The Production of Biodiesel from Waste Cooking Oil Using Calcium Oxide and Barium Oxide Nano Catalysts

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Abstract: A comparative study of biodiesel production at 60°C from waste cooking oils, using calcium oxide (CaO) nano catalyst, barium oxide (BaO) nano catalyst, and catalyst-free methanol was investigated. The waste cooking oils were evaluated for their physicochemical properties, revealing a pH of 2.9± 0.00, moisture content of 1.50 ±0.00%, density of 0.895±0. 00g/cm^3, acid value of 3.66±0.014mg KOH/g oil, free fatty acid value of 1.84±0.01% and saponification value of 196.58±0.32mg KOH/g oil. Biodiesel production was carried out using 1% CaO nano catalyst, 1% BaO nano catalyst, and catalyst-free methanol. The results showed that the BaO nano catalyst yielded the highest biodiesel production (94.56±0.51%), CaO nano catalyst was recorded as 90.53±0.67%, followed by the catalyst free methanol (85.07±0.04%). The fuel properties of the synthesized biodiesels showed very high flash points and higher viscosities than the conventional diesel. However, the flash point of 1% BaO nano catalyst (183^{0}C) and its viscosity (16.0cP) are higher than the flash point (181^{0}C) of 1% CaO nano catalyst and its viscosity (21.0cP), respectively. The study therefore, demonstrates the effectiveness of BaO nanoparticle in biodiesel production using waste cooking oil and highlights potential nano catalysts in the improvement of efficiency and sustainability of biodiesel production.

Keywords: *Biodiesel; Cooking Oil; Calcium Oxide; Barium Oxide; Nano-Catalysts.*

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I. INTRODUCTION

Diesel fuels, which are used for transportation of industrial and agricultural goods play an important role in the industrial economy of a developing country, as well as, in agricultural sector which is accompanied by economic growth due to increase in the transport (Jomir **et al.** 2011). The widespread use of fossil fuels make it necessary to develop renewable energy sources and this has stimulated recent interest in alternative sources for petroleum-based fuels that must be technically feasible, economically competitive, environmentally acceptable, and readily available (Lebedevas **et al.** 2017).

On the other hand, studies have shown that the use of nano particles can be effective in biodiesel production (Salamat & Ahmad, 2022; Sahoo & Panda, 2021). Nanoparticles are increasingly used as catalysts in biodiesel production because they have high surface area and unique properties that enhance reaction efficiency, improve the transesterification process, converting oils and fats into biodiesel. Commonly used nanoparticles include metal oxides (like TiO_2, ZnO, CaO and Al_2O_3) and mixed metal oxides. These catalysts are advantageous because they provide higher reaction rates, better yields and can be reused, making the process more sustainable and cost-effective (Rahman & Hossain, 2018; Srivastava & Prasad, 2017). Sahoo & Panda, 2021; Rahman & Hossain, 2018 asserted that nanoparticles enhance biodiesel production by providing a more efficient catalytic process. Their high surface area allows for better contact between the reactants, leading to increased reaction rates. Additionally, nanoparticles can be engineered to have specific properties, such as acidity or basicity, which can further optimize the transesterification process. Calcium oxide (CaO) nanoparticles are highly effective catalysts in the production of biodiesel due to its strong basicity, catalytic activity and low cost. CaO nanoparticles enhances the transesterification reaction as a result of its high surface area, converting triglycerides in oils to biodiesel efficiently (Srivastava & Prasal, 2000; Ibrahim & Hameed, 2017). Additionally, CaO nanoparticles can be easily synthesized and are relatively non-toxic, making them

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an environmentally friendly option (Liu & Zhao, 2019). CaO could be reused multiple times with minimal reduction in activity, contributing to cost-effectiveness and sustainability in biodiesel production (Zhang & Sun, 2020). Zinc oxide (ZnO) nanoparticles are another promising catalyst for biodiesel production. Yong and Liu (2019) revealed that ZnO nanoparticles offer high surface area and strong catalytic activity, which enhance the efficiency of transesterification process. They found that ZnO are particularly effective in catalyzing the reaction under mild conditions, making the process more energy - efficient. ZnO nanoparticles also exhibit good thermal stability and can be doped with other metals to further improve their catalytic performance. Their reusability and relatively low toxicity contribute to the environmental and economic benefits of using ZnO nanoparticles in biodiesel production (Ayban, 2019). Jurgen et al. 2012 stated that while nanoparticle catalysts offer many advantages, there are still challenges to address. This study therefore, seeks to evaluate and compare the catalytic effectiveness of calcium oxide (CaO) and barium oxide (BaO) nanoparticles in biodiesel production, aiming to determine which catalyst provides higher yields, faster reaction times, and improved fuel quality. Traditional catalysts often require high temperatures and have negative environmental impacts. The use of nano catalysts like calcium oxide and barium oxide may offer more efficient and sustainable biodiesel production, with lower energy requirements and reduced environmental impact. The widespread adoption of biodiesel as an alternative energy source hinges not only on its environmental benefits but also on the economic viability of its production processes. This research therefore, investigates the economic implications of using calcium oxide and barium oxide nanoparticles as catalysts for biodiesel production, providing insights into the feasibility of scaling up their use in industrial settings.

