

# Conversion of Jatropha Curcas Oil to Biodiesel in One-Step Transesterification Using Cowbone Derived CaO/Al<sub>2</sub>O<sub>3</sub> with Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> Catalyst

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## Abstract

Mixture of Ferric sulphate (Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>) and Calcium oxide (CaO) supported with alumina were employed as catalysts in this study. Production of biodiesel from edible sources is a threat to food security globally, to alleviate this problem biodiesel can be produced through the exploration of non-edible oils as raw materials and the use of waste resources. This work contributes to the development of a catalyst from waste cow bone supported with alumina for single step transesterification of jatropha curcas oil. The optimal conditions were observed at catalyst loading of 5 wt %, methanol to oil mole ratio of 15:1, reaction temperature of 60 °C and reaction time of 2 hours. Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> to CaO/Al<sub>2</sub>O<sub>3</sub> ratio of 4:1 gave optimum conversion as indicated by Gas chromatography-mass spectrometry (GCMS) of fatty acid methyl ester (FAME) of 96.72% using cowbone derived CaO and 97.01% using the commercial CaO at optimum conditions. Fourier Transformed Infrared Spectroscopy (FT-IR) analysis showed important peaks in 1750 – 1730 cm<sup>-1</sup> that confirm the presence of biodiesel. From the fuel characteristics of jatropha curcas oil methyl esters were found to be well within the range recommended by ASTM D6751 for biodiesel. Therefore, the waste-bone catalysts produced for transesterification of jatropha curcas oil successfully produced biodiesel from jatropha curcas oil using one-step simultaneous esterification and transesterification with ferric sulphate to CaO/Al<sub>2</sub>O<sub>3</sub>. The study's results suggest that non-edible jatropha curcas oil could be a good substitute for petro-diesel. Brunauer–Emmett–Teller (BET) surface area of CaO/Al<sub>2</sub>O<sub>3</sub> was found to be 533.9 m<sup>2</sup>/g with pore volume of 0.2722 cc/g and width of 2.647nm while that of CaO was found to be 427.5 m<sup>2</sup>/g.

## 1. Introduction

Fossil fuel plays a very vital role as energy in our day-to-day activities. Due to the depletion of fossil fuel reserve and the adverse effect of fossil fuel in the environment, there is need for a renewable source with no negative effect to the environment such as release of greenhouse gases. Biodiesel is an alcoholic ester of various fatty acids,

