

Characterization of Kaolin Clay from Ejigbo, Lagos, South-Western Nigeria

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Abstract

Characterization of kaolin clay from Ejigbo area of Oshodi-Isholo local government area of Lagos state of Nigeria was carried out to deduce its supportive properties when used in the synthesis of catalyst nanoparticles. The kaolin clay samples were subjected to elemental compositional and morphological analysis through scanning electron microscopy test. They were also analyzed by powder diffraction, thermal, Fourier infrared and surface area analysis. The results of the characterization revealed that the kaolin clay contains kaolinite as the dominant clay mineral with decomposition water loss of 14.55% and thermally stable at 600 °C. The morphological studies showed that the clay sample particles are pseudo-hexagonal in shape with a particle size of between 8 nm to 32 nm. The Fourier analysis test depicted the presence of hydroxide bonding ions forming on active sites dominated by the presence of aluminum and silica materials. The BET analysis showed that it has a high surface area of 106 m²/g enough to allow the creation of anchoring sites for nanoparticles. These properties showed that the kaolin clay could be used as a supporting material in catalyst synthesis.

Introduction

Nigeria has an estimated reserve of about two billion metric tons of kaolin [9]; it has attracted both local and foreign investors. Due to its wide applications, characterization of this material has been of considerable interest to investigators. Kaolinite is a clay mineral belonging to layered aluminum silicate minerals of kaolin [13]. It consists of high defected kaolinite with some impurities in form of feldspar and quartz. It is a soft, light weighted often chalklike sedimentary rock that has an earthy odour. It may have a colour of white, pink, and grey depending on its geological formation and composition [9]. It has a large array of uses such as in ceramics, paper filling, coating, refractory, pharmaceuticals and agriculture [8]. The chemical formula of kaolin is Al₂Si₂O₅(OH)₄ and its theoretical composition expressed in oxides are SiO₂: 46.54%, Al₂O₃:

39.50%, and H₂O 13.96% [11]. It is made up of tiny, thin pseudohexagonal flexible sheets of tricline crystal with a diameter of 0.2 – 12µm and a density of 2.1 – 2.6 gcm⁻³ [3,9]. Kaolinite is one of the most abundant aluminosilicate minerals, occurring primarily as a clay particle with the high surface area to volume ratio. [5].

Dehydroxylation of kaolin to produce metakaolin is the thermal decomposition of kaolinite crystal to a completely disordered metakaolin in which the new phase less of amorphous metakaolinite, does not collapse but, rather, retains a layered structure. This takes place at a temperature of between 550 – 900 °C [9]. This process involves the loss of absorbed water at a temperature of 100 – 200 °C, loss of structural water in form of hydroxyl at a temperature of 450 – 500 °C and complete loss of structural water at 600 – 750 °C. This leads to the disintegration of its crystal lattice [12]. It was reported that in general, kaolin clay contains three kinds of water molecules in their structure [2]. The physisorbed and interlayer water which is loosely bound and are mobile. They can be removed by heat treatment below 200 °C. The water molecules present in the first coordination sphere of the interlayer ions are strongly bounded and they require a higher temperature in the range of 300 – 500 °C for their removal. Finally, the structural hydroxyl groups can condense and dehydrate in the temperature range of 500 – 800 °C.

The raw form of kaolin is less reactive and forms hydro sodalite when reacted with sodium hydroxide. The process of metakaolinization involves beneficiation and calcination. Metakaolinization occurs within the temperatures of 550 to 900 °C [15]. This process involves the loss of hydroxyl group, followed by rearrangement of the octahedral layer to tetrahedral shape in the calcined clay [10]. During thermal decomposition of kaolinite crystal, a partially disordered metakaolin is formed. The new phase metakaolinite does not collapse but rather retains a layered structure. This enhances its application for water treatment.

