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Catalytic Performance for Transesterification Reaction Using Waste Cooking Oils Over Nano-Calcium Oxide (n-CaO) Catalyst from Different Waste Bones

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Keywords:
Waste cooking oil,
Animal bone,
Calcinations,
Biodiesel,
Nano-calcium oxide

Abstract

The trans-esterification processes of refined waste groundnut oil (WGO) and sunflower oil (WSO) was optimized using calcium oxide produced from three different animal bones after incineration and characterization. The bone samples were crushed and calcined at 600 °C into fine powders with particulate dimension size of 49 nm. The calcined bones such as, calcined fish bone (CFB), calcine cow bone (CCB), and calcined chicken bone (CCHB) were characterized using the transmission electron microscope (TEM), scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray fluorescence (XRF), Brunnar Emmet Teller (BET), and Fourier transform infrared spectroscopy (FTIR) techniques.The powdered calcium oxide (CaO) was obtained from the waste animal bones to produced methyl ester (FAMEs). The effect of the catalyst dosage ratio on the physicochemical properties such as, yield, specific gravity, density, kinematic viscosity, and acid values of the produced biodiesel were studied and evaluated. The analysis of results using Statistical Package for Social Sciences (SPSS software) showed maximum catalyst dosage and yield for groundnut-based biodiesel (GNB) with CCB at 3.0 wt.% with yield 95.0 % followed by sunflower based biodiesel (SFB) with CCB 4.0 wt. % with yield 95.0 %. The best optimal trans-esterification reaction conditions were a 1:6 molar ratio of oil to methanol, 1 hour reaction period, a 333 K reaction temperature, stirring rate of 1000 rpm, and 3-4 wt. % CaO catalyst loading. This study revealed that waste cooking oils and the obtained calcium oxide were good sources of raw materials that canenhance optimal production of biodiesel that meet the American standards for testing materials ASTM requirements.

DOI: https://doi.org/10.47758/ijn.vi3.54

Introduction

The economic development of every country depends largely on energy. Economy sectors such as, commercial, transport, industrial, and agricultural sectors require energy and the largest sources of energy are conventional fuel such as, coal, natural gas, and petroleum. About 81.1% of energy supplies of the world are obtained from petrochemical sources such as, natural gas, coal and oil, while other sources like, biofuel and renewable energy schemes are around 18.9% [1, 2]. The wide application of fossil fuel in the domestic sector and industrial activities has led to increasing environmental pollution complications as a result of fossil fuel combustion [3].

These concerns have instigated the study and development of alternative safe means of energy generation. Major renewable energy sources are tidal, biofuel, biomass, solar, and wind energy etc. The environmental and economic contribution of these renewable resources is vital, and biodiesel production might be one of the benefits [4]. Methyl ester is a type of fuel derived from the fatty acids contained in vegetable oils or fat. Vegetable oil such as rapeseed, castor oil, and peanut oil can be utilized to make biodiesel using homogeneous or heterogeneous catalysts [3]. The word “waste cooking oil” simply indicates that the edible oil has been used for frying at elevated temperature over a period of time, and could no longer be used for a related purpose. The majority of cities in developing countries dump waste cooking oil into the environment, posing environmental, health, social, and economic difficulties to society. [5]. The biodiesel made from refined waste cooking oil offers eco-friendly fuel with low emission of toxic gases such as,SOx, and NOxetc. Biodiesel obtained from waste cooking oil provides environmental, economic, and waste management benefits. In recent times, solid catalysts have attracted great attention for use in the biodiesel production [6]. Some limitations such as, loss of biodiesel during washing, the impossibility of catalyst reusability, and the use of rigorous separation techniques necessitate the invention of heterogeneous catalysts.

Calcium oxide (solid alkaline catalyst) is easier to handle than homogeneous catalysts, owing to their low toxicity, high basicity, as well as its low cost. The choice of a catalyst is an interesting one because it is green and safe to the ecosystem. [5]. The biological waste materials are abundantly found in our immediate environment. To activate the catalyst surface, it can only be thermally degraded into metal oxides like, calcium oxide and magnesium oxide. These oxides are important for the production of biodiesel due to their activity in the methanolysis of vegetable oil to fatty acid methylester. The nature of conventional fossil fuel, high cost, global warming, and other environmental issues associated to fossils fuel necessitate the search for inexhaustible and sustainable fuel. In the near future, utilization of edible oils as, a feedstock for biodiesel synthesis will competes with food availability, causing the cost of raw materials (feedstock, catalyst, etc.) to account for 60–70% of the actual price of biodiesel fuel, making biodiesel production to be very high.There has been a renewed interest in the exploration and creation of renewable resources that are not competitively demanded for food and non-edible industrial product purposes such as, waste cooking and biological catalysts for low-cost biodiesel production.Thus, waste cooking oil (WCO) and biological catalyst are among the less expensive, unregulated, and renewable resources that can be used to produce biodiesel.Hence it becomes imperative to look for acceptable local alternatives that will probably reduce environmental impact and production cost, thereby creating a feasible economy [7].

In this study, the aim is to evaluate the catalytic performance of calcium oxide obtained from different waste sources (cow, chicken and fish bones) and waste cooking oils in trans-esterification process.

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Experimental procedure

1. Materials collection

The raw fish bone (RFB), cow bone (RChB) and chicken bone (RCB) were collected from Omu-aran in Kwara State, Nigeria. Also, the waste groundnut oil (WGO) and sunflower oil (WSO) were the WCOs randomly collected from local restaurants and groundnut cake fryers.

2. Preparation of nano- CaO particles from selected waste animal bones

The discarded bone was thoroughly washed with water to remove any unnecessary materials adhering to its surface, rinsed twice with distilled water, and dried for 24 hours in a hot air oven at 105°C.The discarded bones were crushed to smaller particle size using a pulveriser. The pulverised waste materials were calcined at 600°C for 2 hours in a vacuum furnace (Box-type resistance furnace).The heat treatment was used to convert organic compounds and carbonates into oxides [7].Afterwards, the solid catalysts (CaO) were stored in a desiccator.

3. Characterization of nano-CaO particles

The morphology, elemental components, crystalline phases, diffraction peak, and functional group of the solid alkaline (CaO) catalysts were determined using scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray fluorescence (XRF), Brunnar Emmet Teller (BET), and Fourier transform infrared spectroscopy (FTIR) on the powdered calcium oxide (CaO) obtained from the waste animal bones.

