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Production and Characterization of Biodiesel from Blended Mobola Plum and Sicklepod Seeds Using Calcined Turkey Eggshell as Heterogeneous Catalyst

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ABSTRACT

Energy remains a significant element of socio-economic development even during the contemporary world energy crisis period. Biodiesel represents an alternative to fossil fuel sources. This study revolves around the production of biodiesel from Mobola plum (*Parinari curatellifolia*) and Sicklepod (*Senna obtusifolia*) seed oil blends via transesterification reaction under Turkey eggshell-derived CaO catalyst produced from calcination between 800°C and 900°C for 2 hours after cleaning, drying, and crushing processes. The proportions of blending for 50:50, 30:70, and 70:30 ratios resulted in the extraction of oil with oil content percentage of 8.59%, 12.30%, and 17.92% correspondingly. Transesterification reaction of oil extracts resulted in biodiesel production with 90% biodiesel content in 60 minutes at 65°C temperature, 1 percent catalyst, and methanol-oil ratio of 3:1. Physical and chemical analyses revealed compliance with ASTM D6751 standards for biodiesel specification. FTIR analysis confirmed the existence of alkanes, ester, carboxylic acid, alcohol, ether, and methyl ester elements. X-ray diffraction (XRD) and x-ray fluorescence (XRF) analysis of the catalyst revealed calcite as the principal element with 76.606% CaO content percentage.

Keywords: Biodiesel, Mobola plum, Sicklepod, Turkey eggshell, Calcium oxide, Heterogeneous catalyst

1.0 INTRODUCTION

The demand for energy is increasing on account of industrialization, mobility, and the increase in population, which cause exhaustion of fossil fuels along with environmental hazards like greenhouse gas emissions [32]. Fossil fuels have posed various challenges and issues within societies, for example, security of supply, financial security, improper burning process, and possible health and environmental threats [20]. Increasing energy needs of the world together with the issue of depleting oil reserves have paved the way for discovering alternative fuel sources [24]. In this regard, biodiesel has become an important source of energy due to its renewability and cleanness. [1] Besides, biodiesel has become an important clean and green energy source as a substitute for conventional diesel (No. 2 diesel) in motor vehicles. It consists of animal fat or vegetable oil methyl esters and can be prepared using catalytic transesterification reactions between oil and methanol.

Mobola Plum (*Parinari curatellifolia*) is an evergreen African native tropical tree, and the oval fruits (about 50 mm long) have edible yellow pulp. The seeds of this plant produce more than 50% of oils and, hence, are potential biodiesel sources [23]. These same properties are demonstrated in the work of [29]. Sicklepod (Senna obtusifolia L) is an annual West African weed belonging to the leguminosae family. Its seeds consist of protein (14-19%), fat (5-6%), and carbohydrates (66-69%). The plant was earlier used in traditional medicine, and its polysaccharide yield has currently attracted industrial attention [28]. Seeds of Mobola plum (*Parinari curatellifolia*) and Sicklepod (*Senna obtusifolia)* are unexploited oil-producing crops. The former has high oil content [9], whereas the latter has considerable amounts of lipids but also contains contaminants that hinder yield [28]. Combining both seeds enhances oil extraction while cutting costs. Using the two crops will lead to increased output at reduced costs without creating a food versus fuel conflict [9].

The heterogeneous catalysts like calcined eggshell-derived calcium oxide are environmental-friendly, recyclable, and economically priced [5]. Turkey eggshells calcined possess high levels of CaO and have proven successful in producing biodiesel [36]. On the other hand, the use of homogeneous catalysts such as KOH and NaOH has proven efficient; however, they generate soaps that are difficult to separate and reduce biodiesel production yields [25]. Low-cost and recyclable environmental catalysts like calcined eggshells-derived calcium oxide can be used in producing biodiesel, but there is less research conducted on the use of this type of catalyst for mixed non-food feedstock.

Conclusion, by [14]. In comparison with diesel fuel, biodiesel is superior to petroleum-based fossil fuels in terms of the following: non-toxicity, lack of sulfur dioxide (SO_2) release, high flashpoint (at least ≥100°C), high cetane index, low smoke and particulates emissions, low carbon monoxide (CO) emissions, and unburnt hydrocarbons.

