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Characterization of Kaolin Deposits in Kutigi, Niger State, Northern Nigeria

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Abstract

Kaolinite is among the most important mineral resource of kaolin clay sample with numerous industrial applications. Its effective characterization in terms of its chemical composition, structure and thermal properties is important for streamline it specific industrial application. In this study, the mineralogical, thermal properties, surface morphology, surface area, structural functional groups and chemical compositions were determined using the X-ray diffraction (XRD), thermogravimetry analyser, scanning electron microscope (SEM), nitrogen adsorption-desorption BET, Fourier transform infrared spectroscopy (FTIR) and X-ray fluorescence (XRF) method. The major phases in the kaolin clay samples was the kaolinite with quartz, orthoclase, and albite as minor phases. the kaolin sample loss in weight was due to dihydroxylation. The kaolin particles were made of agglomeration of its irregular sized particles. The BET surface area of kaolin sample was 181.549 m^2/g, with particle size of 2.98 nm and pore volume of 0.161 cc/g. The FTIR spectra showed presence of peaks for Si-O at positions 2359.48,1217.35, 1032.21, and 753.55 cm^{-1}, while the Al-O group appeared at 1365.84, 912.65 and 668.21 cm^{-1}. Elemental composition content results from XRF showed that SiO_2 was 34.25 wt %, Al_2O_3 was 28.16 wt % as the major content while the minor content such as Fe_2O_3, MgO, TiO_2, CeO_2, K_2O and ZrO_2 were 1.17, 1.63, 1.59, 0.95, 0.37, and 0.27 wt % respectively.

Keywords: *Agglomeration; Kutigi; Kaolin; Mesoporous; Aluminium Silicate*

Introduction

Natural clay and clay minerals are significant raw materials for the ceramic, pottery, building and construction industries (Abubakar *et al.,* 2020; Hernández-chávez *et al.,* 2021). Clay is an abundant fine textured earthly powder produced by the weathering and disintegration of granite and feldspathic rocks(Omang *et al.,* 2019). Clay of varying mineralogical and chemical compositions has been reported to have occurred as sedimentary clays within varying lithological units and at various locations in the world, of which Bida basin, in Niger State is one such location(Alabi *et al.,* 2015). Kaolin is among different types of clays generally regarded as aluminosilicate mineral(Chigondo *et al.,* 2015). It has a high Al content compared to other types of clay such as smectite, illite, and chlorite (Omang *et al.,* 2019).

Kaolin deposits could be classified as primary i.e. formed through hydrothermal, residual or mixed hydrothermal and residual deposits, or secondary i.e. formed by erosion and transportation of clay particles and their deposition in lacustrine, paludal, deltaic and Lagoona environments(Bukalo *et al.,* 2017). The hydrothermal kaolin's are formed from the hydrothermal alteration of alumina-silicate rocks. These types of kaolin's are closely related to the tectonic framework of a deposit and the alteration of parent rocks. Residual kaolin's on the other hand are usually form in high rainfall, subtropical or tropical climate; because elevated temperatures and high rainfall increase the rate of breakdown of the primary minerals to clay minerals(Bukalo *et al.,* 2017; Omang *et al.,* 2019).

In an attempt to diversify the economy of Nigeria, the exploration of Kaolin deposit in Bida and environs was undertaken to understand it chemical composition, structure and thermal properties as important parameters for streamlining its industrial applications. The aim of this study therefore is to characterize the kaolin samples obtained from Bida in Niger State of Nigeria for its mineralogical, structural, morphological, thermal, particle porosity and chemical constituents of the major oxides present with a view to determining its applicability for industrial purposes.

The study area (Kutigi) is situated in Lavun Local Government Area of Niger State, It lies between longitude 5° 35′ E and 5° 39′ E and latitude 9° 10′ N and 9° 13′ N of Nigeria. It covers an area of about 39.88km^2. Kutigi is the major town in the area, while the surrounding villages include Kusogi, Shebe, Makufu, Fazhi and Ruga. Kutigi is within Bida basin located in the west central Nigeria. It is a linear intra-cratonic sedimentary basin of about 350 km long and 75 km to 150 km wide stretching northwest-southeast and starting from Kontagora (in the north) to Lokoja (in the south) and is aligned closely orthogonal to the Benue Trough (Figure 1).

