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The Influence of bifunctional Heterogeneous Catalyst on Biodiesel Production from Oleochemicals

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Abstract- ***Over the years, production of biodiesel has gain momentum due to the advent of novel catalysts like bifunctional heterogeneous catalyst which is capable of catalyzing both esterification and transesterification reaction simultaneously through its multiple sites, and this save time, cost of production, materials utilization and most importantly production of biodiesel from oleochemicals with high free fatty acid. Thus, this study focused on the assessment of the efficiency of bifunctional heterogeneous catalyst (CaO/Al2O3) on biodiesel production using oleochemicals (Bush Mango Seed Oil (BMSO), African Native Pear Seed Oil (ANPSO) and Orange Peels Oil (OPO) as triglycerides. The bifunctional heterogeneous catalysts which were produced at various conditions of temperature, time and components mixing ratio (CaO: Al2O3) were used to catalyze the transesterification reaction in order to ascertain their efficacies. Among the tested catalyst, catalyst B yielded the highest percentage of biodiesel with 67.3%, 35.5 % and 60.2 % for ANPSO, BMSO and OPO respectively. Analysis of physicochemical properties of the biodiesel (flash point, cloud point, pour point, viscosity and density) showed that there are all within the range of acceptable values for biodiesel specified by ASTM6751.***

Keywords: Oleochemical, Biodiesel, Bifunctional Catalyst, Energy, Transesterification Reaction.

I. INTRODUCTION

Energy crisis is of serious concern globally, due to the increasing demand brought forth by industrialization and explosion of human population. Farouk et al (2024) reported that 580TJ of energy is required annually around the globe for sustainable development and good livelihood, but unfortunately this amount has not been met due to inadequate sources and lack of even distribution of the available sources. Aside from the shortage of energy sources; globally, the fossil energy sources which supplied over 80 % of the world available energy pose environmental challenge due to emission of greenhouse gases and it is depleting having used for over decades. Also the exploration and mining processes of some of these fossil energy sources are detrimental to the ecosystem and also causes the released of toxic pollutants which corrode structures; hence the need for supplementary sources of energy which are sustainable, economically viable and environmentally friendly. Production of fuel from biomass (biofuel) is promising in replacing the fossil fuel because it is eco-friendly, sustainable and economically viable. Biofuel production is a developing technology which required a lot of input and materials development. Different grades and types of biofuel have been synthesized over the years, ranging from liquid, solid and gaseous (example biodiesel).

Biodiesel is a biodegradable form of fuel derived from biomass. They are good source of energy and showed a promising potential of been a substitute to fossil fuel. Gerpen, (2005) described biodiesel as a renewable, Environmental-friendly, efficient energy source, replacement fuel which can provide fulfill energy security needs of the world without reducing engine's operational efficient. The high flash point of 423K when compared to that of conventional diesel of 337K makes it to exhibit non-flammable and non-explosive characteristics which makes it easy to be stored, handling and transported with little risk as compared to fossil fuel (Ling et al., 2014). Record from (OECD/FA, 2018) has it that about 36 billion litres of biodiesel was produced globally in 2017; by 2027 the figure is projected to increase by 9 %.

The type of biomass used for biodiesel production is very essential, because it has direct implication on the cost of production, amount of biodiesel obtain, environmental sustainability, quality of biodiesel obtain and the available technology use for the production process. Although there are many biomass used in biodiesel production, yet the most sustainable ones are those biomass that are readily available, does not require complex process for the extraction of the lipids and not ensue food crisis. Aside from the forth

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mentioned criteria of materials selection, region in which the biodiesel would be used may also be a yardstick on the type of material for the production, because lipid with high percentage of saturated fatty acid give rise to biodiesel with better cold properties which make it very suitable for use in temperate region of the world but lipid with high percentage of unsaturated fatty acid produce biodiesel with better oxidative stability which is a good storage property for any substance (Suzihaque et al., 2022). Thus, the choice of raw material is very essential in biodiesel production as it affect social, environmental, economic and technical aspect of the production.