The purpose of this study was to produce biodiesel from blended seeds of Mobola plum and Sicklepod using calcined turkey eggshell as a heterogeneous catalyst

2.0 MATERIALS AND METHODS

2.1 Materials and Reagents

Equipment's used to carry out this research are; Soxhlet extractor, thermometer, Viscometer, Cloud and Pour point Analyzer, Stirrer, density bottle, Refractometer, Hot plate, Weighing balance, Beaker, Oven, Burette, Conical flask, Pipet, Retort stand and Filter paper.

FT-IR spectrophotometer (Vertex 70, Bruker), NYC12 muffle furnace, water bath HHW420 (B-scientific England), heating mantle and Reflux condenser.

All reagents used in this research work were of analytical grade All glass wares used in this research work were properly washed with detergent, rinsed with distilled water and dried.

2.3 Study area

The research was conducted at the Permanent Site of the Federal University of Lafia, located in Lafia town, the capital of Nasarawa State, Nigeria. Situated in the north-central geopolitical zone (Middle Belt region), Lafia lies between latitudes 8°25' N and 8°35' N, and longitudes 8°28' E and 8°34' E. The Local Government Area (LGA) covers approximately 258 km², extending about 18 km from north to south and 14 km from east to west [22]. as shown in Fig 1:

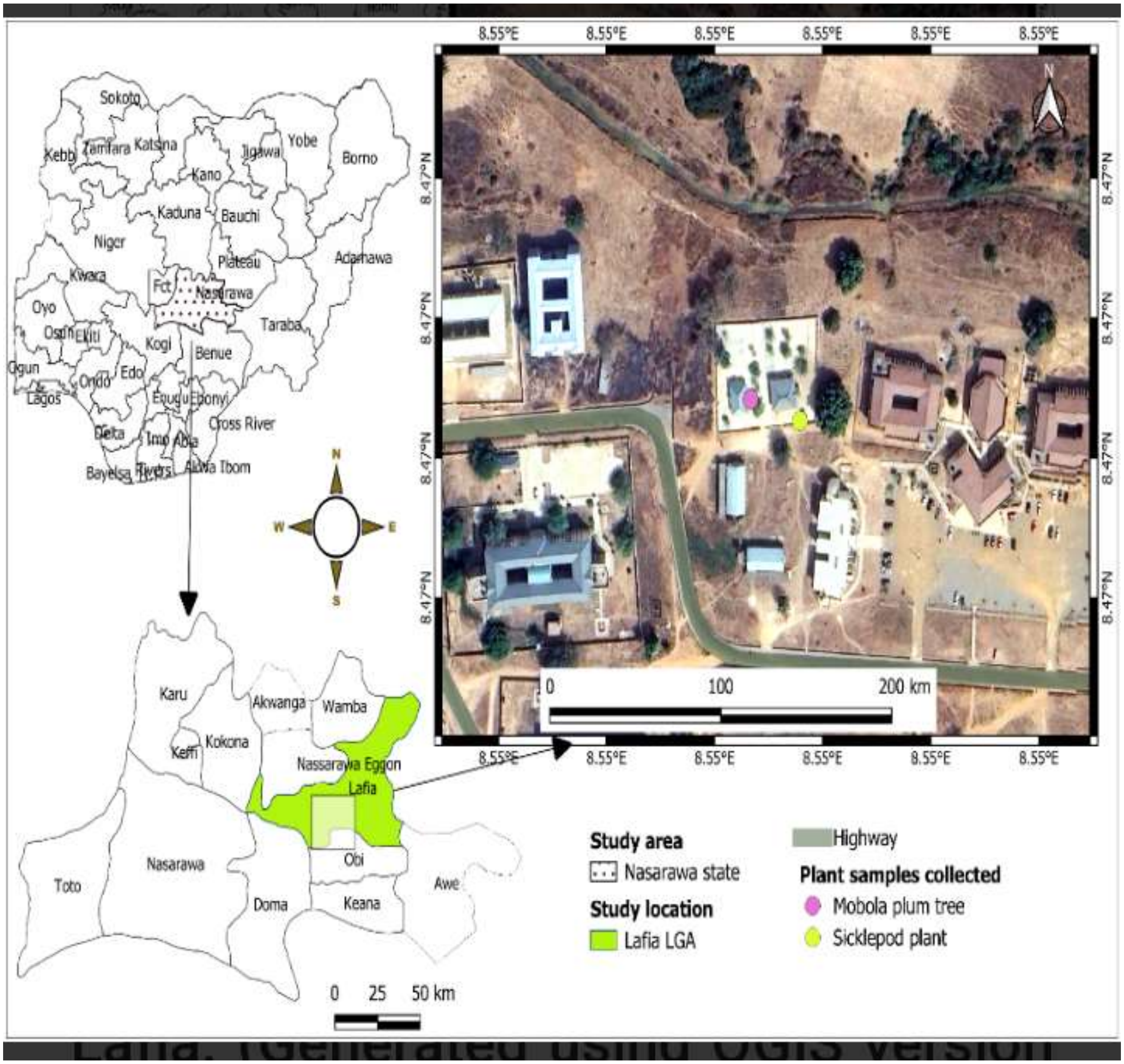

Figure 1: Map of the Plant Species Collection site in Federal University Lafia. (Generated using QGIS version 3.40.1-Bratislava)

2.2 Sample collection and preparation

Fresh seeds of Mobola plum and Sicklepod were obtained from the university's permanent site. A total of 4000 g of seeds were collected in a clean polythene bag and subsequently identified and authenticated at the Department of Plant Science and Biotechnology, Faculty of Science, Federal University of Lafia, Nasarawa State.

Turkey eggshells were obtained locally in Lafia, Nasarawa state, Nigeria. And washed, dried, and ground to powder.

2.4 Methods

2.5 Determination of moisture content

The moisture content was determined using procedure of [14]. A 1.00 g seed sample was weighed(W_1) into a pre-dried crucible and dried in an oven at 105 °C for 1 h. The crucible was cooled in a desiccator and reweighed (W_2). Drying and reweighing were repeated until a constant weight was obtained. Moisture content (%) was calculated in equation 1 below [14].

