cosmetics. This review of the literature examines the nutritional advantages, uses, composition, and extraction techniques of shea oil, emphasizing both its importance and possible drawbacks. Shea oil is a valuable and adaptable resource that can be used in food, cosmetics, and possibly the manufacturing of biodiesel. Although issues with extraction techniques, sustainability, and socioeconomic considerations need to be addressed, its rich content has several health benefits. The future of shea oil in regional and international markets will depend heavily on ongoing research into enhancing extraction technologies and encouraging sustainable practices.

2.3.1 Waste oils

The utilization of waste oils, including restaurant grease and spent cooking oils, presents an opportunity for sustainable biodiesel production. Research by Liu et al. (2014) indicates that using waste feedstocks can lower production costs and minimize environmental waste. These feedstocks often contain high FFAs which can be converted to biodiesel through specific pretreatment methods like acid-catalyzed transesterification. Nonetheless, the variability in quality and composition of waste oils can affect the consistency of the biodiesel produced.

2.3.2 Neem Oil

A growing interest in neem oil as a bio-based natural product is highlighted by this literature review, which examines the oil's chemical composition, health benefits, agricultural uses, and potential limitations. Neem oil is derived from the seeds of the neem tree (Azadirachta indica) and has gained recognition for its many uses in medicine, cosmetics, and agriculture.

1. Chemical Composition The main active ingredient, azadirachtin, is known for its insecticidal activity and ability to disrupt the growth and reproductive cycles of pests. The presence of other triterpenoids and antioxidants also increases the oil's effectiveness in a variety of applications. Neem oil's chemical diversity makes it a valuable resource in both the health and agricultural sectors. Other bioactive compounds that contribute to its therapeutic and pesticidal properties include nimbi din, fatty acids, and azadirachtin.

2 Therapeutic Qualities: For millennia, neem oil has been used in traditional medicine, especially in Ayurvedic procedures. It has well-established antibacterial, anti-inflammatory, and antioxidant qualities. Neem oil is effective against a wide range of pathogens, such as bacteria, fungi, and viruses, according to Raghavan et al. (2019). It is a common component of herbal treatments for skin disorders like psoriasis, dermatitis, and acne because of its broad-spectrum action. Additionally, research by Tiwari et al. (2019) confirms neem oil's potential as a natural medicinal agent by highlighting its function in lowering inflammation and facilitating wound healing.

3. Agricultural Applications: Neem oil is frequently used as a biopesticide in agriculture because it works well against a range of pests, such as spider mites, aphids, and whiteflies. Isman (2006) talks about how neem oil is a safer alternative to synthetic pesticides because it inhibits the feeding, growth, and reproduction of pests, which lowers their populations without harming beneficial insects. Sarma et al. (2020) supports these findings by showing the effectiveness of neem oil in organic farming practices, where ecological balance is crucial.

4. Advantages of Personal Care and Cosmetic Products: Due to its healing and moisturizing qualities, neem oil is now used in a variety of cosmetic and personal care products. Neem oil is commonly used in shampoos, soaps, and lotions because it nourishes skin and hair and protects against microbial illnesses (Dhananjaya et al., 2018). Customers looking for sustainable and organic personal care products will find the oil intriguing due to its natural composition and absence of artificial additives.

5. Restrictions and Difficulties: Even though neem oil has several uses, there are some issues that must be resolved. Its effectiveness may be impacted by variations in content and quality, which are frequently impacted by elements including extraction techniques and place of origin (Hussain et al., 2019). Neem oil's intense smell may also discourage its use in some formulations, therefore odor-masking methods have to be developed for wider cosmetic uses. Additionally, even though neem oil is usually regarded as safe, cautious use is advised due to the possibility of allergic reactions in certain people, highlighting the necessity of extensive research prior to broad use in pharmaceutical and cosmetic goods.

Neem oil is a natural product that can be used in many different fields, including personal care, medicine, and agriculture. Its abundant bioactive makeup offers both powerful health advantages and efficient pest control solutions. However, in order to fully realise its potential, issues with customer acceptability and quality variability must be resolved. Our knowledge of neem oil and its numerous applications in different industries will only grow with more study and development.

2.3.3 Jatropha Oil

Jatropha oil, derived from the seeds of the Jatropha curcas plant, has emerged as a promising renewable resource for various applications, particularly biofuel production and agricultural uses. This literature review examines the chemical properties, production processes, applications, advantages, and challenges associated with Jatropha oil, highlighting its significance in sustainable development.

1. Chemical Composition and Properties

Jatropha oil is composed predominantly of triglycerides, with significant amounts of oleic, linoleic, and palmitic acids. According to Basha et al. (2009), the oil's fatty acid profile is pivotal in determining its suitability as a biodiesel feedstock. The presence of high levels of unsaturated fatty acids, particularly oleic acid, contributes to favorable fuel properties, such as higher cetane numbers and improved cold flow characteristics compared to other vegetable oils.

The physicochemical properties of Jatropha oil also play an essential role in its potential applications. Kaur et al. (2016) note that the oil has a higher viscosity and density than conventional diesel, which can affect its performance in standard diesel engines. This necessitates the exploration of transesterification processes to improve its characteristics for biodiesel production.

2. Biodiesel Production

The transesterification process is the primary method for converting Jatropha oil into biodiesel. Studies by Demirbas (2009) illustrate the process, where Jatropha oil is reacted with alcohol, usually methanol or ethanol, in the presence of a catalyst. The resulting biodiesel has been shown to exhibit properties comparable to fossil diesel, making it a viable alternative fuel. Research conducted by Knothe et al. (2005) confirms that biodiesel derived from Jatropha oil meets international fuel specifications, further supporting its commercial potential.

Jatropha oil's potential for biofuel production is enhanced by its non-food crop status, reducing the competition for food supply often associated with traditional biodiesel feedstocks. A study by Sahoo et al. (2007) emphasizes the oil's economic viability, highlighting that Jatropha cultivation can provide income opportunities for rural farmers while contributing to rural development.

3. Agricultural Benefits

In addition to its energy applications, Jatropha oil boasts several agricultural benefits. The plant is drought-resistant and can thrive in marginal lands, making it a suitable crop for areas with poor soil quality. According to Achten and Verchot (2009), Jatropha can help improve soil conditions through its extensive root system, which may enhance soil structure and fertility. Moreover, the plant's leaves and seeds can serve as organic fertilizers and pest deterrents, promoting sustainable agricultural practices.

4. Environmental and Socioeconomic Implications

Jatropha oil has been regarded as a more sustainable choice compared to fossil fuels, as its cultivation can contribute to carbon sequestration and reduce greenhouse gas emissions. Research by IEA (2010) indicates that cultivating Jatropha for oil production could provide significant environmental benefits if managed correctly, with potential reductions in CO2 emissions.

However, the large-scale cultivation of Jatropha presents several challenges. Concerns regarding its invasive nature and potential impacts on local ecosystems need to be addressed. Furthermore, studies have shown that inconsistent yields and variability in oil quality can hinder its commercial viability (Harun et al., 2014). Implementing effective agricultural practices, along with continued research into breeding high-yielding varieties, is crucial to overcoming these challenges.

5. Economic Viability and Market Potential

The market potential for Jatropha oil as a biodiesel feedstock remains a topic of active research and discussion. According to Dutta et al. (2013), while initial investment costs for Jatropha cultivation can be high, the long-term returns may justify these costs, especially in regions where alternative cropping options are limited. The establishment of sustainable supply chains and processing facilities will be essential for maximizing the economic viability of Jatropha oil.

Jatropha oil represents a multifaceted resource with significant potential in biodiesel production, sustainable agriculture, and environmental benefits. Its favorable chemical properties and suitability for cultivation in marginal lands highlight its importance in renewable energy strategies. However, challenges related to yield variability, oil quality, and ecological impacts must be addressed to fully realize its potential. Continued research, effective agricultural practices, and policy support will be crucial in promoting the sustainable development of Jatropha oil as a bio-based alternative to fossil fuels.