4. Chemical characterization of the waste cooking oils

The refinement and chemical characterization that include acid value (mg/KOH/g) were as determined by AOCS [8], iodine value ($mgI_2/100g$) as determined by AOAC [9], peroxide value (MeqO2/Kg) as determined by AOCS [8], saponification value (mg/KOH/g) as determined by AOAC [10], and free fatty acids as determined by AOAC [10].

5. Trans-esterification process

The trans-esterification process of the two refined cooking vegetable oils ((RGO and RSO) were subjected to a two-step method to obtain a larger yield of ester, by first neutralizing the free fatty acid with an acid catalyst (esterification) and then transesterification with a base catalyst.With a mixture of oils and methanol at a pre-determined molar ratio, reaction temperature, reaction duration, reaction speed, and CaO as a dosage catalyst, trans-esterification procedures were carried out according to Mustapha et al. [11].

6. Statistical analysis

Statistical examination will be conducted with Statistical Package for Social Sciences (SPSS) 13.0.1 and Microsoft excel spread sheet will be used to determine the standard deviation of the data.

Results and discussion

3.1 Characterization of nano-CaO particles

Table 1 (a) displayed the FTIR spectra of raw fish bone (RFB), chicken bone (RChB) and cow bone (RCB), while Table 1(b) showed calcined fish bone (CFB), chicken bone (CChB) and cow bone (CCB).After calcination at 550 - 600°C, all collagen-derived absorption bands, including those at 1545.45 cm^{-1} and 2800–3400 cm^{-1} bands, vanished, signifying that the organic component had been removed from the bone samples.

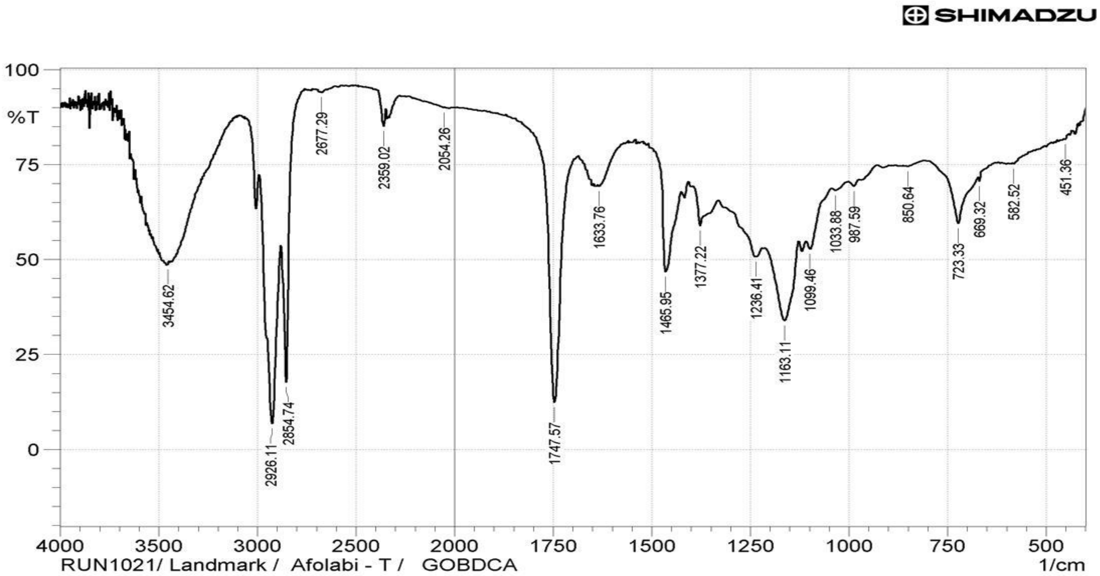
Table 1a Fourier transform infrared spectroscopy analysis of the pre-treated bones (RFB, RChB and RCB)

Wave band frequency (cm^{-1})	Functional group	RFB	RChB	RCB	CaO
3600 – 3200	O – H stretch Vibration (Alcohol)	3288.01	3412.86	3348.00	3446.91
29500 – 2840	C –H aliphatic stretching	2924.53	2917.58	-	-
1750 – 1720	C = O stretching (ester)	1745.63	1787.78	-	-
1680- 1600	C = C stretching of (Alkene aromatic band)	1651.95	-	1655.93	-
1635.69					
1465 – 1440	O-CH$_3$ bend	1464.90	1456.44	-	
1435.09					
1390 -1365	O-CH$_3$ bend	-	-	-	-
1200 – 1020	O-CH$_2$ bend	-			
890 – 885	C=C bend	-	874.41	871.261	
873.78					
730 - 665	C=C bend	721.58	712.40	602.75	
713.69					
545.87		561.81	-	560.56	

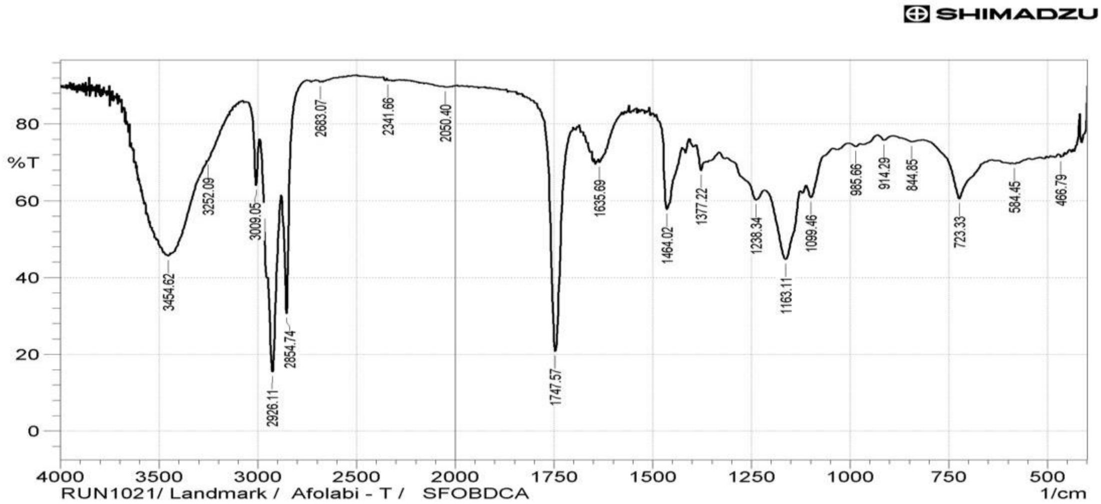
Table 1b Fourier transform infrared spectroscopy analysis of calcined bones (WFB, WChB and WCB)

