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Special Issue

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This special issue is dedicated to Professor Onukwuli Okechukwu Dominic (FAS), marking his retirement and celebrating a remarkable career. His legacy of exemplary scholarship, mentorship, and commitment to advancing knowledge is commemorated in this collection of works.

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Effects of process parameters on methyl ester yield from fruit-peel ash catalyzed lard oil methanolysis

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Abstract

The viability of utilizing ash from fruit-peel of banana, plantain, and hybrid banana-plantain as low-cost hetero-basic solid catalysts in waste-lard-oil (WLO) methanolysis in lard-oil-methyl-ester (LOME) production was explored together with the effects of process parameters on LOME conversion. The catalyst properties were ascertained using SEM-EDX, XRD, and BET. The effects of temperature, catalyst-amount, time, and methanol-to-WLO molar proportion on LOME yield utilizing the various catalysts were investigated. Each of the catalysts exhibited high catalytic ability in WLO methanolysis resulting in over 95% LOME yield at the optimal settings of 60°C, 2.5 wt. % catalyst amount, and 10.5:1 molar proportion at a reduced period of 1.5 h, and mixing rate of 300 rpm. The CBPA-catalyzed WLO transesterification depicted the highest catalytic capability FAME yield of 98.8%. The catalysts' effectiveness in the WLO transesterification was in relation to the potassium content and the surface area. The LOME physicochemical properties were within the specified biodiesel standard. Thus, a route for an ecologically and economically sustainable fuel could be established with bio-derived catalysts.
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Keywords: Bio-catalyst, lard oil methyl ester, methanolysis, surface area, waste lard oil

1. Introduction

The increasing global demand for sustainable and renewable energy sources has propelled significant investigation into methyl ester (biodiesel) production as a substitute to fossil fuels (Ume et al., 2024). Biodiesel, primarily produced by means of oils or fats transesterification with alcohol, is recognized for its environmental gains, for instance relegated greenhouse gas emissions, and biodegradability (Ao et al., 2024; Neupane et al., 2022). The numerous gains of biodiesel also include regenerability, biocompatibility, and low sulfur content (Ezekannagha et al., 2024; Singh *et al.,* 2020). However, the high price of raw materials and catalysts remains a major challenge in the biodiesel industry. To address this, researchers are exploring waste-derived feedstocks and catalysts, such as waste oils, waste animal fats, waste fish, and fruit peel, etc. to cause biodiesel production to be economical and sustainable (Osman et al., 2024; Tsai and Tsai, 2024). Biodiesel production using homogeneous-catalysts is straightforward and less time-consuming however, its utilization in the transesterification reaction is hampered by a number of issues, including soap generation, equipment corrosion, non-renewability, non-recyclability, and difficulty in separation (Niju et al., 2018). Heterogeneous base catalysts offer benefits like low-cost, high-catalytic-activity, non-toxicity, ease of separation from reaction mixtures, and reusability, making them economically viable and environmentally friendly alternatives to homogeneous catalysts (Maroa and Inumbao, 2022). Several process parameters for instance temperature, catalyst concentration, time, and methanol-to-oil molar proportion greatly influence the methyl ester conversion. Temperature rise results in a corresponding rise in methyl ester yield. However, the optimum temperature ought not be exceeded as it will lead to a reduction in yield. The same applies to other process parameters as a function of methyl ester yield in the transesterification of oil. It however becomes imperative to establish the optimum operating conditions for optimum methyl ester yield in catalyzed methanolysis of oil (Etim et al., 2022).

Banana and plantain peels are abundant agricultural wastes generated worldwide, particularly in regions where these fruits are staple foods (Etim et al., 2018; Pathak et al., 2016). The peels constitute environmental hazards when not properly disposed and as such utilization as catalysts will simultaneously create a waste disposal management option and also a value-added product that is beneficial to the environment and society at large (Esonye et al., 2018; Niju et al., 2023). These peels contain high levels of potassium, calcium, and other alkaline earth metals, which can be activated to function in the role of a heterogeneous catalyst for methyl ester production (Dias et al., 2024). Utilizing such bio-derived catalysts aligns with the principles of green chemistry, enabling the valorization of waste materials and reducing reliance on conventional chemical catalysts, which are often expensive, non-reusable, and environmentally harmful (Satapathy et al., 2024; Maroa and Inumbao, 2022).