2.4 Previous Studies on Biomass Catalysts

Biomass catalysts have gained significant attention in recent years due to their potential to facilitate sustainable chemical processes, particularly in the production of biofuels and value-added chemicals. This literature review explores various studies focusing on the development, characteristics, and applications of biomass-derived catalysts, highlighting key findings, methodologies, and implications for future research.

1. Introduction to Biomass Catalysts

Biomass catalysts are derived from renewable biological resources and serve as alternatives to conventional catalysts sourced from non-renewable materials. The advantages of using biomass in catalysis include lower environmental impact, cost-effectiveness, and enhanced sustainability. Chen et al. (2021) emphasize the growing interest in utilizing biomass waste for catalyst production, thereby promoting a circular economy while reducing reliance on fossil fuels.

2. Types and Synthesis of Biomass Catalysts

Various biomass materials have been investigated for use as catalysts, including agricultural residues, forestry by-products, and algae. Cai et al. (2020) review the conversion of lignocellulosic biomass into solid acid catalysts, detailing the thermochemical processes involved in their synthesis. These catalysts demonstrate significant activity in the esterification of fatty acids and transesterification of triglycerides for biodiesel production. Moreover, Torres et al. (2017) explore the potential of using char derived from agricultural residues as a catalyst for biomass gasification, highlighting its effectiveness in enhancing reaction kinetics.

The synthesis methods can significantly affect the physicochemical properties of biomass catalysts. Zhang et al. (2019) investigate different activation processes for producing biochar catalysts from rice husk, showing that temperature and activation agents influence the surface area and catalytic performance. The outcome of these studies underscores the importance of optimization in catalyst design to maximize efficiency and effectiveness.

3. Catalytic Applications in Renewable Energy

Biomass catalysts have shown promise in the production of biodiesel, bioethanol, and other renewable fuels. A study by Sharma et al. (2018) focuses on the use of waste biomass-derived heterogeneous catalysts for the transesterification of vegetable oils, demonstrating high conversion rates and selectivities. The authors suggest that leveraging biomass waste not only enhances economic feasibility but also improves the sustainability of the biodiesel production process.

Similarly, Liu et al. (2020) present a comprehensive review of catalyst applications in biomass pyrolysis, emphasizing the critical role of biomass-derived catalysts in enhancing the yield of bio-oil. They demonstrate that integrating these catalysts into pyrolysis processes can significantly improve the chemical composition and quality of the produced bio-oil, making it more suitable for transportation fuels.

4. Advantages and Challenges

The advantages of biomass catalysts include their renewability, the potential for lower production costs, and the reduction of greenhouse gas emissions. However, challenges remain in terms of catalyst performance, stability, and reusability. According to Ren et al. (2022), while biomass-derived catalysts exhibit good catalytic activity, their long-term performance and longevity under reaction conditions are paramount for practical applications. Strategies for improving catalyst durability include refining the synthesis methods and investigating the interactions between catalyst components.

5. Future Directions in Biomass Catalysts Research

Future research should focus on enhancing the catalytic properties and longevity of biomass-derived catalysts while minimizing their environmental impact. Innovations in catalyst characterization techniques and process engineering may provide insights into catalyst performance mechanisms, as discussed by Kauffman et al. (2020). Additionally, interdisciplinary approaches that integrate materials science, chemistry, and engineering will be crucial in advancing the field of biomass catalysis.

Biomass catalysts represent a promising avenue for advancing sustainable chemical processes. Previous studies have established their efficacy in various applications, particularly in renewable energy production. Continued research and development in this area will be essential to address current challenges and unlock their full potential in the transition toward a bio-based economy.

2.5 Research Gaps

The development of biomass-derived catalysts for the production of biodiesel from non-edible oil sources, such as shea, neem, and jatropha oil, presents a significant opportunity for sustainable energy solutions. While numerous studies have explored catalyst development and optimization, certain research gaps remain that could enhance the efficacy and applicability of these catalysts. This literature review identifies and discusses these gaps based on recent studies.

1. Catalyst Composition and Modification

One major research gap is the optimization of catalyst composition and modification techniques to improve catalytic activity and selectivity. While various biomass-derived catalysts have been synthesized, studies such as those by Zhan et al. (2017) highlight that the performance of these catalysts can vary significantly based on their physicochemical properties. Limited research has focused on systematically varying the catalyst's composition—particularly when using agricultural residues, such as those from shea, neem, and jatropha. For instance, Wang et al. (2015) emphasize the need for further investigation into how differing processing conditions, precursor materials, and activation techniques can influence catalyst efficiency.

2. Long-Term Stability and Reusability

Another critical gap concerns the long-term stability and reusability of biomass-derived catalysts. Many studies, including those by Fadhil et al. (2019), often report short-term experimental results without adequate assessment of catalyst lifespan or deactivation mechanisms. Ensuring that these catalysts retain their functional properties over extended periods is essential for their economic viability. Research focusing on the durability of catalysts derived from specific oils, particularly in realistic operating conditions, is scant. Standardized testing methodologies are needed to evaluate and compare the reusability of catalysts, as highlighted by Kumar et al. (2020).

3. Economic Analysis and Scale-Up Feasibility

The economic aspects related to the production and application of biomass-derived catalysts in biodiesel production remain underexplored. While studies have demonstrated the technical feasibility of using catalysts from shea, neem, and jatropha oils, there is a lack of comprehensive life cycle assessments (LCA) and cost-benefit analyses. Research by Shafiee and Topal (2009) indicates that an understanding of the economic implications of large-scale biodiesel production is critical for its commercial viability. Investigating the costs associated with biomass feedstock collection, catalyst synthesis, and processing will provide insights into the scalability of these technologies.

4. Compatibility with Feedstock Variability

The variability in feedstock quality and composition is another factor that impacts catalyst performance but is often overlooked. Sectional differences in the oil content and fatty acid profile of shea, neem, and jatropha may affect the efficiency of biomass-derived catalysts. Studies such as those conducted by Niaounakis et al. (2019) suggest that feedstock variability can lead to inconsistencies in biodiesel yield and quality. More research is needed to develop adaptable catalysts that can handle variations in feedstock composition, thereby maintaining high performance under diverse conditions.

5. Comprehensive Characterization Techniques

There is a pressing need for more comprehensive characterization techniques to evaluate biomass-derived catalysts' structural, surface, and reactivity attributes. Techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and Brunauer-Emmett-Teller (BET) surface area analysis are widely used; however, they do not always provide a complete understanding of the catalyst behavior during biodiesel production. Studies by Montoya et al. (2016) propose that advanced characterization methods, including in situ techniques, need to be applied to gain deeper insights into catalyst activity and deactivation mechanisms, particularly under real operational settings.

6. Environmental Impact Assessment

Lastly, the environmental impacts related to the production and application of biomass-derived catalysts remain largely understudied. While the use of biomass in catalyst development is touted as environmentally friendly, a more thorough evaluation of the associated emissions, resource consumption, and ecological consequences of large-scale production is necessary. Research by Bringezu et al. (2017) emphasizes the importance of integrating sustainability assessments into catalyst development, ensuring that the overall benefits outweigh potential environmental costs.