Wave band frequency (cm-^{1})	Functional group	**Post-incinerated animal bones**			
		Fish bone	WFBWChB	WCB	CaO
3600 – 3200	O – H stretch Vibration (Alcohol)	3495.35	3496.67	3496.67	3446.91
29500 – 2840	C –H aliphatic stretching	-	-	2925.95	-
1750 – 1720	C = O stretching (ester)	-	-	1744.71	-
1680- 1600	C = C stretching of (Alkene aromatic band)	1648.30	-	-	1635.69
1465 – 1440	O-CH$_3$ bend	-	1456.44	1412.77	1435.09
1390 -1365	O-CH$_3$ bend	-	-	-	-
1200 – 1020	O-CH$_2$ bend	-	-	-	
890 – 885	C=C bend	874.41	873.26	873.78	713.69
730 – 665	C=C bend	-	-	-	-
620 – 501			553		

The bands in figure 1 (a - f) at 721.58 cm^{-1}and 1464.90 cm^{-1} (WFB), 712.40 cm^{-1}and 1440.32 cm^{-1} (WChB), and 602.75 cm^{-1}and 871.261 cm^{-1} (WCB), show the presence of out-of-plane bending, asymmetric stretching, and in-plane bending modes of the carbonate groups that were typical of natural dolomite, respectively. The IR bands of calcined bones corresponded to CaO in figure 1 (g) were visible with the vibrations bands at 3495.35, 3412.86, and 3496.67 cm^{-1}. Furthermore, the organic content in the waste bones at band 2925.95 cm^{-1} indicated the presence of alkyl C-H stretch. The similar peaks from CaO catalysts made from crab shell were observed by Joshi, et al. [12].



(a): Raw cow bone



(b): Calcined cow bone

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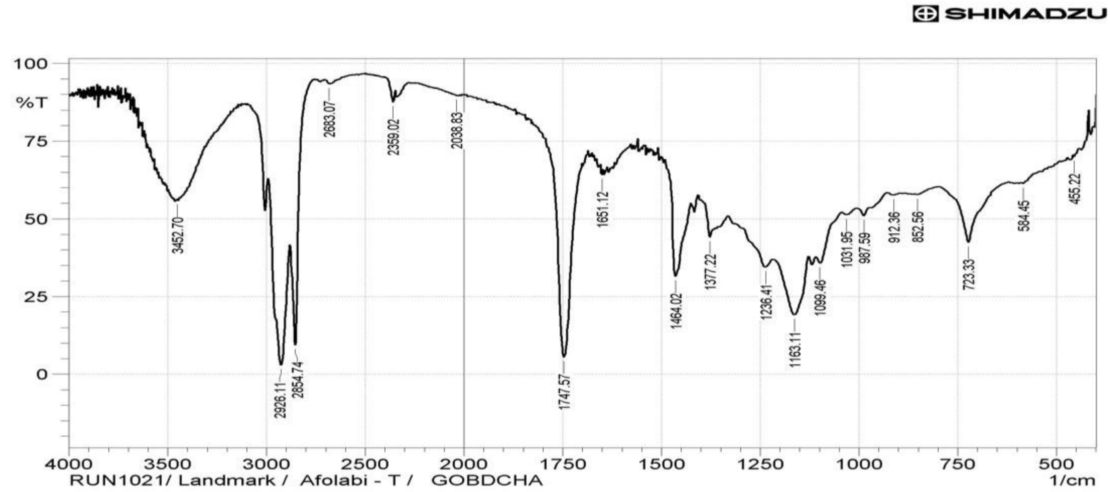
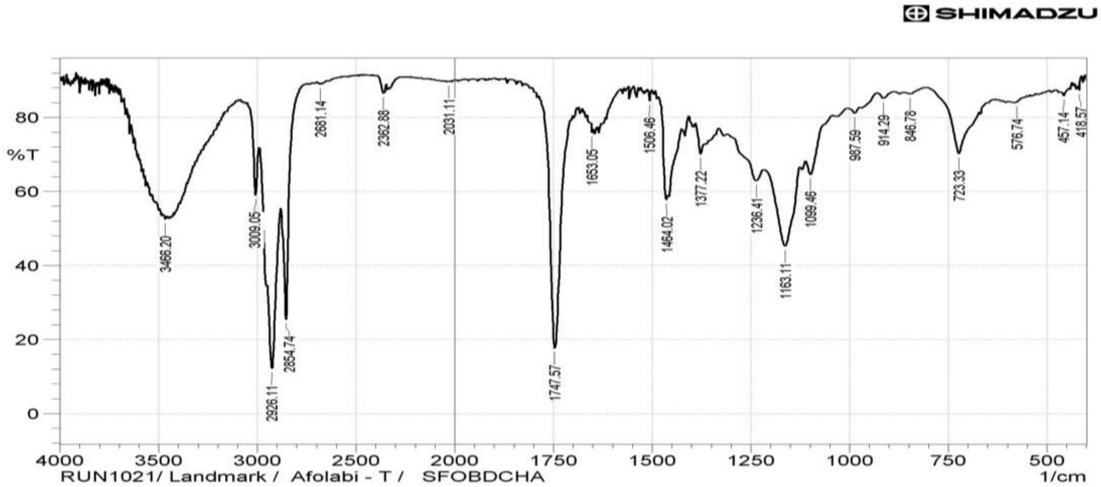
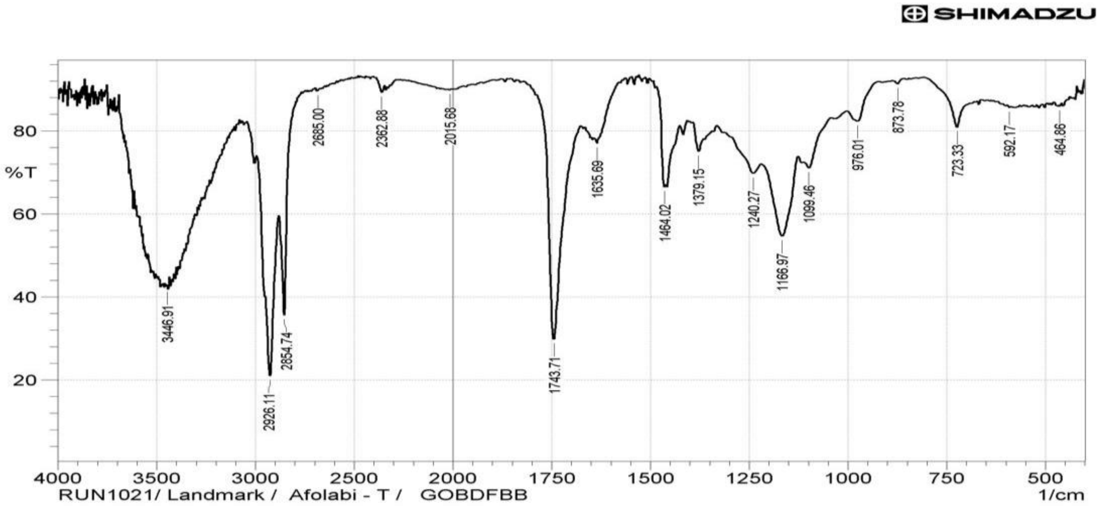


