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Synthesis of Biodiesel Using Native, Nano and Modified Kaolinite Copper Oxide Catalyst

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Biodiesel is fuel composed of monoalkyl esters of long chain fatty acid derived from lipid feedstock, such as vegetable, animal fat and waste oils. The objective of the study was to investigate the potential of oil from seeds of *luffa cylinderica* as raw material for the production of biodiesel using native, nano and modified nano kaolin as catalyst. The oil used in this work was extracted from the seed of *luffa cylinderica* seed by solvent extraction method using N-hexane at 60°C for one hour. The physicochemical analysis of the oil such Acid value, viscosity, saponification value, specific gravity, and free fatty acid, were evaluated. The native, nano, and modified nano catalysts were characterized using Fourier Transform Infrared (FT-IR), Scanning Electron Microscope (SEM), Brunaur-Emmett-Teller (BET), X-ray Diffraction Crystallography (XRD), Thermogravimetric Analysis (TGA), and X-Ray Fluorescence (XRF). Biodiesel was produced using native, nano, and modified nano kaolinite clay as heterogeneous catalyst. The results gave oil yield (30.20%), Acid value (6.31 mg/KOH), FFA (3.15%), Flash point (167,128,124,132, and 130 respectively), pour point (-12,-13,-0.90, and -11.6), viscosity (6.22), and saponification value (184.6), The results show that the fuel properties conform to the standard values of ASTM method which give the evidence that luffa seed could serve as good source of oil for the production of biodiesel.

Keywords: *Luffa cylindrica*, Kaolin, Transesterification, Biodiesel, ASTM

1. Introduction

Nigeria, a major exporter of crude oil, is facing the rapid depletion of its crude oil reserves along with other challenges (Asiegbu *et al*, 2024). As a result, there is a growing need to explore and adopt alternative sources of fuel, such as vegetable oils from underutilized seeds, to produce economical, clean, renewable, and environmentally friendly biofuel. Biofuel is produced through contemporary biological processes, such as agriculture and anaerobic digestion, rather than geological processes involved in the formation of fossil fuels, like coal and petroleum, from prehistoric biological matter. Examples of biofuels include bioethanol, biodiesel, green diesel, and biogas. The demand for biofuels as alternatives to petroleum fuels is increasing, leading to the implementation of policies in various countries and regions to promote their adoption and utilization (Kumar, *et al* (2013). Existing vegetable oils used in biofuel production are competing with local consumption, so it is important to identify non-edible oils, such as luffa seed oil, which can be used in place of edible vegetable oils. The production of biodiesel offers several benefits, including the availability of a cheap renewable source of fuel on the farmstead to power vehicles and machines, nonpolluting of the environment, increased efficiency and lifespan of diesel engines, reduced dependency on petroleum fuel, and reduced costs. Major problems associated with petroleum-derived diesel include the depletion of natural resources, CO2 and SO2 emissions contribution to photochemical smog. Therefore, biodiesel production appears to be a promising endeavour. Biodiesel oil is derived from cultivated plant seed oil, and studies emphasize the use of vegetable oils from both edible and non-edible sources (Almady, *et al*, 2024).

2. Materials and Methods

2.1 Materials

The materials used include all the titration apparatus, Soxhlet extractor, oven, weighing balance, plastic tray, and mortar and pestle. The reagents include acetone, caesium oxide, hydrochloric acid, n-hexane etc.

2.2 Sample Collection and Pretreatment

Mature and dry luffa sponges were collected from abandoned, incomplete buildings in Lapai, Niger State. The sponges were opened, and approximately 30 to 50 dry seeds per sponge were shaken out and collected in a bowl. Defective seeds and dirt were removed. The dry seeds have a black coat that is difficult to break, peel off, or remove. White kaolin was obtained from Kankara Local Government, Katsina State. The kaolin was then ground and sieved into fine powder using a standard sieve with a mesh size of 120 mm.

2.3 Experimental Methods

2.3.1 Purification of the native kaolinite clay into Nano kaolin

About 1 kg of native kaolinite clay was mixed with 1000 L of water and 200 mL of hydrogen peroxide (H_2O_2) for 3 hours to complete hydration. Hydrochloric acid (HCl) was added step by step and the mixture was left overnight to settle. The finest kaolinite particles were removed by decanting the suspension. The suspension settled for 42 hours, after which the supernatant liquid was separated from the fine kaolinite particles at the bottom. The particles (nano kaolin) were dried, sieved, and stored for further analysis.