The exploitation of waste lard oil as a biodiesel feedstock further enhances the sustainability of this approach (Ezekannagha et al, 2024). Waste lard oil is a byproduct of the cooking and food processing industries, often discarded or underutilized despite its significant lipid content. It is as well a striking economic substitute in the circumstance of explosion of pork ailments or wherever portions of the animal are barred for human use owing to wellbeing conditions. Converting this waste into biodiesel not only alleviates environmental pollution but also delivers an economically viable solution to biodiesel feedstock scarcity (Farouk et al., 2024).

Despite the promising potential of banana and plantain peels as catalysts, limited studies have systematically explored their catalytic performance in biodiesel synthesis, particularly employing waste lard oil for a feedstock. Existing exploration primarily focuses on individual peels, neglecting the potential synergistic effects of combining different types of peels, such as banana-plantain mixtures, as heterogeneous catalysts (Dias et al., 2023). Furthermore, there is insufficient information on the optimization of reaction parameters, catalyst characterization, and reusability in such systems.

This study purposes to tackle these research gaps by exploring the catalytic efficacy of banana peels, plantain peels, in addition to their mixtures in waste-lard-oil transesterification to methyl ester. It additionally seeks to optimize process parameters providing insights into their economic and environmental feasibility. By leveraging agricultural waste materials as catalysts and feedstocks, this research adds to advancing the affordability, and sustainability of biodiesel synthesis. It offers a novel approach to waste valorization, addressing critical challenges in renewable energy development.

2.0 Materials and methods

2.1. Materials

Ripe banana and plantain fruit peels were gathered together from household waste. Waste pork lard was obtained from New Market, Enugu. Methanol-(CH_3OH, 99.8% purity) was procured from Conraws Company Ltd. The chemical reagents expended in this work are of analytical grade.

2.2. Catalyst preparation and characterization

Each of the disposed ripe banana and plantain peels were slashed into tiny bits to speed up the drying route. The tiny fragments of the peels were carefully rinsed with purified H_2O thrice to eliminate dirt and further sun-dried. Each of the dried-out banana and plantain peels respectively was afterwards charred entirely to form ash which was ground-to-powder catalyst by a mixed-blender. The banana-plantain peel ash mix catalyst was fixed by integrating equal measures (60 g each) of smooth grains of charred banana and plantain peel ash with one hundred ml of purified water. Then, the mixture was desiccated in an oven at 100^{o}C: 2 h. Each of the catalysts was activated thermally at 700^{o}:4 h in a muffle-furnace to achieve calcined-banana-peel ash (CBPA), calcined-plantain-peel ash (CPPA), and calcined-banana-plantain peel ash (CBPPA) mix catalysts. Finally, all the synthesized catalysts were put in storage separately in tight containers.

The CBPA, CPPA, and CBPPA catalysts were evaluated by X-ray diffraction (XRD: Rigaku miniflex-600 diffract meter) to ascertain the crystalline phases of the samples. Energy dispersive X-ray (EDX) and scanning electron microscopy (SEM) were used to establish the elemental compositions and morphological structure, respectively. Brunauer-Emmett-Teller analysis-(BET) was operated to define the surface-area, pore-volume, and pore-size of the samples.

2.3 Synthesis of WLO and its characterization

In order to generate WLO, leftover hog fat was heated to 110 degrees Celsius for one hour in a pan without water. The approach followed Ezekannagha et al., 2024. Subsequently, the liquefied fat was strained to get rid of the unsolvable materials. To counteract oxidation, the WLO was put away in a tight plastic jar. The lard oil was also characterized to establish the physico-chemical properties.