II. MATERIALS AND METHODS

The waste cooking oils used were obtained from fast food vendors within Trans-Amadi Industrial Layout, Port Harcourt Local Government Area of Rivers State. Collected waste cooking oils were put in plastic bottles and transported to the laboratory for further analysis. The sample was filtered using filter paper to remove unwanted food chunks and placed in beakers. The Waste Cooking Oils were characterized using the following parameters.

Density of the oil was measured when a clean dry graduated cylinder was placed on a mettler balance and the mettler balance was adjusted to zero. Waste cooking oil was gently poured into the graduated cylinder until 5cm^3 mark was reached. The corresponding weight at the mark was noted. The density was calculated using **equ. 1** below.

$$Density\left(\frac{g}{cm^3}\right) = \frac{mass\ (g)}{volume\ (cm^3)} \quad (1)$$

Viscosity of the waste cooking oil was determined by using a viscometer after calibrating viscometer with distilled water. 50ml of the waste cooking oil was poured into a beaker, and the viscometer spindle was dipped into the sample. The viscometer spindle was rotated at a speed of 6.0 rpm at a temperature reading of 30.4^{o}C, and the reading was recorded as cP.

In the determination of the moisture content, a clean dry empty beaker was weighed and 5ml of the waste cooking oil was poured inside it and reweighed. The beaker was then placed in a drying oven at 105^{0}C till constant weight was obtained. This weight is recorded in %.

The moisture content was determined using **equ. 2** below.

$$moisture\ content(\%) = \frac{m_2 - m_1}{m_3 - m_1} \times 100 \quad (2)$$

Where, m_1 = mass of beaker (g), m_2 = mass of beaker and oil before drying (g), and m_3 = mass of beaker and oil after drying (g).

Acid value was determined when 10g of waste cooking oil was weighed into a conical flask using a mettler balance, to which 100ml of ethanol was added and swirled gently till complete mixing. Then, 10 drops of phenolphthalein was added, and the solution was titrated with 0.1N KOH until a light pink color was obtained that persisted for about 30 seconds. The acid value was calculated using **Equ. 3** below.

$$\text{Acid value, } AV = \frac{V \times N \times m_w}{m_s} \quad (3)$$

Where, AV = Acid value (mg KOH/g), V = Volume of titrant (ml), N = Normality of KOH = 0.1N, m_w = Molecular weight of KOH, and m_s = Weight of Waste cooking oil (g).

Free fatty acid (FFA) value was calculated from the determined acid value as given in **Equ. 4** below.

$$\text{Free Fatty Acids, } FFA(\%) = \frac{AV}{2} \quad (4)$$

Where, FFA (%) = free fatty acid, AV (mg KOH/g) = acid value.

In the determination of the Saponification Number, a dry empty conical flask was weighed to which 5g of waste cooking oil was added. Also, 50ml ethanol was added to the flask and swirled gently to ensure proper dissolution. Two (2) drops of phenolphthalein indicator was added to the solution and was titrated with 0.5N KOH until a pink color that persisted for 30 seconds was achieved. A blank solution was prepared using ethanol and the procedure performed. The saponification value was calculated using **Equ. 5** below.

$$\text{Saponification Value, } SV = \frac{(V_1 - V_0) \times N \times m_w}{w_s} \quad (5)$$

Where, SV = Saponification Value (mg KOH/g oil), V_1 = Volume of KOH solution used for the oil sample (ml), V_0 = Volume of KOH solution used for the blank (ml), N = Normality of KOH solution (usually 0.5 N), and W_s = Weight of Waste Cooking Oil (g).

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Calcium oxide nanoparticles were synthesized by separately dissolving 0.5M calcium nitrate and 0.7M Sodium Hydroxide in 50ml de-ionized water and mixed to a 100ml solution. The mixture was stirred at 50rpm for 10 minutes at ambient temperature. A white gel precipitated which was dried, vacuum filtered, and washed with de-ionized water and absolute ethanol. It was then dried at 80^0C for 1hr to obtain the calcium oxide nano powder.