In summary, while there is substantial progress in the development of biomass-derived catalysts for biodiesel production from shea, neem, and jatropha oil, several research gaps persist. Addressing these gaps by focusing on catalyst optimization, long-term stability, economic feasibility, compatibility with feedstock variability, advanced characterization techniques, and environmental assessments will be crucial in advancing this.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1. Materials and Reagents

The major raw materials used in this research were jatropha seed oil, waste cooking oil and neem seed oil purchased from a local market in Benin city Edo state. They were obtained fresh and used as source of feedstock for the synthesis of fatty acid methyl ester. The chemicals were purchased from local chemical store in Benin City, Edo state. They were obtained in a high analytical grade, and they are listed as follow.

a) Sodium Hydroxide; 96% pure, produced by CDH, New Delhi, India.
b) Potassium Hydroxide; 96% pure, produced by CDH, New Delhi, India.
c) Sulphuric acid; 98% pure, produced by Fisons, Loughborough England.
d) Benzene; 98% pure, produced by BDH Chemicals ltd, Poole, England.
e) Ethanol; 99.7% pure, produced by JHD, Shatou, Guondghuo China.
f) Methanol; 99.7% pure, produced by JHD, Shatou, Guondghuo China.
g) Phenolphthalein Indicator; produced by Kermel Chemicals Reagent Company Ltd, Tianjin, China.
h) Chloroform; 99.7% pure, produced by JHD, Shatou, Guondghuo China.
i) Acetic acid; 99.7% pure, produced by JHD, Shatou, Guondghuo China.

3.1.1 Equipment

The glass wares used during the experimentations are measuring cylinders of various sizes, beakers of various sizes, conical flasks, separating funnel. The specific equipment are as presented in Table 3.1

Table 3.1: Equipment List

Property	Equipment and Manufacturer
Stirrer	MS300 Constant Temperature Magnetic Stirrer. B. Brian, England
Drying	Vacutherm VT 6025 Air-Dry Vacuum Oven. Thermo Scientific NJ, USA
Calcination	Uniscope SM9080 Muffle Furnace. Surgifriend, England
Density	SVM 3000 50 millilitre Pycnometer (Density Bottles) Anton Paar, UK
Dynamic Viscosity	NDJ-5S Viscometer. Anton Paar, UK
Calorific Value	C2000 Oxygen Bomb Calorimeter. IKA, UK
Flash Point Tester	Pensky Martins SYD-261 Closed Cup Flash Point Tester. Chitao, Hongkung
Design Expert	Design Expert version 13 .0.6. Stat-case@, Inc. Minneapolis, USA)
SEM	Scanning electron microscope
Weighing Balance	OHAUS AV264 Adventurer Pro Analytical balance.

3.2 Preparation and Characterization of Catalyst

Biomass (coconut husks and plantain peels) as a catalyst was prepared by calcination method (Jazie et al. 2012; Buasri et al. 2013). Coconut husks and plantain peels obtained from a local market wastes bins were washed clean with water and spread in an open sun light to remove excess water. This was then followed by oven drying at 105ºC for 3 hours. The dried sample were ground and sieved through a 250µm mesh before calcination at 750°C in a muffle furnace with a heating rate of 10°C/min for 3 hours (Rohim et al. 2014). The product was obtained as greyish powder and was cooled in desiccator and later were kept in closed vessel to avoid its reaction with carbon dioxide (CO_2) and humidity in air before used.

3.2.1 Characterization of the Catalyst

After calcination, the porosity of the catalyst was determined experimentally by performing the following experiment: The sample was transferred to a 100cm^3 measuring cylinder and it was well shaken up to 50cm^3 mark. 50cm^3 of distilled water was mixed with the sample and the experiment left for 1 hour for water to percolate and fill the pores between the sample. The porosity of the sample was calculated as the difference between the theoretical volume expected and the actual volume observed. The final volume of the mixture was then read.

3.3 Biodiesel Equipment Set-Up

A liquid reaction mixture was charged into a 1000ml flask fitted with a stir bar to achieve a homogeneous mixture at a constant mixing speed of about 380rpm. This flask will be connected to a Braham condenser, such that any vapour given off will be cooled back to liquid, and fell back into the reaction flask. The flask was heated with the aid of the constant temperature magnetic stirrer for the reaction time. Different temperatures will be maintained for different runs of the experiment so as to determine the effects of temperature on the yield and the kinetics of the transesterification reaction will be determined.

Figure 3.1: Experimental set up for biodiesel synthesis

3.3.1 Esterification Reaction

Exactly 500g of jatropha oil, neem oil and waste cooking oil blend at a blending ratio of 50:50:50 was added to a round bottom flask and was esterified with 25wt% of methanol using 1.0wt% H_2SO_4 as catalyst to reduce the free fatty acids concentration to about 1% FFA. The mixtures were placed on a constant temperature magnetic stirrer set to heat at a constant temperature 60^oC for 1.5hour esterification reaction. The procedure was repeated until %FFA was about 1%.

3.3.2 Transesterification Reaction

The production of biodiesel from jatropha oil and methanol was carried out in a 1000ml round bottom flask reactor equipped with condenser and placed on a constant temperature magnetic stirrer at atmospheric pressure. 500g of esterified oil was weighed into the reactor and preheated to the required temperature and then added a mixture catalyst and methanol, while stirring at 300rpm for a set reaction time. The product from the reactions was poured into a separating funnel and allowed to settle into two very distinct layers of biodiesel and glycerol.

3.3.3 Optimization of biodiesel Synthesis from Jatropha, Waste cooking oil and Neem Oil

The production of biodiesel from jatropha, neem and waste cooking oil blend was optimized using Design Expert version 13.0.6. Response surface methodology (RSM) was adopted in a box behnken design (BBD). The statistical program was used for regression and model fit analysis of the data obtained and used to estimate the coefficient of the regression equation and the analysis of variance (ANOVA) of selected factors. Factors conducted were reaction temperature (40 – 80^{o}C), reaction time (30 – 150 minutes), catalyst load (1 – 10wt%) and methanol to oil mole ratio (3:1 – 10:1). The deign generated 29 experimental runs. The selected process parameters to produce biodiesel were coded and actual variable in levels as displayed in Table 3.2

Table 3.2 Coded and actual levels of four factors Box Behnken Design (BBD)

Variables	Symbol	Coded and actual levels		
		-1	0	+1
Reaction temperature (^{o}C)	A	40	60	80
Reaction time (minutes)	B	30	90	150
Catalyst load (wt%)	C	1	5.5	10
Methanol to oil mole ratio	D	3	6.5	10

3.3.4 Crude Biodiesel Purification

After obtaining the maximum separation, the crude biodiesel was purified by warm water washing with distilled water using a separating funnel. Since both glycerol and methanol are highly soluble in water, crude biodiesel was mixed with distilled water and agitated gently to avoid formation of emulsion, then slowly percolating droplets of water through the ester (Atadashi et al. 2011). The process was repeated until colourless wash water was obtained, indicating complete removal of impurities.

3.4 Biodiesel Characterization

ASTM method was be used to characterize the physical and chemical properties of biodiesel. The various tests to be done are described below.

3.4.1 Acid Value Determination (ASTM D 664)

Exactly 0.1M of KOH solution was prepared by dissolving 5.61g KOH (pellet) with 1000ml distilled water. Furthermore, a mixture of 99.7% pure ethanol and 98% pure benzene in a ratio of 1:1 by volume was prepared by mixing 50 ml benzene and 50 ml of ethanol. About 1g of the oil was weighed and dissolved in the mixture of ethanol and benzene. The solution was titrated with 0.1N KOH solution in presence of 2 drops of phenolphthalein as indicator until the end point with the appearance of a pale permanent pink. The titre volume of 0.1N KOH (V) was noted. The total acidity (acid number) in mgKOH/g was calculated using the following equation

$$AV = \frac{MW \times N \times V}{W}$$

Where:

MW ≡ Molecular weight of potassium hydroxide (56.1g).

N≡ Normality of potassium hydroxide solution (0.1 N).

V≡ Volume of potassium hydroxide solution used in titration.

