

**COMPARATIVE STUDIES ON THE USE OF MAGNESIUM OXIDE AND
MANGANESE MAGNESIUM BINARY OXIDE NANO PARTICLES AS A CATALYST
IN THE PRODUCTION OF BIODIESEL FROM WASTE COOKING OIL**



BY

ENOYOGIERE EDENAYAGOSA GODSON

PSC2007975

DEPARTMENT OF CHEMISTRY

FACULTY OF PHYSICAL SCIENCES

UNIVERSITY OF BENIN

BENIN CITY

FEBRUARY, 2025

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EDENAYAGOSA GODSON ENOYOGIERE

PSC2007975

A RESEARCH PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMISTRY,
FACULTY OF PHYSICAL SCIENCE, UNIVERSITY OF BENIN IN PARTIAL
FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF BACHELOR OF
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FEBRUARY, 2025.

CERTIFICATION

This is to certify that this project work was carried out by Edenayagosa Godson Enoyogiere with the matriculation number PSC2007975 in the department of Chemistry, Faculty of Physical Sciences, University of Benin, Edo State, Nigeria.

EDENAYAGOSA GODSON ENOYOGIERE

(Student)

DATE

PROF J. M. OKUO

(Project supervisor)

DATE

PROF. E.E.E I. IRABOR

(Head of department)

DATE

DEDICATION

I dedicate this project, first and foremost, to God Almighty, whose grace, strength, and unfailing mercy have carried me through this journey. In times when I felt helpless, He remained my source of hope and guidance, making a way where there seemed to be none. To my wonderful parents, Mr. and Mrs. Enoyogiere Agbomnwanre, whose unwavering love, sacrifices, and support have been my greatest motivation. Your belief in me has shaped my journey, and for that, I am forever grateful. To my Amazing Siblings, Mr Godspower Enoyogiere, Mrs Faith Osasumwen and Mr Favour Enoyogiere, whose encouragement and companionship have been a constant source of strength. Lastly, I dedicate this project to myself—for not giving up against all odds, even when the journey was tough. Despite the challenges, I persevered, stayed committed, and pushed forward. The challenges may have been many, but so were the lessons, the growth, and the victories along the way.

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ABSTRACT

The production of biodiesel from waste cooking oil presents a sustainable approach to waste management and alternative fuel generation. This study investigates the use of Manganese Magnesium Binary Oxide (MMBO) nanoparticles as a catalyst for biodiesel synthesis. The physicochemical properties of waste cooking oil were analyzed before and after esterification, revealing significant reductions in acid value (2.973 mg KOH/g to 1.85 mg KOH/g) and free fatty acid content (1.4865 mg KOH/g to 0.925 mg KOH/g), which improved feedstock suitability for biodiesel production.

A comparative analysis of biodiesel produced using MgO and MMBO nanoparticles demonstrated the superior catalytic efficiency of MMBO-NP, yielding 84.6% biodiesel compared to 65% with MgO. The biodiesel produced with MMBO-NP exhibited favorable fuel properties, including a lower kinematic viscosity (3.89 mm²/s), reduced acid value (0.35 mg KOH/g), and improved density (875 kg/m³), aligning with ASTM D6751 and EN 14214 standards.

Despite meeting most standard requirements, the flash point (77°C) and cold flow properties (cloud point: 32°C, pour point: 39°C) indicate potential areas for further optimization. These results highlight the potential of MMBO nanoparticles as an efficient catalyst in biodiesel production, promoting a more effective and environmentally friendly conversion of waste cooking oil into biodiesel.

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CHAPTER ONE

1.1 INTRODUCTION

Energy security, environmental protection, and the reduction of greenhouse gas emissions have become critical concerns as global fossil fuel demand continues to rise. The urgency of these issues has accelerated the development of renewable alternatives, which offer more environmentally friendly solutions (Wang *et al.*, 2023). Among these alternatives, biodiesel has emerged as a promising option, offering several advantages. Derived from renewable sources, it is biodegradable, non-toxic, and emits fewer hazardous pollutants compared to conventional fossil fuels (Malik *et al.*, 2024).

Waste Cooking Oil has become one of the most attractive feedstocks used in biodiesel production due to the fact that it offers a cost-effectively feasible route and serves as an environmentally friendly way of recycling what would have otherwise been a waste product burden to the environment (Abdullah *et al.*, 2013). However, the transesterification process needs an efficient catalytic system for Waste Cooking Oil biodiesel production in spite of high Free Fatty Acid content.