$$\text{Percentage moisture} = \frac{W1 - W2}{W1} \qquad (1)$$

2.6 Extraction of oil

Seeds were blended in ratios of 50:50, 30:70, and 70:30 (Mobola plum:Sicklepod). Oils were extracted via maceration using n-hexane. Extracted oils were filtered, and solvent removed under reduced pressure. Oil yield was calculated by modification method [13].

The physiochemical parameters such as acid value, free fatty acid, specific gravity, moisture content, oil yield, color, kinematics viscosity flash point, cloud point, pour point and pH were determined.

2.7 Catalyst Preparation

Turkey eggshells were washed with distilled water, dried at 110°C for 2 hr, ground to fine powder, and calcined at 800–900°C for 2 hr to produce CaO. Catalyst characterization was carried out using X-ray Diffraction (XRD) and X-ray Fluorescence (XRF) [35].

2.8 Transesterification

Biodiesel was synthesized by reacting extracted oils with methanol in the presence of the CaO catalyst. Reaction conditions varied to optimize yield:

Reaction time: 30, 60, 90 min, Catalyst concentration: 0.5, 1.0, 1.5 wt%, Methanol-to-oil molar ratio: 3:1, 6:1, 12:1 and Temperature: 65°C. The mixture was allowed to settle for glycerol separation. Biodiesel was washed with warm distilled water and dried at 60°C.

2.9.1 Physiochemical properties

Extracted crude oils and biodiesel were analyzed for color, odor, flash point, pour point, cloud point, density, pH, moisture, viscosity, free fatty acids, acid value, and saponification value following ASTM D6751 and AOAC methods. [14].

2.9.1 Flash point

The flash point was determined according to [7]. Five milliliters (5.00 mL) of oil were heated in a 50 mL beaker at a constant rate on a hot plate. The flash point was recorded as the lowest temperature at which vapors above the sample ignited upon exposure to a test flame.

2.9.2 pH

Following [1].5.00 mL of oil was transferred to a 50 mL beaker, and the pH was measured using a clean, dry electrode of a calibrated pH meter.

2.9.3 Cloud point

The method of [14] was employed. Five milliliters (5.00 mL) of oil were placed in a cylindrical test tube with a thermometer, clamped, and immersed in an ice bath. The cloud point was recorded as the temperature at which a visible cloud first formed at the bottom of the tube.

