

**INTERNATIONAL JOURNAL OF SCIENCE FOR GLOBAL SUSTAINABILITY****Animal Bone Ash Catalyzed Upgrading of Mango Seed Oil into Biodiesel through Heterogeneous Transesterification****\*<sup>1</sup>A. Galadima, <sup>1</sup>A. M. Lawal, <sup>1</sup>R. Muhammad, <sup>1</sup>M. Abubakar, <sup>1</sup>A. Lawal, <sup>1</sup>J.M. Nahuche, <sup>1</sup>S. Mustapha, and <sup>2</sup>S. S. Abdussalam**<sup>1</sup>Department of Chemistry, Federal University Gusau, P.M.B. 1001, Gusau, Nigeria<sup>2</sup>Department of Chemistry, Federal University Dutsin Ma, P.M.B. 500, Katsina State, Nigeria\*Corresponding Author's E-mail: [ahmadgldm@yahoo.com](mailto:ahmadgldm@yahoo.com)

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**ABSTRACT**

Biodiesel production via catalytic transesterification of non-edible oils derived from plants is one of the priority areas considered by researchers as an approach to reduce the over-dependence on fossil fuels. However, the development of most appropriate catalyst system is still under investigation. The current study therefore investigated the potentials of bone ashes derived from cow, camel and goat as catalytic materials for upgrading mango seed oils into biodiesel under a range of reaction conditions. Irrespective of the bone ash type, the XRF data revealed the presence of CaO, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, MgO, TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub>. Among the nine oxides, CaO and P<sub>2</sub>O<sub>5</sub> were found to play a competitive role during the upgrading reaction. However, reactions influenced by active basic sites in CaO were very favourable and accounted for the production of high biodiesel yields reaching maximum at ~94.5%. Further studies on properties characterization showed the produced methyl esters to have properties in line with those approved internationally for the designing of B100 and other grades of biodiesel.

**Keywords:** Bone Ash; Biodiesel; Base Catalysis; Yield; CaO, P<sub>2</sub>O<sub>5</sub>.**1.0 INTRODUCTION**

Energy supply, demand and consequent utilization are key important parameters that substantially determined a country's economic standard and livelihood of the inhabitants. There is no any iota of doubt, energy serves as the fundamental factor required to assess human life status (Guan et al., 2023). The general civilization status of a nation has linear relationship with adequate energy supply. It is an indisputable fact that the growth of industries, agricultural and health sectors could not be possible without sufficient application of external energy. Globally today, fossil fuels (i.e. petroleum, coal and natural gas) remained the major sources of energy for both domestic and industrial applications. Statistically, the fuels are responsible for 35.6, 28.6 and 23.8% of the global energy supply, respectively. Thus, accounting for a cumulative share of 88% for the worldwide energy supply. The balance 12% comes from other sources such as renewable (i.e. including biomass based and hydroelectric sources) and nuclear power.

Over several decades before now, Scientists around the world have raised serious concerns on the continuous and over-dependence on the fossil fuels as the main energy sources. These could be attributed to certain fundamental and inevitable factors (Liu et al., 2023; Zhang and Hao, 2023; Zhu et al., 2023). The distribution of these fuels is non-linear with some countries having huge reserves whereas others have little or none (Donaghy et al., 2023). Specifically, > 66% of petroleum reserves is housed in the countries around the Middle East. Another factor is the non-renewable nature of the fossil fuels. Their formation requires a very long time (i.e. unarguably hundreds of millions of years). Thus, could be considered as irreplaceable energy feedstock. Coupling these parameters with the fact that the available reserves could be exhausted in the next four to six decades caused threats in the direction of energy sustainability. Despite these factors, serious environmental degradation had been attributed to these fossil fuels (Garba et al., 2012). The emission of CO<sub>2</sub>, CH<sub>4</sub> and NO<sub>x</sub> into the atmosphere from the combustion processes is a global issue with