Recent advancements in nanotechnology have paved the way for the development of highly efficient and selective catalysts for biodiesel production (Togun *et al.*, 2025). Among these, metal oxide nanoparticles have garnered significant attention due to their high surface area, enhanced catalytic activity, and tunable properties (Uyiosa *et al.*, 2024). In particular, manganese magnesium oxide (MnMgO) nanoparticles have shown promise as a bifunctional catalyst,

offering both basic and acidic sites necessary for effective transesterification and esterification reactions (Farouk *et al.*, 2024)

The use of MnMgO nanoparticles as a catalyst addresses several challenges in biodiesel production. These nanoparticles exhibit high thermal stability, reusability, and resistance to saponification, making them suitable for processing low-grade feedstocks like Waste Cooking Oil. Moreover, their unique physicochemical properties enable the conversion of Free fatty acids and triglycerides into biodiesel with high yield and purity under optimized reaction conditions (R. Dayana, 2023)

This research will involve the production of biodiesel from waste cooking oil using MnMgO nanoparticles as a catalyst. The project looks into the efficiency, reusability, and environmental impact that MnMgO nanoparticles create in biodiesel synthesis. In this study, the use of Waste Cooking Oil as feedstock and MnMgO nanoparticles as a catalyst will help in achieving the aim of contributing toward the development of cost-effective and environmentally sustainable technologies for biodiesel production.

1.2 BACKGROUND OF STUDY

The global reliance on fossil fuels has led to significant environmental concerns, including greenhouse gas emissions, air pollution, and climate change (Wang *et al.*, 2023). As energy demand continues to rise, the depletion of non-renewable resources has become a critical issue, prompting researchers to explore sustainable energy alternatives. Among various renewable energy sources, biodiesel has gained attention as an eco-friendly substitute for petroleum diesel due to its biodegradability, lower carbon footprint, and compatibility with existing diesel engines

(Demirbas, 2022). Biodiesel is primarily produced through transesterification, a chemical reaction that converts triglycerides in oils or fats into fatty acid methyl esters (FAMEs) using alcohol and a catalyst (Atadashi *et al.*, 2020).

One of the major challenges in biodiesel production is the high cost of feedstock, which accounts for approximately 70–80% of the total production cost (Balajii & Niju, 2020). To address this issue, researchers have focused on low-cost and sustainable feedstocks such as waste cooking oil (WCO), which is abundantly available and reduces environmental pollution (Kumar *et al.*, 2021). The utilization of Waste Cooking Oil not only minimizes disposal problems but also contributes to a circular economy by converting waste into valuable biofuel. However, typically contains high levels of free fatty acids (FFAs), leading to soap formation when using traditional homogeneous catalysts like sodium hydroxide (NaOH) or potassium hydroxide (KOH), which reduces biodiesel yield and requires additional purification steps (Lee & Saka, 2021).

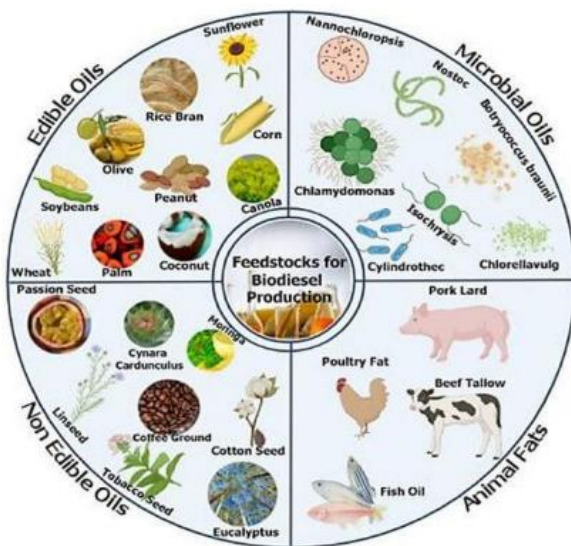


Figure 1. Biodiesel from different feedstock (Source: Google images)

To overcome these limitations, heterogeneous nanocatalysts have been proposed as an efficient alternative due to their reusability, stability, and environmentally friendly properties (Sharma *et al.*, 2022). Among these catalysts, Manganese Magnesium Oxide (Mn-MgO) nanoparticles have shown great potential due to their strong basicity, high surface area, and thermal stability, which enhance catalytic performance in transesterification reactions (Malik *et al.*, 2024). Magnesium oxide (MgO) is a well-known solid base catalyst with high efficiency in biodiesel synthesis, while the incorporation of manganese (Mn) enhances its structural and catalytic properties, improving biodiesel yield and reaction rates (Patil *et al.*, 2023). The nanostructured form of these catalysts further increases their efficiency by providing a larger surface area and more active sites for the reaction, making the process more economically viable (Zhang *et al.*, 2023).

This study aims to explore the production of biodiesel from waste cooking oil using Mn-MgO nanoparticles as a catalyst, focusing on optimizing reaction conditions, evaluating catalyst efficiency, and assessing the environmental benefits of using a heterogeneous nanocatalyst. The findings will contribute to the development of cost-effective, sustainable, and high-performance biodiesel production methods, reducing dependence on fossil fuels while addressing waste management challenges.

1.3 STATEMENT OF PROBLEM

Growing demand for energy worldwide, together with increasing concerns regarding environmental pollution and climate change, has made the search for cleaner, more sustainable fuel alternatives all the more pressing. Biodiesel is one such renewable, biodegradable fuel that promises a very good solution. Despite its advantages, large-scale production of biodiesel still

faces numerous challenges, including high production costs, inefficient catalytic processes, and sourcing of affordable raw materials.