2.4 Waste lard oil transesterification reaction to LOME

The transesterification process of WLO with methanol was catalyzed by each of these catalysts, CBPA, CPPA, and CBPPA-mix respectively to generate glycerol and LOME. The reaction was carried out with 50 g of WLO in a 500 ml capacity flat-bottom flask equipped with a reflux condenser. A mixture of a measured amount of the calcined heterogeneous catalyst and the requisite molar proportion of methanol-to-WLO was slowly moved into the reactor with the heated oil. The reaction was timed at a set temperature. Agitating the reactor content continued for the recommended time at the optimum mixing- rate of 300 rpm established on literature data to realize highest transformation (Ezekannagha et al., 2023). On the transesterification reaction completion, the resultant mixture was poured into a separating funnel. It was permitted to stand for 1 h for phase separation. Three distinctive layers were spotted after the settle down process; LOME was existent at the top followed by the glycerol layer (middle) while the catalyst was at the bottom. The catalyst and glycerol strata were drawn-off from the bottom while the remaining LOME was measured and the volume recorded. The biodiesel was heated for ten minutes at a temperature marginally exceeding the vaporization point of methanol in order to evaporate the surplus methanol. The percentage LOME yield was computed based on the measured weight of LOME and WLO with equation 1 (Tshizanga et al., 2017).

$$\text{(\%) LOME Yield} = \frac{Weight\ of\ LOME}{Weight\ of\ lard\ oil}\ x\ 100 \quad (1)$$

2.5 Effect of process parameters on LOME yield with the different calcined heterogeneous catalysts

Factors affecting the transesterification reaction of WLO catalyzed by CBPA, CPPA, and CBPPA catalysts were varied. The reactions were conducted at ambient pressure and an optimum rotation speed of 300 rpm based on literature by varying a particular parameter and keeping the others constant. The optimum reaction conditions were chosen based on the percentage yield obtained. The effects of temperature, catalyst-amount, time, and methanol-to-WLO molar proportion on LOME yield were taken into consideration. The procedure for the production of LOME listed above was carried out repeatedly for each of the calcined heterogeneous catalyzed-based WLO transesterification reaction processes.

The reaction-parameters values were selected as follows; temperature: 45^{o}C to 65^{o}C, catalyst weight ratio-to-WLO: 1.0% to 3.0%, time: 1 h to 3 h, molar proportion of methanol-to-WLO: 6:1 to 12:1.

3.0 Result and Discussion

3.1 Catalysts characterization

The single catalysts coupled with the bio-mixture catalyst were investigated to ascertain the properties. The surface morphologies of the CBPA, CPPA, and CBPPA catalysts detected through SEM were captured at 500x and 750x magnifications and illustrated in **Figure 1**. Owing to calcination influence, several aggregates of micro-porous structures were seen on each of the catalysts owing to calcination influence. This observation is akin to those of Etim et al., (2022) and Rajkumari et al., (2020).

XRD patterns of each of the fruit peel catalysts exhibited similar results as shown in the superimposed graph (**Figure 2**). Various sharp peaks were detected in the samples calcined at 700ºC- 4 h within 2ϴ range 2 - 70º. Each of the catalyst samples exhibited identical clear and sharp peaks. The observed peaks in the samples designate the presence of potassium, calcium, silicon magnesium, etc. with potassium exhibiting a distinct peak in all. This could be a result of the similar crystalline compounds each of the samples possesses. These peaks are related to those reported in most calcined catalytic biomass resources with enhanced peak intensities relating to potassium compounds after calcination (Adedayo et al., 2020; Falowo and Betiku 2021; Mendonca *et al.*, 2019).

The EDX analysis (Table 1) shows the summary of the elemental compositions of the different calcined catalysts as detected through the EDX analysis. As evaluated in Table 1, each of the fruit peel-derived catalysts is largely comprised of K, O, Cl, C, Ag, P, Si, and Mg with K possessing over 60% weight concentration for each of the catalysts. The descending order of K content in the catalysts was hierarchical along these lines: CBPA (63.71%) > CBPPA (62.17%) > CPPA (60.33%).