Several routes have been studied on how to convert biomass to biodiesel; such route includes fast pyrolysis, gasification, catalytic fast pyrolysis and transesterification reaction, among all these routes, transesterification is the most widely used method. Transesterification is a reaction in which alcohol reacts with glyceride in the present of catalyst to form fatty acid alkyl ester (biodiesel) and alcohol. It is the major method in which biodiesel can be produced and the reaction is aided by a catalyst. Catalysts are substances that alter the rate of chemical reaction by reducing the energy of activation. This is done by rearranging the reacting molecules for easy bonding. Table 1 shows the comparison between petro-diesel and biodiesel as prescribed by American Standard of Testing Materials (ASTM D975 and ASTM D5751).

Table 1. Similarities in Properties of Petrodiesel and Biodiesel (Latif et al, 2017)

Fuel Property Fuel standard	Diesel ASTM D975	Biodiesel ASTM D5751
Higher heating value, Btu/gal	137,640	127,042
Lower heating value, Btu/gal	129,050	118, 170
Kinematic viscosity, mm^2/s@ 40°C	1.3 - 4.1	4.0 – 6.0
Specific gravity kg/I @15.5°C	0.85	0.88
Density, Ib/gal @ 15.5oC	7.1	7.3
Carbon, wt %	87	77
Hydrogen, w%	13	12
Oxygen w%	0	11
Sulphur, w%	0.0015max	0.0024max
Boiling point, °C	180 - 340	315 - 350
Flash point, °C	60 - 80	100 - 170
Cloud point, °C	-35 to -5	-3 to 15
Pour point, °C	-35 to -15	-5 to 10
Cetane Number	40 - 55	48 - 65

There are two categories of catalysts that can be used for biodiesel synthesis; these include homogeneous and heterogeneous catalysts. All other forms of catalysts fall within these two categories. Example mixed oxides, enzymatic, algae, acidic, basic catalysts and the bifunctional catalyst which is the most novel form of catalysts and the subject of interest in this work

Bifunctional catalyst is a type of catalyst that possess both acidic and basic sites this makes it to catalyze both esterification and transesterification simultaneously during biodiesel production. The need for this type of catalyst arises because most biomass synthetic oils comes with unacceptable amount of free fatty acid which makes it impossible for basic catalyst alone to effectively catalyze the transesterifiction reaction. Acid heterogeneous catalyst specie could be more effective in biodiesel production than homogeneous specie as the former will reduced significantly purification challenges, environmental pollution due to disposal of spent catalyst, heterogeneous catalysts are reusable and this make the entire process economically viable. Thus, in this work, bifunctional heterogeneous catalyst (CaO/Al_2O_3) formulated from calcium oxide derived from periwinkle shell and fused with aluminum oxide is used to catalyze tranesterification reaction using lipids extracted from Bush Mango Seed, African Native Pear Seed and Orange Peel as triglycerides as recorded in the previous work (Akpan et al., 2025) in order to validate its potential in catalyzing biodiesel production from oils with slight high free fatty acid.

II. MATERIALS AND METHOD

2.1 Material

The main materials used for this study are Bush Mango Seeds Oil (BMSO), African Native Pear Seeds Oil (ANPO) and Orange Peels Oil (OPO). The African

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Native Pear Seeds and Orange Peels were collected from dumpsite in Choba, River State with the aid of sack bags, while Bush Mango Seeds were bought in market in Aluu. The oils from the biomass were extracted as recorded in the previous work by Akpan et al. (2025) and CaO/Al_2O_3 bifunctional catalyst prepared via incipient wet impregnation method as described in previous work by Akpan et al. (2026).

2.2 Method

2.2.1 Production of biodiesel

The efficiency of the bifunctional heterogeneous catalysts formulated under different experimental conditions as described in the previous work (Akpan et al., 2026) and shown in Table 2 were tested in transforming the oils from the biomass to biodiesel. The production done via transestrificaton reaction was conducted in 500 mL three-neck round bottom flask equipped with magnetic stirrer, thermocouple for temperature regulation and reflux condenser for cooling and recycling of the methanol. The reaction conditions were 6:1 methanol to oil molar ratio, 1:20 catalyst to feedstock (oil) ratio, reaction temperature of 60 ºC and reaction time of 60 min.