3.9.4 Pour point

Five milliliters (5.00 mL) of oil were placed in a cylindrical test tube with a thermometer and cooled below 0 °C in an ice bath. The tube was periodically tilted, and the lowest temperature at which the oil still flowed was recorded as the pour point [14].

2.9.5 Specific gravity

Specific gravity was determined using a clean, acetone-rinsed, oven-dried bottle [2]. The bottle was weighed empty (W_1), filled with oil (W_2), and filled with water (W_3). Specific gravity was calculated using equation 2 below.

$$\text{Specific gravity} = \frac{W1 - W2}{W3 - W2} \qquad (2).$$

Where; W_1= weight of bottle + oil, W_2 = weight of empty bottle, W_3 = weight of equal volume of water + bottle

2.9.6 Kinematic viscosity

Kinematic viscosity was determined using a viscometer [7]. The oil was introduced into one arm of the viscometer, drawn into the bulb, and allowed to flow between calibration marks. The flow time (s) was recorded with a stopwatch, and viscosity was calculated in equation 3.

. $KV = t \times K$ (3)

Where, = kinematic viscosity, t = time in seconds, K= viscometer constant = 0.00768.

2.9.7 Free fatty acid (FFA)

Following [2].5.00 g of oil were mixed with 50.00 mL methanol and warmed. Two drops of phenolphthalein indicator were added, and the sample was titrated with 0.2 M NaOH until a faint pink color persisted for 1 min. FFA (%) was calculated as follows in the equation 4.

$$\% FFA = \frac{Titre\ value \times M \times 28.2}{weight\ of\ sample} \qquad (4)$$

Where; M = Molarity of the base.

2.9.8 Acid value

The acid value was calculated from the FFA using the formula follows in equation 5 [2].

Acid Value = % FFA x 1.99 (5)

2.9.9 Saponification value

The saponification value was determined by the method of [2]. Five grams (5.00 g) of oil were mixed with 20.00 mL of 0.10 N ethanolic KOH and boiled gently under reflux for 30 min. After cooling slightly, phenolphthalein was added, and the sample was titrated with 0.50 M HCl until the pink color disappeared. A blank was also run. The saponification value was calculated using equation 6.

$$S.V = \frac{56.1 \times N \times (Vo - Vi)}{m} \qquad (6)$$

Where: V_O – the volume of the solution used for the blank test, Vi – the volume of the solution used for determination, N– Actual normality of HCl used and m– mass of the sample

2.9.10 Density

Density was determined using a specific gravity bottle as described by [2]. The mass of oil was divided by its volume to obtain the density (g/mL). and calculated in equation 7.

$$\text{Density} = \frac{(\text{weight of bottle} + \text{oil}) - \text{weight of empty bottle}}{\text{Volume of oil}} \quad (7)$$

2.9.11 Fourier transform infrared spectroscopy (FT-IR)

FT-IR spectra were recorded using a spectrometer equipped with a Michelson-type interferometer and a ZnSe ATR crystal (shear angle 45°, 10 internal reflections). The chamber was purged with dry air for 1 h prior to analysis. Spectra were collected in the 400–4000 cm^{-1} range at 2 cm^{-1} resolution [2].

3.0 RESULTS AND DISCUSSION

3.1 Oil of Extraction

The oil produced from the blend of Mobola plum (Parinari curatellifolia) and sicklepod (Senna obtusifolia) seeds, shown in Table 3.1. Yields varied depending on the ratio at which they were blended. At the ratio of 70:30, oil yield was the highest, i.e., 17.92%. It was higher than those of avocado (3%) [7] and mango (15%) [20] but lower than castor seeds (55%) and rubber seed (68%) as noted by [6]. This shows that blend ratio 70:30 can be used to produce biodiesel. In agreement with [17], who stated that Mobola plum is abundant in oil (50%) and can be used as biodiesel feedstock. As per [20], feedstocks containing more than or equal to 17% of oil are suitable to produce biodiesel and according to [6], the most economical one will be with high oil content. The order of oil yield at different ratios 30:70 < 50:50 < 70:30.