(**c**): Raw chicken bone



(**d**): Calcined chicken bone

(e): Raw fish bone



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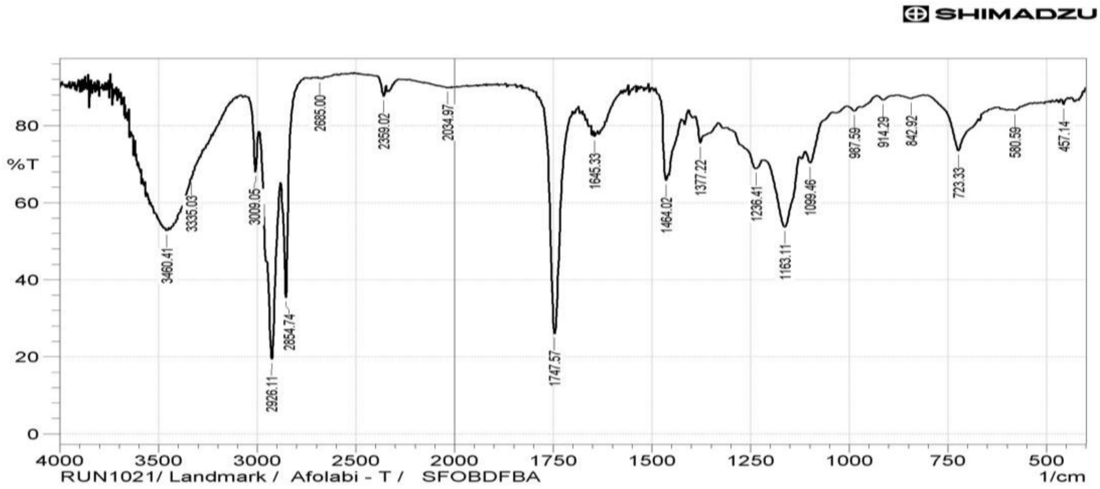
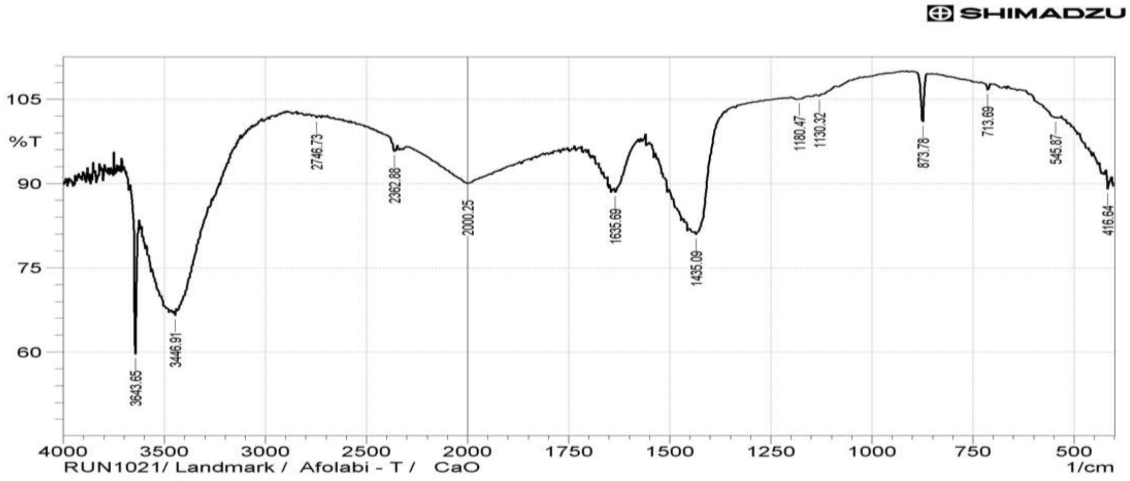


(f): Calcined fish bone



(g): Commercial calcium oxide

Figure 1 The raw and calcined bones of (a-b) cow, (c-d) chicken, (e-f) fish and (g) commercial calcium oxide

The morphology figures 2 (a - f) show the SEM micrographs of the natural and calcites of fish, cow, chicken bones, and compared with standard commercial calcium oxide. The micrograph displays show the structures of irregular and heterogeneous distribution of particle sizes of less surface area of $CaCO_3$ and organic matter), as compared to the calcined bones at 600 ^oC after 2hrs, which show some alterations in its morphology.

The SEM micrographs of the calcined animal bones exhibit particles with lower particle size and more homogeneous distribution probably due to exothermic process that took place in the de-carbonation processes. The TEM morphology of nano-CaO particles is shown in figure 3. It demonstrates that the particles are spherical. The average particle size measured was 35–49 nm.