dangerous consequences (Galadima and Garba, 2011; Galadima et al., 2012). Specifically, CO<sub>2</sub> and CH<sub>4</sub> are two greenhouse gases produced daily with negative effects of global warming (Belaid et al., 2023; Humphrey et al., 2023; Parris et al., 2023; Pfeifer et al., 2023). Statistical evidences have shown that during the period of 31 years (i.e. 1990 to 2021), the effects of global warming associated with the

greenhouse gases increased by 50%, with CO<sub>2</sub> alone responsible for 80% of the cumulative escalations. Similarly, the CO<sub>2</sub> levels in the year 2021 hit about 416 ppm whereas CH<sub>4</sub> and NO<sub>x</sub> hit 1908 and 334.5 ppb, respectively. Figure 1 provides a typical trend of CO<sub>2</sub> and CH<sub>4</sub> emissions for the decades between 1985 and 2020.

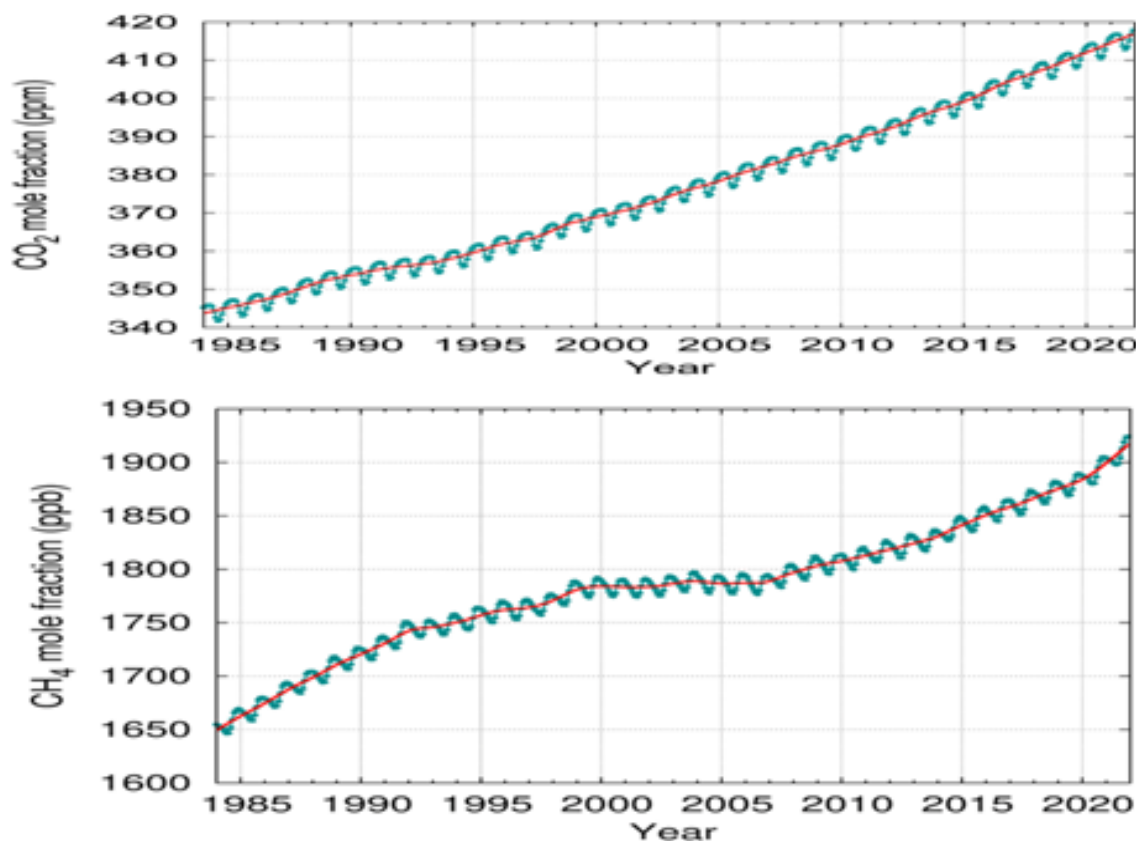
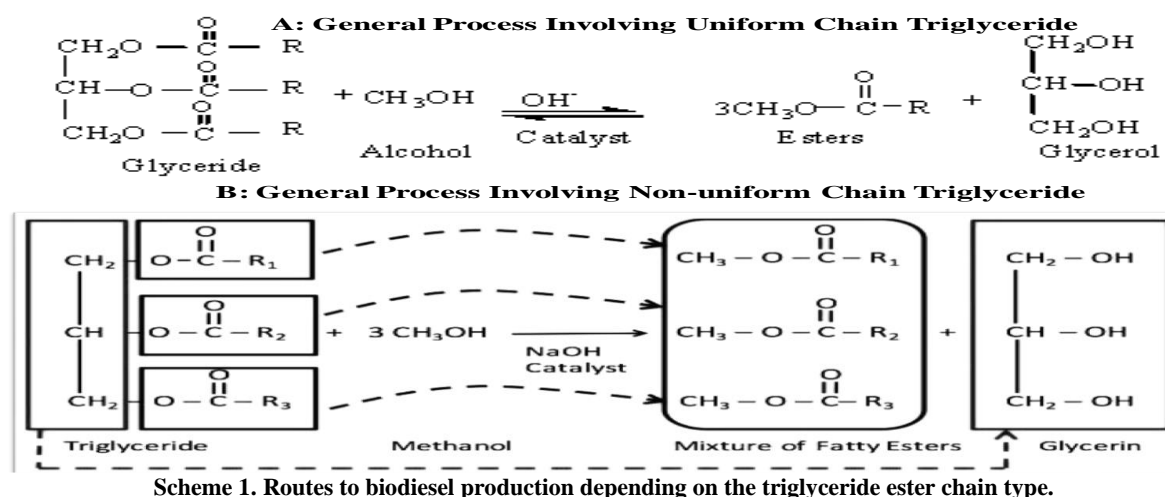