One of the major challenges for biodiesel production is the feedstock cost, which comprises a great share of the overall cost. Conventionally, edible vegetable oils have been used, but their reliance is in direct competition with food supply and results in increased prices. A more feasible and sustainable alternative is waste cooking oil (WCO), which is generated in households and the food industry and is usually disposed of improperly, causing environmental nuisance. By reusing Waste cooking oil for biodiesel production, waste is reduced while the cost of production decreases. However, Waste cooking oil also has its challenges. The high content of free fatty acids and impurities in Waste cooking oil interferes with the chemical process and reduces efficiency, making purification more complicated. Another major problem includes the type of catalysts being used in the biodiesel production process. Conventional liquid catalysts such as sodium hydroxide (NaOH) and potassium hydroxide (KOH) are highly effective but due to their very high costs. These might result in the formation of unwanted byproducts like soaps, which make purification cumbersome with wastewater generation, hence increasing costs and the environmental impact. To solve such challenges, more efficient, reusable, and eco-friendly solid nanocatalysts should be considered.

Examples of such catalysts are : Manganese Magnesium Oxide nanoparticles (Mn-MgO). These materials have peculiar properties, including high surface area, strong basicity, excellent thermal stability, and enhanced catalytic activity, which improve the yield and efficiency in biodiesel. In

spite of such promising characteristics, scant studies on the effectiveness of such a catalyst in converting WCO into biodiesel are still at hand.

It intends to fill this knowledge gap through studying the performance of Mn-MgO nanoparticles as a catalyst in biodiesel production. Precisely, this present study shall:

assess the catalytic efficiency of Mn-MgO nanoparticles towards the enhancement in the yield and/or improvement in reaction rate in the production of biodiesel; find the optimal conditions concerning temperature, dosage of the catalyst, ratio between methanol and oil, and reaction time.

- Check the quality of the biodiesel produced to meet the standards for the fuel.
- Check the reusability and stability of the catalyst for economic and ecological viability.

This work addresses these challenges and wants to contribute to cleaner and more sustainable biodiesel production. If Mn-MgO nanoparticles prove to be efficient as catalysts, then it may make biodiesel cheaper and greener but at the same time give a practical way to manage waste cooking oil. This work could bring us yet another step closer to a greener future where renewable energy sources will play an ever-increasing role in satisfying global energy demand.

1.4 AIM AND OBJECTIVE OF THE STUDY

The aim of this project is to synthesize and evaluate biodiesel production from waste cooking oil (WCO) using manganese magnesium oxide (Mn-MgO) nanoparticles as a heterogeneous

catalyst. This study seeks to explore the catalytic efficiency of Mn-MgO nanoparticles in the transesterification process, optimizing reaction parameters to achieve high biodiesel yield while ensuring cost-effectiveness and environmental sustainability.

To achieve this aim, the following objectives were set:

- **Catalytic Performance** – Investigate the effectiveness of Mn-MgO nanoparticles in catalyzing the transesterification of waste cooking oil into biodiesel.
- **Optimization of Reaction Conditions** – Study the impact of variables like catalyst concentration, reaction time, temperature, and methanol-to-oil molar ratio on biodiesel yield.
- **Biodiesel Quality Analysis** – Assess the physicochemical properties of the produced biodiesel, including viscosity, density, acid value, and fuel properties, to ensure compliance with ASTM or EN standards.
- **Sustainability & Reusability** – Examine the reusability and stability of the Mn-MgO catalyst across multiple reaction cycles to determine its economic and environmental viability.

1.5 JUSTIFICATION OF THE STUDY

The world is facing a critical need for cleaner, renewable energy sources, especially as concerns over climate change, rising fuel prices, and environmental pollution continue to grow. At the same time, millions of liters of waste cooking oil (WCO) are improperly disposed of every day, clogging drainage systems, polluting water bodies, and harming ecosystems. This study bridges the gap between these two challenges by exploring a sustainable way to transform

waste into energy - producing biodiesel from Waste Cooking Oil using manganese magnesium oxide (Mn-MgO) nanoparticles as a catalyst.

Turning Waste into a Valuable Resource

Instead of allowing used cooking oil to go to waste or cause pollution, this study focuses on giving it a second life as an alternative fuel. Biodiesel made from Waste Cooking Oil can power diesel engines just like traditional fossil fuels, but with significantly lower emissions and a smaller carbon footprint (Demirbas, 2009). This approach not only reduces environmental damage but also helps to conserve valuable resources by reusing what would otherwise be discarded.