Numerous researchers reported the physicochemical characteristics of kaolin clay of various origins. They achieve these by characterization of raw, upgraded or treated kaolin. For instance, Biljana *et al.* (2010) reported the production of metakaolin by calcination of kaolin at various temperatures of 550, 600, 650 and 700 °C and time of 30, 60, 90, 120, 150 and 180 min. The authors showed that the optimum calcination parameters were the temperature of 650 °C, the heating time of 90 min and the degree of calcination of 0.97. The produced metakaolin has pozzolanic (reaction rate of aluminous and siliceous materials) activity of 0.65 g Ca(OH)₂/g

metakaolin. The result of their work also indicated that the main parameters for the synthesis of high reactivity pozzolana from kaolin clay are calcination temperature and heating time. These parameters affect both bound and unbound water removal. However, since kaolin can produce kaolinites of good pozzolanic activity, the pozzolans could serve as support to nanoparticles by producing active sites for anchoring these particles. The active sites in kaolin contain negative ions that can attract positive ions from nanoparticles of metal ions to create a strong support for proper improvement in the catalytic properties.

Aroke *et al.* (2013) reported the characterization of Alkaleri kaolin clay using cation exchange capacity (CEC), X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Fourier Transform Infrared (FTIR), Thermogravimetric Analysis (TGA) and Brunauer-Emmet-Teller (BET). The result of their findings showed that kaolinite is the dominant clay mineral of about 76 % and other constituents such as quartz and mica in trace amount. Their report also showed that aluminous and silicate layers in kaolin are well arranged and coordinated to allow for adsorption of substrates, the kaolinite was stable at 500 °C. One layer structural formula for a unit of kaolin as presented by the authors revealed that Alkaleri kaolin clay has potentials for use in areas of pharmaceuticals, drugs, paints, paper, catalysis, energy and ceramic industries.

It was reported the potentials of calcined Nigerian kaolin as paint pigment to serve as a replacement to titanium dioxide in the paint formulation [2]. The kaolin was subjected to many empirical tests to find out its suitability for the purpose. Their results showed that calcined kaolin has some properties such as brightness, and opacity. it can equally scatter light because its structure is porous and this porosity allows reflections of light off the planar surfaces as titanium oxide. The structural arrangement of calcined kaolin due to increase in silica and alumina content is similar to titanium oxide, hence it can replace it in paint pigments. In the synthesis of nanomaterials, silica and alumina are among the widely used support materials in heterogeneous catalysis [2]. Their usage leads to the excellent performance of the produced catalysts [1]. When kaolin is calcined, the moisture contents, as well as structural changes, take place. This lead to the release of silica and alumina of considerable functional groups which provide anchoring sites for substrates and hence increase the surface yielding a supported catalyst of better characteristics [14,9,4].

The use of kaolin as a catalyst support was studied by Sanchi *et al.* (2013). They investigated the effect of acid and alkaline treatments on the structural and chemical properties of kaolin. This was done by characterization of the acid and alkaline treated kaolin clay using XRF, XRD, FTIR, TGA, and SEM. The results of their findings showed that the removal of some impurities was aided by the acid treatment leading to increasing Si-Al ratio, surface area and pore volume of the clay. Therefore, the treated kaolin clay can be used as catalyst support. This work was supported by the report of Karim *et al.* (2015). Bawa *et al.* (2016) also reported the thermal effect on the surface properties of gamma alumina synthesized from Kankara kaolin. The results of their findings revealed that kaolin contains a considerable amount of alumina, with a considerable functional group that provides active sites for anchoring nanoparticles.

Many researchers have worked on kaolin characterization, but few documented literature are available on kaolin clays obtained from southwestern Nigeria especially those from oshodi- isolo area of Lagos state. Therefore the aim of this study was to characterize kaolin clay from Ejigbo, a suburb of the city of Lagos in Oshodi-Isole local government area of Lagos State, to deduce its suitability as a support in the synthesis of the catalyst.

Materials and Methods

Materials

Kaolin clay sample was obtained from Ejigbo area of Oshodi-Isholo LGA in Nigeria. The sample was utilized as received - characterized to monitor the changes in physical, chemical and microstructural properties. This was done to determine its suitability as catalysts support for Carbon Nano Fibres (CNFs) synthesis.