Figure 1. Global trends in the emissions of CO<sub>2</sub> and CH<sub>4</sub> for the decades between 1985 and 2020 (see also Belaid et al., 2023).

To address the various inconsistencies attributed to the fossil fuel feedstocks, a shift to renewable energy alternatives was considered feasible by scholars around the world. While hydroelectric and solar energy options have key advantages, there are indications that the transformation of biomass resources into fuels such as bioethanol, biomethane (i.e. biogas), biopetrol, biodiesel and even the biojet fuels would have greater benefits for the transportation industry (Canavarro et al., 2023; Hassan et al., 2023). Specifically, the biodiesel, which is the main subject of this research, can provide good alternative to the

fossil diesel fuel. It represents a diesel fuel derived from the upgrading of plants/animals oils and/or fats (used or fresh) by reaction with low level alcohols (i.e. commonly C<sub>1</sub> to C<sub>3</sub>) through the incorporation of active catalyst species under variable reaction conditions. A typical reaction profile is provided in scheme 1.

The renewable nature of biodiesel, availability of the feedstock and the possibility to formulate its various grades (i.e. B5, B10, B20, B50 and B100) are key advantages that could not be neglected.



Catalysts based on homogeneous alkalis like NaOH, KOH and their NaOR, KOR derivatives (i.e. R = CH<sub>3</sub> or CH<sub>2</sub>CH<sub>3</sub>) and acids covering HCl, H<sub>2</sub>SO<sub>4</sub>, organic counterparts and allied soluble catalysts were the catalytic systems earlier explored for the industrial biodiesel production (Okoronkwo et al., 2012; Galadima and Muraza, 2014; Galadima and Muraza, 2020). However, the recovery dangers, associated environmental problems and the purity challenges of both products (i.e. biodiesel and glycerol) infer that new catalyst materials must be designed. Oxides, zeolites and supported metallic catalysts have all been tested in the recent times (Basumatary et al., 2023; mIweka et al., 2023; Mulkan et al., 2023; Zhang and Sun, 2023). One important finding is the need for a great shift to the exploration of heterogeneous waste materials from domestic or industrial activities. Sequel to this, the current research targeted the evaluation of three different ashes derived from animal bones (i.e. cow, camel and goat bones) as prospective catalytic systems for upgrading oil derived from mango seed.