W ≡ Weight of oil sample.

$$\%FFA \cong \frac{AV}{2}$$

3.4.2 Peroxide Value Determination Method

About 5g of the sample was weighed into a conical flask. 50ml of 3:2 acetic acid and chloroform was added. This was stirred (swirl) to dissolve. 0.5ml of saturated (10% in water) KI solution was then added with constant shaking for about 1 minute. 30ml distilled water was then added. The mixture was immediately titrated with 0.1N sodium thiosulphate with constant and vigorous shaking until the disappearance of the yellow iodine colour. 0.5ml starch indicator was added and the titration was continued with constant agitation to liberate all the iodine from the solvent layer. Thiosulphate solution was then added drop wise until the disappearance of the blue colour. Blank titration was then conducted on the reagents with exactly 0.1ml of the 0.1N sodium thiosulphate solution. The peroxide value was thus estimated from the formula in M*eq*/Kg

$$PV = \frac{(S - B) \times N \times 1000}{weight.of\ .oil}$$

Where:

S = Sample titre value

B = Blank titre value

N = mol of thiosulphate

3.4.3 Moisture Content

The moisture content of oil was quantitatively determined by oven drying method at 110^{o}C for about 1 hour. 5g of oil was weighed in a crucible using the electronic mass balance. The weight of the crucible and oil obtained together was placed in an oven at 110 degrees. At time intervals of 10 minutes, the crucible with oil was taken out and weighed with a new mass for both oil and crucible obtained. The process was continued till constant weight of the oil was obtained respectively. The moisture content was calculated using the following equation.

%moisture $= \frac{Wm - Wd}{wm} x100$

Wm = weight of moist sample

Wd = weight of dry sample

3.4.4 Density and Specific Gravity Determination

The density was measured according to the ASTM D1298 standard while the specific gravity was determined according to the ASTM D1217 standard. A 50ml SEDI-M pycnometer bottle was washed thoroughly with detergent, water and petroleum ether, it was then oven dried and weighed. The bottle was filled with distilled water and weighed, the bottle was then dried and filled with the oil sample and weighed. The specific gravity is the mass of the oil weighed divided by the mass of water weighed and the density of the oil was equal to mass of the oil per unit volume.

3.4.5 Flash Point Determination (ASTM D 93)

The flash point of biodiesel was determined using Pensky Martens Closed Cup method (Figure 3.1). The cup was filled with the biodiesel up to the mark (about 75 ml) and placed in the tester. The machine was then set to heat at about 5^oC/min until a rise in temperature with simultaneous stirring was observed. Small open flame was maintained from an external supply of petroleum gas. Periodically, the flame was passed over the surface of the oil. When the flash temperature is reached the surface of the oil catch flame, the temperature at the moment was noted and thus reported as flash point temperature.

Figure 3.2: SYD-261 Pensky Martens Closed Cup Flash Point Tester

3.4.6 Viscosity Determination (ASTM D 445)

Brookfield NDJ-5S Rotary viscometer was used in the determination of viscosity. The appropriate spindle number was identified selected for the test sample and gently mounted on the machine. A 250ml beaker was cleaned and the sample was poured up to the 200ml mark. The beaker was then placed on a water bath with temperature preset at constant 30^{o}C and allowed to equilibrate for 10 minutes. The spindle and the temperature sensor of the machine were then lowered into the sample and the power button was turned on. The appropriate spindle number and speed were selected on the display screen and followed by the run button. The machine was then allowed to read the viscosity until a stable value is obtained and recorded.

Figure 3.3: Digital rotary viscometer

3.4.7 Fatty Acid Composition Determination (ASTM method D1983-90)

Fatty acids are of two types: saturated and unsaturated. The composition of fatty acids in synthesized biodiesel will be determined using gas chromatograph mass spectrometer (GC-MS).

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Results of Physical and Chemical Analysis of Jatropha, Neem and Waste cooking Oil Blend

The results of oils blend analysis are as shown in Table 4.1. The amount of FFA in the oil was found to be 20.65% for jatropha, neem and waste cooking oil blend. Studies have shown that high FFA reduces catalyst effectiveness and decreases the production yield; therefore, the recommended amount of FFA should not exceed 3wt% (Kawentar and Budiman 2013; Sarno and Iuliano 2019). In contrast, Bharti et al. (2019) stated that heterogeneous catalysts have proven to catalyze oil with higher FFA content of about 6 - 15% without any pre-treatment of oil. The effective transesterification in the current study on the oils without any pretreatment can be attributed to the high basic strength of CaO (Sarno and Iuliano 2019). The moisture content of the oil blend (0.21wt%) were less than the amount (≥0.5wt%) indicated in the literature to reduce biodiesel yield (Okullo et al. 2012).

Table 4.1: Physical and Chemical Properties of Oil Blend

Properties (Unit)	Oil Blend
Density (g/cm^3)	0.93
Moisture content (wt%)	0.21
Flash point (^{o}C)	176
Acid value (mgKOH/g)	41.3
FFA content (%)	20.65
Saponification value (mgKOH/g)	188.652

4.2 Properties of Biomass Derived Catalyst

The physical properties of the waste biomass derived catalyst are summarized in Table 4.2. It was observed that the properties resulted in a strong activity in the reactions. Table 4.6 shows the properties of the biodiesel produced using biomass derived catalyst at the optimum conditions of all variables.

Table 4.2 Properties of Biomass Catalyst

Calcined Biomass catalyst	Value
Surface Area	86.10 m^2/g
Bulk Density	1.285 g/cm^3
Particle Size	< 100µm
Porosity	48%

The catalyst was characterized to have large surface area of 86.1m^2/g which allow reactants to diffuse easily into the interior of the catalyst (Bharti et al. 2019). Jain and Sharma (2010) reported that a high surface area is desirable for better diffusion of reactant and product molecules. The observation is in agreement with that of Ordóñez and Díaz (2009) with a slightly higher (0.76cm^3/g) pore volume of biomass derived catalyst.

Table 4.3: Box Behnken Experimental Design

Run	Catalyst (wt%)	Time (minute)	Temperature (℃)	Methanol Ratio	Biodiesel Yield (wt%)
1	10	90	60	10	78.54
2	5.5	90	60	6.5	82.37
3	10	90	60	3	37.76

4	5.5	90	60	6.5	92.37
5	5.5	150	60	10	65.83
6	5.5	30	60	10	32.04
7	5.5	90	80	3	38.62
8	10	150	60	6.5	63.37
9	5.5	90	80	10	78.82
10	1	90	60	3	24.14
11	1	150	60	6.5	33.16
12	1	30	60	6.5	16.63
13	5.5	150	40	6.5	55.24
14	5.5	150	80	6.5	69.17
15	5.5	90	40	10	37.69
16	5.5	30	40	6.5	31.22
17	5.5	30	60	3	35.52
18	1	90	80	6.5	28.39
19	10	90	40	6.5	54.51
20	5.5	90	40	3	42.97
21	5.5	90	60	6.5	92.37
22	5.5	30	80	6.5	36.45
23	1	90	40	6.5	31.18
24	5.5	90	60	6.5	92.37
25	10	90	80	6.5	67.08
26	5.5	150	60	3	38.95
27	10	30	60	6.5	30.15
28	1	90	60	10	22.05
29	5.5	90	60	6.5	92.37

Table 4.4: BIODIESEL YIELD ANOVA for Quadratic model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	16293.24	14	1163.80	45.35	< 0.0001	significant
A-Catalyst load	2577.23	1	2577.23	100.43	< 0.0001	
B-Reaction time	1721.05	1	1721.05	67.07	< 0.0001	
C-Temperature	359.93	1	359.93	14.03	0.0022	
D-Mole Ratio	784.25	1	784.25	30.56	< 0.0001	
AB	69.64	1	69.64	2.71	0.1217	
AC	58.98	1	58.98	2.30	0.1518	
AD	459.46	1	459.46	17.90	0.0008	
BC	18.92	1	18.92	0.7374	0.4050	
BD	230.43	1	230.43	8.98	0.0096	
CD	517.11	1	517.11	20.15	0.0005	
A^2	5101.16	1	5101.16	198.78	< 0.0001	
B^2	4200.35	1	4200.35	163.68	< 0.0001	
C^2	1985.54	1	1985.54	77.37	< 0.0001	
D^2	3224.82	1	3224.82	125.67	< 0.0001	
Residual	359.26	14	25.66			
Lack of Fit	279.26	10	27.93	1.40	0.4002	not significant
Pure Error	80.00	4	20.00			
Cor Total	16652.51	28				

The Model F-value of 45.35 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Probability value (P-values) less than 0.0500 indicate model terms are significant. In this case A, B, C, D, AD, BD, CD, A^2, B^2, C^2, D^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The Lack of Fit F-value of 1.40 implies the Lack of Fit is not significant relative to the pure error. There is a 40.02% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Table 4.5: Fit Statistics

R^2	0.9784
Adjusted R^2	0.9569
Predicted R^2	0.8959
Adeq Precision	20.8464
Std. Dev.	5.07
Mean	51.77
C.V. %	9.79

The Predicted R^2 of 0.8959 is in reasonable agreement with the Adjusted R^2 of 0.9569; i.e. the difference is less than 0.2.

Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 20.846 indicates an adequate signal. This model can be used to navigate the design space.

Final Equation in Terms of Coded Factors

$$\begin{aligned} Biodiesel\ yield\ (wt\%) \\ &= 90.37 + 14.65A + 11.98B + 5.48C + 8.08D + 4.17AB + 3.84AC \\ &+ 10.72AD + 2.17BC + 7.59BD + 11.37CD - 28.04A^2 - 25.45B^2 - 17.50C^2 \\ &- 22.30D^2 \end{aligned}$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

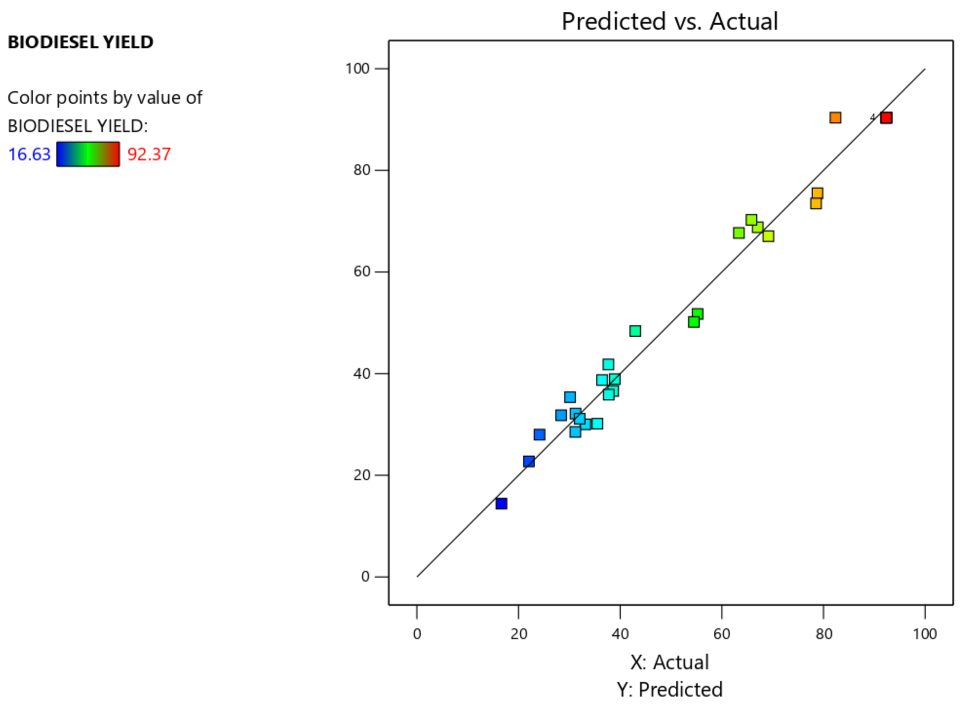


Figure 4.1: graph of predicted against actual

This **predicted** vs. **actual** plot evaluate the accuracy of the model used to predict biodiesel yield(wt%) based on the experimental data. The data point are closely aligned with the diagonal line (y = x), indicating a high predictive accuracy. This suggests that the model effectively captures the relationship between process variables and biodiesel yield. The model is highly accurate, with only minor deviations. A high coefficient of determination(R^2) would likely confirm strong predictability. The presence of some outliers suggests that refining the model (e.g. considering additional factors or non-linear interactions) could further improve accuracy.

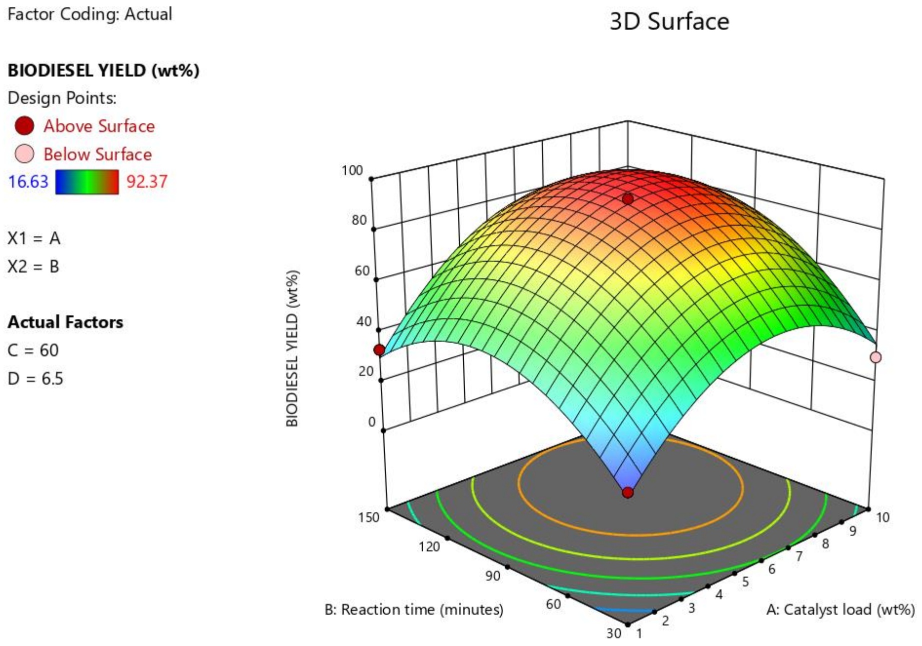


Figure 4.2 Response surface plot of reaction time and catalyst load effects on biodiesel yield

The figure above is an image of a 3D surface plot representing the effect of two factors- reaction time(mins) and catalyst load(wt%) on biodiesel yield in a chemical reaction. The surface follows a parabolic shape, indicating an optimal region where biodiesel yield is maximized. The red zone(top region) represent higher yields(~92.37wt%) and the blue zone(bottom region) represent lower yields(~16.63wt%). The concentric contour lines indicate how biodiesel yield changes with different combinations of reaction time and catalyst load. The central bright-coloured region(yellow-orange) represents the highest yield. Interpreting the graph: the biodiesel yield increases with both catalyst load and reaction time reaching an optimum point. After a certain limit, excessive catalyst load or reaction time does not further improve the yield. This graph helps optimize the biodiesel production process by determining the ideal conditions.