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also known as FAMES (fatty acid methyl esters); it is synthesized from plant oil, lipids of microalgae, animal fat, and sewage sludge via the transesterification process [1]. Biodiesel is a renewable fuel which serves same purpose as conventional diesel fuel. Biodiesel is environmentally friendly because it has low carbon dioxide emission, biodegradable fuel, high cetane number and combustion efficiency, lower aromatic and sulphur content unlike petroleum diesel [2]. Biodiesel can be made from either edible or non-edible oils in the presence of a catalyst. However, the high price of the feedstock used to produce biodiesel is the primary obstacle to its widespread use as a fuel replacement. Therefore, the cost of the final product can be reduced significantly through careful consideration of the feedstock [3]. Depending upon used feedstocks for production and several production technologies, biodiesel is categorized into four generations. Using edible feedstocks like rapeseed, palm oil, corn etc gives first generation biodiesel, whereas non-edible feedstocks like rubber seed, neem, jatropha etc and waste oil can be used for biodiesel production of second generation. Feedstocks that do not compete with crops for land include microalgae, which is used for third-generation biodiesel. The fourth generation comprises genetically modified algae, electro-fuels, and photo-biological solar fuels. This generation represents an emerging field of research that requires extensive studies for further exploration in the future [4]. Apart from the first three common generations of biodiesel mentioned, the fourth generation has also been identified to be made from genetically modified algae [5]. Addition of Alumina support is to affect its surface area and increase the basic sites. Biodiesel is usually produced through esterification and transesterification reactions of vegetable oils and/or animal fats with an alcohol. Methanol or ethanol is usually the alcohol for biodiesel preparation in the presence of either homogeneous or heterogeneous catalyst. The use of CaO for transesterification of oil with high free fatty acid (FFA) (above 1%) is difficult. The amount of FFA is important to select the appropriate catalyst [6]. One step simultaneous esterification and transesterification is a method of producing biodiesel in just a single reaction. The conventional method of biodiesel production requires a first step reduction of high FFA content of oils by acid-catalysed esterification followed by an alkali-catalysed transesterification which is the second step [9]. Homogeneous catalysts are difficult to recover, hence increasing the cost of biodiesel production [10]. Heterogeneous acid catalysts are an alternative to avoid the alkaline pathway for producing alkyl esters from oils with a high content of free fatty acids [9]. In the conventional two steps, Sulphuric acid which is a homogeneous catalyst and not easily recoverable is often used in the first step because of its high conversion and low cost [7]. These disadvantages and high cost of metal oxides have caused research for different sources of heterogeneous catalysts [10]. Bones among other raw materials are several sources of calcium such as eggshells and mollusks. Bone consists of organic and inorganic materials [10]. Cattle bones is containing 42.8% calcium, 16.85% phosphor, 1.08% sodium and 0.71% magnesium. Based on the content contained in cow bones, cow bones can be used as a source of calcium [11]. It also consists of inorganic materials such as calcium phosphate (65-70%), protein and collagen matrix (30-35%). To deal with the higher cost of fatty acid methyl ester and make it a viable fuel on a broad scale, creating a cheap and heterogeneous catalyst from waste resources could be good way [12]. Alumina was added to CaO via wet impregnation method to enhance the catalytic performance of the catalyst by increasing its surface. Ferric sulphate is a substitute for sulphuric acid for the esterification of FFA since it is heterogeneous hence, easy to recover. It is environmentally friendly and easy to separate. Ferric sulphate has much higher catalytic activity compared to sulphuric acid [13]. Jatropha Curcas is an oil-bearing plant that has high oil yield compared to other non-edible plants, and it is drought resistant [14] and [15]. Jatropha Curcas is non-edible and can grow in non-arable land without causing food crisis since it's non-edible [16].

In this study, one step esterification and transesterification of Jatropha curcas oil and methanol was carried out using ferric sulphate and cow bone ash as source of CaO. Previous research has reported on attempts to improve the characteristics of CaO (obtained from discarded bones) by various chemical and physical means. Numerous studies show that CaO can be effectively mixed with other metals to enhance its catalytic property [17,18]. Hence, the objective of the present study was to improve the catalytic activity of the CaO catalyst that had been created using discarded bones by impregnating the catalyst with alumina as the active species. Consequently, the current study's contribution/novelty consists of the development of catalyst CaO/Al<sub>2</sub>O<sub>3</sub> synthesized from waste bones and its utilization in fatty acid production from Jatropha Curcas oil. The products obtained were analyzed with GC-MS and FTIR for esters yield and viscometer for viscosity. Also, the acid value, iodine value, saponification value, cloud point, pour point and density were analyzed using the accepted American Society for Testing and Materials (ASTM) D6751 and EN 14214. Procedure. Characteristics of biodiesel were evaluated, including its Flash Point, Cloud Point, Kinematic Table 1 displays the estimated results, which show that all the properties fall within acceptable ranges for biodiesel.

## 2. Materials and Method

### 2.1 Materials and Sample Preparation

The *Jatropha Curcas* oil (JCO) used for this research was supplied by the National Research Institute for Chemical Technology (NARICT). Cow bones were sourced from Abattoir, Zango, Zaria, Kaduna State. Methanol, calcium oxide and ferric sulphate, in the analysis phase 99% and pure were obtained from Cardinal Chemicals, Zaria. Alumina was collected from the Department of Chemical Engineering. Equipment used are Gallen Kamp hot plate magnetic stirrer, 500 ml one neck flat bottom flask, funnel, filter paper and thermometer. Calcium Oxide was prepared from cow bone. The Cow bones were first washed with water to remove dirt from the bones. The washed bones were boiled with water for 1 hour at 120 °C to aid the removal of oil, fats, tissues and flesh from the bones. The purified cow bones were oven dried at 105 °C for 24 hours, crushed using an electric crushing machine, ball milled, and sieved with 125 µm. The sieved cow bone was calcined at 900 °C for 2 hours.