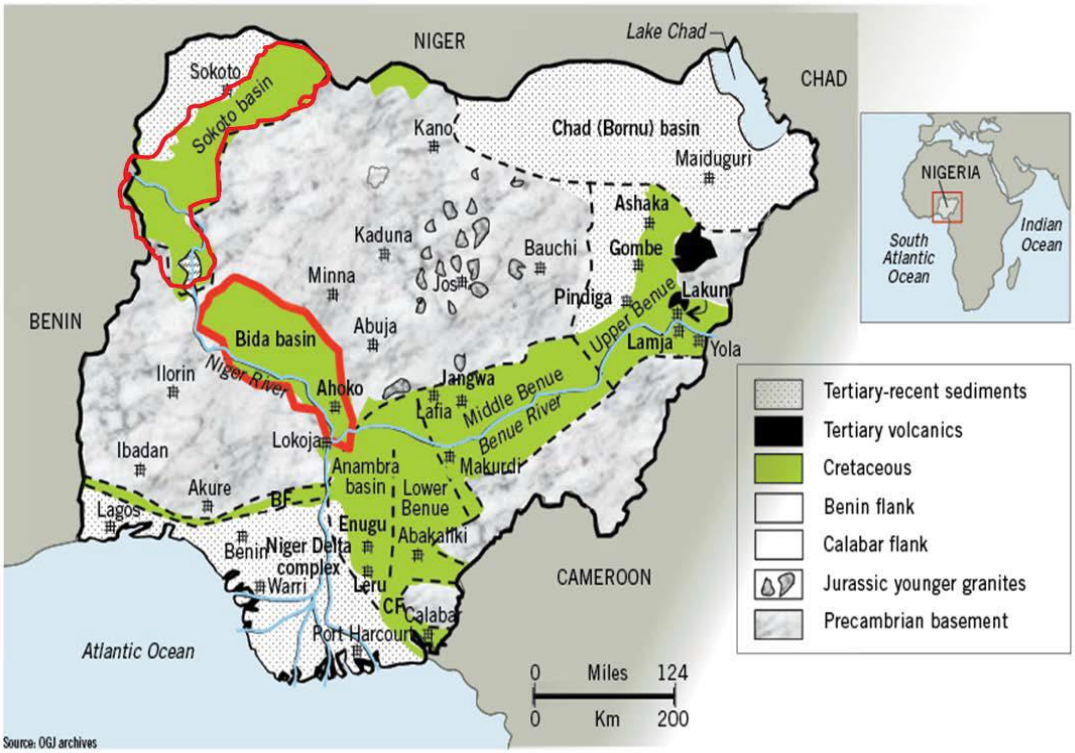

Figure 1. Geologic map of Nigeria (Auduson & Onuoha, 2020)

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Methods

About 10 kg of the raw kaolin samples were collected in a bag and transported to the laboratory, where it was spread on clean flour to air dried for 48 hours. The dried kaolin was then crushed with a hammer mill and sieved to obtain particle size of 300 µm. A portion of the samples were characterized with Thermo Fisher Scientific Energy Dispersive X-ray Fluorescence (EDXRF), Rigaku Miniflex-XRD, Perkin-Elmer TGA 4000, EDX Scanning Electron microscope SEM (PRO X) Phenom world, Quantachrome NOVA 4200e nitrogen adsorption-desorption and Fourier transform infrared analysis (FT-IR) Thermo-scientific Nicolet 5i using the ATR mode with spectral range of 400–4000 cm^{-1}.