Table 2. $CaO-Al_2O_3$ Bifunctional catalysts formulated at different conditions

Notation for the catalyst	Calcination Temp (ºC)	Time of calcination (hr)	Component ratio ($CaO:Al_2O_3$)
A	900	2	6:4
B	900	5	6:4
C	600	2	6:4
D	600	5	6:4
E	750	3.5	5:5
F	900	2	4:6
G	900	5	4:6
H	600	2	4:6
I	600	5	6:4

First, the catalyst was added to methanol in the round-bottom flask and heated to the reaction temperature with continuous stirring. The oil was then added to the catalyst/methanol mixture with continuous heating and stirring still 60 min (reaction time). When the reaction was completed, the catalyst was filtered out using Whatman no.1 filter paper with pore size of 25µm place in vacuum filter. The filtrate which comprises of glycerol and FAME was transferred into separating funnel and left overnight to allow phase separation. After which the top layer which is FAME was collected and washed with hot distilled water and then dried in oven at 50 ºC to remove water and unreacted methanol. The yield of the biodiesel was obtained using Equation (1)

$$\text{FAME yield} = \frac{\textit{weight of biodiesel obtained}}{\textit{weight of oil sample used}} \times 100 \quad (1)$$

2.2.2 Determination of physicochemical properties of the produced biodiesel

a). Cloud point, pour point and flash point

To determine the cloud and pour points of the biodiesel, a test tube filled with biodiesel was inserted into a beaker filled with ice-chips and then clamped to a retort stand. A thermometer and stirrer were inserted to measure the reaction temperature and proper mixing of the reactants respectively. The stirring persisted as the temperature increases. The sample was closely observed until it became cloudy. The temperature at which the biodiesel sample became cloudy was taken as the cloud point. The pour point was obtained at the temperature the biodiesel began to pour after solidification when the test tube was slightly bent.

The flash points were determined using 5 mL of the biodiesel sample in a crucible placed on a hot plate. A thermometer clamped to a retort stand was inserted into the crucible in such a way that the mercury bulb was slightly immersed in the sample. The temperature in which a flash was noticed from lighted match placed close to the crucible was recorded as the flash points. The procedure was carried out in duplicates.

b). Determination of Specific Gravity (Density)

A clean 50 mL specific gravity bottle was weighed (W_o), then filled to the brim with water and cover with a stopper. The water on the stopper was carefully wiped off and reweighed (W_1). The procedure was repeated, but with the sample (biodiesel) instead of water and weighed again (W_2). The specific gravity of

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the biodiesel samples were calculated using Equation 2.

Specific gravity of the sample = $\frac{W2-WO}{W1-WO}$ (2)

c). Determination of Viscosity

The viscosity was determined using OSTWALD-TYPE viscometer shown in Figure 1. The viscometer was thoroughly washed and dried. The sample was filled to the point marked G through the arm 1 using a long pipette to minimize wetting of the tube above the marked point. Then the tube was placed vertically in a water- bath maintained at temperature of 40 °C and a allow to stand for 30 min to allow the temperature to reach equilibrium.

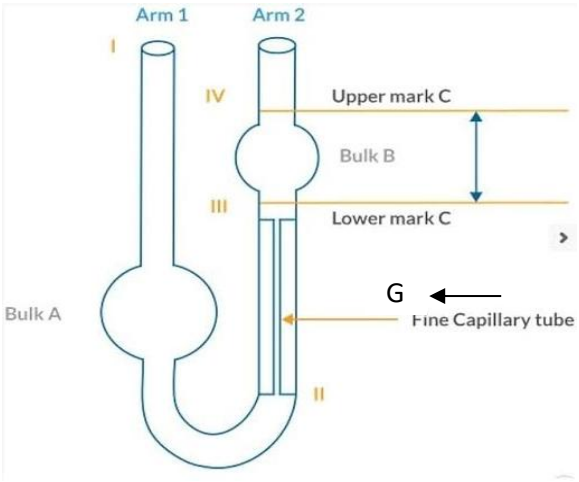

Figure 1: Ostwald-type viscometer

The volume of the sample was adjusted so that the bottom of the meniscus settles at the mark G. The liquid was sucked to a point about 5 mm above the upper mark C. After releasing pressure of suction, the time taken for the bottom of the meniscus to fall from the top edge of upper mark C to the lower mark C was measured.

The kinematic viscosity (v) in square millimetres per second (mm^2s^{-1}) was calculated using Equation 3.

$v = Kt$, (3)

where t = time in seconds for the meniscus to fall from upper mark C to lower mark C,

The constant (k) of the instrument is determined using a liquid of known viscosity.