#### 2.1.1 Preparation of CaO/Al<sub>2</sub>O<sub>3</sub> Catalyst

The wet impregnation method was employed for the preparation of CaO/Al<sub>2</sub>O<sub>3</sub> catalyst [19]. Alumina was used as support on the prepared catalyst because of its high surface area and high thermal stability. 80 % Alumina was mixed with 20 % CaO in 40ml distilled water. The solution was first evaporated at 70 °C for 60 min on a water bath, then oven dried at 120 °C for 90 minutes and calcined at 700 °C for 90 minutes.

#### 2.1.2 Determination of FFA

1.0 g of *Jatropha Curcas* oil sample was dissolved in 25 ml of propan-2-ol with addition of two drops of phenolphthalein to the solution. The solution was titrated against 0.1 M potassium hydroxide until appearance of pink colour is seen. A separate titration (blank) was performed by titrating 25 ml of propan-2-ol against 0.1M potassium hydroxide. The difference of the two titre values (V) was used to determine the FFA of the oil.

$$FFA = \left(0.1M \times V \times \frac{56.1g}{mole}\right) / (1.0g) \quad (1)$$

$$\%FFA = \frac{FFA}{2} \quad (2)$$

## 2.2 One-Step Esterification and Transesterification

One step esterification and transesterification were carried out for methyl ester production from *Jatropha Curcas* oil. The use of one step as against the conventional two steps is to reduce the overall time of reaction, reduce cost of materials and energy consumption [20]. In this work, reaction temperature of 60°C, reaction time of 120 minutes, methanol to oil mole ratio of 15:1 and agitation rate of 400 rpm were kept constant while ferric sulphate to CaO/Al<sub>2</sub>O<sub>3</sub> ratio were varied. Increasing the methanol to oil ratio particular critical value does not promote the reaction rate or percentage yield [21] hence this was kept constant. 50 g of oil was weighed and placed in a 500 ml flat bottom flask and was heated using a Gallen Kamp hot plate magnetic stirrer at 60°C for 60min. 750g of methanol was then added to the heated mixture, stirred with magnetic stirrer and the reaction mixture was allowed to react for 120 minutes. After the end of each reaction time, the catalyst was filtered off and the filtrate was transferred into a separating funnel and allowed to separate overnight. The glycerol which is denser than the biodiesel was collected at the bottom while the biodiesel was washed with hot water to remove impurities after which it was taken for FTIR and GCMS analysis.

### 2.2.1 Catalyst Characterization and Biodiesel Analysis

The catalyst CaO/Al<sub>2</sub>O<sub>3</sub> was characterized for BET surface area, pore size and pore volume using a NOVA 4200e BET machine. FTIR and GCMS were used for analysis of the biodiesel produced.

## 3. Results and Discussions

### 3.1 BET Surface Area of Calcined Cow Bone Supported with Alumina

Table 1 show the catalyst surface area analyzed using NOVA 4200e BET machine. The multipoint BET surface area of the CBA/Al<sub>2</sub>O<sub>3</sub> was found to be 533.9 m<sup>2</sup>/g with pore volume of 0.27 cc/g and width of 2.647nm while that of pure cow bone without support was found to be 427. 5 m<sup>2</sup> /g. The high surface area of the CBA/Al<sub>2</sub>O<sub>3</sub> catalyst was due to high ratio (80% alumina) of alumina on the precursor catalyst. [22] and [23] reported that high surface