Analysis Techniques

Scanning Electron Microscope (SEM)

High-Resolution Scanning Electron Microscope coupled with Electron Diffraction Spectrometer (HRSEM-EDS; JEM 100S, JEOL Ltd., Japan) was used to verify the morphology of the samples. 1 g of the sample on wet pasty graphite was placed on a sample holder and left for about twenty-four hours to dry. The SEM holder was then used to hold the specimen with the aid of pasty graphite while tightening the screw on the holder to ensure proper grip on the sample holder. A

sample exchange rod was then used to transfer the sample onto the SEM chamber. The SEM images were then taken at different magnifications.

X-Ray Diffraction (XRD)

The patterns and phase identifications of the samples were carried out using X-ray Diffractometer (XRD; PW 1800 diffractometer, Philips, Netherlands), with graphite monochromatized copper K α -radiation. The equipment was operated at a voltage of 40 kV and a current of 20 mA with the geometry 2θ values varying from 0 to 80° at a scan rate of 1.0 s/step, after which the background was removed and peak positions identified. The background and peak positions were identified after X-ray scanning of the samples. Then the matching routine was performed based on the peak positions and intensities. The analysis of the samples was carried out using High Score Plus software. The particle diameter of the kaolin sample was calculated using the Debye-Sherrer Equation;

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1.0)$$

Where λ is the wavelength of the x-ray, (0.1541nm) β is the full width at half maximum (FWHM), θ is the diffraction angle in radian, and D is the particle diameter size. The interspacing between the atoms (d) was calculated using the Bragg's law given as;

$$d = \frac{\lambda}{2\sin\theta} \quad (2.0)$$

Thermogravimetric analysis (TGA)

Thermogravimetric Analysis (TGA 4000, Perkin Elmer, USA) was used to monitor the thermal stability of kaoline. 0.3 g of sample was weighed and placed in the sample holder in the furnace section. The temperature was selected between 30°C to a maximum of 700°C at a heating rate of 10 °C/min. Nitrogen gas (N₂) was allowed to flow at the rate of 20 ml/min at a pressure of 3 bar. Pyris manager software was used for the smoothing to obtain the TGA curve and generate the corresponding data.

Brunauer-Emmett-Teller (BET)

Surface area and pore sizes of the sample were determined through the method of Brunauer-Emmett-Teller (BET). Brunauer-Emmett-Teller (BET; NOVA 4200e, Quantachrome

Instruments, USA) performs sample preparation and the analysis. N₂ gas was used for degassing of samples while liquid nitrogen was used for sample analysis. This equipment has an exhaust pump that suctions gases from the system into the external environment. To carry out BET analysis of samples, 0.1 g of sample was weighed and was fed into the sample cell after which the flow rod was set in. The sample placed in the cell was degassed under N₂ gas at the desired temperature for 5 hrs in the BET degassing station. The cell with the sample was re-weighed to record its post-degassing mass. The degassed sample was then transferred to the sample analysis section where analysis was carried out in liquid N₂.

2.2.5 Fourier Transform Infrared (FTIR)

Perkin Elmer 69 Frontier FTIR Spectrometer was used to study the structural changes in the samples. 10 mg wafer consisting of 1 % of the sample and 99 % KBr window was prepared. The mixture was pressed with a pressure of 12 MPa to produce a thin film wafer. The wafer was inserted in the sample holder of the FTIR equipment. FTIR spectra of the sample were then recorded from 400 to 4000 cm⁻¹ with a 1 cm⁻¹ resolution operated at a scan speed of 0.125 cm⁻¹s⁻¹. Prior to sample analysis, a background spectrum of air at ambient condition was run.

Results and Discussions

The phase identification and the crystallinity of the kaolin clay sample were carried out using XRD. The result of the analysis is shown in Figure 1.0.