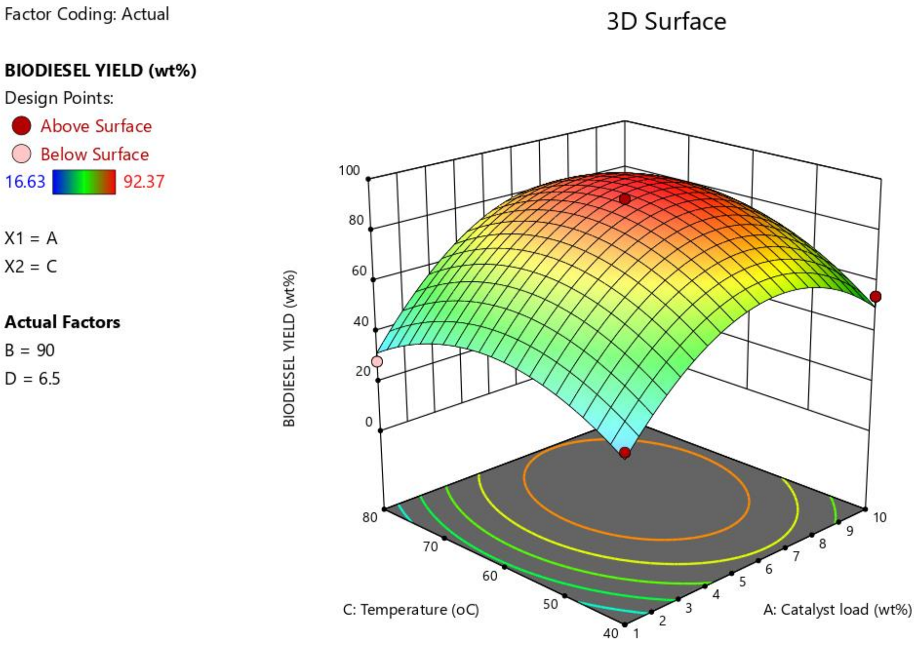


Figure 4.3 Response surface plot of reaction temperature and catalyst load effects on biodiesel yield

From the figure above , the image presents a 3D surface plot showing the relationship between biodiesel yield(wt%), temperature(^{0}C) and catalyst load(wt%). The colour gradient on the surface indicates the yield values, ranging from 16.63 wt% (blue) to 92.37 wt% (red). The bodieesel yield increases as both temperature and catalyst load increase, reaching a peak before slightly declining at extreme values. The highest yield(~92.37wt%) is found in the upper-center region(red zone), suggesting an optimal range for reaction conditions. The lowest yield(~16.63wt%) appears in the lower-left region(blue zone), where both the catalyst load and temperature are at their minimum. The contour lines at the bottom provide a 2D representation of the response surface. The closer the contour lines, the steeper the gradient, meaning a more significant change in biodiesel yield. Interpreting the graph: the plot suggests that moderate to high temperatures(around 70-80^{0}C) and catalyst load(6-8wt%) optimize biodiesel yield. Too low or too high values of either factor may result in reduced efficiency. This visualization is useful for process optimization in biodiesel production, guiding researchers in selecting the best operating conditions.

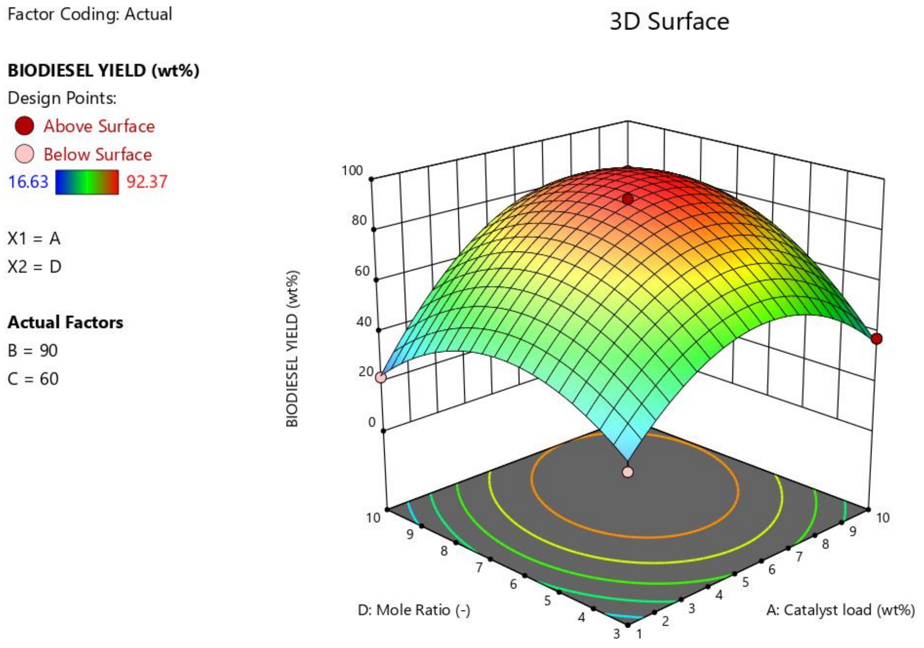


Figure 4.4 Response surface plot of methanol to oil mole ratio and catalyst load effects on biodiesel yield

The figure is a 3D surface plot that illustrates the relationship between biodiesel yield(wt%), catalyst load(wt%) and mole ratio(D) while keeping B=90 and C=60 constant. The yield follows a parabolic trend, increasing with higher catalyst load and mole ratio but reaching a peak before slightly declining. The highest biodiesel yield(~92.37wt%) occurs at a moderate to high values of both parameters(red region). The lowest yield(~16.63wt%) is observed in the lower left region where both variables are at their minimum(blue zone). Increasing the catalyst load improves biodiesel yield to an optimum point. Excess catalyst might cause soap formation, which could reduce efficiency. The mole ratio(D) likely methanol to oil ratio has a similar effect. A higher ratio enhances transesterification but beyond a certain limit, excess methanol can dilute the reaction and make product recovery difficult. The contour lines at the bottom indicate the gradient of change, densely packed contour lines suggest a steep change in biodiesel yield, while wider spacing indicates a more gradual response. Interpreting the plot: the optimum biodiesel yield is achieved at a balanced catalyst load and mole ratio. Too low values lead to poor conversion, while too high values may cause side reactions or inefficient separation. This study helps refine conditions for maximum biodiesel production.

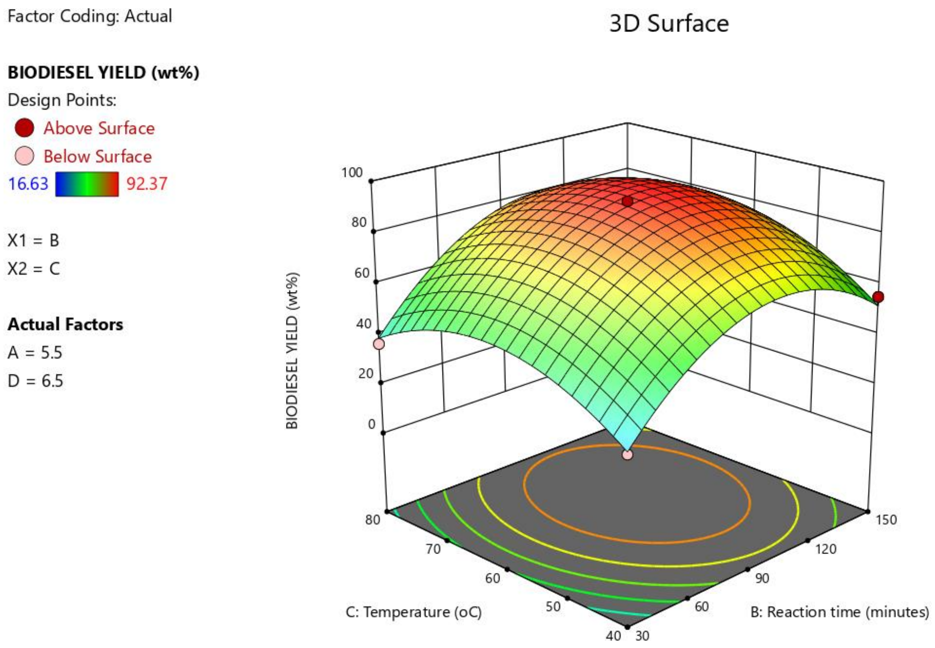


Figure 4.5 Response surface plot of reaction temperature and reaction time effects on biodiesel yield

From the figure above the 3D surface plot explores the impact of reaction time(B, mins) and temperatures(C ^{0}C) on biodiesel yield(wt%), while keeping catalyst load(A=5.5wt%) and mole ratio(D=6.5) constant. Biodiesel yield increases with bith temperature and reaction time up to an optimum level. Beyond this peak, further increases in reaction time might lead to side reactions such as soap formation or degradation of biodiesel components. Similarly, too high temperatures may promote undesired thermal degradation of reactants. The highest yields(~92.37wt%) appears in the upper center region(red zone), where temperature and reaction time are in their optimal range. The lowest yield(16.63wt%) is found in the lower left region(blue zone), where both factors are at their minimum. The concentric contour lines indicates a clear peak, suggesting a well-defined optimal reaction condition. Tightly packed contour lines mean a rapid change in yield with respect to temperature and time. Interpreting the plot: optimal biodiesel yield is achieved at moderate to high temperature(~70-80^{0}C) and reaction time(~90-120 mins). Excessive reaction time does not necessarily improve yield and may even cause side reactions. This plot helps in determining the best operating conditions to maximize biodiesel production efficiency.