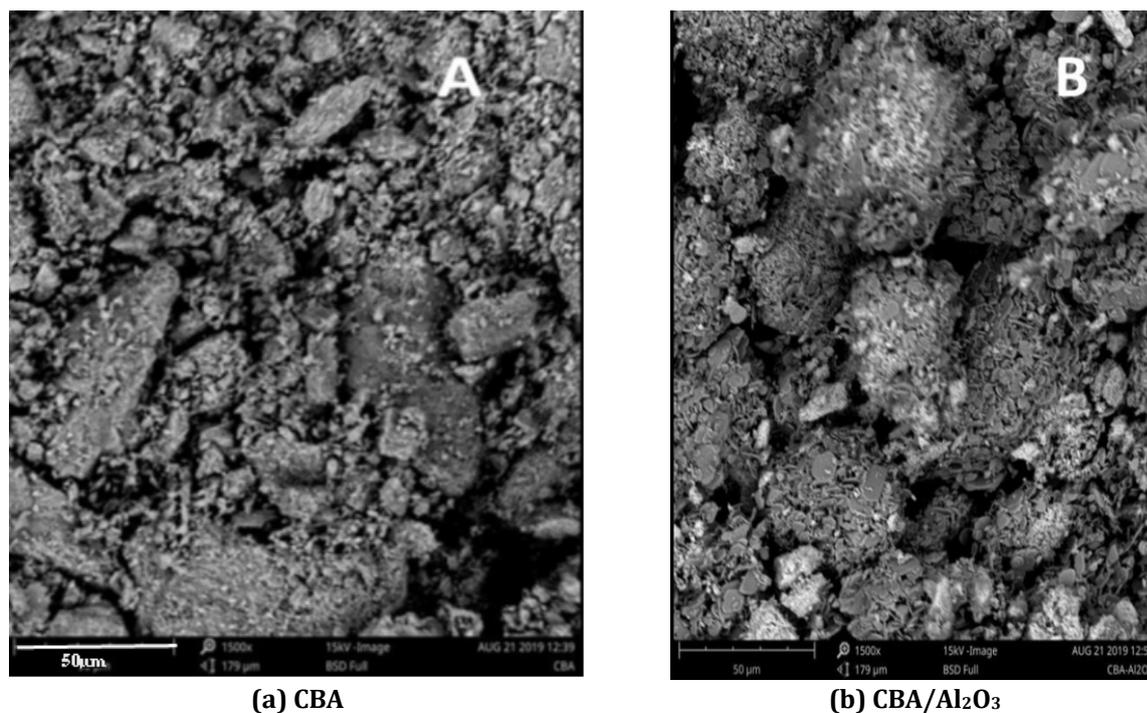
area reduces leaching of precursor to medium and produced a high fatty acid methyl ester conversion. The pore diameter of the composite catalyst of 2.647 nm indicate a mesopores while the pore volume was 0.27 cc/g. A high surface area and pore volume contribute to the catalytic activity of solid catalysts [24]. The high fatty acid methyl ester conversion is due to alumina's large specific area which enhances high dispersion of active sites [25] and [26] reported that alumina-supported potassium iodide gave the highest catalytic activity and highest conversion compared to CaO catalyst. [23] used an alumina support on heterogeneous catalyst and confirmed the catalyst supported with alumina has the highest conversion. High surface area, large pore volume and smaller pore sizes allow triglycerides of different sizes to enter the pores of the catalyst, which gives a large surface for proper transesterification [27].