Making Biodiesel More Affordable

One of the biggest barriers to biodiesel production is cost. Traditional raw materials like vegetable oils can be expensive, making biodiesel less competitive with petroleum-based diesel (Nur *et al.*, 2024). However, Waste Cooking Oil is a low-cost and widely available alternative, making the production process far more economically viable. This means that biodiesel could become a more accessible and affordable fuel option for individuals, businesses, and industries alike. (None Monika, 2023)

Additionally, the use of Mn-MgO nanoparticles as a catalyst offers an exciting advantage. Unlike conventional chemical catalysts that wear out quickly and generate excess waste, Mn-MgO nanoparticles can be reused multiple times, cutting down on material costs and making the process even more cost-effective and environmentally friendly (Maria-Anna Gatou, 2024)

Advancing Green Technology with Nanoparticles

Nanotechnology is changing the way we approach energy solutions. The Mn-MgO nanoparticles used in this study have unique properties that speed up the biodiesel production process, improve fuel quality, and increase overall efficiency (Maria-Anna Gatou, 2024). By harnessing this cutting-edge technology, this study paves the way for more advanced, high-performance catalysts that could revolutionize the biofuel industry.

Contributing to a Sustainable Future

This study aligns with global sustainability efforts, including the United Nations Sustainable Development Goals (SDGs):

- **SDG 7 (Affordable and Clean Energy):** By promoting a cost-effective and renewable fuel source.
- **SDG 12 (Responsible Consumption and Production):** By turning waste into a valuable energy resource.
- **SDG 13 (Climate Action):** By reducing greenhouse gas emissions and promoting cleaner fuels.

1.6 SCOPE OF THE STUDY

This study focuses on the production of biodiesel from waste cooking oil (WCO) using manganese magnesium oxide (Mn-MgO) nanoparticles as a catalyst. The scope encompasses various aspects, including catalyst synthesis, biodiesel production optimization, product

characterization, and catalyst reusability analysis. The study will be conducted under controlled laboratory conditions to ensure the accuracy and reliability of results.

The research is structured to align with the key objectives as follows:

1. Synthesis & Characterization of Mn-MgO Nanoparticles

- The study involves the synthesis of Mn-MgO nanoparticles using a suitable method (e.g., sol-gel, co-precipitation).
- The structural, morphological, and textural properties of the catalyst will be analyzed using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Fourier Transform Infrared Spectroscopy (FTIR), and BET surface area analysis.
- This ensures that the catalyst possesses high surface area and active sites necessary for biodiesel production.

2. Catalytic Application in Biodiesel Synthesis

- The synthesized Mn-MgO nanoparticles will be applied as a heterogeneous catalyst in the transesterification of waste cooking oil into biodiesel.
- The study will optimize reaction parameters, including:
 - Methanol-to-oil molar ratio
 - Catalyst loading (%)
 - Reaction temperature & time

- Agitation speed
- The impact of each parameter on biodiesel yield and reaction efficiency will be investigated.

3. Biodiesel Characterization & Quality Assessment

- The produced biodiesel will be analyzed to determine key fuel properties, such as:
 - Viscosity
 - Density
 - Acid value
 - Ester content
 - Flash point
 - Cetane number
- The results will be compared with international biodiesel standards (ASTM D6751 and EN 14214) to evaluate its suitability for commercial use.

4. Catalyst Reusability & Sustainability Analysis

- The study will evaluate the recyclability of the Mn-MgO catalyst by using it in multiple transesterification cycles.
- Catalyst deactivation and possible structural changes will be examined using XRD and SEM after several reaction cycles.

- The economic and environmental sustainability of using Mn-MgO nanoparticles in biodiesel production will be assessed, with a focus on cost-effectiveness and waste reduction.

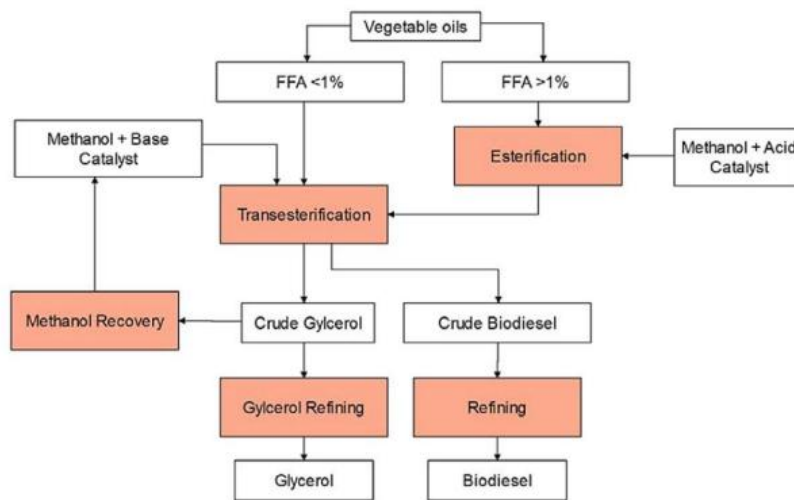
1.6 LITERATURE REVIEW

In recent years, the search for renewable and sustainable energy sources has been a major focus, and biodiesel production is at the forefront of this movement. One exciting development in this area is the conversion of waste cooking oil (WCO) into biodiesel. Not only does this approach help reduce waste disposal issues, but it also turns what is often considered pollution into a valuable resource. Waste Cooking Oil, typically discarded after use, can contain a significant amount of free fatty acids (FFAs) that make it harder to use in traditional biodiesel production methods (Demirbas, 2009). However, nanotechnology—particularly the use of nanocatalysts—has brought new possibilities to the process, offering a more efficient and environmentally friendly way of converting Waste Cooking Oil into biodiesel. One such nanocatalyst that is gaining attention is manganese magnesium oxide (Mn-MgO) nanoparticles, known for their high stability and effectiveness in promoting transesterification reactions. (Islam *et al.*, 2022)