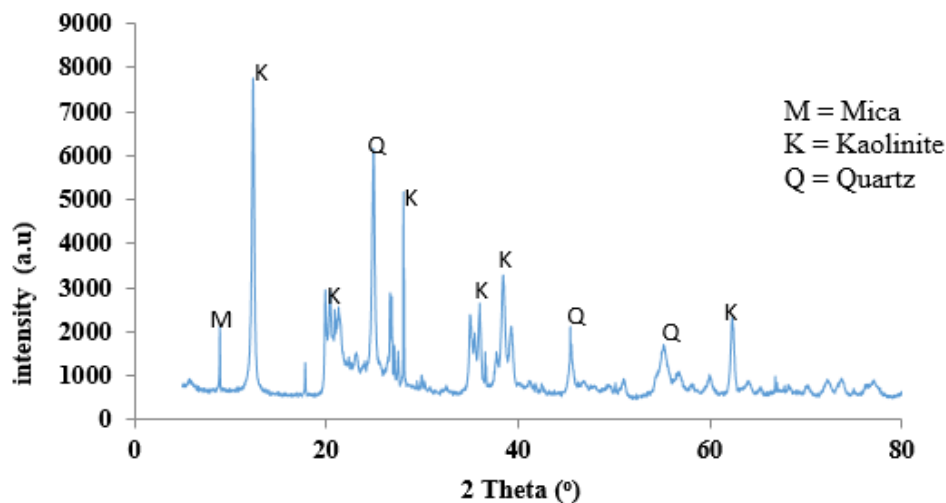


Figure 1.0: XRD Spectra of kaolin sample

The result shows ten peaks at diffraction angles 2θ of 8.9, 12.4, 19.9, 24.9, 26.6, 28.0, 35.0, 38.3, 50.1, 62.2°. The presence of mica and quartz was as a result of silicate minerals present in the kaolin sample. The XRD study confirmed that the resultant kaolinite particles for this study are simple cubic (100), body-centered cubic (110) and combinations of face-centered cubic (220). The average lattice parameters from indexing of the peaks in the XRD were calculated to be 9.9882Å as presented in table 1.0.

Table 1.0: Indexing Results for Kaolin Characterization

Peak No	2θ	β (nm)	d(nm)	D (nm)	an (Å)
1	8.9	0.0962	0.9926	8.182	9.93
2.	12.4	0.0824	0.7135	9.964	10.09
3.	19.9	0.0824	0.4458	10.058	9.97
4.	24.9	0.0549	0.3574	14.203	10.11
5.	26.8	0.0549	0.3324	14.257	9.97
6.	28.0	0.0823	0.3184	10.209	10.07
7.	35.0	0.0274	0.2559	29.085	10.25
8.	38.5	0.0275	0.2337	29.706	9.92
9.	45.5	0.0543	0.1992	16.710	9.96
10.	62.4	0.0275	0.1487	32.429	9.98

From Table 1.0, the average interspacing between the atoms, average crystal size and average lattice parameters of the study kaolin sample are 0.3764 nm, 17.480 nm and 10.03 Å respectively. This characteristic indicates less interplanar spacing which supports reflections at higher orders. The wider angle between the unit cell of the kaolin crystal of 10.03Å is more than general well-crystallized kaolinites having a graphitic line of 001 d spacing of 7.1 to 7.2 as reported by Keller (2005). The difference could be connected to the small amount of interlayer water within the kaolin crystals. Moreover, there are some incompleteness and imperfections in some patterns in Figure 1.0 indicating the overshadowing amounts of other minerals of clay-size with the kaolin. Some peaks related to crystallized phase were also detected, which is attributed to quartz, and mica initially present in the kaolin. This is in line with the work of Shani *et al.* (2011). The kaolin clay sample showed well-defined diffraction angles at 2θ values of 12.4° and 19.9° corresponding to the d crystal particle size of 9.964 nm and 10.058 nm respectively. These

reflections also correspond to reflections (100), (110) graphitic lines respectively. This is a little bit different from that obtained by Olaremu (2015) who reported the typical characteristic peaks of kaolinite at a 2θ value of 12 and 25. The difference may be due to the presence of pristine phase of mica and quartz in the sample not detected at the diffraction angle of less than 12.4. However, the graphitic line of this sample observed at 12.4 and 19.9 may be as a result of low contents of quartz and mica impurities present in the sample at 2θ values of 24 and 31 as reported by Diko and Ekosse (2013).