**Table 1** BET surface area and pore size of CBA and CBA/Al<sub>2</sub>O<sub>3</sub>

Sample	Specific surface area (m <sup>2</sup> /g)	Average pore diameter (nm)	Pore volume (cm <sup>3</sup> /g)
CBA	427.500	6.503	0.191
CBA/Al <sub>2</sub> O <sub>3</sub>	533.900	5.839	5.839

### 3.2 Morphology of Cow Bone Ash

The SEM-EDX data is presented in Figure 1, the SEM image (Figure 1a) of the thermally treated bone shows that the collagens and other organic components originally present in the bone matrix were expelled, resulting in a more porous material [28]. The impregnation of Alumina onto the calcined bone, followed by further thermal treatment, led to a noticeable change in morphology, as illustrated in Figure 1b. The treated bone in Figure 1b displays molecules that have bonded together to form aggregates. These aggregates likely contribute to a higher specific surface area, facilitating better contact between the catalyst and the reactant mixture. This observation aligns with the findings reported by [29]. In contrast, the unsupported catalyst exhibited a rough, layered surface with low porosity and a more compact texture compared to the supported catalyst. The elemental composition of the CaO/Al<sub>2</sub>O<sub>3</sub> catalysts by EDX analysis is summarized in Table 2.



**Fig. 1** Scanning electron microscope (1500X magnification) of (a) Calcined cow bone ash; (b) Cow bone ash/Alumina

**Table 2** EDX analysis CaO/Al<sub>2</sub>O<sub>3</sub> catalyst

Elements	Composition (wt.%)
Al	50.41
Ca	34.07
P	13.91
S	0.52
Mg	0.50
Na	0.30
Si	0.28

In Table 2 The major elements found in the composite catalyst were aluminum, calcium, phosphorous silicon and magnesium. This represents a mix of alkaline (calcium, magnesium and potassium) and acidic elements (silicon and iron) indicating that the catalyst has bifunctional capability. This means that the catalyst has the capacity to simultaneously catalyze esterification and transesterification during biodiesel production. They are also highly active elements and could be responsible for the catalytic activity of the catalyst [30].

The FFA of the raw JCO was calculated to be 6.3 using Equation 1 above hence %FFA is 3.15. FTIR and GCMS were used to analyses the biodiesel produced for conversion obtained. GCMS analysis gave FAME conversion of 96.72% for CaO from cattle bone ash and 97.01% for commercial CaO at 2-hour reaction time, 15:1 methanol to oil ratio, temperature of 60°C and 5wt% catalyst at 4:1 Ferric sulphate: CaO/Al<sub>2</sub>O<sub>3</sub> ratio.

One step transesterification of JCO using CaO/Al<sub>2</sub>O<sub>3</sub> and ferric sulphate as catalyst was done using CaO sourced from cow bone ash and commercially sourced CaO. The FTIR peaks of JCO at 1744.4 cm<sup>-1</sup> confirm the presence of fatty acid methyl ester. These methyl esters were also confirmed in a GCMS analysis where all the esters and the masses of the esters present were listed in Table 4. The fatty acid profile revealed that tridecanoate, linoleic, palmitate and arachidic acids were the dominant fatty acids (Table 4). The results also showed that the biodiesel contained more saturated fatty acid methyl esters. Previous research has shown that biodiesel with more saturated fatty acid methyl esters has higher thermal efficiency and lower emission loads [31]. Saturated fatty acid methyl esters are helpful in maintaining the oxidative stability of biodiesel. The biodiesel samples had specific gravity of 0.89 and 0.88 which are within range of 0.86-0.9 EN standard [32]. Their viscosity was 4.78 also fall within the range 3.5 -5.0 EN standard and 1.6-6.0 ASTM standard [32]. Their acid values are lower than the minimum standard for both EN and ASTM 0.5 mg KOH/g [32]; hence they are suitable for use in any diesel engine. The physicochemical properties are shown in Table 1. The GCMS analysis gave a FAME conversion of 97.01% for biodiesel using commercial CaO and 96.72% biodiesel using cow bone ash supported on alumina. The biodiesel produced from synthesized cow bone and commercial CaO meets minimum ASTM standard of 96.5 % for conversion.