Table 3.1 Oil Extraction

S/N	Sample	Ratio	% Yield
1st	Mobola pod Sicklepod	50:50	8.95
2nd	Mobola pod: Sicklepod	70:30	17.92
3rd	Mobola pod: Sicklepod	30:70	12.30

3.2 Physicochemical Properties of Crude Oil

The physicochemical properties showed that the seed oil was coffee brown in color with fruity smell due to the presence of pigments in the sicklepod, as presented in Table 3.2. The flash point of the oil was 170 °C, which is less than that of castor oil (228 °C) but greater than that of conventional diesel (69 °C), implying its safety during biodiesel processing [20]. Its cloud point (4 °C) and pour point (−4 °C) indicate that the oil can perform well in low temperatures [20]. Density (0.85 g/mL) and pH (5.5) were consistent with those previously recorded for vegetable oils [4], as well as [8]. Moisture content (10%) is relatively high and thus may adversely affect the transesterification process as it favors hydrolysis and soap formation [6]. However, viscosity (0.43 mm^2/s) was quite low compared to that of castor oil (228.93 mm^2/s) [21] and hence favorable for biodiesel production [20]. Free fatty acid (4.5 mg KOH/g) and acid value (9.0 mg KOH/g) indicated that the heterogeneous catalyst is the best option, although caution should be exercised when using the homogeneous catalyst because it forms soap [27].

Table 3.2 Physicochemical Properties of Crude Oil

S/N	Properties	Unit	Oil	ASTMD Standard
1.	% yield	%	8.95, 17.92, & 12.30	-
2.	Color	-	Coffee brown	-
3.	Odor	-	Fruity	-
4.	Flash point	°C	170	230-170
5.	Pour point	°C	-4	-15 - 10
6.	Cloud point	°C	4	-3 - 12
7.	Density	g/mL	0.85	0.86 – 0.90
8.	pH	-	5.5	-
9.	Moisture	%	10	-
10.	Viscosity	mm^2/s	4.2	1.9 – 6.0
11	% FFA	mgKOH/g	4.5	25
12.	Acid value	mgKOH/g	9.0	0 – 8
13.	Saponification value	mgKOH/g	145	-

M:R= Molar ratio

Cat. Con.= Catalyst concentration

3.3 Biodiesel Production Parameters

Optimization of the reaction time on biodiesel yield revealed that the reaction time should be sixty minutes, giving a yield of 82 %, while shorter (thirty minutes, 57 %) and longer reaction times (ninety minutes, 71 %) resulted in low yields because of incomplete reactions and back reactions/decoupling, respectively in Table 3.3. Loading rate of catalyst was investigated and revealed in Table 3.4 that the use of 1.0 wt% of CaO gave the highest yield (90 %), whereas low (0.5 wt%) or high concentrations (1.5 wt%) lowered the yield because of inadequate active sites or emulsification reactions, respectively [19, 34].

In Table 3.5 Ratio of methanol-to-oil molar was optimized and it was found that the ratio of three to one gave the highest yield (86%), while higher ratios such as six to one or twelve to one reduced the yield, perhaps because of inhibition of glycerol phase separation [18, 10].

Table 3.3: Effect of Different Reaction Time on Biodiesel Yield at 30 Min, 60 Min, 90 Min.

S/N	Batches	Time (Min)	Catalyst (wt%)	Temp (°C)	Molar ratio	% Yield
1.	First	90	1.5	65	6:1	71
2.	Second	60	1.5	65	6:1	82
3.	Third	30	1.5	65	6:1	57

Table 3.4: Effect of Various Catalyst on Biodiesel Yield at 0.5, 1, And 1.5 Wt % Catalyst Loading.

S/N	Batches	Time (Min)	Catalyst (wt%)	Temp (°C)	Molar ratio	% Yield
1.	First	60	0.5	65	6:1	64
2.	Second	60	1	65	6:1	90
3.	Third	60	1.5	65	6:1	83

Table 3.5: Effect of Methanol-to-Oil Molar Ratio on Biodiesel Yield at 1:3, 1:6, and 1:12 Mol/Mol.