The FTIR spectra of kaolin as presented in Figure 2.0 shows three characteristic bands at 2992, 3697 and 3622 cm^{-1} . These bands correspond to the general features of OH stretching which is well established for kaolin samples. This is in agreement with the works of Saikia *et al.* (2010), Lenka *et al.* (2011) and Aroke *et al.* (2013). It is an indication that the sample contains OH stretching with an inner layer of Al-OH and (H-O-H) all arranged and well-coordinated. It also shows high structural ordering of the sample which will allow the proper base for anchoring nanoparticles for effective performance. The spectra of FTIR presented also show that the overall spectra are divided into three general regions: 4000-2500 cm^{-1} (functional group region) and 2000-1500 cm^{-1} (double bond region) and 1500-600 cm^{-1} (fingerprint region). This frequency assignment approach is used for the interpretation of the spectra as reported by Aroke *et al.* (2013). The bands placed at 1666 cm^{-1} , is a double bond region revealing the presence of bonding of H-O-H. This is somewhat far from 1637 cm^{-1} as indicated by Burhan and Emin (2009). The difference may be due to the source and treatment of the kaolinite. The absorption band at 1117 cm^{-1} is assigned to Si-O normal the plane stretching.

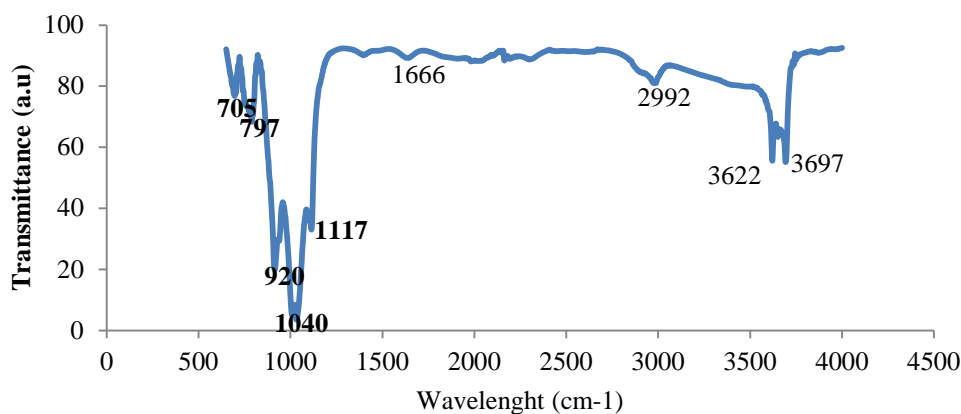


Figure 2.0: Infrared spectrum of kaolin

The bands placed at 1117-1000 cm^{-1} region corresponds to Si-O planar stretching which agrees closely with 1027-1009 cm^{-1} obtained by Burhan and Emin (2009). The peak at 920 cm^{-1} corresponds to inner-surface Al-OH deformation. The frequency vibration at 797 cm^{-1} is assigned to OH deformation linked to Fe^{3+} and Al^{3+} . This shows the substitution of Fe in the structure of the octahedral sheet in this clay as assigned to Fe^{2+} -OH and Fe^{3+} -OH stretching which is a sign of impurity. The peak at 705 cm^{-1} corresponds to Si-O deformation. All these points to the fact that the sample contains the substitution in both tetrahedral silica and octahedral alumina arranged in an orderly manner. The presence of these functional groups in this kaolin sample as revealed by FTIR is an indication that anchoring sites for substitution of substrates are present in the sample. Therefore, it could serve as support for said catalyst synthesis. The wave numbers and assignments for the kaolin are shown in Table 2.0.