**Table 3** Properties of JCO and derived biodiesel

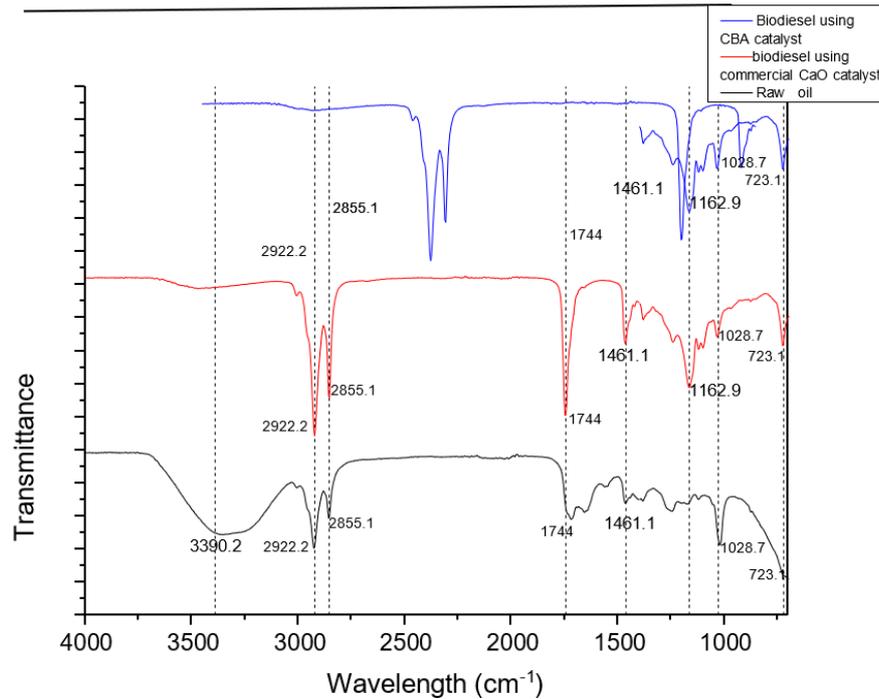
Property	JCO	CBA derived Biodiesel	Commercial CaO derived Biodiesel	ASTM D6751 -02 Standard/ EN 14214
Ester content (%)	-	96.720	97.010	96.500
Density at 40°C (g/cm <sub>3</sub> )	0.920	0.89	0.880	0.875 - 0.900
Viscosity at 40°C (mm <sup>2</sup> /s)	32.500	4.78	4.780	1.900 – 6.000
Acid value (mg KOH/g)	12.600	0.264	0.120	0-0.500
Pour point (°C)	-	4.000	3.000	-15 to 16
Cloud point (°C)	-	13.000	10.000	-3.0 to 12
Iodine value (mg I/100g oil)	102	104	105	120

**Table 4** Fatty acid profile of the produced biodiesel

Peak	Compound	Molecular Formular	Molecular Mass	Conversion%
1	3Hexene2,5diol	C <sub>6</sub> H <sub>12</sub> O <sub>2</sub>	116.16	1.44
2	*Methyl Tridecanoate	C <sub>14</sub> H <sub>28</sub> O <sub>2</sub>	228.38	0.23
3	1,4Dimethoxybenzene	C <sub>8</sub> H <sub>10</sub> O <sub>2</sub>	138.17	0.91
4	2,6Dimethoxyphenol	C <sub>8</sub> H <sub>10</sub> O <sub>3</sub>	154.17	0.41
5	* Methyl Linoleate	C <sub>19</sub> H <sub>34</sub> O <sub>2</sub>	294.26	2.75
6	* Methyl Linolenate	C <sub>18</sub> H <sub>30</sub> O <sub>2</sub>	280.45	3.20
7	*Methyl Hexadec9enoate	C <sub>16</sub> H <sub>30</sub> O <sub>2</sub>	254.22	3.40
8	* Methyl Oleate	C <sub>19</sub> H <sub>36</sub> O <sub>2</sub>	296.27	38.54
9	*Methyl Myristate	C <sub>15</sub> H <sub>30</sub> O <sub>2</sub>	242.40	1.08
10	*Methyl Arachidate	C <sub>21</sub> H <sub>42</sub> O <sub>2</sub>	326.57	3.06
11	* Methyl Palmitate	C <sub>17</sub> H <sub>34</sub> O <sub>2</sub>	270.26	22.15
12	* Methyl Stearate	C <sub>19</sub> H <sub>38</sub> O <sub>2</sub>	298.29	10.25
13	*Methyl Tetracosanoate	C <sub>24</sub> H <sub>48</sub> O <sub>2</sub>	368.63	0.14
14	*Methyl Heneicosanoate	C <sub>22</sub> H <sub>44</sub> O <sub>2</sub>	340.59	3.30
15	*Methyl Lignocerate	C <sub>25</sub> H <sub>48</sub> O <sub>2</sub>	380.14	3.11
16	*Methyl Margarate	C <sub>18</sub> H <sub>36</sub> O <sub>2</sub>	284.48	2.50
17	*Methyl Melissate	C <sub>31</sub> H <sub>62</sub> O <sub>2</sub>	466.84	2.82
18	1Heptadecene	C <sub>17</sub> H <sub>34</sub>	238.46	0.09
19	Hexadecanenitrile	C <sub>16</sub> H <sub>31</sub> N	237.43	0.24
20	Squalene	C <sub>30</sub> H <sub>50</sub>	410.72	