## 2.0 EXPERIMENTAL METHODS

### 2.1 Materials Collection and Treatment

**Mango Seed:** the mango seeds were obtained from three different locations (i.e. Sokoto central market, Sokoto State; Basawa mango plantation in Zaria, Kaduna State and Sabon Gida village, Zamfara State). The seeds were thoroughly washed with detergents and distilled water, sundried for 72 h, grounded into powder and stored under laboratory conditions in Pyrex glass-containers for five (5) days prior to further

experiments. Oil was extracted from the powered mango seeds with the aid of Soxhlet extraction procedure. Generally, for each cycle of extraction, 50g of the powdered seeds and 300 cm<sup>3</sup> of n-hexane were employed and the process maintained at 60°C for 5 h. Residual seed powder was removed from the set-up and discarded whereas the extracted oil was further heated to remove n-hexane.

**Bone Ash:** On the other hand, all the bone samples were collected in polyethylene bags from abattoir of Gusau in Zamfara State. They were separately soaked in detergents for 2 h and thoroughly washed to remove attached meat debris, followed by distilled water and sundried for five (5) hours and ashed at 1000°C with the aid of heating oven for 6 h. The ashed bones were further grounded into powder using pestle and mortar and stored separately in labelled plastic containers.

### 2.2 Characterization

The chemical compositions of the powdered bone ashes were established using the x-ray fluorescence (XRF) method as previously reported in our earlier publication (Galadima et al., 2023). On the other hand, properties such as specific gravity and free fatty acid contents (mgKOH/g) for the extracted oil were evaluated in line with the ASTM procedures.

### 2.3 Transesterification and Fuel Properties

Biodiesel production was generally performed using mango seed oil and methanol as the reactants whereas bone as the catalytic material. The separate reactions were conducted in

accordance with Galadima et al. (2023). Fuel properties such as acid number, oxidation stability, methanol content, water and sediments and sulphated ash content were determined using the coded ASTM methods of D664, EN 14112, EN 14110, D2709 and D874, respectively.

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Characterization Results

The results of catalyst characterization as derived from the x-ray fluorescence (XRF) studies are reported in Table 1. All the three bone ash types exhibited similar forms of oxide minerals (i.e. CaO, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, MgO, TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub>). However, the ratio/relative abundance of the two main oxides detected (i.e. CaO : P<sub>2</sub>O<sub>5</sub>) depends on the bone ash type. Bone ash from camel has the highest CaO content of 54.5% whereas the bone ash from goat has the lowest CaO content of 51.8%. In terms of P<sub>2</sub>O<sub>5</sub> concentration, the goat based bone ash exhibits 46.2% which represents the highest detected P<sub>2</sub>O<sub>5</sub> composition for all the three catalytic materials characterized.

Generally, the contents of all the oxides detected are within the ranges found for most domestic animals by different investigators (Bih et al., 2022; Wang et al., 2023; Yessimbekove et al., 2023).

Table 2 presents details on the yield of oil derived from mango seeds via the solvent-based extraction method. Specifically, the yield of 16.50% obtained is in good agreement with 15-17% reported in the literature (Okonkwo and Omenihu, 2021). Oils with good potentials for upgrade to fuels or direct energy applications must exhibit certain physicochemical properties. For instance, the specific gravity of 0.8-1.0 is suitable for oil upgrade into energy fuels. This is due to the fact that side reactions interference and diffusion problems would be strongly suppressed. On the other hand, low free fatty acid composition is beneficial in limiting the neutralization of active catalytic sites when basic materials are employed as the catalyst systems. In this direction, the free fatty acid content of 2.1 mgKOH/g found with the mango seed oil could be considered as prospectively promising