Table 2.0: Wavenumbers and assignments for the kaolin

Wave Number (cm^{-1})	Assignments
3697	Free OH stretching
3622	Inner layer OH (Al-O...H) stretching
2992	(H-O-H) stretching
1117	(Si-O) normal to the plane stretching
1040	(Si-O) planar stretching
920	Inner surface Al-OH deformation
797	Deformation of OH linked to Fe^{3+} and Al^{3+}
705	Fe^{2+} and Fe^{3+} stretching
654	Si-O deformation

The suitability of using kaolin as a support for the synthesis of catalyst was investigated through studying of its thermal stability. This was carried out by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). The result as presented in Figure 3.0 shows two weight loss regimes. The first occurred between the temperatures 26.75 and 180.06 $^{\circ}\text{C}$ which corresponds to weight loss of about 1.94 % and represents the loss of hygroscopic water. The onset temperature at which the kaolin sample started metamorphosing into kaolinite was found to be 367.12 $^{\circ}\text{C}$.

This happened gradually because of the presence of some impurities in the sample in form of quartz and mica as shown in Figure 1.0. This result is in close agreement with the report of Kovo (2011). The main weight loss was observed at zone b at the peak temperature of 440.88 °C representing weight loss of 12.61 %. The higher weight loss witnessed might be associated with the process of dehydroxylation of the kaolin sample when the structural OH component of kaolin is lost in form of water. This is in good agreement with the work of Francisca (2014).

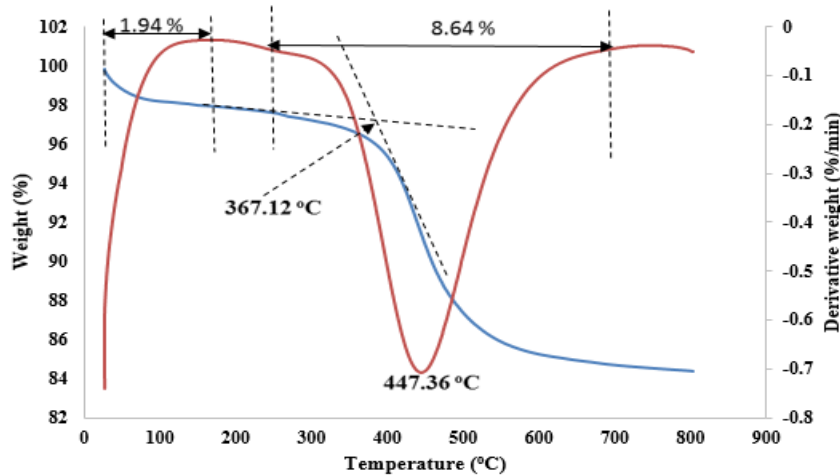


Figure 3.0: TGA/DTA curve of kaolin

who reported that during heat treatment of kaolin, it transforms into kaolinite and water in form of moisture are released. however, the stoichiometry of the dehydroxylation of kaolin shows that the mass of water loss at that temperature was calculated to be around 25%. This indicated that the complete loss of hydrate water has not taken place in accordance with the work reported by Oluremi (2015). Oluremi (2015) reported that complete loss of structural water takes place at a temperature of 600 – 750 °C. The dehydroxylation of kaolinite occurred at 420°C, which is in reasonable agreement with Aroke *et al.* (2013) and Francisca (2014). The degree of dehydroxylation of kaolin used for this study was calculated to be 0.98. This is in close agreement with the literature value of 1 (Biljana *et al.*, 2010) which is the mass in bound hydroxyl ions in kaolinite. The result as presented further shows that high percentage content of kaolinite in the clay mineral present in the sample is stable at 600 °C. this indicated that the kaolinite could be used as a support to catalyst nanoparticles at a temperature of less of equal to 600 °C.

Table 3.0: BET results of kaolin sample

Sample	Surface area (m ² /g)	Pore volume (cm ³ /g)	Pore size (nm)
Kaolin	106.2	0.89	0.031

The BET analysis as revealed in Table 3.0 showed that the kaolin clay was majorly a mesoporous material of high surface area of 106.2. m²/g. 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 in the sample. This resulted in extensive internal and external specific surface as reported in the work of Carter *et al.* (2006). It has also been reported by Bawa *et al.* (2016) that porous materials having a surface area value above 100 m²/g is considered having a high surface area and a potential material for catalyst support. The results in Table 3.0 also indicated that the materials also contain the Dubinin-Radushkevich (DR) Method micropore with size and volume of 0.031nm and 0.89 cm³/g respectively.