Table 1: The elemental compositions of CBPA, CPPA, and CBPPA catalysts

Elements	Concentration-(wt. %)		
	CBPA	CPPA	CBPPA
Potassium-(K)	63.71	60.33	62.17
Oxygen (O)	8.75	9.12	9.05
Chlorine (Cl)	6.31	6.63	6.87
Carbon (C)	3.03	3.86	5.13
Calcium (Ca)	5.26	3.74	4.08
Silver (Ag)	2.26	2.86	2.95
Phosphorus (P)	1.63	1.84	1.55
Silicon (Si)	1.53	1.47	1.55
Magnesium (Mg)	1.38	1.08	1.46
Others	6.14	9.07	5.19

Table 2 shows the BET surface area (m^2/g), pore volume (cc/g), and pore diameter (nm) of the CBPA, CPPA, and CBPPA. Each of the fruit peel-derived catalysts exhibited trace pore volume and as such, are considered to be less-porous materials. Each of the catalysts is termed mesoporous owing to their observed pore diameters (nm). This is because mesoporous size is associated with pore-diameters of 2 to 50 nm (Laskar et al., 2020). The SEM analysis corroborates the mesoporous nature of the catalysts. The lesser the size of the aggregates, the higher the surface area translating to higher effectiveness of the catalysts. This is following the observation of Tshizanga et al., (2017) who recorded that catalytic activity is a function of the catalyst's surface area, alkaline strength, and the alkaline site concentration. This explains the increased catalytic efficacy of each of the synthesized catalysts (CBPA, CPPA, and CBPPA) in the waste lard oil transesterification reaction.

Table 2: BET surface-area, pore-volume, and pore-diameter of the fruit peels-derived catalysts

Catalyst source	Surface area (m^2/g)	Pore volume (cc/g)	Pore diameter (nm)
CBPA	870.766	0.482	2.123
CBPPA	697.422	0.392	2.131
CPPA	547.372	0.303	2.133