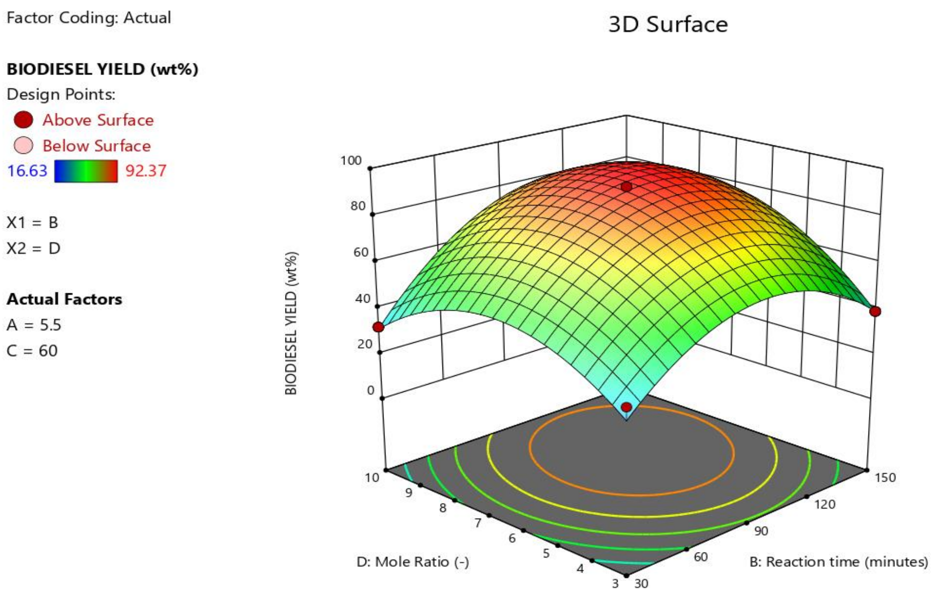


Figure 4.6 Response surface plot of methanol to oil mole ratio and reaction time effects on biodiesel yield

From the above image, the 3D surface plot examines the effect of mole ratio(D) and reaction time(B) on biodiesel yield(wt%), while keeping catalyst load(A=5.5wt%) and temperature(C=60^0C)constant. Biodiesel yield increases initially with both reacton time and mole ratio. However, beyond a certain point, excessive reaction time or mole ratio does not lead to further improvement in yield and may cause side reactions or losses. The peak yield(~92.37wt%) is observed in the red zone, which represents the beest combination of mole ratio and reaction time. The lowest yield(~16.63wt%) is observed at the lowest mole ratio abd reaction time. The concentric contours at the bottom indicate a clear optimal region. The presence of widely spaced contour lines suggests a gradual change inn biodiesel yield with respect to mole ratio and reaction time. Interpreting the plot: optimal biodiesel yield is obtained at moderate to high mole ratio (6-9) and reaction time(~90-120 mins). Excessive mole ratio may lead to difficulties in phase separation, increasing costs for methanol recovery. Longer reaction time do not always improve yield, and prolonged exposure may cause biodiesel degradation. This plot provides insight into how reaction time and mole ratio interact to maximize biodiesel yield.

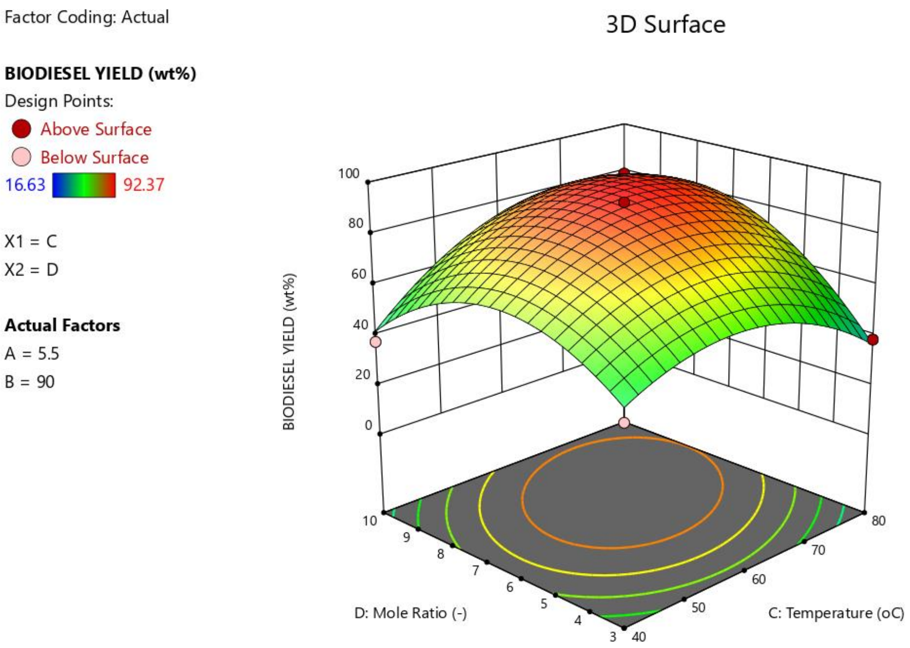
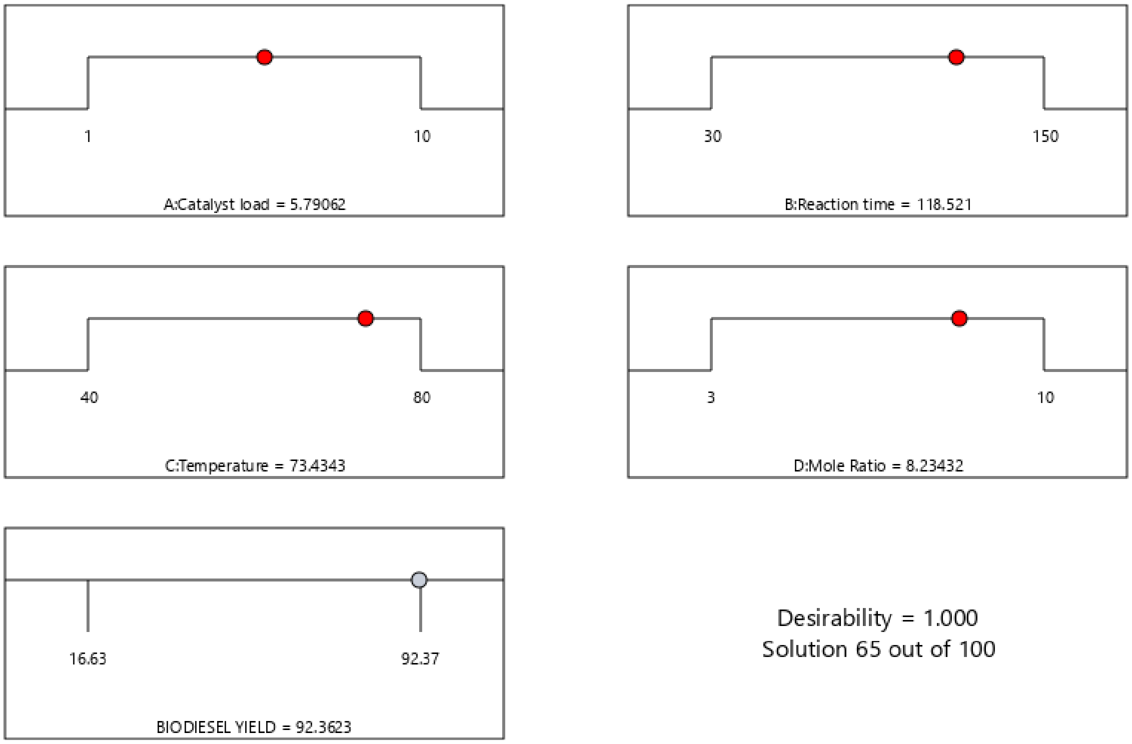