Results and Discussion

Chemical Composition

The elemental composition of the raw kaolin sample (Table 1) indicates that it contains 34.25 wt.% SiO_2 and 28.16 wt. % of Al_2O_3. With a silica to alumina ratio of 1.21. Higher silica to alumina ratio has been reported due to much higher content of the silica in respect to the alumina for several kaolin sample investigated in other regions. For instance, the values reported by Yaya *et al*. (2017) indicates that the SiO_2 content was 49.79 and 58.59 wt % for Assin-Fosu kaolin and Kumasi kaolin while the Al_2O_3 contents was 35.17 wt. % and 22.19 wt. % respectively, which implies a 1.42 and 2.64 silica to alumina ratio. The quality of kaolin is determined by the chemical composition which translate to higher silica to alumina ratio. The differences in content may be attributed to difference geographical and vegetation of the sites.

Table 1: Chemical Composition of Raw Kaolin

Oxide	Kaolin wt %	Oxide	Kaolin wt %
Fe_2O_3	1.1697	Cr_2O_3	0.0143
SiO_2	34.250	V_2O_5	0.0362
Al_2O_3	28.160	PbO	0.0176
MgO	1.630	Cl	0.0145
P_2O_5	0.0951	ZrO_2	0.2715
SO_3	0.0867	CeO_2	0.9470
TiO_2	1.5851	BaO	0.0943
MnO	0.01126	Bi_2O_3	0.0258
CaO	0.0332	Others <0.01	0.0504
K_2O	0.3663	LOI @ 800 °C	5.0120

Ibrahim *et al.* (2018) also reported higher content of 53.90 wt.% for SiO_2 and 29.90 wt.% of Al_2O_3 with 1.80 silica to alumina ratio. While Gougazeh and Buhl, (2020) reported 58.02 wt. % for SiO_2 and 28 wt. % for Al_2O_3 with silica to alumina ratio for 2.07. The usual range of SiO_2/Al_2O_3 ratio in kaolinite mineral was reported as 1 to 2 by Babalola *et al.* (2015) while Garcia-Valles *et al.* (2020) reported a range of 1.2 to 5.21. Higher content of SiO_2 in kaolin sample may also implies the presence of free silica in the form of quartz(Ibrahim et al., 2018) which affects the silica to alumina ratio. The iron, titanium and magnesium content of 1.1697, 1.5851 and 1.63 wt.% respectively may be considered as high impurities in the Bida Kaolin which may affect the purity of end product.

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Mineralogical Content of The Kaolin

The major mineral phase of the kaolin sample is shown in Figure 2. Which indicate that the dominant phase is the kaolinite mineral and quartz. The Muscovite, Albite and Orthoclase are hydrated phyllosilicate of potassium or sodium aluminosilicate minerals which may also contains elements such as iron and titanium. This observation is supported by the presences of iron, titanium, magnesium and potassium content of 1.1697, 1.5851, 1.63 and 0.37 wt.%.

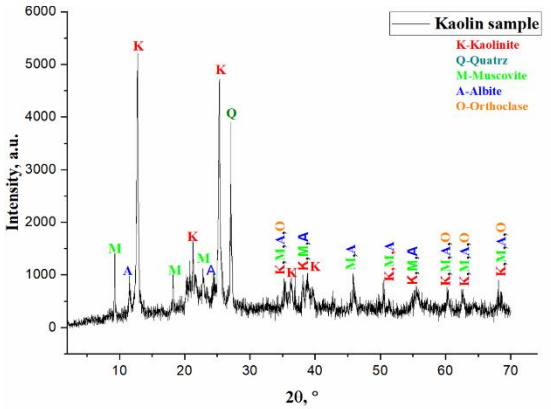

Figure 2. XRD pattern of Bida kaolin sample with indexed peaks.