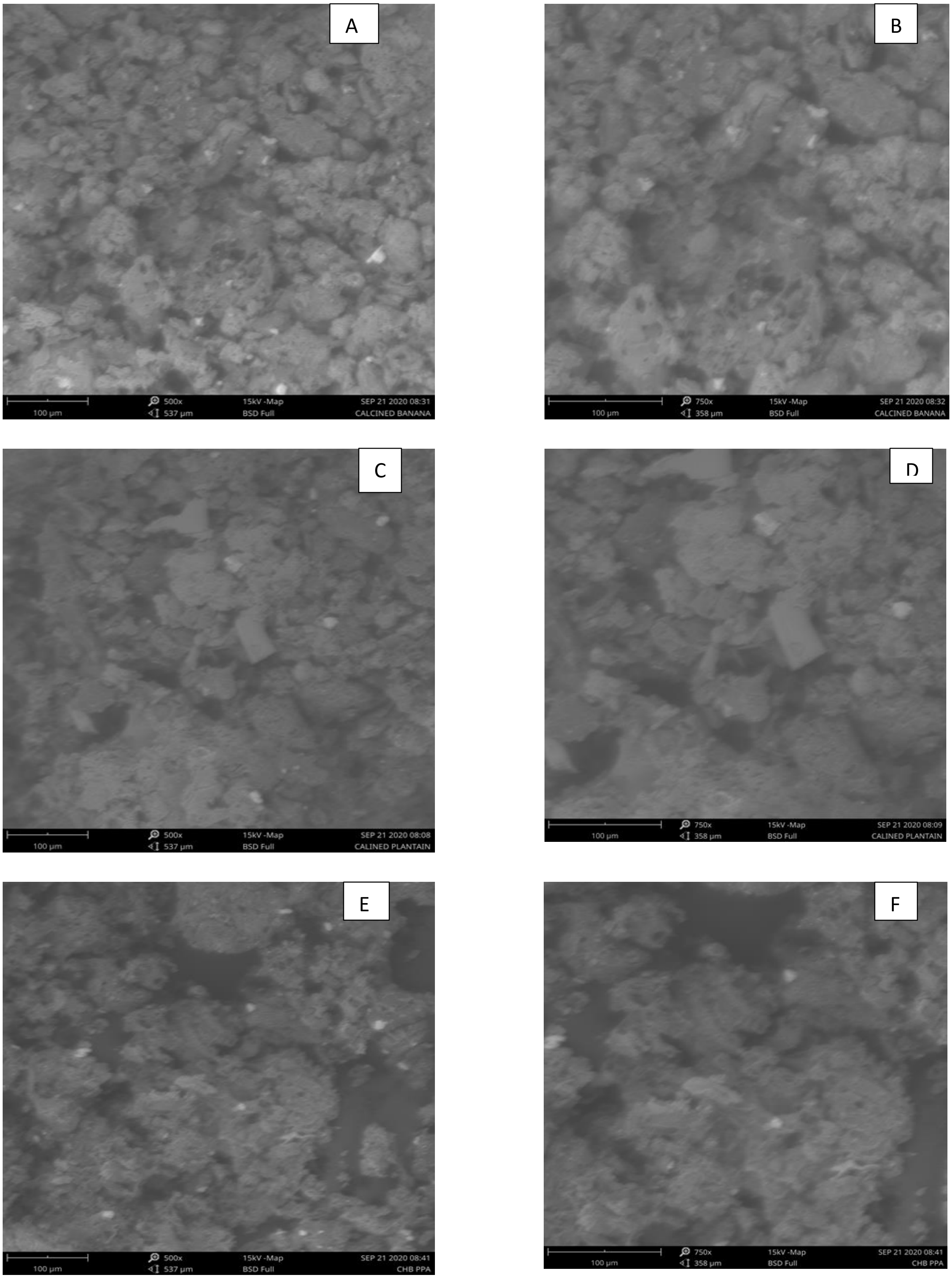

Figure 1: SEM images of (A & B) CBPA, (C & D) CPPA, and (E & F) CBPPA

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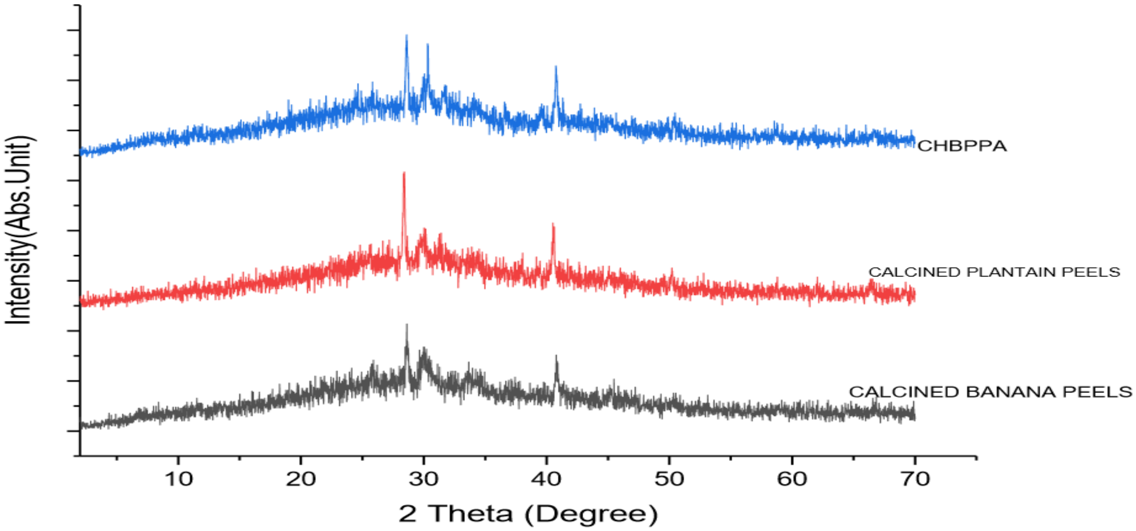

Figure 2: XRD patterns of CBPA, CPPA, and CBPPA

3.2 Effect of process parameters on LOME yield

The LOME yield was affected by temperature, catalyst concentration, time, and methanol-to-WLO molar proportion which was adjudged based on the obtained experimental results. According to Ma and Hanna, (1999), reaction variables' impact on methyl ester yield is a function of the type of catalyst utilized. Therefore, the effects of process-parameters on LOME yield was studied in the presence of CBPA, CPPA, and CBPPA mix catalysts. The catalysts' effectiveness was tested in a lard oil transesterification reaction with a methanol-to-WLO ratio of 10.5:1, catalyst-concentration of 2.5 wt. %, and mixing rate of 300 rpm at 60^{o}C for 1.5 h.