Barium oxide (BaO) nanoparticles were also synthesized by separately adding 0.4M potassium hydroxide (KOH) into 0.2M barium nitrate solution under vigorous stirring. The solution was placed in a centrifuge and spilled at 3000rpm for 20 minutes, after which, the supernatant solution was discarded. The white gel at the bottom was washed with distilled water and absolute alcohol and calcinated at 500^0C for 3 hours to obtain the barium oxide nano powder.

The waste cooking oil was pretreated before it was used for biodiesel production. One hundred milliliters (100ml) of waste cooking oil was put in a 250ml conical flask and warmed gently to a temperature of about 50^0C. One percent (1%) calcium oxide nano particles by weight of oil were added to the waste cooking oil and stirred for proper mixing. Two grams (2g) of KOH was properly dissolved in 33ml methanol and gently poured into the warmed waste cooking oil and set at 60^0C. It was mixed using a magnetic stirrer. The reaction proceeded for 60minutes after which the produced biodiesel was washed by spraying warm water over it to remove unreacted methanol and residual catalysts. The procedure was repeated using 1% barium oxide nano particle. Thirdly, biodiesel was produced at 60^0C without adding any nano particle but only methanol. The amount of biodiesel produced was calculated as %yield shown in **equ. 6** below.

$$\%\,yield = \frac{weight\ of\ biodiesel}{weight\ of\ waste\ cooking\ oil} \times 100 \qquad (6)$$

Some of the fuel properties were also characterized as shown below:

In the Cloud Point determination, 5ml of produced biodiesel was measured into a test tube and placed in a beaker containing ice blocks. After some time, it was observed that wax crystals were noticed at the surface of the sample and the temperature was recorded at this point.

Flash Point was determined by adding 2.5ml of produced biodiesel into a porcelain dish of a predetermined weight. The biodiesel was heated for some minutes and a piece of paper was lit and was waved across the biodiesel being heated. The point at which the diesel became ignited was recorded as the flash point. When a flame was observed, the temperature was immediately recorded with the use of thermometer.

Pour point was determined by adding 30ml of produced biodiesel into a clean and dry glass test tube. The sample was heated slowly and was cooled at a specified rate and examined at intervals of 5 °F (3 °C) for flow characteristics. The lowest temperature at which the movement of the sample was observed was recorded as the pour point.

III. RESULTS AND DISCUSSION

Results for physicochemical properties of waste cooking oil, biodiesel yield and fuel properties of the biodiesel are presented in Tables 1.0, 2.0 and 3.0, respectively. Table 1.0 presents the results for the mean and standard deviation of the physicochemical properties of the waste cooking oil samples. The parameters evaluated were pH, moisture content, density, acid value, free fatty acid, and saponification value. The values obtained were 2.9±0.00, 1.50±0.00%, 0.895±0.00g/cm^3, 3.66±0.014mgKOH/g, 1.84±0.01% and 196.58±0.32mgKOH/g.

Table 1 Mean Physicochemical Properties of Waste Cooking Oil

S/no.	Parameter	Value	Value	mean± SD
1.	pH	2.9	2.9	2.9±0.00
2.	Moisture content (%)	1.5	1.5	1.50±0.00
3.	Density (g/cm^3)	0.895	0.895	0.895±0.00
5.	Acid value (mg KOH/g)	3.65	3.67	3.66±0.014
5.	Free fatty acid (%)	1.70	1.84	1.84±0.01
6.	Saponification value (mg KOH/g oil)	196.35	196.8	196.58±0.32

Table 2 presents the results for the biodiesel yield when 1% calcium oxide nanoparticles, barium oxide nanoparticles and catalyst free methanol were used. The results obtained were 90.53±0.67, 94.56±0.51, and 85.07±0.04% for CaO, BaO and catalyst free methanol biodiesel yields, respectively. The results proved that the use of 1% BaO nano catalyst resulted in the highest biodiesel yield of 94.56±0.0.51%.

Table 2 Mean Biodiesel Yield

Biodiesel Yield at 60^0C for 1hr				
Nano catalyst	**Amount of nano catalyst (%)**	**Yield (%)**	**Yield (%)**	**%Mean±SD**
Calcium oxide (CaO)	1.0	90.05	91.00	90.53±0.67
Barium oxide (BaO)	1.0	94.92	94.20	94.56±0.51
Catalyst-free methanol	0.0	85.04	85.10	85.07±0.04

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Table 3 presents the fuel properties of the synthesized biodiesel with 1.0% CaO nano catalyst, 1.0% BaO nano catalyst, as well as methanol without nano catalyst, all at 60^0C for a period of 1 hour. The fuel properties of the produced biodiesel analysed were cloud point, pour point, flash point, and viscosity. The values obtained without nano catalyst were -2^0C, -15^0C, 178^0C, and 19.0cP, those obtained with CaO nano catalyst were -4^0C, -13^0C, 181^0C, and 21.0 cP, while the values obtained with BaO nano catalyst were -6^0C, -13^0C, 183^0C, and 16.0 cP, respectively. All values were within the range for conventional diesel except the flash point and viscosity values.