Several kinds of minerals may occur in natural kaolin clays depending on the geographical locations. For instance, the kaolin sample analysed by Ibrahim *et al*. (2018) shows the presence of kaolinite, quartz and muscovite mineral phase in the xrd study of kaolin samples from Riyadh area in Saudi Arabia, used for extraction of alumina. Omang *et al.* (2019) reported the presence of kaolinite, quartz and muscovite mineral phase in the kaolin samples from Okpella area (Ajego and Anegha) with the kaolinite phase accounting for 10 - 40 % clay content. While, Garcia-Valles *et al.* (2020) in their report showed that kaolinite was the major clay mineral phase, with quartz, illite, hematite, minor K-feldspar and calcite phases. Oyetade *et al.* (2021) also reported the mineralogical investigation of composition of the Gerinya claystone of Southern Bida Basin, to comprise of kaolinite as the major clay mineral within the range of 10.8 - 67.6 wt%, while quartz, anatase, diopside, goethite, hematite, rutile, muscovite and microcline constitute the minor mineral phase. The presence of the other phases; maybe attributed to minor impurities associated to the clay as seen in the chemical composition. For instance, traces of Na_2O in association with the aluminium silicate give rise to feldspar, in form of albite ($NaAlSi_3O_8$) and Anorthite ($CaAl_2Si_2O_8$), SiO2 is also associated to the phases of quartz, cristobalite. While K_2O is associated with the forms of feldspar such as orthoclase ($KAlSi_3O_8$)]. Abubakar *et al.* (2020) have also reported Kankara kaolin clay to contains kaolinite as the major constituent phase, with traces of mullite, illite and quartz phases.

Morphological Features of Bida Kaolin Clay

The surface morphology of the kaolin depicts (Figure 3) an uneven surface with condensed pores formed as a result of continuous agglomeration of the irregular flakes of the kaolin particles. There was no sign of clear distinction between the mineral phases as suggested by the study reported by Dewi *et al.* (2020) which shows that the morphology of Jaboi kaolin are hexagonal platelet with uniform size, having distinct cluster of quartz and cristobalite impurities on the kaolin surface. The surface of the kaolin sample also appeared indented all over, suggesting it may be suitable for adherence of particles on the surface through adsorption process.

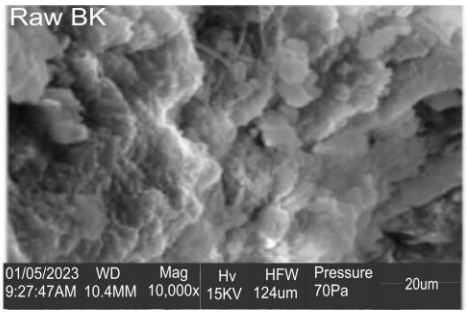

Figure 3. Surface morphological microgram of kaolin sample

The kaolin particle surface also shared similar irregular shape, porosity and big agglomeration of the particles surround by smaller particles measuring few micron sizes as observed by Shaaibu *et al.* (2020). A plate-like stacked layer particles with a pseudohexagonal shape of kaolin particles was similarly, reported by Mudi *et al.* (2018) and linked the particles to compartments of aluminosilicate $[Al_2(Si_2O_7)(OH)_4]$ as the dominant component of the kaolin studied. Ihekweme *et al.* (2020) reported a well-formed hexagonal stacks flaky surface of kaolinite layers for Kutigi kaolin sample, while the morphology of Nsu clay was pseudohexagonal sharp-edgy platelets with a face-to-face arrangement. But for the kaolin clay sample from Oboro and Obowo, surface morphology was that of a honeycomb spherical and sheet-like flakes particles respectively. This suggestively implies that the particle surface and morphological feature of kaolin particle differs from one region to another and also depends on the distribution of the chemical content of the kaolin particle as often confirmed by the XRF and XRD results.