3.2.1 Effect of temperature on LOME yield

Temperature effect on LOME conversion was studied using the observed optimal values of a methanol-to-oil molar proportion of 10.5:1, time of 1.5 h, agitation rate of 300 rpm, and 2.5 wt. % of each of these heterogeneous catalysts (CBPA, CBPPA, and CPPA). The temperature was altered from 45^{o}C to 65^{o}C at a space of 5^{o}C. The lowest chosen temperature was 45^{o}C as very low temperatures prolong the reaction period which is not realistic for large-scale production. In general, high reaction temperatures below 65^{o}C accelerated the reaction speed thus shortening the duration. The temperature effect on LOME yield is displayed in **Figure 3a**. It was found that LOME yield increased with temperature increase but declined with temperature increase exceeding 60°C, and effervesces decreasing the LOME conversion. This is due to methanol-evaporation which possibly preferred saponification reaction (side reaction). This result follows the conclusions of Etim *et al.*, (2022) and Silitonga *et al*., (2015) who related that extended exposure to temperature beyond 60^{o}C gives rise to soap and glyceride formation prior to the completion of the transesterification reaction. Reaction-temperature of 60^{o}C was seen to be optimum as it gave the maximum biodiesel yield for each of the catalysts; CBPA (99%), CPPA (94%), and CBPPA (97%) used. It was maintained to investigate other parameters.

3.2.2 Effect of catalyst amount on LOME yield

The influence of CBPA, CBPPA, and CPPA catalysts was investigated on the LOME yield. The effect of catalyst amount on yield was examined at a catalyst amount range of 1% (weight of the oil used) to 3%, temperature of 60^{o}C, methanol to oil molar proportion of 10.5:1, duration of 1.5 h, agitation speed of 300 rpm. **Figure 3b** displays the correlation relating the forms of heterogeneous catalysts (CBPA, CBPPA, and CPPA) used and their concentrations on the yield of lard oil methyl ester (LOME). Although each of the catalysts used gave a yield of above 95% at 2.5 wt. % catalyst concentration, CBPA (98.8%) yielded more LOME than CBPPA (98.2%), and CPPA (97.8%). However LOME yield using CBPA was marginal when compared with that of CBPPA thus CBPPA was chosen over the two catalysts (CBPA and CPPA) for being readily available and in abundance and also for the sustainability of the biodiesel production process. The yield of LOME increased with an increase in catalyst amount of 2 wt.% for CBPA, 2.5 wt.% for CBPPA, and 2.5 wt.% for CPPA and then remained constant when increased beyond the optimum levels of the catalysts amount. A similar result of 2 wt. % as the optimum catalyst concentration of CBPA was recorded by Gohain *et al*., (2017). The intensification in oil to LOME transition with an increase in catalyst amount before reaching the optimum level could be a result of more basic sites that were made available for the reaction which accelerated the reaction rate by reducing the reaction energy. Increasing it beyond the optimum level resulted in no significant changes as a result of elevated concentration of catalyst making the three-phase solution more viscid which resisted the mass transfer between them and also the saponification side reaction that could be stimulated in the process (Etim *et al*., 2022a; Foroutan et al., 2022). This catalyst produced from waste can be easily made and also separated from the product effortlessly hence cost effective.

3.2.3 Effect of time on LOME yield

Figure 3c portrays the effect of time on LOME yield with the heterogeneous catalysts (CBPA, CBPPA, and CPPA). The reaction time was varied within the range of 1 h to 3 h at an interval of 30 minutes while other parameters were kept constant. It was noted that the yield increased considerably from 1 h to 1.5 h indicating the occurrence of majority of the transesterification reaction at 1.5 h beyond which the yield decreased. The LOME yield declination after 1.5 h is as a result of the changeable nature of the transesterification process that resulted in loss of esters. Further increase in time resulted in constant yield. The extended time from 1.5 h to 3 h is not cost-effective as the improvement was negligible. CBPA, CBPPA, and CPPA-catalyzed WLO methanolysis gave an optimal yield of 99%, 98.2%, and 96% respectively at the optimum reaction condition.