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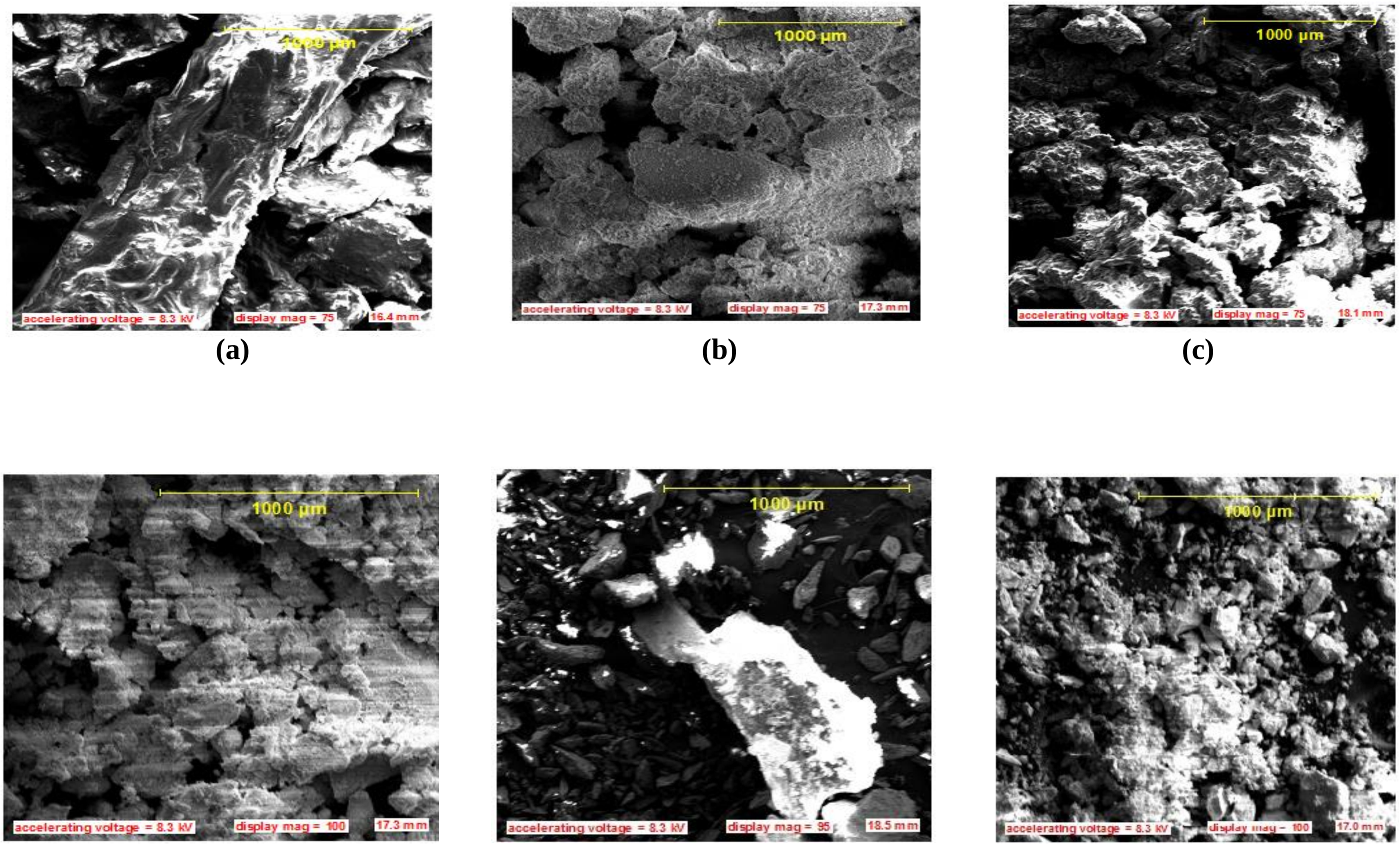

(a) (b) (c)

(d) (e) (f)

Figure 2 Scanning electron microscopy (SEM) analysis: (a) RFB – raw fish bone (b) CFB – calcined fish bone (c) RCB - raw cow bone (d) CCB – calcined cow bone (e) RChB – raw chicken bone (f) CChB – calcined chicken bone

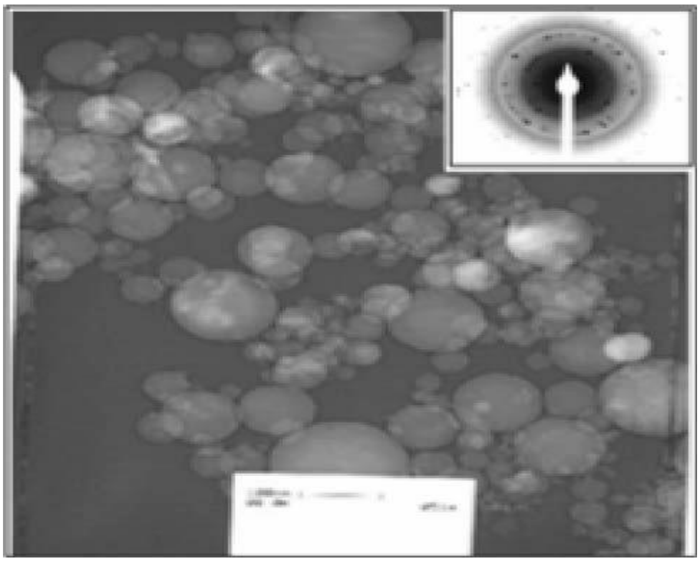

Figure 3: TEM image of nano-CaO showing the diffraction pattern

Figures 4 (a – b) show the XRD- spectra of the raw and calcined animal bones.The peaks observed were referenced using a standard Joint Committee on Powder Diffraction Standards file with the conversion of aragonite ($CaCO_3$) and chlorapatite (carbonated apatite) to calcium oxide that has been enhanced by thermal treatment at 600°C for 2 hours [13]. The formation of hydroxyapatite and other phases on the surface of calcined fish and chicken bones are caused by carbonation and hydroxylation reactions that occur as a result of direct exposure to atmospheric air [14]. Peaks attributable to hydroxyapatite in calcined samples of ostrich bones derived catalyst showed a similar pattern [15].

The determination of the oxide compositions of the alkaline solid catalysts were performed using x-ray fluorescence (XRF).