Surface area and porosity of kaolin sample

The results of the nitrogen adsorption-desorption study to determined the surface area of the kaolin particles by the multi-point isotherm BET method showed that the surface area of the kaolin particles was 181.549 m^2/g (Figure 4). the fairly large surface area higher than the 106 m^2/g values obtained by Mudi ***et al***. (2018), implying that the high specific surface area observed for the kaolin may be due to the presence of expanding layer silicates such as montmorillonites, smectites and other vermiculites. A very low surface area of 2.30 – 16.2 m^2/g for raw kaolin and 2.78 to 17.5 m2/g for thermally treated kaolin were reported by Adeniyi *et al.* (2020) for the sites; Ijero-Ekiti, Ikere-Ekiti, Isan-Ekiti, Abusoro and Odigbo. Similar low surface area of 15.96 m2/g was reported for alkeleri kaolin by Deju *et al.* (2021). This implies that the surface area obtained for the present studied kaolin had large surface area enough to support varying application requiring large surface materials such as adsorption and catalytic studies. High specific surface area of kaolin particles and its environmental friendliness have been suggested for wide environment decontamination application. *Chen et al.,* (2023) in their review studies, highlighted the utilization of kaolinite as a photocatalysts' carrier for ZnO/kaolinite, g-C_3N_4/kaolinite, g-C_3N_4-TiO_2/kaolinite, and CeO_2-g-C_3N_4/kaolinite composites. By focusing on their synthesized methods, photocatalytic performance and intrinsic mechanisms effects on organic pollutant degradation and disinfection. The efficient performance of the materials with increasing specific surface area, was shown to expand the light absorption from UV light to visible light.

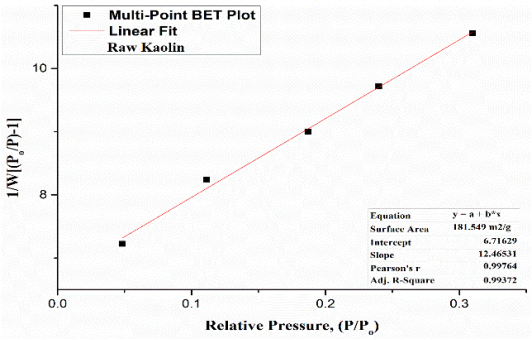

Figure 4. Nitrogen adsorption -desorption multi-point BET plot of Kaolin sample

The kaolin particle can also be classified as mesoporous material largely due to the pore value of 2.98 nm obtained (Figure 5) which is within the range of 2 to 50 nm IUPAC classification of pores (Thommes *et al.*, 2015). Knowledge and control of the pore size distribution and surface area of porous materials is of utmost importance in many applications, including medical and dental implants, filtration, catalysis, next-generation high-pressure manufacturing, geological applications (including storage of gas, oil, and water in reservoirs (Shi *et al.*, 2021).

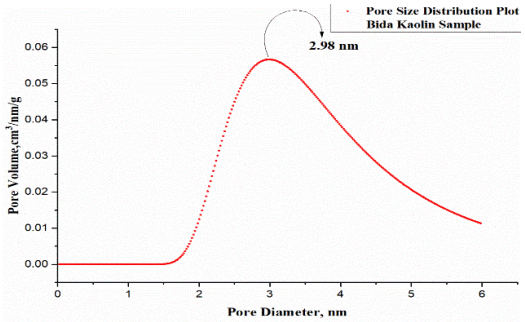

Figure 5. Pore size distribution plot of kaolin sample

Thermal stability behaviour of kaolin sample

The thermal stability study of kaolin investigated by the thermogravimetry analyser indicated that weight loss occurred in three stages. The first weight loss was the weakest, which was observed between the 100 to 300 °C and represents the loss of moisture. Thus, the dehydration stage results in the loss of 0.457 wt % of the kaolin particles (Figure 6).