III. RESULTS AND DISCUSSION

3.1 Production of biodiesel from the oleochemicals

The catalysts produced at different conditions as reported in the previous work Akpan et al. (2026) were tested to ascertain their efficiencies in catalyzing production of biodiesel via transesterification reaction using the following reaction conditions 1:6 oil to methanol molar ratio, 60°C reaction temperature and 1:20 catalysts to feed ratio. This was done to ascertain the best formulated catalyst as biodiesel yield has a direct relationship to the efficiency of the catalysts. The results from different oils are as presented in bar charts in Figure 2 to 4 in term of percentage yield.

a. African Native Pear Seeds Oil

The catalysts efficiencies in catalyzing biodiesel production from African Native Pear Seed oil (ANPSO) were determined to ascertain the best formulation for the oil. The result presented in Figure 2 with the plot of biodiesel yield (%) against catalysts indicates that catalyst B (6:4, 900 °C, 5 h) produced the highest percentage yield of 67.3 % follow by catalyst A with 65.4 %.

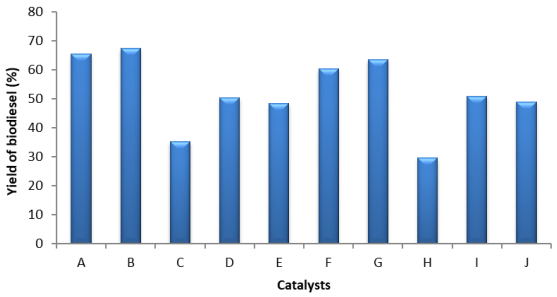

Figure 2. Determining the efficiencies of the catalysts on biodiesel production from ANPSO

African Native pear seed oil yielded good percentage of biodiesel with some of the formulated bifunctional catalysts. Catalyst B gave the highest yield of 67.3 %. Five of the formulated catalysts performed above average and above the conventional catalyst which gave a yield of 48.9 %. The influence of temperature could be noticed here in the performance of the catalysts. Those that were calcined at high

temperatures gave a better yield than those formulated at lower temperatures. The result also indicates that a high ratio of the basic component (CaO) gave a better performance than high ratio of the acid species (Al_2O_3) even at the same temperature. This could be attributed to a well arranged crystal structure occasioned by high percentage of calcium oxide at high temperature given rise to larger surface area which promotes reactants interaction and rapid mass transfer (Akpan et al, 2026; Nizah et al. 2014).

b. Bush Mango Seeds Oil

The catalysts efficiencies in catalyzing biodiesel production from Bush Mango Seed oil were determined to ascertain the best formulation for the oil. The result presented in Figure 3 with the plot of biodiesel yield (%) against catalysts shows the highest yield of 35.5 %. from catalyst B(6:4, 900 ºC,5h) where 6:4 is the calcium oxide to aluminum oxide ratio, 900 ºC is the calcination temperature and 5 h is the time of calcination.

The efficiencies of the catalysts in catalyzing biodiesel production from bush mango seed oil were very low. The highest yield obtained was 35.5 % from catalyst B (900 ºC, 5 h and 6:4). No yields was obtained from catalyst H (4:6, 600 ºC and 2 h) and from the conventional catalyst (J).

The poor performance of the catalysts in the reaction maybe due to high percentage of saturate fatty acid present in the oil which slow down reactivity of the reactants and weaken the efficiencies of the catalysts through pore blockage. This type of oil requires special treatment to dismantle the complex bond structure. The saturated nature reduces the rate of mass transfer and phase contact between the reacting species during transesterification/esterification which occurs simultaneous when bifunctional catalyst is used to catalyze the reaction. The triglycerides from saturated fatty acid are less polar leading to poor miscibility with methanol during the reaction.(Miyuranga et al., 2023). Thus, for effective reaction to occur, cosolvent like acetone with higher polarity is use to break the multiphase created by poor miscibility to single phase for effective mass transfer (Mohadesi et al., 2020).