2.3.2 Preparation of modified nano kaolinite

Before modification is done there is need to know the cation exchange capacity of the nano kaolin, The cation exchange capacity (CEC) of a soil is a measure of the quantity of negatively charged sites on soil surfaces that can retain positively charged ions (cations) such as calcium (Ca^{2+}), magnesium (Mg^{2+}), and potassium (K^+), by electrostatic forces. Cations retained electrostatically are easily exchangeable with cations in the soil solution so a soil with a higher CEC has a greater capacity to maintain adequate quantities of Ca^{2+}, Mg^{2+} and K^+ than a soil with a low CEC. A soil with a higher CEC may not necessarily be more fertile because a soil's CEC can also be occupied by acid cations such as hydrogen (H^+) and aluminum (Al^{3+})). However, when combined with other measures of soil fertility, CEC is a good indicator of soil quality and productivity.

2.3.3 Determination of cation exchange capacity (CECs)

The methods of measuring CECs include: CEC at pH with ammonium acetate (chapman, 1965), Determination by the $BaCl_2$ compulsive exchange method, Agronomic soil tests, ammonium acetate method. The ammonium acetate method was used in this work.

2.3.4 Prepation of reagents used

1M ammonium acetate (NH_4OAc)

5.7 ml glacial acetic acid (99.5%) was diluted with 800ml of distilled H_2O in a 1L volumetric flask. 68 of concentrated NH_4OH, was added and it was mixed and cooled. pH to 7.0 was adjusted with NH_4OH and dilute to 1litre.

2.3.5 1M KCL replacing solution

74.5 g KCL was dissolved completely to dissolve water and was diluted to a final volume of 1 litre.

2.3.6 Ethanol 95%

2.4 Procedure for CECs

25 g of the nano kaolin was added to a 500ml Erlenmeyer flask, 125 ml of the 1ml NH_4OAc was added, it was then shaken thoroughly and was allow to 16 hours(or overnight), 5.5 cm Buchner funnel was fitted with retentive filter paper, the paper was moisten and the sample was transferred, the nano kaolin was washed four times with 25 ml additions of the NH_4OAc,allowing each addition to filter through, the leached was decanted, the nano kaolin was washed with eight separate addition of 95% ethanol to remove excess saturating solution, it is then allow to filter through respectively, adsorbed NH_4 was extract by leaching the nano kaolin with eight separate 25 ml addition of 1ml KCl, it was then discarded and the leachet was transferred into a 250 ml volumetric and was then diluted to volume with KCl. The concentration of NH_4-N in the KCl extract was determine by colorimetry.

2.5 Procedure for colorimetry

The solution of 1 ml KCl and 1 ml ammonium acetate prepared was measured into 2ppm, 4ppm, 6ppm, 8ppm and 10ppm and the readings obtained are taken from the colorimeter. A plot of graph of concentration against absorbance at 220 nm was used to calculate the CEC as follows:

NH_4-N reported in mg N/L: CEC ($cmol_c$/kg) = (NH_4-N in extract –NH_4-N in blank)/18

CEC= 2.89mg/l -0.500/18=0.133$Cmol_c$/kg

To get the value in g we divide the value in kg by 1000 i.e

0.133$Cmol_l$/kg/1000 = 0.00013mol_c/g

Note: The value of the cation exchange capacity is used to calculate the concentration the amino acids used in g/cm^3 i.e

If 1mol of the amino acid (phenylalanine) =165.19g

Xg=0.000133mol ×165.19=0.0210g

2.6 Modification of Nano kaolin

40 ml of HCl was measured into a beaker, 200ml of amino acid (e.g. leucine) was added to the solution, the pH of the solution was adjusted to 1-2 and 20 g of the kaolin was added to the mixture and was stirred on a mechanical stirrer for 2hours at ambient temperature. The resulting product was washed until the washed water was free of chloride ions (Cl^-) i.e. no precipitate with $AgNO_3$ solution. The modified kaolin on the filter paper was then air dried overnight, it was then oven dried at 105^o C for 60 minutes, grinded, and sieved intone a fine powder of modified nano kaolin of various amino acids.

2.7 Preparation of Catalysts (Native, Nano, and Modified Nano Kaolinite Catalyst)

30 g of each cleaned dried kaolin type was treated with copper nitrate trihydrate solution, stirred, filtered, and dried before being calcined.

2.8 Characterization of Kaolin.

The kaolin was characterized using SEM, XRD, XRF, FT-IR, BET and TGA analysis.