S/N	Batches	Time (Min)	Catalyst (wt%)	Temp (°C)	Molar ratio	% Yield
1.	First	60	1.5	65	3:1	86
2.	Second	60	1.5	65	6:1	83
3.	Third	60	1.5	65	12:1	66

3.4 Physicochemical Properties of Biodiesel

The obtained biodiesel had a yellowish green color and a moderate pungent fruity smell. Table 3.6 shows that the flash point (185 °C) of the biodiesel was much higher than that of diesel fuel (69 °C) and met safety requirements. The pour point (−3 °C) and cloud point (5 °C) suggest good cold flow properties under temperate weather conditions [21]. The density (0.877 g/mL) and specific gravity (0.87) meet the requirements of ASTM D6751 standard specifications (0.86–0.92 g/mL) [8]. The viscosity (4.2 mm^2/s) falls into the range of recommended biodiesel (3.5–5.0 mm^2/s) values and is similar to avocado and Terminalia mantaly biodiesel viscosities [35]. The moisture content (3%) and free fatty acid (FFA) (3.38 mg KOH/g) contents of the biodiesel were higher compared with other biodiesel types [32]. The acid value (6.78 mg KOH/g) exceeded the ASTM standards (0.8 mg KOH/g) [14].

Table 3.6 Physicochemical Properties of Biodiesel

S/N	Properties	Unit	Biodiesel	ASTMD6751
1.	% yield	%	82, 90, & 86	-
2.	Color	-	Yellowish green	-
3.	Odor	-	Pungent	-
4.	Flash point	^{O}C	185	230-170
5.	Pour point	^{O}C	-3	-15 - 10
6.	Cloud point	^{O}C	4	-3 - 12
7.	Density	g/mL	0.87	0.86 – 0.90
8.	pH	-	4.8	-
9.	Moisture	%	3	-
10.	Viscosity	mm^2/s	1.7	1.9 – 6.0
11	% FFA	mgKOH/g	3.38	25
12.	Acid value	mgKOH/g	6.78	0 – 8

M:R= Molar ratio
Cat. Con.= Catalyst concentration

3.5 Functional Group Analysis by FT-IR

The FT-IR spectrum of the blended seed oil as well as biodiesel showed the presence of functional groups as shown in Fig. 2 and Fig. 3, respectively with the wave numbers in Table 3.7. Functional groups were observed in the oil spectrum in the form of absorption bands including those at 2918-2849 cm^{-1} (stretching vibration of C-H bond), 1742 cm^{-1} (ester C=O group), 1235-1159 cm^{-1} (C-O bond), and bends corresponding to alkanes and alkenes [31]. Characteristic peaks of methyl esters in biodiesel were observed at 2942 and 2830 cm^{-1} (stretching vibration of C-H bond), 1705-1653 cm^{-1} (stretching vibration of C=O group), and 1200

Table 3.7 FTIR spectra confirmed:

Wave number (cm^{-1})	Types of vibration	Nature of functional group
2918.56, 2849.57 cm^{-1}	(C–H stretching)	Alkanes
1742.54 cm^{-1}	(C=O)	Esters/carboxylic acids
1235.68, 1159.27 cm^{-1}	(C–O)	Alcohols, ethers, esters
9896.73 cm^{-1}		Methylene
7212.78 cm^{-1}		Alkenes