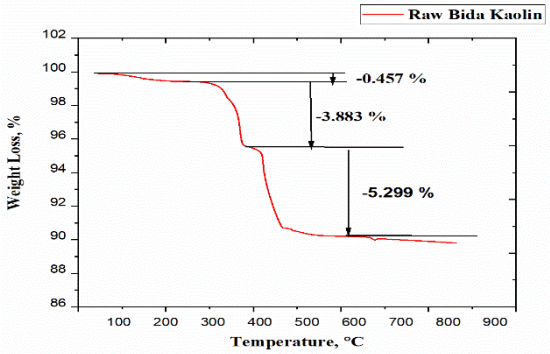

Figure 6. Thermal analysis of Raw Bida kaolin sample

The second and third stages of weight loss which occurs 300 to 400 and 400 to 500 °C is regarded as the crystallite transformation. Shaaibu et al. (2020) reported the loss of organic matter between 500-550 °C due to organic matter oxidation to carbon dioxide. As well as the endothermic lost OH- group from the kaolin structure in the form of water of an alkaleri kaolin sample studied. This stage of loss, is similar to the observed loss in the current study which are associated to dihydroxylation of kaolin i.e. loss of the hydroxyl loss (-OH) in $Al_2(Si_2O_7)(OH)_4$ to form $Al_2Si_2O_7$.

$$Al_2Si_2O_7(OH)_4 \rightarrow Al_2Si_2O_7 + 2H_2O \quad (1)$$

So, between the temperature of 600 to 1000 °C only oxide metal would be retained in a thermally treated kaolin sample. This will imply, that the total loss of volatile in the kaolin samples are closely related to the loss of ignition value of the kaolin sample. Adeniyi et al. (2020) reported similar three stage mass losses for kaolin samples obtained from five site; Ijero-Ekiti, Ikere-Ekiti, Isan-Ekiti, Abusoro and Odigbo. With the first loss between the temperatures of 100–200 °C were an insignificant weight loss of 0.04 % for Ikere-Ekiti and Isan-Ekiti samples, 0.06 % for Ijero-Ekiti sample, 0.12 % for Odigbo and 0.26 % for Abusoro sample was observed to implies the loss due to moisture in the samples. The second and third weight losses occurred between 390 – 700 °C which represent the start and end of the dehydroxylation of the kaolinite in the clay's samples.

Structural Functional Group in Kaolin

The functional group present in the kaolin sample investigated by the Fourier transform infrared spectroscopy (FT-IR) was depicted in Figure 7. The strong peak stretch at 1030.28 cm^{-1} position represents the Si-O bond of the SiO_2 which exist in the kaolin sample as indicated by the XRF and XRD techniques. The existence of peak at the 1030.28 cm^{-1} position was suggested as an in-plane stretching of the Si-O bond in the Si-O-Si by Vaculikova *et al.* (2011). While the moderate peak stretch at the 912.65 cm^{-1} position can be attributed to Al-O bond due to the bending vibration of Al-O-H kaolin sample. Tantawy & Alomari, (2019) similarly observed and reported existence of these peak positions of Si-O and Al-O respectively at 1025 cm^{-1} and 908 cm^{-1}. The observed weak stretch at position 1365.36 cm^{-1} is also attributed to Al-O bond.