3.2.4 Effect of methanol-to-WLO molar ratio on LOME yield

At the optimum reaction condition (temperature 60ºC, time 1.5 h, catalyst amount 2.5 wt.%, agitation speed 300 rpm), the effect of methanol-to-WLO molar proportion of 6:1 – 12:1 at an interval of 3 moles was examined on the transesterification reaction of WLO with methanol with the heterogeneous catalysts (**Figure 3d**). It was observed that LOME yield increased with an increase in the quantity of methanol used up to 10.5:1 methanol/oil molar ratio. LOME yield was spotted declining beyond 10.5:1 molar proportion. This may well be ascribed to the statement that surplus methanol neutralized the catalyst thus decreasing its efficiency (Ude and Onukwuli, 2019). The noted optimal value was upheld during the course of the experiments to examine the influences of other process-variables.

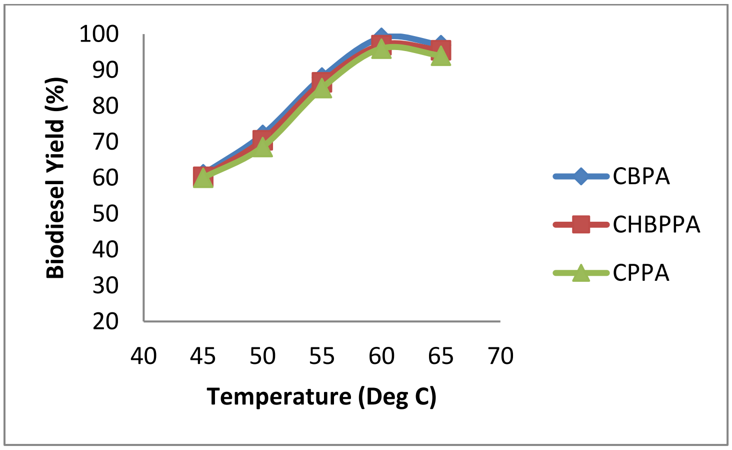

Figure 3a: Plot of LOME yield against temperature

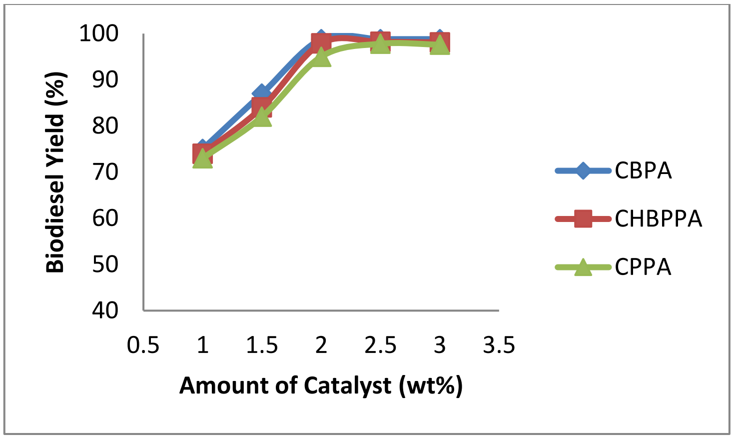

Figure 3b: Plot of LOME yield against Catalyst amount

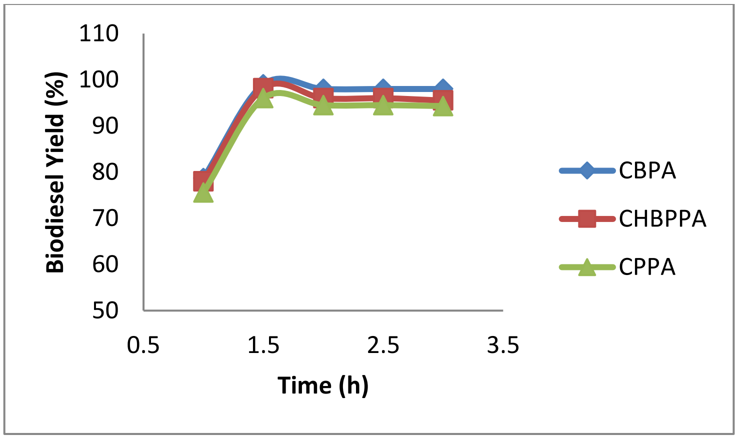

Figure 3c: Plot of LOME yield against time

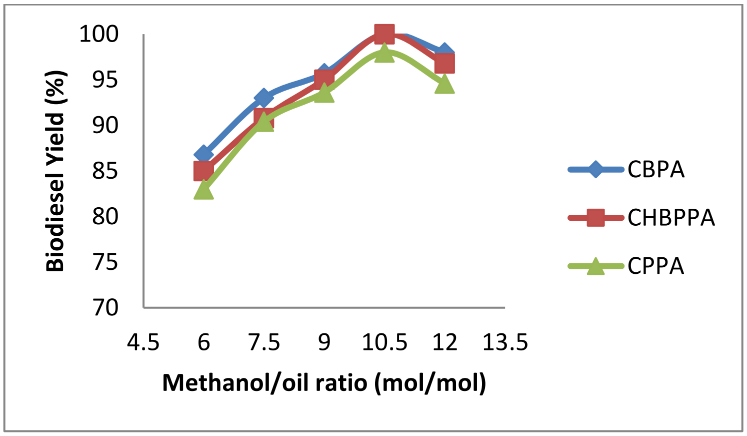

Figure 3d: LOME yield against methanol-to-WLO molar ratio

3.3 WLO and LOME characterization

The properties of the WLO and the optimal LOME yield synthesized at optimum conditions via CBPA-catalyzed WLO methanolysis are depicted in Table 3. The results of the LOME compared with ASTM D6751, and EN14214 standards. All the LOME properties conform to the biodiesel standard specifications. Improved fuel quality was seen after the catalyzed WLO transesterification reaction to LOME when compared with the parent waste lard oil.

Table 3: Physico-chemical properties of WLO and LOME

Properties	Units	WLO	LOME	ASTM-D6751	EN-14214
Acid-value	MgKOH/g oil	2.2	0.28	0.50 max	0.50 max
Specific-gravity@30 ^{o}C	-	0.915	0.873	0.86 – 0.90	0.85