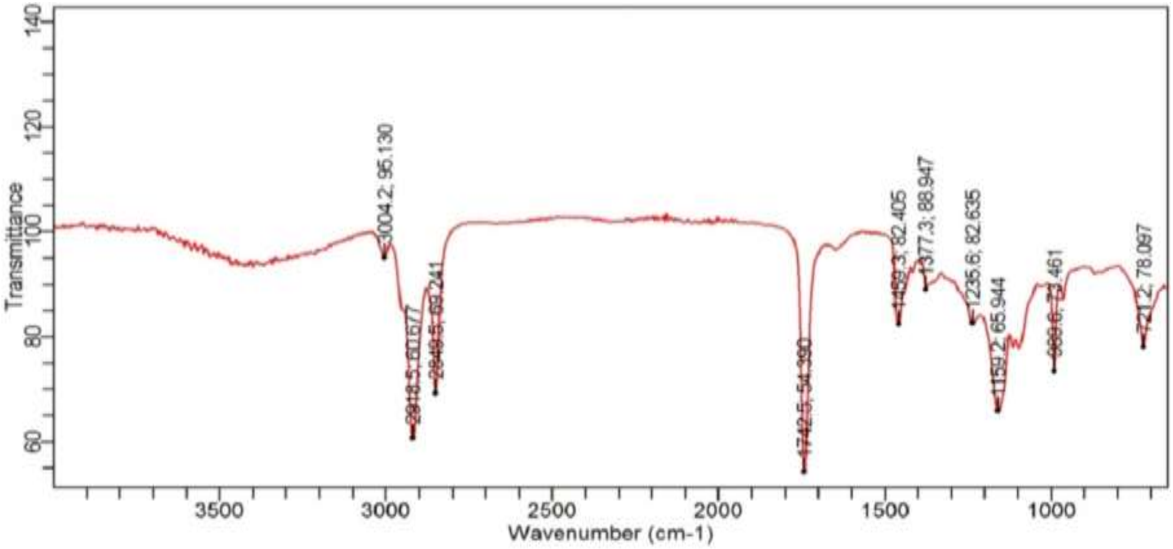

Figure 2: FTIR spectra of crude oil of Blended seeds of Mobola Plum and Sicklepod Oil

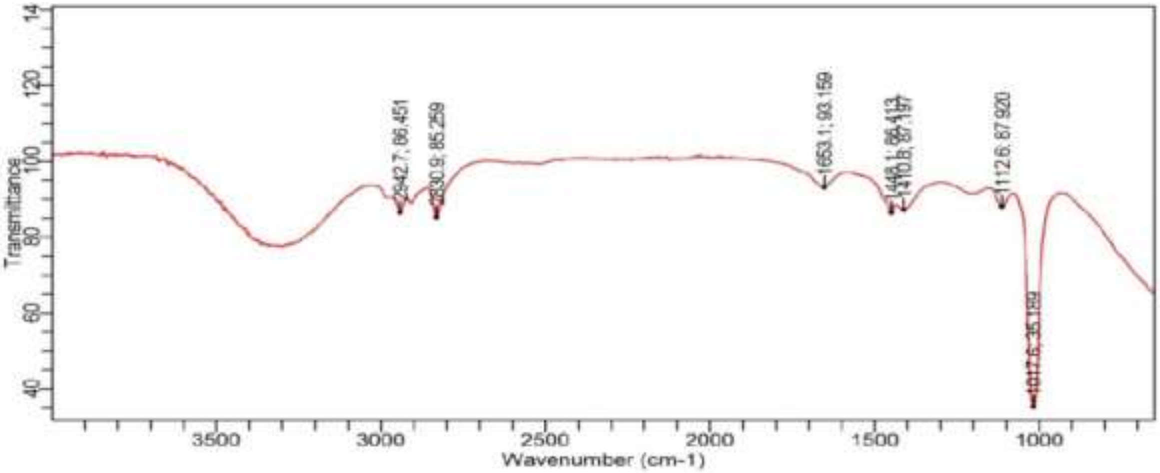

Figure 3: FTIR spectra of biodiesel from Blended Powdered Seeds (Mobola Plum and Sicklepod) Oil

3.6 Characterization of Turkey Eggshell Catalyst

Calcite, as the principal mineral phase in turkey eggshells, together with quartz, albite, and orthoclase, was detected from XRD spectra shown in Fig. 4 and Fig. 5. Most intense peaks (36–49.5° 2θ) were detected for calcite ($CaCO_3$). Calcite is readily converted to CaO by heating that acts effectively as a basic catalyst in transesterification reactions and other base catalysis processes [36]. The minerals, quartz, albite, and orthoclase add mechanical strength and act as sources of slight acidity [16]. CaO (calcined eggshells) content in Turkey eggs calculated through XRF analysis was found to be 76.606% (Fig. 6; Table 3.8; similar to [3, 26]). This implies significant catalytic capability. It is economically and environmentally advantageous to use turkey eggshells as heterogeneous catalysts for biodiesel production.