**Table 1. Chemical compositions of the bone ashes as obtained from XRF studies.**

Bone Ash Type	Chemical Compositions
Cow	CaO (52.1%), P <sub>2</sub> O <sub>5</sub> (40.3%), Others (7.06%): SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , K <sub>2</sub> O, Na <sub>2</sub> O, MgO, TiO <sub>2</sub> and Fe <sub>2</sub> O <sub>3</sub>
Camel	CaO (54.5%), P <sub>2</sub> O <sub>5</sub> (39.4%), Others (6.1%): SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , K <sub>2</sub> O, Na <sub>2</sub> O, MgO, TiO <sub>2</sub> and Fe <sub>2</sub> O <sub>3</sub>
Goat	CaO (51.8%), P <sub>2</sub> O <sub>5</sub> (46.2%), Others (2.0%): SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , K <sub>2</sub> O, Na <sub>2</sub> O, MgO, TiO <sub>2</sub> and Fe <sub>2</sub> O <sub>3</sub>

**Table 2. Fuel-related physicochemical properties of the mango seed oil.**

Property	Quantity/Value (average for triplicate analysis)
Percentage Yield (%)	16.50
Specific gravity	0.89
Free fatty acids (mgKOH/g)	2.1

#### 3.2 Heterogeneous Upgrading of the Oil

Biodiesel production from oils and/or fats is a well-established industrial technology considered feasible by companies and policy makers around the world. The application of homogeneous catalysts proved very efficient in producing high yields of the mono-alkyl esters (i.e. the biodiesel products), but the recovery of the catalyst materials and possible re-utilization in subsequent reactions became a serious

difficulty. This specific challenge prompted a devoted research effort on the evaluation of heterogeneous replacement catalysts. Thus, the selection of natural catalysts from animal bones is in this suitable direction. All the oxides detected from the XRF analysis (i.e. CaO, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, MgO, TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub>) have previously been employed for biodiesel production by different scholars under different reaction conditions (Galadima et al., 2020).

Therefore, this combination in a single catalytic material could be beneficial in terms of numerous active sites generation and favouring the production of high biodiesel yields. As demonstrated in Table 3, the yields of biodiesel realized from the current study are in the range of 90 to 95%, and followed the order Camel Bone Ash > Cow Bone Ash > Goat Bone Ash. As illustrated in Scheme 1 above, the biodiesel production process requires three moles of monohydric alcohol for every one mole of triglyceride ester. However, to obtain high yield, the associated equilibrium must be shifted in the forward direction by increasing either the concentration of the alcohol or creating abundant catalytic active sites that will elevate the reaction speed. It is our belief here that the latter factor played a formidable role in achieving yields > 90% as also agreed in some previous literature reports.

Table 3 presents the various fuel properties of the synthesized mango seed oil-based biodiesel

as compared to approved international standards for B100 biodiesel feedstock. These parameters must be maintained at the specified levels to avoid problems such as partial combustion, catalytic converter damage, phase separation, side chain reactions and engine corrosion. Interestingly, all the values obtained are within those limits accepted by the ASTM guidelines for B100 biodiesel fuels. Another advantage observed was the strong inhibition of the possible interference by the free fatty acids detected with the parent oil (see Table 2). Galadima and Garba (2009), earlier demonstrated that when the level of water and sediments exceeds the permitted international limit the catalytic converter could be destroyed due to reversed reactions interference. Accordingly, when acid number exceeds 0.50 mgKOH/g numerous side reactions can override the fuel-to-energy transformation and consequently trigger engine inconsistencies

**Table 3. Physicochemical and fuel properties of the derived biodiesel.**

Parameter	ASTM Limit	Cow Bone Ash	Camel Bone Ash	Goat Bone Ash
Yield (%)		92.15	94.50	90.38
Specific Gravity	0.8-0.9	0.85	0.84	0.88
Acid Number (mgKOH/g; D664)	0.50 max	0.20	0.15	0.23
Oxidation Stability (h, EN 14112)	3.0 min	30.0	28.5	27.2
Methanol Content (%mass, EN 14110)	0.2 max	0.08	0.10	0.04
Water and Sediments (% vol, D2709)	0.05 max	0.02	0.015	0.016
Sulphated Ash (% mass, D874)	0.02 max	0.01	0.013	0.011