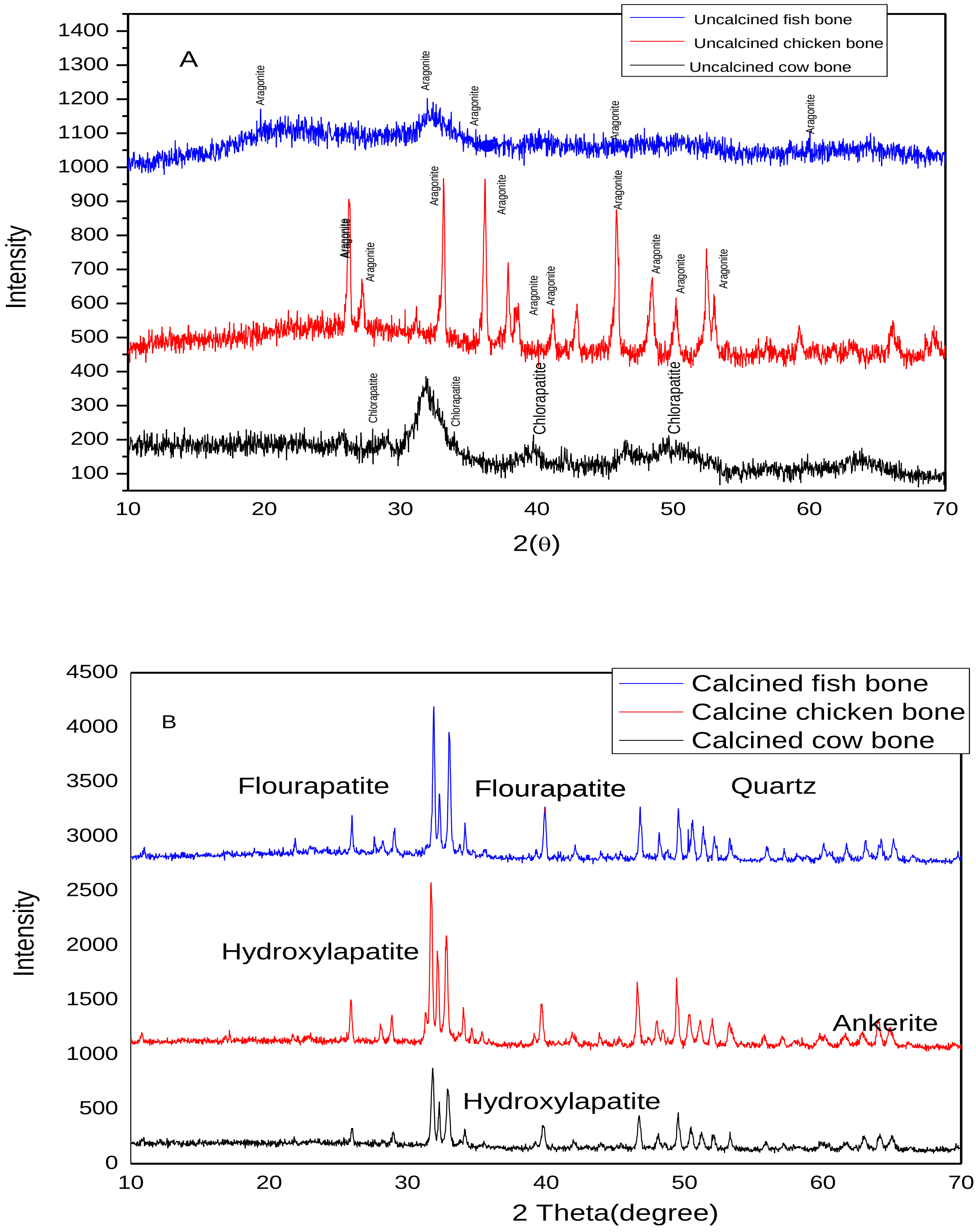

Figure 4 X – ray diffraction analysis of (a) uncalcined and (b) calcined animal bones

In table 2, the oxide composition of calcites (animal bones) wereCaO (70.87, 64.19, and 70.20) for CFB, CCB, and CChB, respectively, and that it could be utilized as a biodiesel base catalyst with high basic strength, low toxicity, and simple water reactions (Joschek, 2016). Trace amounts of other metal oxides made up the remaining 48.8% (MgO, SiO, P_2O_5, Na_2O, Al_2O_3, K_2O, FeO, Cr_2O_3, TiO_2, Na_2O and SO_3). These oxides are active transesterification materials, according to research [16].

Table 2 Oxide composition of the calcined animal bones

Oxide composition (%)	CFB	CCB	CCHB
SiO_2	24.84	26.77	23.83
Al2O3	1.20	1.78	1.09
Fe_2O_3	0.09	1.53	0.09
MnO	0.02	0.01	0.01
CaO	70.87	64.19	70.20
P_2O_5	2.00	3.90	2.05
K_2O	0.12	0.12	0.15
TiO_2	0.04	0.06	0.06
MgO	0.01	0.57	0.80
Na_2O	0.22	0.23	0.20
SO_3	0.21	0.34	0.24

Acidic oxides (SiO_2, SO_3, P_2O_5) mediate the esterification of the feedstock's FFA content, as basic oxides (MgO, K_2O, Fe_2O_3) enhance the catalyst's basic strength.

Effects of catalyst dosage on the yield of biodiesels

1. The catalyst dosage performances on the refined sunflower oil-based biodiesel (RSOBD) and refined groundnut oil-based biodiesel (RGOBD) yields

In figure 5, an increase in the catalyst percentage dosage of post-incinerated cow bone shows a significant effect on the sunflower oil-based biodiesel, with over 80 % yield across. In comparison, 1, 2, 3, and 5 wt.% post-incinerated chicken bone catalysts have comparable higher yields in groundnut oil-based biodiesel. Increased catalyst percentage dosage resulted in the formation of a highly viscous mixture, which caused mixing difficulties and a higher demand for power consumption.

The catalyst dosages for sunflower oil-based biodiesel at 1.0 wt.% of CaO produced 95, 70, 60 % for CFB, CCB, CChB respectively, also 1.0 wt.% of CaO produced 70, 86, 85 % for CFB, CCB, CCHB for groundnut oil-based biodiesel respectively. The 2.0 wt% of CaO produced 95 % yield for sunflower biodiesel. The 4.0 wt.% of CaO for CFB, CCB, CChB produced 88, 95, 86 % respectively for sunflower biodiesel, 4wt.% of CaO produced 75, 80, 95 % for CFB, CCB, CChB for groundnut biodiesel and the 5 wt.% of CaO produced 75 and 80 % for CFB and CCB, respectively. The data were in agreement with the results obtained by Shu, et al [17]

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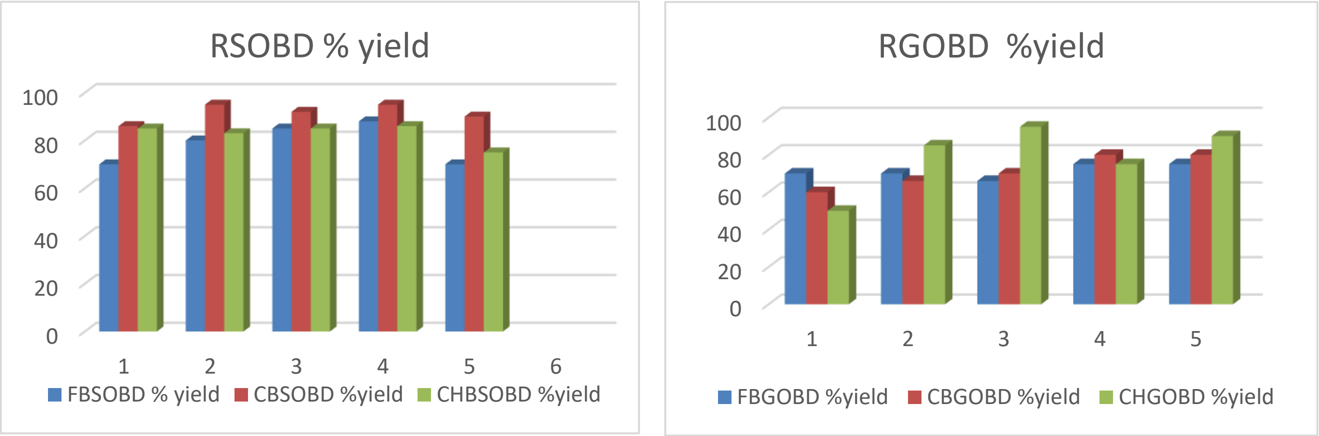

Figure 5 The effect of catalyst dosages on the yields of RSOBD and RGOBD

2. The catalyst dosage performances on the RSOBD and RGOBD specific gravity

The 3.0 wt.% of CaO for CChB has specific gravity of 0.959 for sunflower biodiesel, and the 3.0 wt.% of CaO for CChB was 0.959 for groundnut biodiesel. The specific gravity values were within the ASTM standards (Figure 6).