2.9 Extraction of Seed Oil

Oil from Luffa cylindrica L. seeds were extracted using N-hexane under cold extraction conditions.

2.9.1 Physicochemical Characterization of Seed Oil

Properties of the extracted oil such as color, pH, refractive index, viscosity, specific gravity, acid value, free fatty acids, iodine value, and saponification values were analyzed according to AOCS 1990 methods.

2.10 Preparation of Biodiesel using CuO/Kaolinite as Catalysts

CuO/Kaolin catalysts were used to convert oil into biodiesel through a specific procedure.

%Yield of methyl esters

$$= \frac{\text{Glycerol + Biodiesel}}{\text{Total oil}} \times 100$$

2.10.1 Determination of Fuel Properties of Biodiesel from Luffa cylindrica Seed Oil

The fuel properties of the biodiesel produced were determined according to the procedure reported by Ogungbele and Omosola (2015) and AOAC (1975), respectively. These properties include color, refractive index, kinematic viscosity, specific gravity, acid value, flash point, cloud point, and pour points.

3. Results and Discussion

3.1 Results

The results for the synthesis, modification, and preparation of the copper oxide/kaolinite catalysts, and the physicochemical parameters of the biodiesel are presented in Table 1 below.

Tables 1*: Physicochemical properties of luffa cylindrical oil and comparism with ASTM standard*

Parameter	Experimental value	Standard method	Standard value
Percentage yield (%)	30.20		25-47
Acid value (mgKOH^{-1})	6.316		10.200
Colour	Dark brown		Brown
Peroxide value (mMol/kg)	3.78	ASTM D95-90	0.200
Viscosity	6.22		
Saponification value (mg.g-$^{-1}$)	184.62	AFNORT60-206	200
Refractive index (25%)	1.46		1.458
Ester value	188.79		10286.72
Iodine value (g.100^{-1})	63.28	AFNOR60T-203	72.820
Specific gravity (kgcm^3)	0.937		0.908-0.9334

Table 2*: Fuel properties of luffa cylindrica methyl ester with the ASTM standard*

Properties	Native kaolin	Nano kaolin	Alanine modified nano kaolin	Leucine modified nano kaolin	ASTM Standards
Relative density(g/cm)	0.89	0.87	0.83	0.89	0.86-0.90
Kinematic viscosity (40%)	1.2	2.5	5.0	4.2	1.9-6.0
Refractive index (30 %)	1.26	1.50	1.3	1.51	1.24-1.62
Acid value (mgKOH/g)	4.21	0.90	0.79	0.81	0.80
Flash point	167	128	128	132	130
Cloud point	6	4	3.5	4.2	3-12
Pour point	-12	-13	-0.90	-11.6	-15-10

Table 3: *BET surface Area of prepared catalyst*

Samples	Surface Area(m^2/g)
NAKC	16.729
NKC	71.646
AMN	136.559
LMN	179.233
TMN	370.497
PMN	154.912

Keywords: NAKC, NKC, TMKC, AMKC, LMKC, PMKC and SLMKC represents Native kaolinite catalyst, Nano kaolinite catalyst, Tyrosine-modified Nano kaolinite catalyst, Alanine-modified Nano kaolinite catalyst, Leucin-modified Nano kaolinite catalyst, Phenylalanine-modified Nano kaolinite catalyst and Leucine -modified kaolin catalysts.

Table 4: *Chemical composition of the catalysts*

Composition (%)	NAKC	NKC	TMKC	AMKC	LMKC	PMKC	SLMKC
Al_2O_3	23.487	25.975	24.169	28.310	26.761	28,203	27.047
SiO_2	49.310	61.647	61.200	60.472	61.740	59.816	59.971
V_2O_5``	0.084	0.009	0.02	0.012	0.011	0.016	0.019
Cr_2O_3	0.018	0.034	0.038	0.037	0,037	0,044	0.028
MnO	0.042	0.112	0.038	0.037	0.098	0.091	0.012
Fe_2O_3	10.373	0.919	1.140	0.098	0.938	0.988	1.142
Co_3O_4	0.0048	0.000	0.007	0.938	0.002	0.008	0.001
NiO	0.013	0.004	0.005	0.002	0.0060	0.088	4.698
CuO	7.441	3.914	4.189	0.006	3.110	3.633	0.042

Keywords: NAKC, NKC, TMKC, AMKC, LMKC, PMKC and SLMKC represents Native kaolinite catalyst, Nano kaolinite catalyst, Tyrosine-modified Nano kaolinite catalyst, Alanine-modified Nano kaolinite catalyst, Leucin-modified Nano kaolinite catalyst, Phenylalanine-modified Nano kaolinite catalyst and Leucine -modified kaolin catalysts**.**