Waste Cooking Oil: A Viable Feedstock for Biodiesel Production

Using Waste Cooking Oil as a feedstock for biodiesel production presents a dual benefit: it helps solve the issue of waste oil disposal and provides a sustainable alternative to conventional fossil fuels. Waste Cooking Oil is often improperly discarded, contributing to pollution, especially in water and soil (Demirbas, 2009). Converting it to biodiesel can reduce the harmful

environmental effects and offer a renewable energy source at the same time. However, Waste Cooking Oil typically contains high amounts of Free Fatty Acids, which complicate the transesterification process (None Monika, 2023). This is where heterogeneous catalysts, like Mn-MgO nanoparticles, come in, offering a solution to this problem by improving the reaction efficiency and biodiesel yield.



Transesterification process flow chart (Source: Google images)

Nanocatalysts: The Future of Biodiesel Production

The use of nanoparticles in catalysis has revolutionized many industries, and biodiesel production is no exception. Nanoparticles, due to their small size and high surface area, offer increased reactivity compared to traditional catalysts. This leads to faster reaction times and higher yields (Pandit *et al.*, 2023). Metal oxide nanoparticles, in particular, have shown great promise in biodiesel production due to their stability, reusability, and environmentally friendly nature (Bidir *et al.*, 2021). One of the most attractive features of nanoparticles is that they are

heterogeneous catalysts, which means they can be easily recovered and reused, reducing waste and operational costs (Mandari *et al.*, 2021)

Manganese Magnesium Oxide Nanoparticles: A Promising Catalyst

Manganese magnesium oxide (Mn-MgO) nanoparticles combine the properties of manganese (Mn) and magnesium (Mg), two elements that together create a catalyst that is particularly efficient in biodiesel production. The manganese oxide component contributes to the catalytic activity, while the magnesium oxide enhances the basicity and alkalinity, making the catalyst especially effective in Free Fatty Acids-rich feedstocks, like Waste Cooking Oil (Vishal Gadore, 2023). The combination of these two oxides gives Mn-MgO nanoparticles a unique advantage over other types of nanocatalysts, especially in terms of stability and performance under high temperatures.

Some key advantages of Mn-MgO nanoparticles include:

- **Thermal Stability:** These nanoparticles are thermally stable, meaning they can withstand high reaction temperatures without breaking down, making them suitable for industrial-scale applications
- **Reusability:** Mn-MgO nanoparticles have demonstrated excellent reusability, meaning they can be used in multiple reaction cycles without a significant loss of performance. This makes them a more cost-effective option compared to traditional homogeneous catalysts, which often need to be replaced after each cycle
- **Environmental Friendliness:** As heterogeneous catalysts, Mn-MgO nanoparticles can be easily separated from the reaction mixture, reducing the need for harsh chemicals and waste disposal (Sharma *et al.*, 2018).

Catalytic Performance of Mn-MgO Nanoparticles in Biodiesel Production

Compared to conventional catalysts like potassium hydroxide (KOH), the use of Mn-MgO nanoparticles results in faster reaction times and higher biodiesel yields. Additionally, Mn-MgO nanoparticles have demonstrated excellent reusability, with studies showing no significant loss of catalytic activity after several cycles (None Monika, 2023). This long-term stability makes them a reliable choice for biodiesel production, even in industrial settings.

Challenges and Future Directions

While Mn-MgO nanoparticles hold great promise for biodiesel production, there are still challenges that need to be addressed. Scaling up the process for industrial use is one such challenge, as maintaining uniformity in the size and shape of nanoparticles during large-scale production can be difficult. Additionally, the cost of synthesizing the catalyst must be reduced to make it a viable option for widespread use (Khan *et al.*, 2021). However, given the environmental benefits and efficiency of Mn-MgO nanoparticles, these challenges are worth addressing, and ongoing research will likely continue to improve their applicability in biodiesel production.

CHAPTER 2

MATERIALS AND METHODOLOGY

2.1 MATERIALS USED

Waste Cooking Oil (Wco), Hydrochloric Acid, Potassium Hydroxide, Ethanol, CarbonTetrachloride, Benzene, Sodiumthiosulphate, Starch Indicator, Methanol, Wijs Solution (Used in the analysis of waste cooking oil to determine its suitability for biodiesel production), Distilled water.