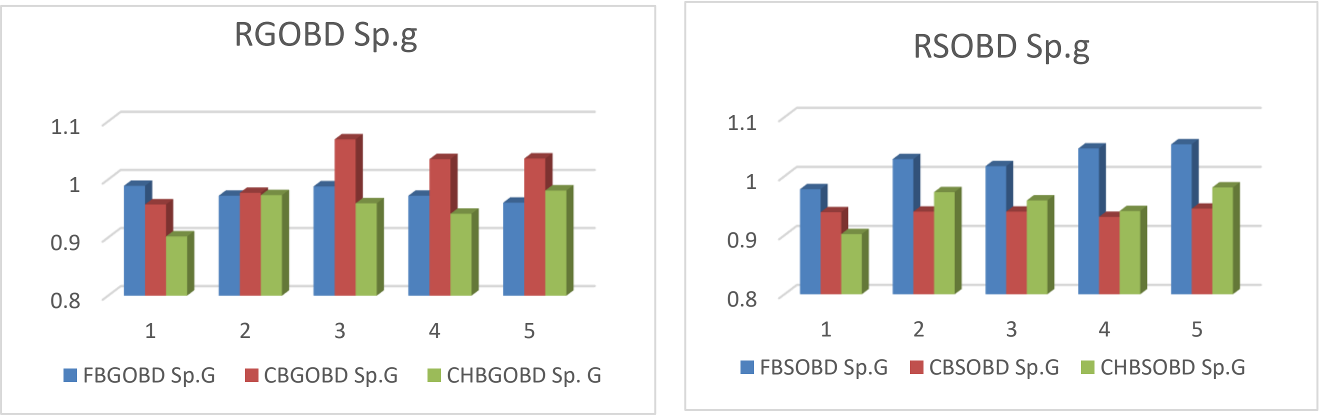

Figure 6 The effect of catalyst dosages on the specific gravity of RSOBD and RGOBD

3. The catalyst dosage performances on the RSOBD and RGOBD density

In figure 7, higher density values can be undesirable as it can affect the atomization during biodiesel combustion, resulting in lower engine performance and increased NOx emission [17].

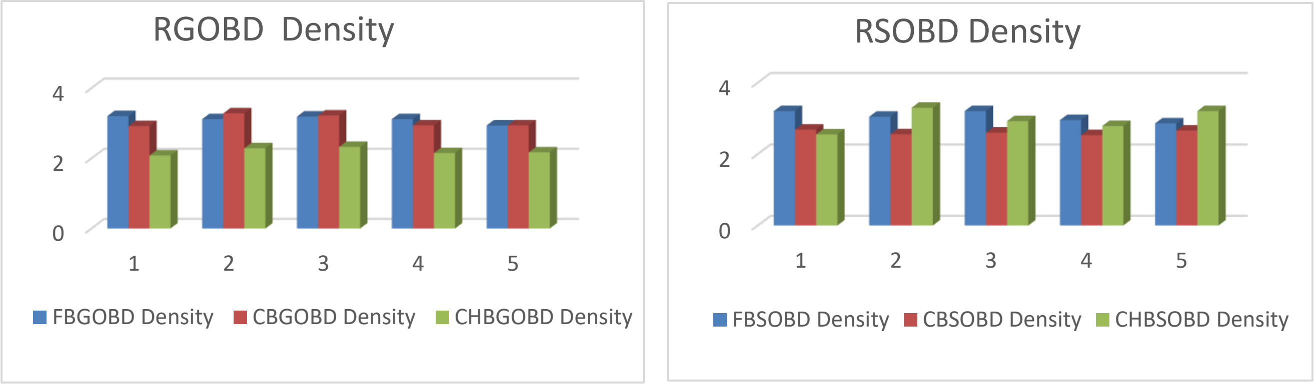

Figure 7: The effect of catalyst dosages on the density of RSOBD and RGOBD

4. The RSOBD and RGOBD kinematic viscosity

In figure 8, the biodiesel synthesized satisfies the kinematic viscosity standard. Highly viscous fuel can shorten the life of an engine. Unburned hydrocarbon deposits can cause problems on engine lubricants, triggering engine damage. For 5.0 wt.% of CaO for CFB, CCB, CChB kinematic values were 5.211, 5.003, and 4.557 for groundnut biodiesel respectively. The 4.0 wt.% of CaO for CFB and CCB kinematic values are 4.942 and 5.034, respectively for sunflower biodiesel. The kinematic viscosity values were within the ASTM limits. Highly viscous fuel can shorten the life of an engine as unburned hydrocarbon deposits can cause problems on engine lubricants, triggering engine damage.

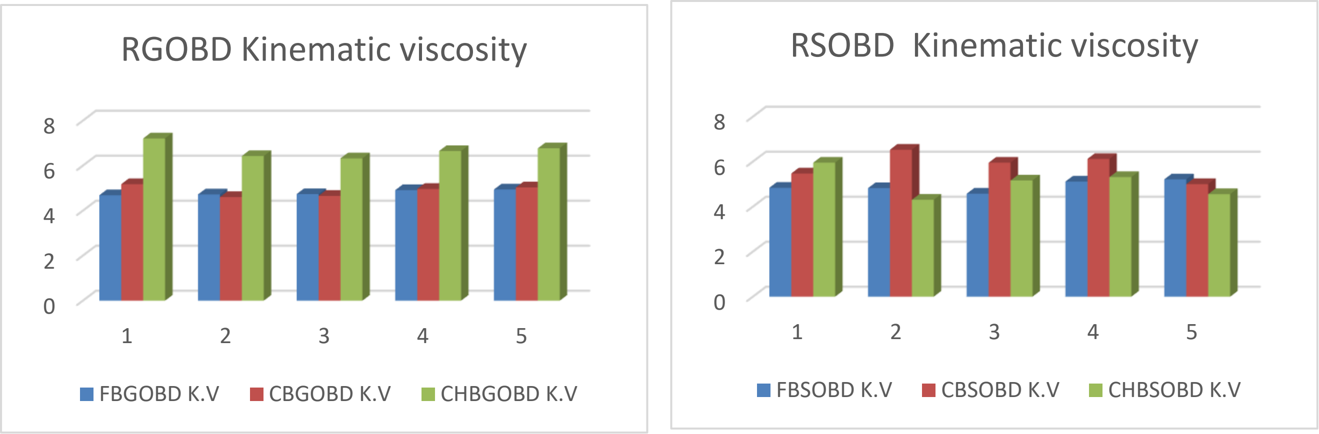

Figure 8 The effects of catalyst dosages on the kinematic viscosity of RSOBD and RGOBD