### 3.3 Effects of CaO Composition on Biodiesel Yield and Properties

Among the various oxide materials (i.e. CaO, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, MgO, TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub>) detected from the XRF analysis, CaO exists in very high abundance. It present can therefore strongly influence the activity of the

overall bone ash material during the catalytic transesterification process. Specifically, Figure 2 reports a trend on the effect of CaO content on the yield of fatty acid methyl esters (i.e. biodiesel) from the mango seed oil upgrading. It could be established that the yield increased linearly with increased in the CaO composition

in the catalyst. The camel bone ash having the highest CaO content of 54.5% produced the largest biodiesel yield of 94.5%. Accordingly, bone ash from goat which possessed 51.8% of CaO yielded the lowest amount of biodiesel (i.e. 90.38%). CaO had been employed in several reactions due to its possession of strongly active basic sites with potentials to promote catalytic transformations (Li et al., 2022; Yamina et al., 2022; Wang and Li, 2023; Xiong et al., 2023). For example, Lizuka et al. (1971), demonstrated that between 0.57 and 1.0 mmol/g of active sites could be established in bulk CaO material when calcined at temperatures exceeding 500°C. A later review article by Kouzu and Hidaka (2012), showed that these active sites could influence the successful conversion of different

grades of vegetable oils into biodiesel via the solid base-catalysed mechanism (see also Galadima and Muraza, 2014). The work similarly demonstrated that the higher the concentration of this sites the faster the reaction rate and consequently the larger the biodiesel yield. Another important parameter for consideration is the overall purity of the derived biodiesel product irrespective of the CaO in the bone ash. Thus, it is suggested here that the basic sites in CaO have the ability to transform not only the triglyceride esters in mango seed oil but also the free fatty acids detected during pre-transesterification characterization of the oil. Accordingly, the active basic sites in CaO could be defined as very stable under the reaction conditions employed.

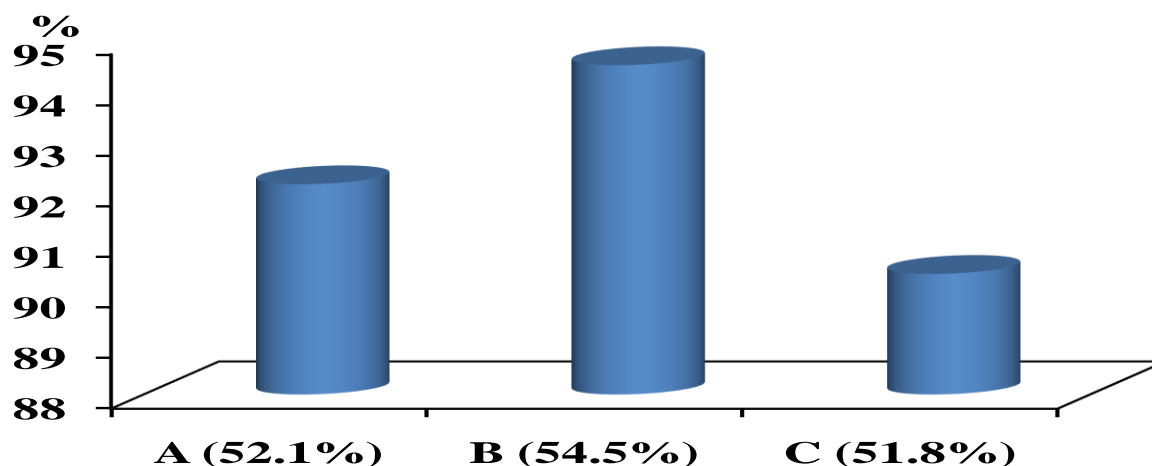


Figure 2. Effect of CaO content on percentage yield of biodiesel for the various bone ash: A = Cow, B = Camel and C = Goat.