### 3.3 FTIR Analysis

The vibrational characteristics and determination of the functional groups of *Jatropha curcas* oils and the derived biodiesel was done via FTIR spectroscopy. The spectra of raw oil and biodiesel produced using CaO/Al<sub>2</sub>O<sub>3</sub> and CBA/Al<sub>2</sub>O<sub>3</sub> is shown in Figure 2. The most intense and broad peaks occur at 2922 cm<sup>-1</sup>, 2855 cm<sup>-1</sup>, 1744 cm<sup>-1</sup>, 1461 cm<sup>-1</sup>, 1162 cm<sup>-1</sup> and 723 cm<sup>-1</sup>. From the plot, the peaks corresponding to presence of metal carboxylates were absent as depicted by a flat profile within the expected range of 1580 – 1541 cm<sup>-1</sup>, independently reported that peaks corresponding to 1750 – 1730 cm<sup>-1</sup> represent the presence of fatty acid methyl esters which are essentially biodiesel. Peaks at 1744 cm<sup>-1</sup> are the case of produced biodiesel which has low peak for the raw oil. The differences observed between the spectra of raw oil and biodiesel is small since the product of the transesterification process is chemically like its precursor JCO. The peaks at 1461 cm<sup>-1</sup> correspond to the asymmetric stretching of -CH<sub>3</sub> present in the biodiesel spectrum and absent in the raw oil spectrum [32]. The stretching of O-CH<sub>3</sub>, represented by the absorbance at 1162 cm<sup>-1</sup> is typical of biodiesel. Region between 1075–1100 cm<sup>-1</sup> is present only in the raw oil spectrum and absent in biodiesel. These findings are like previous findings for products of biodiesel



**Fig. 2** FTIR analysis of JCO at optimum

### 3.4 Effect of Ferric Sulphate to CaO/Al<sub>2</sub>O<sub>3</sub> Ratio on Conversion

The effect of ratio of ferric sulphate to supported calcium oxide was studied on conversion of biodiesel at reaction condition of 60 °C reaction temperature, 120 min reaction time and 15:1 methanol to oil ratio. At 5 wt % catalyst loading, ratio of ferric sulphate to CaO/Al<sub>2</sub>O<sub>3</sub> at 1:1 gave a conversion of 89.72% for biodiesel using commercial CaO and 88.23% using CaO synthesized from cow bone. Ferric sulphate to CaO/Al<sub>2</sub>O<sub>3</sub> ratio was adjusted to 2:1 and a noticeable increase in yield of biodiesel was confirmed and soap formation was reduced from product formed. The optimum conversion was at 4:1 ferric sulphate to CaO/Al<sub>2</sub>O<sub>3</sub> ratio as shown in Figure 3. Alumina supported CaO with ferric sulphate enhanced high fatty acid methyl ester conversion for single step transesterification. This finding concurs with [25] who reported that alumina has very large specific area which enhances high dispersion of active sites. The high specific surface area of alumina provides a larger interface for interactions between the catalyst and the reactants. This property is particularly advantageous in catalytic processes, as it facilitates better adsorption of reactants onto the catalyst surface and increases the likelihood of chemical reactions occurring at the active sites [20].