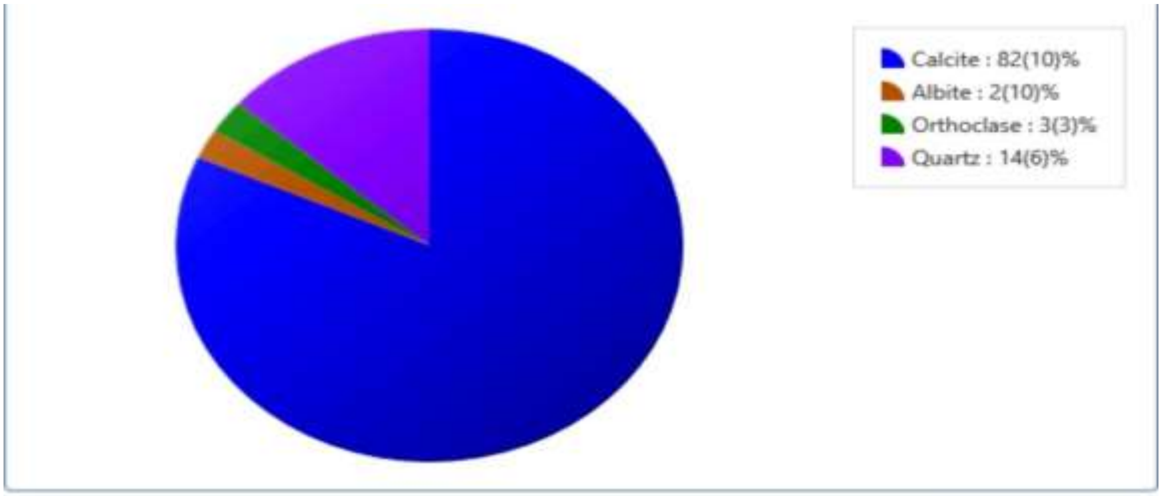

Figure 4: Percentage composition of the functional compound in turkey eggshell sample.

Table 3.8 XRF: CaO dominant (76.606%), confirming effective catalyst

S/N	Oxide	Concentration (%)
1	Fe_2O_3	0.021
2	Al_2O_3	0.719
3	SiO_2	0.236
4	MgO	1.650
5	CaO	76.60
6	CeO_2	4.794
7	BaO	0.229
8	SO_3	0.539
9	TiO_2	0.004
10	P_2O_5	0.412
11	ZnO	0.001
12	MnO	0.001
13	K_2O	0.085
14	PbO	0.007
15	Ga_2O_3	0.000
16	Ta_2O_5	0.002
17	WO_3	0.038

18	Y_2O_3	0.000
19	Nb_2O_5	0.515

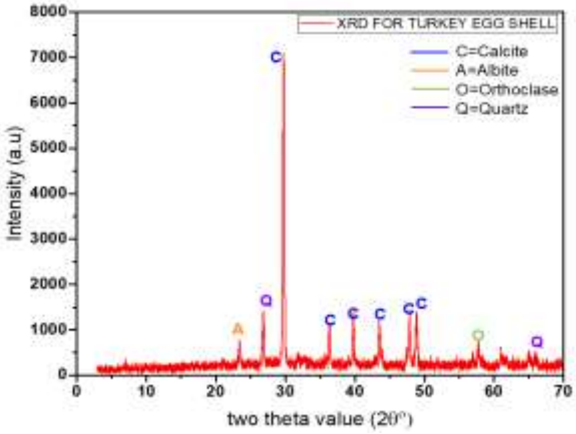

Figure 5: XRD pattern of turkey eggshell

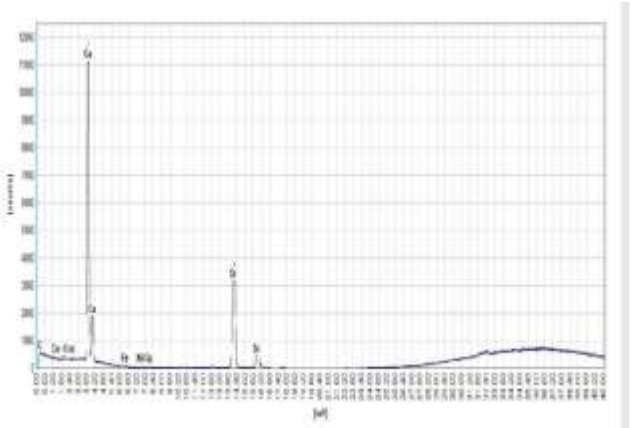

Figure 6: XRF spectrum of turkey eggshell

CONCLUSION

Combination of mobola and sicklepod seed oil is suitable for biodiesel production. Calcined turkey eggshell-based CaO catalyst was efficient in the synthesis of biodiesel. Optimum results showed maximum 90% yield of biodiesel meeting the ASTM D6751 specifications. Results from FTIR, XRD, and XRF techniques revealed that biodiesel was synthesized using the catalyst.

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