Kinematic-viscosity@40 ^{o}C	mm^2/s	80.994	4.63	1.9 – 6.0	3.5 – 5.0
Saponification-value	MgKOH/g oil	226.459	199.7	-	-
Iodine-value	gI$_2$/100g oil	34.491	32.42		3 min
Cetane-number	-	-	68	47 min	51 min
Higher-heating-value	MJ/kg	-	41.1	40 - 42	
Flash-point	^{o}C	240	135	130 min	120 min
Cloud-point	^{o}C	-	+9	-3 to 12	-
Pour-point	^{o}C	-	+6	-15 to 10	-

4.0 Conclusion

The influence of temperature, catalyst concentration, time, and methanol-to-WLO molar proportion on LOME yield via CBPA, CPPA, and CBPPA catalyzed WLO transesterification respectively were observed. The catalysts confirmed the existence of high alkali-elements with potassium exhibiting dominance with CBPA possessing the highest potassium content. Each of the calcined heterogeneous catalysts exhibited high catalytic activity in the WLO transesterification reaction with over 96% LOME yield. The CBPA-catalyzed WLO methanolysis displayed the highest LOME yield of 99% followed by CBPPA (97%) and CPPA (94%); CBPA > CBPPA > CPPA at the optimum reaction condition. The properties of the optimum product achieved are within the biodiesel international standard. Thus, the fruit peel-derived catalysts are a possible source of an active, ecologically benign, and sustainable feedstock for biodiesel synthesis.

5.0 Recommendation

Further studies on the effect of process parameters on methyl ester yield should include the techno-economic analysis of the production process.

Acknowledgements

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Nomenclature

BET = Brunauer-Emmett-Teller analysis
EDX = Energy dispersive X-ray
SEM = Scanning electron microscopy
XRD = X-ray diffraction
CBPA = Calcined banana peel ash
CPPA = Calcined plantain peel ash
CBPPA = Calcined banana plantain peel ash
LOME = Lard oil methyl ester
WLO = Waste lard oil

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