Table 5: *Summary of XRD result based on 2ϴ and d-spacing (Å)*

Sample	2ϴ	d-spacing (Å)
NAKC	8.77	10.08
NKC	8.40	10.52
AMN	8.17	10.81
SLMN	8.13	10.87
LMN	8.29	10.64
TMN	8.46	10.43
PMN	8.37	10.55

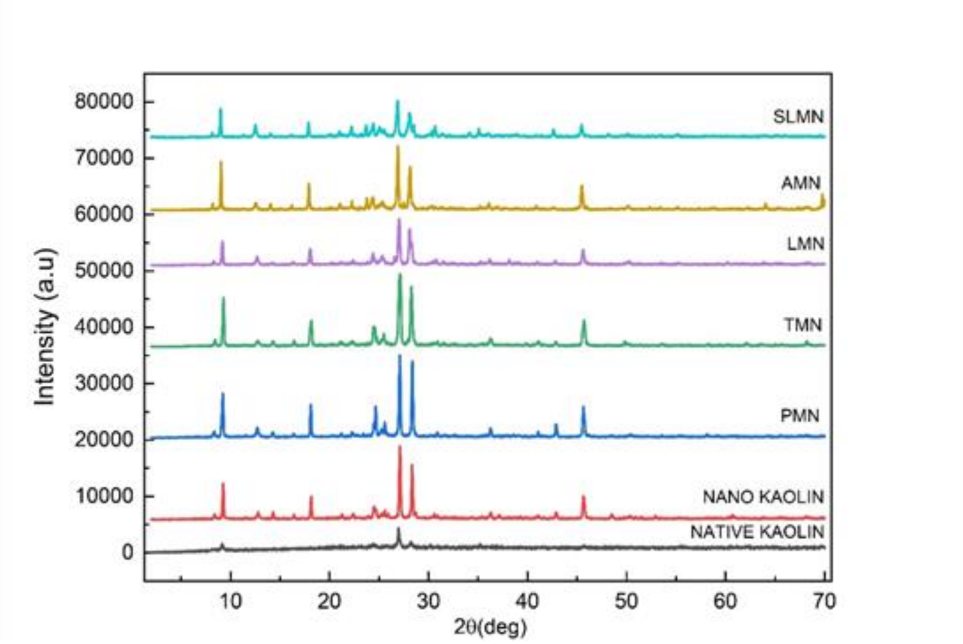

Figure 1: *XRD Peaks of Samples.*

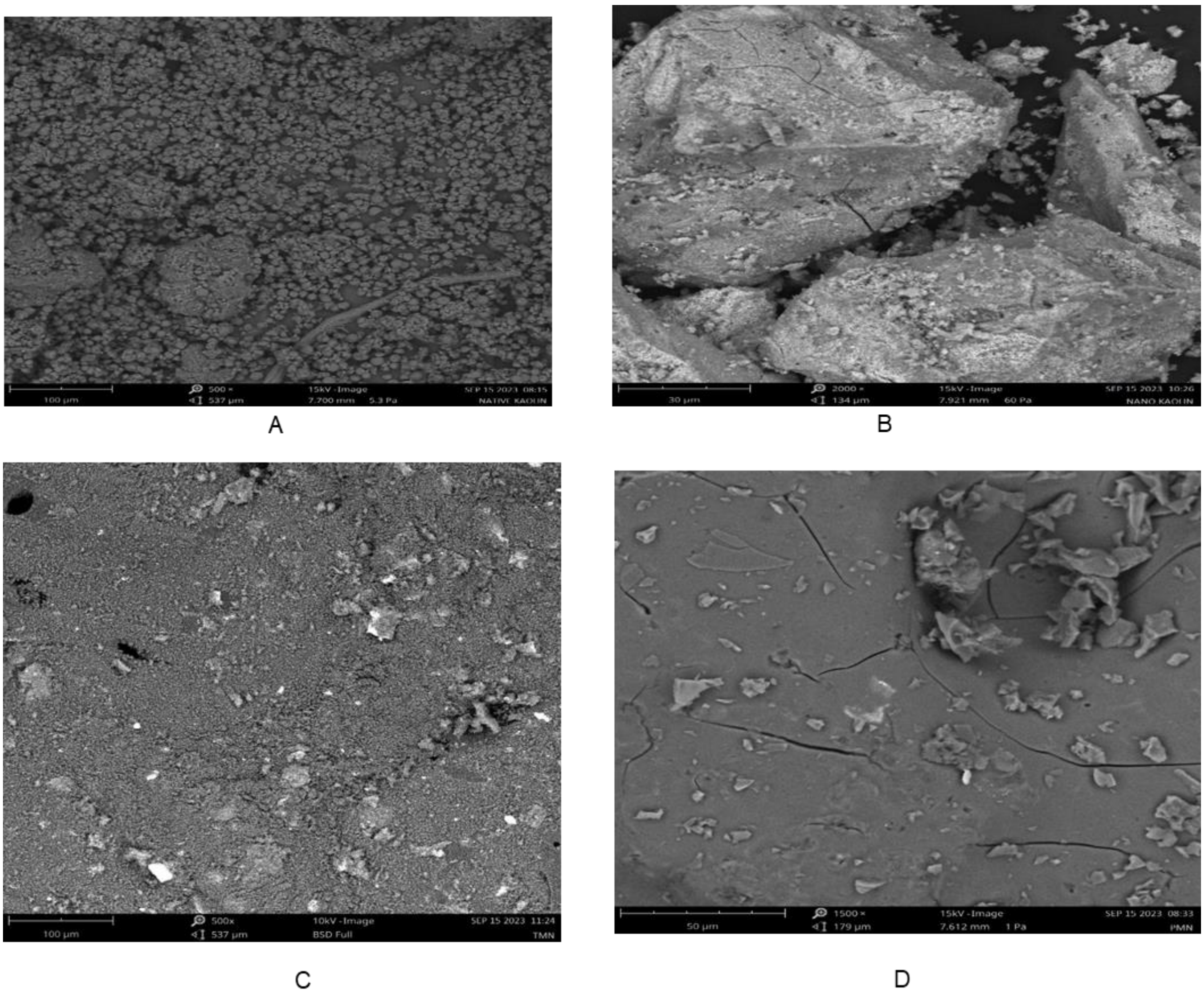

Figure 2: *SEM images of (A) Native kaolin catalyst (B) Nano kaolin catalyst (C) Tyrosine modified nano kaolin catalyst And (D) phenylalanine modified kaolin catalyst*

3.2 Discussion

3.2.1 Physicochemical Parameters of *Luffa cylindrica* Seed Oil

The Percentage oil yield of (30.20%) agrees with the standard (25-47%) indicating that the seed is economical for biodiesel production (Nwosu *et al,* 2022). It shows a relatively high acidity which was in correlation with the high acid value obtained and acid value is an indicator of inedibility of oil and its suitability for industrial activities. The high acid value shows that the sample oil need to be esterified before Transesterification. Saponification value measures the free as well as well as chain bond fatty acids, high saponification decreases catalyst concentration, increases viscosity which promotes gel formation, making it difficult to separate glycerol (Wong, *et al* (2023). It show that the oil is good for production of soap and shampoo. The proportion free fatty acids of the oil 3.158 was more than the limit of 2.0% indicating that the oil must be esterify to reduce the acid value to minimum before Trans esterification to produce biodiesel. Viscosity at 40°C was gotten to be 5.7mm^2/s; this value is lower than the value of 43.4mm^2/s at 40°C reported by Oniya (2010). The Ester value obtained from