5. The catalyst dosage performances on the RSOBD and RGOBD acid value

In figure 9, the acid value can also be used as a performance index. For 4.0 wt. % of CaO for CFB, CCB, CChB acid values produced 0.841, 0.392, 0.305 mg/KOH for groundnut biodiesel respectively. The 4.0 wt.% of CaO for CFB, CCB, CChB acid values produced 0.841, 0.561, 1.122 mg/KOH, respectively for sunflower biodiesel. Hence, the biodiesels produced were lower than breadfruit seed oil's 7.38 mg/KOH as the lower the oil's acidity, the lower its rancidity [18-20].

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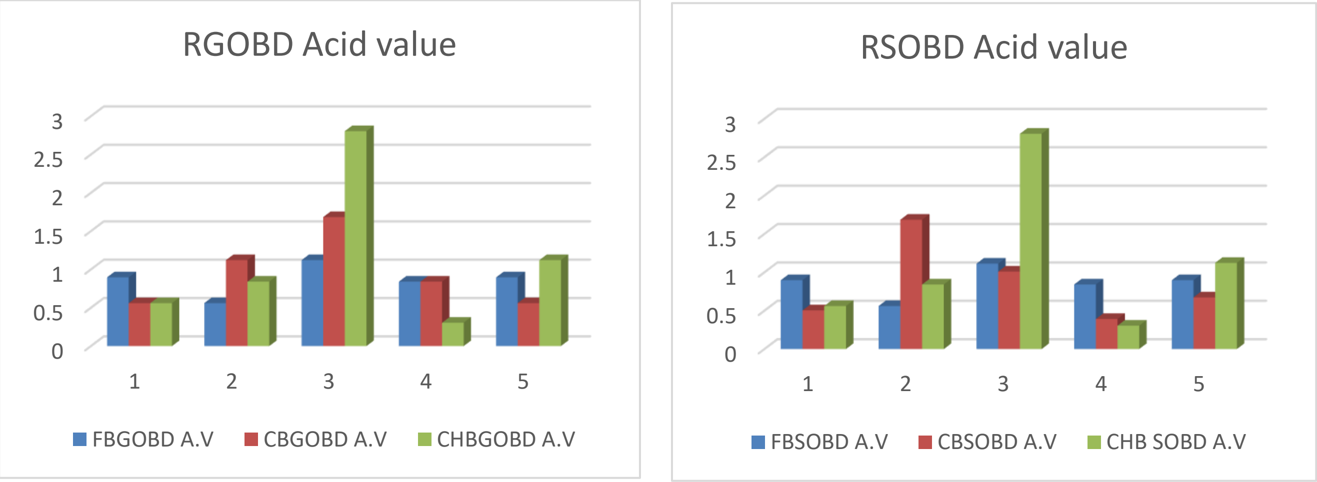

Figure 9: The effect of catalyst dosages on the acid value of RSOBD and RGOBD

6. The catalyst dosage performances on the RSOBD and RGOBD %FFA

In figure 10, for 4.0 wt% of CaO for CFB, CCB, CChB, the FFA values were 0.423, 0.2973, 0.0153 % for groundnut biodiesel respectively. The 4.0 wt% of CaO for CFB, CCB, CChB %FFA values are 0.338, 0.1975, 0.282% respectively for sunflower biodiesel.

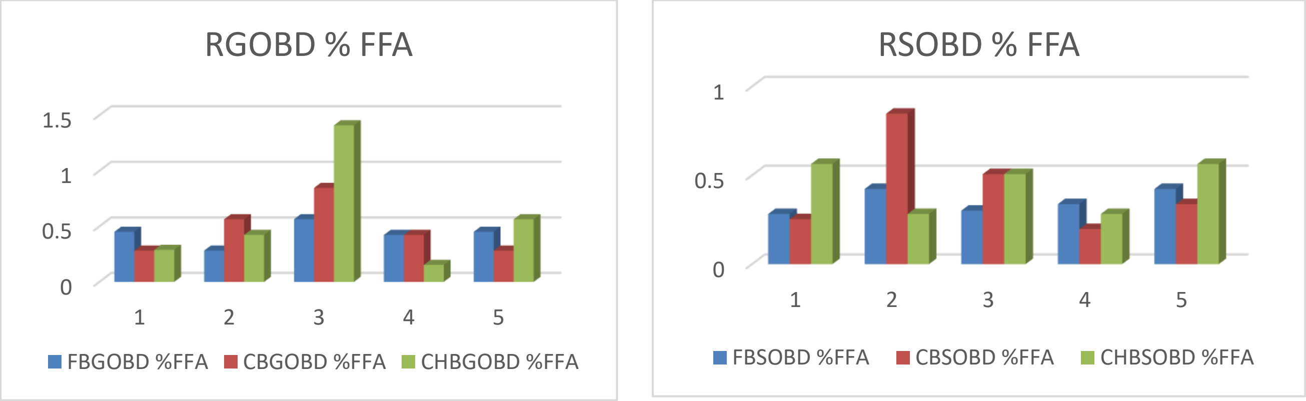

Figure 10 The effect of catalyst dosages on the %FFA of RSOBD and RGOBD

Conclusion

The Calcium oxide as catalyst from different natural sources and doses (waste sunflower and groundnut cooking oils and methanol) were investigated in the production of biodiesel to determine their catalytic performance after characterization. The results have shown that the refined waste cooking oils and calcium oxide from calcined animal bones can be successfully used as a heterogeneous catalyst to produce biodiesel. Our work further showed that, at a moderate methanol to oil molar ratio, the catalyst developed demonstrated promising reusability, and high specific surface area, with considerable biodiesel yield.

Using different catalyst doses from three animal bones and two waste cooking oils, the results showed the optimum catalyst dose of 4 wt. % with better yields and specific gravity, density, kinematic viscosity and acid values that fall within the recommended standards of ASTM. The catalyst results in great outputdue to its low cost, accessibility, its minimal solubility in methanol and ease of handling.The calcium oxide obtained from the calcined animal bones have relative lower environmental impacts that can reduce the cost of biodiesel production.

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