Figure 4.7 Response surface plot of methanol to oil mole ratio and reaction temperature effects on biodiesel yield

This 3D surface plot explores the effect of temperature(C) and mole ratio(D) on biodiesel yield(wt%), while keeping catalyst load(A=5.5wt%) and reaction time(B=90 mins) constant. Biodiesel yield initially increases with temperature and mole ratio, reaching a peak in the red zone. Beyond an optimal range, further increases in temperature or mole ratio do not significantly enhance yield and may lead to undesirable side reactions. The highest yield(~92.37wt%) occurs at moderate to high mole ratio(6-9) and temperature(60-70^{0}C). lower values for these parameters result in reduced biodiesel yield(~16.63wt%). The circular contours at the base indicate a distinct optimal region. More widely spaced contours suggest a gradual effect, meaning the process is not highly sensitive to small variations in these factors. Interpreting the plot: the best biodiesel yield is achieved at temperature between 60-70^{0}C and a mole ratio of around 6-9. Excessively high temperature(>70^{0}C) can cause methanol evaporation, reducing the reaction efficiency. A very high mole ratio may increase unreacted methanol, complicating biodiesel purification. This analysis helps determine the optimal transesterification conditions.

4.3: Result of Optimum Biodiesel Production from Oil Blend



4.4 Physical and Chemical Properties of Jatropha, Neem and Waste cooking Oil Blend Biodiesel

The quality of biodiesel after water washing the properties determined using the American Society for Testing and Materials standard (ASTM). ASTM identifies the parameters that pure biodiesel (B100) should fulfil before being used as a pure fuel or blended with petroleum diesel fuel (Silitonga et al. 2013). The summarized results (Table 4.4) show that all of the measured values were in the range of test limit.

Table 4.6 Physical and chemical properties of Jatropha, Neem and WCO Blend Biodiesel

Properties Measured (Unit)	Jatropha, Neem and WCO Blend Biodiesel	Standard ASTM
Density @ 30^{o}C (g/ml)	0.8794	NA
Sp. Gravity	0.8681	0.88
Dyn. Viscosity (mPa.s)	4.01	1.9 – 6.0
Kin. viscosity @ 30^{o}C (mm^2/s)	5.97	NA
Flash Point (^{o}C)	133	100 – 170
Moisture Content (%)	0.102	NA
Acid value (mgKOH/g)	0.418	<0.5
FFA (%)	0.264	NA

The transesterification reaction which was conducted using the esterified oil blend yielded optimum biodiesel with an acid value and water content of 0.418 and 0.431 mgKOH/g. Flash point was within the standard range at 133^{o}C. The viscosities and other properties (Table 4.6) of the biodiesel quality assessment revealed that the values were within range.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

This study successfully demonstrated the potential of biomass-derived catalysts for the production of biodiesel from a blend of non-edible oils, specifically neem, shea, and jatropha. The research addressed key challenges in biodiesel production, such as the reliance on non-renewable catalysts, high production costs, and environmental concerns, by utilizing agricultural waste materials (plantain peels and coconut husks) to develop sustainable and cost-effective catalysts. The optimization of the transesterification process using the Box-Behnken Design (BBD) revealed that the optimal conditions for maximizing biodiesel yield were a reaction temperature of 60°C, a reaction time of 90 minutes, a catalyst load of 5.5 wt%, and a methanol-to-oil mole ratio of 6.5:1, achieving a yield of 92.37%.

The physical and chemical properties of the produced biodiesel, including density, viscosity, flash point, and acid value, were found to comply with ASTM standards, confirming the high quality and suitability of the biodiesel for use as an alternative fuel. The biomass-derived catalysts exhibited excellent catalytic activity, with a high surface area and porosity, which facilitated efficient transesterification reactions. This study underscores the environmental and economic benefits of using biomass-derived catalysts, as they not only reduce waste but also promote the utilization of locally available agricultural by-products.

In conclusion, this research highlights the feasibility of producing biodiesel from non-edible oil blends using sustainable biomass-derived catalysts. The findings contribute to the broader goal of transitioning to renewable energy sources by providing a practical and eco-friendly approach to biodiesel production. Future research should focus on scaling up the process, exploring additional oil blends, and conducting lifecycle assessments to further validate the sustainability and economic viability of this approach. Overall, this study offers valuable insights into the development of innovative and sustainable biodiesel production methods, paving the way for a greener and more sustainable energy future.

5.2 RECOMMENDATIONS

Based on the findings and implications of this research, the following recommendations are proposed:

1. Optimization of Oil Blends:

Future studies should focus on exploring additional oil blends beyond those tested in this research. Evaluating the performance of other non-edible oil sources could further improve biodiesel yield and quality while promoting sustainable agricultural practices.

2. Enhancement of Catalyst Efficiency:

Continued research into optimizing biomass-derived catalysts is crucial. Investigating modifications to enhance the surface area and catalytic properties could lead to more efficient transesterification processes and better yield outcomes.

3. Long-Term Performance Testing:

Further research should assess the long-term stability and performance of biodiesel blends produced from various oil types stored under different environmental conditions. Understanding how storage affects biodiesel properties will be essential for commercial applications.

4. Lifecycle Analysis:

Conducting a lifecycle analysis of biodiesel production processes will provide insights into the environmental impacts associated with the entire production cycle. This could inform policy decisions and investment strategies aimed at promoting renewable energy sources.

5. Policy Support:

Stakeholders should advocate for policies that encourage the use of biomass-derived catalysts and non-edible oils in biodiesel production. This can help support local farmers and promote sustainable practices in the agricultural sector.

5.3 Future Research Directions

The findings of this research open up multiple avenues for future studies. Some potential areas for exploration include:

1. Enzymatic Transesterification:

Investigating the application of enzymes as a catalyst for transesterification may provide insights into more sustainable and economically viable biodiesel production methods.

2. Blending Techniques:

Further analysis of blending methods and technologies, such as double transesterification, could enhance the quality and yield of biodiesel from feedstock with high free fatty acid content.

3. Integrated Waste Management:

Exploring the integration of biodiesel production with waste management systems can lead to innovative solutions that optimize resource utilization while minimizing environmental impacts.

4. Economic Feasibility Studies:

Conducting comprehensive economic assessments of biodiesel production processes will help determine the market viability and acceptance of biodiesel derived from various biomass sources.

5. Policy and Implementation Studies:

Investigating the ramifications of current policies on biofuel production and identifying potential strategies for favorable legislation that supports the growth of sustainable biodiesel markets.

APPENDIX

Calculations

Density of oil blend:

Density = mass/volume

Mass of empty bottle = 31.03g

Mass of oil + bottle =77.53g

Volume of bottle = $50cm^3$

Density = $(77.53-31.03)g/50cm^3$

Density = $0.93g/cm^3$

Acid value of oil blend:

A.V = X(M.W)N/W

Where

X= S-b

S= titre value

b= normality

W = weight of oil

N= no of moles

A.V = (24.5-0.1) *56.1*0.05/1.66

A.V = 41.3mgKOH/g

%FFA of oil blend:

%FFA = A.V/2

%FFA = 41.3/2

%FFA = 20.65

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