the table conformed to that of the standard value showing that the oil can be used for biodiesel production. The iodine value of *luffa cylindrica* seed oil was 96.40mgI$_2$/100g, which indicates degree of unsaturation and suggests that the oil belongs to a category of non-drying oil because a non-drying oil has iodine value not up to 100..It additionally show that the oil may be used for production of polish. Values compared favorably with the work of Barnwal and Sharma (2005) who reported a specific gravity and density values of luffa oil to be 0.92 and 0.91g/cm3 respectively; Oniya (2010) also reported a value of 0.88 and 0.88g/cm3 for specific gravity.

3.2.2 Physicochemical characterization of *luffa cylinderica* methyl ester

The results of the characterization of the biodiesel produced by the process of transesterification are tabulated in table 4.3 below. The methyl ester was produced in three batches and an average yield of 35% of was obtained. Other researchers have reported a higher yield of biodiesel such as the work of Oniya (2010) with loofah ethyl ester yield of 80%. Alamu et al., (2007) worked on palm kernel oil and reported a yield of 95.80% ester and Obibuzor et al., (2003) reported a yield of 88 – 97% ester from the transesterification of raphia hookeri mesocarp oil. Kinematic viscosity at 40^{o}C results for the native, Nano, alanine and leucine modified Nano methyl esters were 0.89, 0.87, 0.83 and 0.89 respectively. These values falls within the standard ASTM standard range of 0.86 to 0.90. Flashpoint values obtained from this work as shown in table 4, as 167,128,128,132 and 130 respectively, these values are all within the standard of 130nm.