Table 2.1: Apparatus used

Beakers, Heating mantle, Measuring cylinder, Volumetric flask, Magnetic stirrer, Foil Paper, Weighing Balance, Oven, Muffle Balance, Separating Funnel, Conical Flask, Burette, Retort Stand, Droppers, Viscometer

2.2 CHARACTERIZATION OF THE OIL

The waste cooking oil used for this study was obtained from Upper Uwa, Benin City Edo State. The oil was characterized to determine its suitability for use in the production of biodiesel; the following analyses were carried out:

2.2.1 Acid Value

The amount of free fatty acid present in the oil sample was determined by the method of titration (Gunstone *et al.*, 2007). A measured weight of the oil (1g) was dissolved in a mixture of benzene and ethanol (10ml each) and 2 drops of phenolphthalein indicator was added, the mixture was titrated with a 0.05 standard solution of KOH. The KOH solution was prepared by dissolving 2.805g of KOH pellet in 1000ml of distilled water. The acid value of the oil which is described as the amount of KOH required, in milligram (mg) needed to neutralize the free fatty acid in one gram of the oil was calculated thus:

$$\text{Acid value} = \frac{56.1 \times M \times \text{titre value}}{\text{Mass of oil}}$$

Where, 56.1 represent the molecular weight of KOH

M represents the molarity of KOH (0.05)

The *free fatty acid* (FFA) of the oil is half the acid value

$$\text{FFA} = \frac{\text{Acid value}}{2}$$

2.2.2 Peroxide Value

The peroxide value is the milliequivalent of peroxide per kilogram of oil, which oxidize potassium iodide under the test (Ma and Hanna, 1999). A mixture of acetic chloroform was prepared in the ratio 3:2 (900ml: 600ml). 1g of oil was measured in a conical flask and mixed

with 12ml of acetic-chloroform mixture and shook for 60 seconds, 5ml of iodide solution was added to the mixture and shook to obtain uniformity. 12ml of distilled water was also added. The mixture was titrated against 0.1M Na₂S₂O₃ (sodium thiosulphate) using 1ml of starch as indicator.

$$\text{Peroxide value} = \frac{10 (N_1 - N_2)}{\text{Mass of oil}}$$

Where N₁ represents titre value for oil)

N₂ represents titre value for blank (i.e. no oil)

2.2.3 Iodine Value

Waste Cooking Oil and transesterified oil sample, 0.15g each, was weighed and dissolved in 15ml of **carbon tetrachloride** and agitated thoroughly in a stopper flask and 25ml of Wij's solution was added and agitated and then allowed to stand for about 30minutes in the dark.

100ml of distilled water and 20ml of 10% (w/v) potassium iodide was added to the mixture and filtered with 0.1M standard sodium thiosulphate using 0.5% (w/v) starch solution as indicator.

Blank determination was also carried out in a similar manner. The iodine value was then calculated thus;

$$I.V = \frac{12.69 \times M (u - v)}{W}$$

W

Where,

M = Molarity of standard Na₂ S₂O₃

W = Weight of samples in gram used

U = Volume of millimeters of Na₂ S₂O₃ titrated in blank

V = Volume of millimeters of Na₂ S₂O₃ titrated in test.

2.2.4 Saponification Value

This is the number of milligrams of KOH needed to saponify 1g of fat; it's a measure of the average molecular weight of all fatty acids present. To get the saponification value of 1g of the waste cooking oil sample, alcoholic KOH was prepared. Alcoholic KOH was prepared by mixing 5.51ml of KOH solution in 200ml of ethanol. 50ml of the alcoholic KOH was added to the weighted mass of oil in a round bottom flask, and refluxed for one hour using a condenser and a heating mantle, allowed to cool and titrated with 0.5 M solution of HCl using phenolphthalein indicator.

$$\text{Saponification value} = \frac{(B-S) \times M \times 56.1}{\text{mass of oil}}$$

Where B represents the titre value for the blank solution

S represents the titre value for the oil solution

2.2.5 Kinematic Viscosity

The kinematic viscosity of the waste cooking oil sample measured in centistokes (Cst), represents the resistance to the flow of the oil. The viscosity of the waste cooking oil sample was measured using a viscometer.

$$\text{Kinematic Viscosity, } v = \frac{\eta}{\rho}$$

Where η = *dynamic viscosity in Cp*

ρ = *density of substance*



Figure2.1: Viscometer

2.2.6 Density

This is the mass of a substance per unit volume. The density of the waste cooking oil sample was calculated by weighing the mass of a given volume of the oil contained in a 50ml density bottle using the weighing balance. The mass of empty density bottle was subtracted from that of the bottle and the oil contained to get the mass of the oil, which was divided by the volume of the bottle, to get the required density.

$$\text{Density, } \rho = \frac{m}{v}$$

Where m is the mass of the oil

v is the volume of the oil

2.3 BIODIESEL PRODUCTION

According to literature and based on calculations, the number of free fatty acid present in the waste cooking oil has already been proven to be higher than 2wt% (This refers to used oil, typically from cooking processes, which may contain impurities, including a higher content of free fatty acids compared to fresh oils.)