Table 3 Fuel Properties of Synthesized Biodiesel

Parameter	Biodiesel without Nano catalyst	Biodiesel with 1.0% CaO Nano catalyst	Biodiesel with 1.0% BaO Nano catalyst	Conventional Diesel
Cloud point (^{0}C)	-2	-4	-6	-15 to 5
Pour point (^{0}C)	-15	-13	-13	-15 to -5
Flash point (^{0}C)	178	181	183	60 to 80
Viscosity(cP)	19.0	21.0	16.0	3.87

IV. DISCUSSION

From Table 1.0 above, it was discovered that the waste cooking oil is acidic. This is expected, as waste cooking oil can undergo hydrolysis and oxidation reactions, leading to the formation of acidic compounds. The moisture content of the waste cooking oil is relatively low. High moisture content can lead to microbial growth and spoilage, but in this case, the moisture content is within acceptable limits. The density of the waste cooking oil is slightly lower than the typical density range of vegetable oils (0.9-0.95 g/cm³). This could be due to the degradation of the oil during the cooking process. The acid value of the waste cooking oil is relatively high. This indicates that the oil has undergone significant hydrolysis and oxidation reactions, leading to the formation of free fatty acids (FFA). The FFA content of the waste cooking oil is relatively high. This is consistent with the high acid value observed earlier. High FFA content can affect the quality and stability of the oil. The saponification value of the waste cooking oil is relatively high. This indicates that the oil has a high content of triglycerides, which can be hydrolyzed to form soap during the saponification reaction. Overall, the physicochemical properties of the waste cooking oil indicate that it has undergone significant degradation during the cooking process. The high acid value, FFA content and saponification value suggest that the oil may not be suitable for human consumption or other applications where high-quality oil is required. However, the oil may still be suitable for other applications, such as biodiesel production or industrial uses.

The results in Table 2.0 shows that the lower biodiesel yield of CaO nano catalyst may be due to the lower catalytic activity of CaO compared to the BaO nano catalyst. The use of only methanol (without any catalyst) resulted in a biodiesel yield of 85.07$\pm$0.04%. This is likely due to the self-catalytic effect of methanol, which can still catalyze the transesterification reaction to some extent. The results show that BaO nano catalyst is more effective than CaO nano catalyst in terms of biodiesel yield. This may be due to the lower basicity among other characteristics of BaO, which can more effectively catalyze the transesterification reaction. Table 3.0 presents the fuel properties of the synthesized biodiesel with 1.0% CaO (calcium oxide) nano catalyst, 1.0% BaO (barium oxide) nano catalyst, as well as one without nano catalyst. The values obtained for conventional diesel were within the range except the flash point and viscosity values. The very high flash point suggests that the diesel is far above the acceptable limit which is not suitable for use. The viscosity of 16.0 – 21.0 (cP) is too high for conventional diesel fuel and is not suitable for use in standard diesel engines, reasons being that it may clog or strain the fuel injection systems, cause incomplete combustion due to poor atomization and also lead to carbon deposits in the engine. Due to the high flashpoint and viscosity the biodiesel fuel can be used as a blend.

V. CONCLUSION

In this research the synthesis of biodiesel from waste cooking oil by comparing the use of calcium oxide (CaO) nano catalyst, Barium oxide (BaO) nano catalyst and catalyst-free methanol was evaluated. The comparative analysis of the produced biodiesel shows that BaO nano catalyst is more effective in producing biodiesel. The results indicate that the use of BaO nano catalyst can achieve a high biodiesel yield of 94.56%, which is significantly higher than the yields obtained using CaO nano catalyst (90.53%) and that of catalyst-free methanol (85.07%). The study highlights the potential of nano catalysts in improving the efficiency and sustainability of biodiesel production from waste cooking oil. Further studies could be conducted to optimize the catalyst loading, reaction conditions, and other parameters to achieve even higher biodiesel yields and quality. This research work is justified by several key factors. Firstly, the need for sustainable and renewable energy sources is critical in reducing reliance on fossil fuels and migrating environmental impacts. Biodiesel from waste cooking oil offers a valuable solution by recycling waste into energy. Secondly, optimizing the catalytic process is essential for improving biodiesel yield and reducing production costs. Nano catalysts such as CaO and BaO have shown promising enhancement reaction efficiency due to their high surface area and reusability.

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