Cloud point values obtained were 6, 4, 3.5 and 1.2 respectively, these values ranges within the ASTM biodiesel standard for cloud point (-3^{o}C TO 12^{O}C) which indicate that the biodiesel will have greater performance in cold weather because it will flow more easily at lower temperature due to the low cloud point. The values of the pour point are also in accordance with the standard biodiesel pour point range, however these values differs slightly when compared with the work of Oniya(2010) who reported a cloud point of 7^{o}C for luffa ethyl ester. The refractive index values obtained ALSO falls under the standard recommendation. The acid values were 4.21, 0.90, 0.79, and 0.89 respectively but the values in the native kaolin show values above the standard, this might be due to modification of the native kaolin.

3.2.3 BET Analysis

The specific surface areas and pore diameter calculated by BET method for NAKC,NKC,AMN,LMN,TMN and PMN was 16.729 m^2/g,71.646 m^2/g,136.559 m^2/g,179.233 m^2/g,370.497m^2/g and 154.912m^2/g respectively. It can be noticed that the surface area of modified nano kaolin catalyst is much higher than that of kaolin alone, evidence of more binding sites in the modified nano kaolin catalysts. This was because the higher the surface area of a material the larger it adsorptive capacity. This suggests that the addition of porous and polar nanoparticles which further open the pores on kaolin was responsible for the increased surface area of the modified nano kaolin.

3.2.4 XRF

XRF was performed to know the metal oxides composition in the prepared catalyst. The metal oxides include silica oxide, vanadium oxide, aluminum oxide, copper oxide, zinc oxide, lead oxide and magnesium oxide with silica oxide having the highest composition as shown above.

3.2.5 XRD

XRD analysis was performed to understand the minerals and phase composition of the prepared catalyst and to check for the success of modification. The minerals include: Quartz, anorthite, albite, and muscovite. Success of the modification was determined by the appearance of 2ϴ peak down-field and increase in d-spacing as in Table 5.

The sample's XRD pattern for the native kaolinite clay reveals that it has very strong kaolinite (K) peaks and weak quartz (Q) and anatase (A) peaks. The peaks attributed to kaolinite exhibit an organized structure and are sharp and intense (Kurbanov*, et al*, 2024).

Peaks 1 and 2 represent the low-angle peaks associated with the kaolinite mineral while Peaks 3 and 4 correspond to higher-angle peaks, possibly indicating the presence of other minerals or crystallographic features.
The peaks, especially at higher 2θ angles, continue to show characteristics indicative of nano-sized features. Broad peaks and significant integrated intensities suggest the presence of smaller crystallites for the Nano kaolin.

The peaks of the modified nano kaolin continue to show broadening, indicating the presence of nano-sized features Also From quantitative analysis report of the nano kaolin sample showed 5.7%orthoclase as this could be as a result of treatment process of nano Kaolin leading to a drop in the orthoclase concentration as shown in the figure 5.

3.2.6 Scanning electron microscopy (SEM)

The SEM images shows that the catalysts (i.e. native, Nano, TMN, PMN, SLM, AMN, and LMN) have particles which are irregular in shape and having porous texture. Agglomeration of particles occurred. Nano kaolin clay mineral had larger and compacted particles compared to the modified derivatives, which had smaller and porous particles.

4. Conclusion

The study involved the extraction and analysis of *Luffa cylindrica* oil, focusing on its viscosity, FFA, iodine value, peroxide value, acid value, saponification value, ester value, and specific gravity. Additionally, nano kaolinite particles were prepared and modified using amino acids. The CuO/kaolinite catalysts derived from the native and modified nanoparticles were utilized to convert *Luffa cylindrica* oil into biodiesel. The resulting biodiesel met ASTM standards for fuel properties. Furthermore, the *Luffa cylindrica* oil displayed qualities suitable for applications in soap, shoe polish, and cosmetics production. Modification of the catalysts improved the transesterification process. In conclusion, native, nano, and modified nano kaolinite catalysts show significance for the heterogeneous transesterification of *Luffa cylindrica* seed oil.

Conflict of interest

The authors declare no conflict of interest.

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