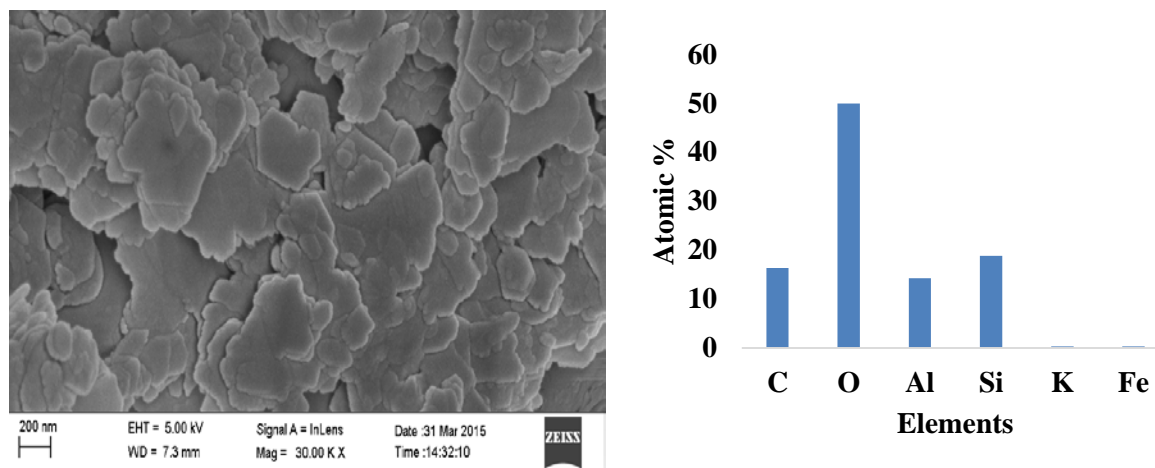


Figure 4.0: (a) HRSEM of Kaolin sample (b) Corresponding EDX showing atomic weight percentage of the components of kaolin

The HRSEM micrograph as presented in Figure 4.0 depicts plate-like nature of the pseudo-hexagonal shape of kaolin. This contains compartments of aluminosilicate [Al₂(Si₂O₇)(OH)₄] constituent which is the dominant component of kaolin. This is in line with the works of Auwar *et al.* (2009), Lenka *et al.* (2011), Francisca, (2014) and Aroke *et al.* (2014). Their works showed that kaolin contains predominantly pseudo-hexagonal crystals which depicted the strong interactions existing in aluminosilicate material. The EDX revealed the

presence of C, O, Al, Si, K and Fe with percentage weight of 16.35, 49.95, 14.25, 18.83, 0.32 and 0.3 % respectively.

Table 5.0: Comparison of EDX percent elemental composition with original Kaolin

Elements	Al	Si	O	Others
Literature kaolin (%)	18	19	61	2
Studied kaolin (%)	14	19	50	17

The percentage composition of elements in kaolin sample as reported by Lenka *et al.* (2013); Francisca (2014) and Aroke *et al.* (2014) also show that (Al: Si: O = 18:19:61) are in ratio 1:1:3. This is in close agreement with what is obtained by the EDX results of the studied kaolin presented in Table 5. The presence of a higher percentage weight of O₂ was due to the presence of oxides of the elements in the kaolin sample. Meanwhile, the carbon content is attributed to the presence of organic constituents due to the source of the kaolin and the carbon grid used during the SEM analysis. Also from Figure 5, the composition of aluminum and silicon is significant. Therefore, there is a need for effective leaching technique of alumina and silica from the kaolin, then it can be used as support for synthesis of catalysts.

Conclusion

Characterization of Ejigbo kaolin clay showed that the kaolin possesses some characteristics such as high crystallinity with orderly arranged structures which can provide a base for supporting nanoparticle. There is also the presence of hydroxyl group that provides active sites for anchoring substrates like a catalyst. It has high thermal stability, good morphology, and high surface area. Therefore, the kaolin can perform well as support for synthesis of the catalyst.

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