Higher than 2wt%: This indicates that the free fatty acid content in the waste cooking oil is more than 2% of the total weight of the oil.)

Hence the need for a catalyst that would carry out simultaneous esterification and transesterification processes in the oil. The catalyst was mixed in a volume of alcohol and stirred for 6-8minutes. A set up of a magnetic stirrer and a reflux condenser was prepared; on the magnetic stirrer was a 1000ml conical flask (reactor) containing the oil, fit to a reflux condenser which was used to condense methanol escaping in the gaseous state back to the reactor. The oil was slightly heated using the magnetic stirrer. The catalyst + alcohol mixture was added to the oil and stirred continuously using the magnetic stirrer at a temperature range of 45-60⁰C for 30mins. The resulting mixture was allowed to cool and sent to a separating funnel where the biodiesel and glycerol separated into two unique phases by gravity, the biodiesel which is less dense was on top and the glycerol below, the tap was opened and the glycerol was separated into another container, leaving the biodiesel.

2.3.1 Biodiesel Purification

The produced biodiesel was washed with water to remove any form of dissolved glycerol, excess methanol, separated in a funnel from the removed waste water, and dried. The purified biodiesel was taken for analyses to see if it met standard specifications.

The weight of the produced biodiesel after washing was measured and the percentage yield calculated thus:

$$\text{Percentage yield of biodiesel} = \frac{\text{Mass of Biodiesel produced} * 100}{\text{Mass of Waste cooking oil}}$$

CHAPTER THREE

RESULTS AND DISCUSSION

3.1 RESULTS

Table 3.1: Physicochemical Properties of Waste Cooking Oil before and after Esterification

| Parameter | unit | Waste cooking oil | Esterified waste cooking oil |
|----------------------|--------------------------|-------------------|------------------------------|
| Acid value | mg KOH/g | 2.973 | 1.85 |
| Free fatty acid | mg/kg | 1.4865 | 0.925 |
| Saponification value | mg KOH/g | 99.0165 | – |
| Peroxide value | mg/kg | 26.6 | – |
| Iodine value | mg/100g | 86.93 | – |
| Specific gravity | g/ml | 0.92 | 0.892 |
| Viscosity | mPas(millipascal-second) | 44.30 | – |

Table 3.2: Physicochemical Properties of Biodiesel from Waste Cooking Oil using MgO and MnMgO - NP

| Property | unit | Biodiesel from MgO | Biodiesel from MnMgO- NP | Standard(ASTM D6751/EN 14214) |
|---------------------|----------------------|--------------------|--------------------------|-------------------------------|
| Density | (kg/m ³) | 893 | 875 | 860 - 900 |
| Kinematic Viscosity | (mm ² /s) | 4.63 | 3.89 | 1.9 - 6.0 |
| Acid Value | mg KOH/g | 0.42 | 0.35 | 0.25 - 0.5 |
| Flash Point | (°C) | 82 | 77 | > 93 |
| Cloud Point | (°C) | 37 | 32 | ~42 |
| Pour Point (°C) | (°C) | 36 | 39 | > 47 |

Table 3.3: Biodiesel yield

| Property | Biodiesel from MgO | Biodiesel from MnMgO - NP |
|--------------------------------|--------------------|---------------------------|
| Mass of oil used (g) | 50 | 50 |
| Mass of catalyst used (g) | 2 | 2 |
| Mass of biodiesel produced (g) | 32.5 | 42.3 |
| Yield (%) | 65 | 84.6 |

$$\text{Biodiesel Yield} = \frac{\text{MASS OF BIODIESEL PRODUCED (g)}}{\text{MASS OF OIL USED (g)}} \times 100$$

3.2 DISCUSSION

The physicochemical properties of waste cooking oil (WCO) before and after esterification, as presented in Table 3.1, highlight the effectiveness of the purification and transesterification processes in improving the quality of the feedstock for biodiesel production. The acid value of waste cooking oil decreased from 2.973 mg KOH/g to 1.85 mg KOH/g, indicating a significant reduction in free fatty acids (FFA). This reduction is particularly important as a high acid value can lead to soap formation during transesterification, which negatively affects biodiesel yield and quality. By lowering the acid value, esterification ensures a more efficient conversion of triglycerides into biodiesel.

Additionally, the free fatty acid (FFA) content dropped from 1.4865 mg KOH/g to 0.925 mg KOH/g after esterification, further confirming the effectiveness of the process in reducing acidity. A lower FFA content is crucial for achieving a high biodiesel yield since excessive FFAs react with the alkaline catalyst to form soap, leading to losses during the separation phase.

The saponification value of waste cooking oil was determined to be 99.0165 mg KOH/g, which reflects the presence of triglycerides in the oil. A higher saponification value typically indicates a higher content of glycerides, which are essential for biodiesel production. However, the presence of oxidation products in the waste oil can compromise the quality of biodiesel. This is evident in the peroxide value of 26.6 mg/kg, which suggests some degree of oxidation in waste cooking oil. Oxidation leads to the formation of peroxides and other degradation compounds that can impact biodiesel stability and performance.