### 3.4 Effects of P<sub>2</sub>O<sub>5</sub> Composition on Biodiesel Yield and Properties

P<sub>2</sub>O<sub>5</sub> is another key oxide detected in the various bone ashes characterized and employed for the transesterification process. Apparently, the lower the P<sub>2</sub>O<sub>5</sub> composition the faster the rate of the reaction. Camel bone ash with the lowest P<sub>2</sub>O<sub>5</sub> content of 39.4% produced the highest yield of biodiesel (i.e. 94.5%) whereas the goat ash with the highest P<sub>2</sub>O<sub>5</sub> amount of 46.2% yield the lowest quantity of biodiesel (i.e. 90.38%). These results are consequently opposite to the

role of CaO during the reaction. The existence of P<sub>2</sub>O<sub>5</sub> produced active basic sites that underwent competitive interaction by synergy with those of the CaO material. This type of phenomenon had been found to affect the overall catalytic performance of catalysts containing the P<sub>2</sub>O<sub>5</sub> material (Ebadinezhad et al., 2023; Galadima et al., 2023). In terms of fuel properties, the net biodiesel product exhibited approved properties irrespective of the P<sub>2</sub>O<sub>5</sub> material composition.

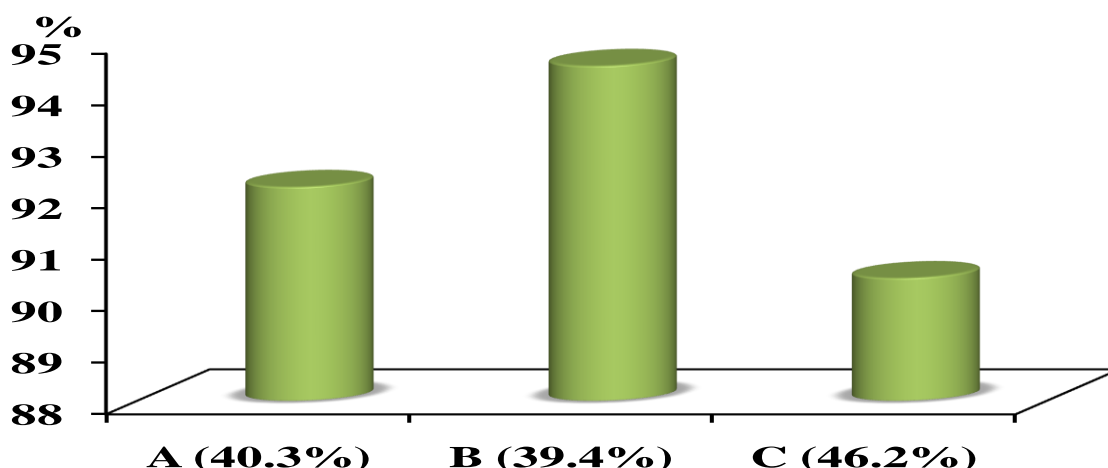


Figure 3. Effect of  $P_2O_5$  content on percentage yield of biodiesel for the various bone ash: A = Cow, B = Camel and C = Goat.

#### 4.0 CONCLUSION

We have carefully employed animal bone ashes to catalyze the upgrading of mango seed oil into biodiesel through heterogeneous transesterification. Irrespective of the originating source, the bone ash comprised of  $CaO$ ,  $P_2O_5$ ,  $SiO_2$ ,  $Al_2O_3$ ,  $K_2O$ ,  $Na_2O$ ,  $MgO$ ,  $TiO_2$  and  $Fe_2O_3$ . Among these oxides,  $CaO$  and  $P_2O_5$  were considered to show a greater influence on the basic-sites catalysed upgrading process. The two oxides similarly displayed competitive activity for the test cycles. Overall, the biodiesel yield of 90 to 95% was achieved as favoured by high  $CaO$  content. Similarly, the obtained methyl esters are good candidates for designing B100 and associated biodiesel grades due to the established properties.

#### 5.0 ACKNOWLEDGEMENT

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