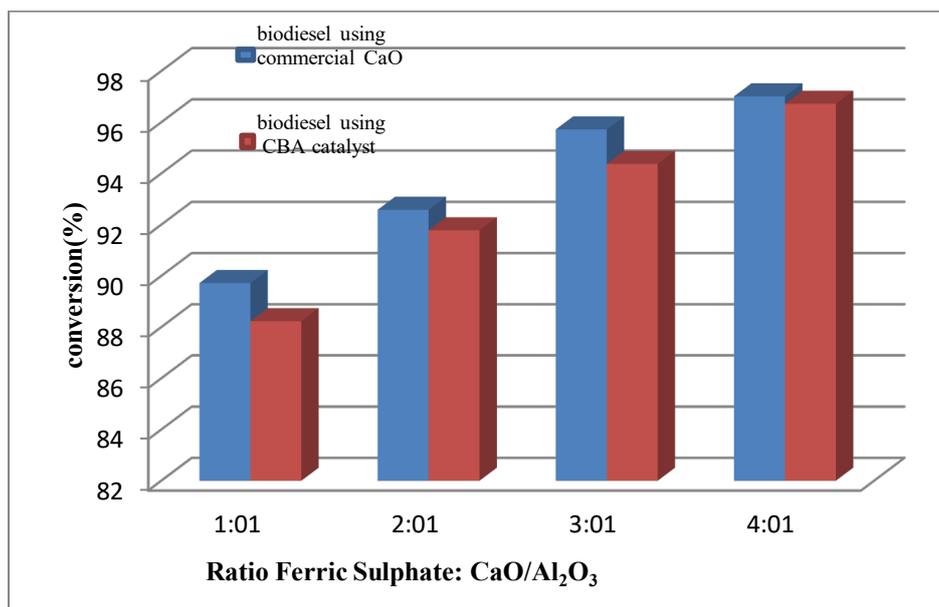


Fig. 3 Effect of ferric sulphate: CaO/Al<sub>2</sub>O<sub>3</sub> ratio on conversion

#### 4. Conclusions

One-step simultaneous esterification and transesterification method using heterogeneous catalysts (ferric sulphate, commercial calcium oxide and synthesized calcium oxide from cow bone) both anchored on alumina was carried out on JCO. High biodiesel quality of 97.01 and 96.72 % methyl esters yields were obtained from that catalysed by commercial and synthesized calcium oxide catalysts respectively. These were achieved at 2 hours with 15:1 methanol to oil ratio at 60°C. This method reduces cost and time as the catalysts used are recoverable by simple filtration. Biodiesel of high ester yield was successfully produced using one-step simultaneous esterification and transesterification with ferric sulphate to CaO/Al<sub>2</sub>O<sub>3</sub> of 4:1. BET analysis revealed large surface area and pore volume of the CaO/Al<sub>2</sub>O<sub>3</sub> catalyst which provides many accessible active sites. The FTIR peaks of JCO in 1744.4cm<sup>-1</sup> confirm the presence of fatty acid methyl ester. The high surface area, pore volume, and bifunctionality as revealed by the SEM contributed most to the high efficiency of the catalyst in converting JCO to biodiesel.

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#### Conflict of Interest

The authors declare that there is no conflict of interests regarding the publication of the paper.

#### Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Haruna Ibrahim; **data collection:** Abdulkareem Abubakar; **analysis and interpretation of results:** Nelson Evangnum Jack; **draft manuscript preparation:** Suleiman Yunusa. All authors reviewed the results and approved the final version of the manuscript.

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