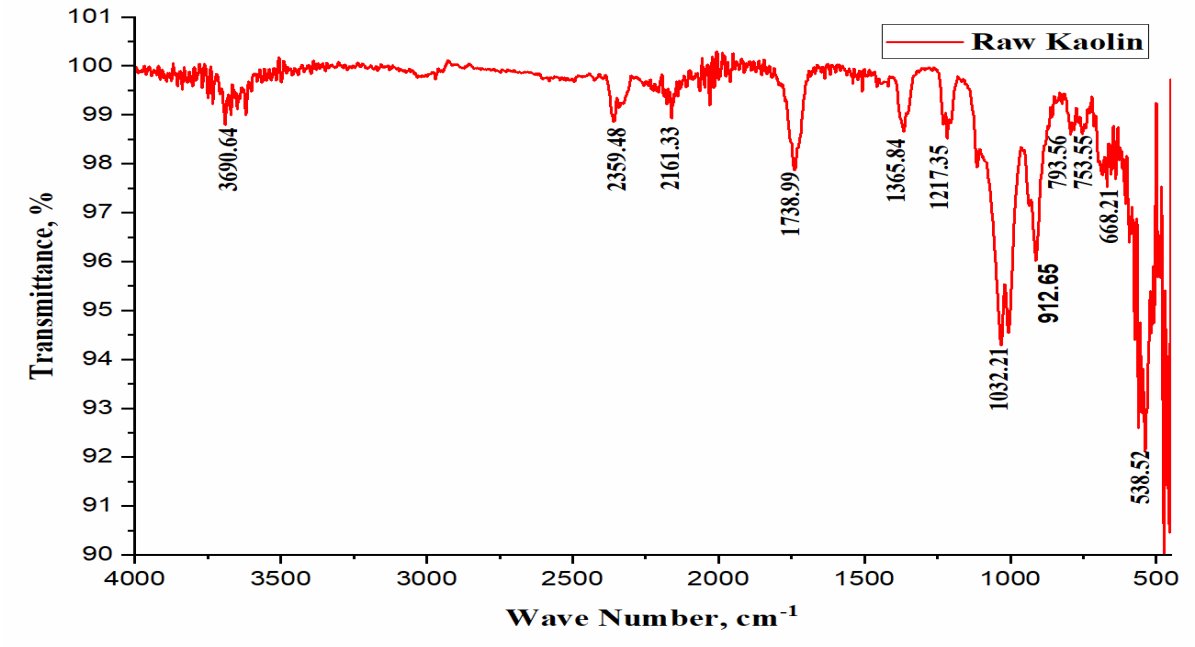

Figure 7. IR spectrum of Bida Kaolin Sample

The peak at 795.49 may also be attributed to the Si-O bond of quartz in the kaolin sample (Saikia and Parthasarathy, 2010) as well as Deformation of OH linked to Fe^{3+} and Al^{3+} according to Mudi *et al.* (2018), while the stretching vibration of position 538.52 cm^{-1} is attributed to Si-O-Si deformation as similarly reported by (Aragaw & Angerasa, 2020) to occur at 547 and 545 cm^{-1} for before and after kaolin treatment. The peak stretch at position 3690.64 cm^{-1} may be attributed to -OH functional group attached to aluminium atom in the octahedral mineral sheet (Aroke & Osha, 2013; Maina *et al.,* 2015). Though, the presence of only the 1738.51 cm^{-1} peak at position suggest minimal organic impurities in the kaolin sample. However, the peak at 2359.48 cm^{-1} position is attributed to the asymmetric stretching vibration of the Si-O bond in the SiO4 tetrahedral. The peak at 2161.33 cm-1 position may be attributed to a C=O from aldehyde or ketone which may be link to organic matter impurities such as leaf or roots of plants. The peak at 1365.3 cm^{-1} which is commonly attributed to the bending vibration of the methyl (CH_3) group may also be link to organic matter impurities in the kaolin samples. These observations are expected since during the pretreatment process of kaolin i.e. drying and grinding process, there is the possibility of organic matter such as leaves of plants been part of the fine grinded kaolin powder. Though Aroke *et al.* (2013) suggested that the peak at 1365.3 cm^{-1} may be an attributed to oxide of sodium and potassium present in kaolin mineral.

Conclusion

The chemical composition analysis shows that, the Kutigi Kaolin contains substantial quantity of aluminium which may be extracted, converted to alumina for use as catalyst support, ceramic insulators, nanoparticle base material and other applications. The kaolin may also be purified and used for both industrial and pharmaceutical applications such as fillers in papermaking, paint thickeners, drug fillers, ceramic tiles, Portland cements etc. The presence of muscovite, albite and orthoclase mineral phases from the XRD result also suggest the possibility of utilizing the kaolin as source fertilizer additives for agricultural soil enrichment to enhance crop production output. In addition, novel ceramics made from the combination of mullite, anorthite

and albite sourced from natural kaolin and chemical additives such as calcium carbonate and sodium carbonate have also been sufficiently explored as a sponge-like materials with high porosity suitable for absorbent studies. The low content of iron, magnesium, potassium, calcium, titanium, barium, chromium etc as impurities in the kaolin samples can be sufficiently remove in the preconcentration stage for enhanced aluminium extraction and conversion to alumina by mild to strong acid treatment.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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