The iodine value of waste cooking oil was recorded as 86.93 mg I₂/100g, which provides insight into the level of unsaturation in the oil. A higher iodine value indicates a greater degree of unsaturation, which can influence oxidation stability and cold flow properties. Unsaturated fatty acids are prone to oxidative degradation, leading to polymerization and the formation of deposits in engines. However, they also contribute to better low-temperature flow properties in biodiesel, which is beneficial for cold-weather performance.

The specific gravity of waste cooking oil decreased slightly from 0.92 g/mL to 0.892 g/mL after esterification. This change falls within the acceptable range for biodiesel feedstock and suggests that the purification and transesterification processes effectively modified the physical properties of the oil to align with biodiesel standards. Similarly, the viscosity of waste cooking oil, which was initially measured at 44.30 mPas, was significantly higher than the acceptable limits for biodiesel. This underscores the necessity of transesterification, as reducing viscosity is critical for improving fuel flow properties and ensuring efficient combustion in diesel engines.

A comparison of biodiesel produced using MgO and MMBO -NP catalysts reveals notable variations in key fuel properties (Table 3.2). The density of biodiesel produced using MgO (893 kg/m³) and MMBO -NP (875 kg/m³) falls within the ASTM standard range (860-900 kg/m³), confirming that both biodiesel samples have suitable densities for engine applications. Density is a crucial parameter, as it influences fuel injection, atomization, and combustion efficiency in diesel engines.

The kinematic viscosity of biodiesel produced with MMBO -NP was lower (3.89 mm²/s) compared to that produced with MgO (4.63 mm²/s). This suggests that nanoparticle catalysts

enhance reaction efficiency, leading to improved fuel flow characteristics. Lower viscosity contributes to better fuel atomization and combustion, reducing engine deposits and improving overall performance.

The acid value of the biodiesel samples was found to be 0.42 mg KOH/g for MgO and 0.35 mg KOH/g for MMBO-NP. Both values fall within the standard range (0.25-0.5 mg KOH/g), indicating good biodiesel quality with minimal residual free fatty acids. A lower acid value is desirable, as it prevents corrosion in fuel systems and extends the shelf life of biodiesel.

The flash point, which is a measure of fuel volatility, was slightly lower than the standard (>93°C) for both catalysts, with 82°C for MgO and 77°C for MMBO -NP. A lower flash point suggests higher volatility, which can pose safety concerns during handling and storage. This indicates the need for improved purification methods to remove residual alcohols and other volatile components from the biodiesel.

The cold flow properties of the biodiesel were evaluated based on the cloud point and pour point. The cloud point was measured at 37°C for MgO and 32°C for MMBO -NP, while the pour point was 36°C for MgO and 39°C for MMBO -NP. These values indicate that biodiesel produced using MMBO -NP has slightly better cold flow properties, making it more suitable for use in colder climates. However, further modifications or the addition of cold flow improvers may be required to enhance low-temperature performance.

The efficiency of the two catalysts in biodiesel production was assessed through a yield comparison (Table 3.3). The same amount of oil (50 g) and catalyst (2 g) was used for both

reactions, but the mass of biodiesel produced was significantly higher for MMBO -NP (42.3 g) compared to MgO (32.5 g). This resulted in a yield of 65% for MgO and 84.6% for MMBO -NP, demonstrating the superior catalytic efficiency of MMBO -NP.

The improved yield achieved with MMBO -NP can be attributed to the increased surface area and enhanced reactivity of nanoparticles. Nanoparticle catalysts provide more active sites for the transesterification reaction, facilitating a more efficient conversion of triglycerides to biodiesel. Additionally, MMBO -NP likely promotes better separation of biodiesel from glycerol, further contributing to the higher yield.

3.3 CONCLUSION

The findings of this study confirm that both esterification and transesterification processes significantly enhanced the quality of waste cooking oil, making it more suitable for biodiesel production. The use of the MMBO -NP catalyst proved to be highly effective, outperforming traditional MgO in terms of biodiesel yield and critical fuel properties. Notably, the MMBO -NP catalyst contributed to a substantial reduction in viscosity and acid value, which are essential parameters for fuel efficiency and engine compatibility.

Furthermore, while the produced biodiesel met the majority of ASTM/EN fuel standards, certain refinements may be necessary to optimize specific properties. In particular, adjustments to the production process could be explored to enhance the flash point and cold flow properties, ensuring improved performance in a wider range of environmental conditions. Addressing these aspects would further solidify biodiesel's suitability as a reliable alternative fuel source.

Overall, these results highlight the promising potential of nanoparticle-based catalysts in the biodiesel industry. By significantly improving the conversion efficiency and fuel characteristics of biodiesel derived from waste cooking oil, the MMBO-NP catalyst contributes to the development of a more sustainable and economically viable biofuel. The successful implementation of such catalysts could help reduce dependence on fossil fuels, mitigate environmental pollution, and support the transition towards cleaner energy solutions.

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