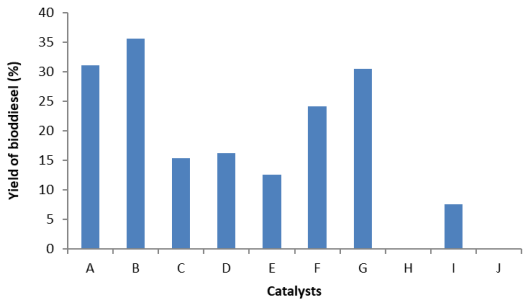

Figure 3. Determining the efficiencies of the catalysts on biodiesel production from BMO

c. Orange Peels Oil

The efficiencies of the formulated catalysts in catalyzing biodiesel production from Orange Peel oil were determined to identify the best formulation for the oil. The result is as presented in Figure 4 using a plot of biodiesel yield (%) against catalysts. The yields of biodiesel from orange peel oil using the formulated catalysts were significant. The highest yield of 60.2 % was obtained from catalyst B with more than six of the formulated catalyst performing above 50 % and better than the conventional catalyst which gave 33.1 %. The lowest yield of 25.4 % was obtained from catalyst H (600 ºC, 2h and 4:6-CaO: Al_2O_3).

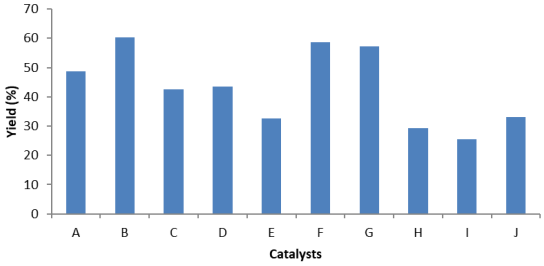

Figure 4. Determining the efficiencies of the catalysts in biodiesel production from OPO

This could be as a result of high percentage of unsaturated fatty acid in the oil which promote miscibility due similarity in polarity between triglyceride and methanol during the reaction (Miyuranga et al., 2023). The large surface area, pore size and volume of catalyst B may also lead to increase in the rate of mass transfer which in turn increase the overall rate of reaction.

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3.2 Physicochemical properties of the produced biodiesel

The physicochemical properties of the produced biodiesel were analysis to ensure that the produced biodiesel meet up with the specified standard by regulatory bodies. The result is shown in Table 3. Cloud point is the temperature at which dissolve particles in a fluid precipitate, ASTM6751 for biodiesel is between (-3 to 12 ^{o}C) from Table 3 only biodiesel from ANPSO meet up the recommended standard, but biodiesel from BMSO and OPO are above the recommended standard which mean they may not be good in temperate region.

Table 3. Physicochemical Properties of the produced biodiesel from each Oil

Properties	WCO	ANPSO	BMSO	OPO	ASTM 6751
density (g/ml)	0.79	0.814	0.880	0.801	0.880
Viscosity (mm2/s) @40^{o}C	3.4	3.2	6.7	4.7	1.6 to 6.0
Flash point (^{o}C)	165.2	145.5	156.5	168	>96
Cloud point (^{o}C)	12.3	11.9	23	17.5	-3 to 12
Pour point (^{o}C)	-2.4	-3.5	3.4	2.5	-15 to 16

The exceptional cloud point ANPSO may be due to high degree of unsaturation of its fatty acid, nevertheless for BMSO and OPO the percentage of unsaturation is not very significant as compare to ANPSO (Akpan et al., 2025). Pour point is the temperature at which fluid start to flow after solidification. For biodiesel the range is between -15 to 16 ^{0}C, all the produced biodiesel met the ASTM6751 specification. Flash point is the measure of flammability of fluid, for biodiesel ASTM6751 is >96^{o}C. From the result obtained all the biodiesel met the recommended standard. Kinematics viscosity entails the tendency of a fluid to flow at a particular temperature (ASTM6751 for biodiesel @40 ^{o}C is 1.6 – 6.0). Aside from BMO, all other ones met the required specification.

IV. CONCLUSION

From the analysis of the oils for biodiesel production using CaO-Al_2O_3 bifunctional heterogeneous catalysts formulated at different condition as described in the previous work (Akpan et al., 2026), it is obvious that the oils are viable for biodiesel production. The highest yield obtained were 67.3 %, 35.5 % and 60.2 % for African Native pear oil, Bush mango seed oil and orange peel oil respectively. The catalyst formulated at the following conditions 900 ^{o}C calcination temperature, calcination time of 5 hours and component mixing ratio of 6:4 (CaO:Al_2O_3) gave the best result. The analysis of the physicochemical properties of the biodiesel (flash point, cloud point, pour point, viscosity and the density) showed that the fall within the acceptable range specified by ASTM6751 for biodiesel. Thus, the use of these oils and the catalyst could reduce the overall cost of production, process time and environmental hazard caused by the used of homogeneous acidic catalyst for esterification when oil with high free fatty acid is used for biodiesel production.

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