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Physicochemical Characteristics of Clay Mineral from Ikpeshi in Akoko-Edo Local Government Area, Edo State, Nigeria

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ABSTRACT: Clay mineral has become increasingly relevant for industrial application due to its environmentally friendly properties, low cost, and relative abundance. Several solid mineral deposits could be found in Edo State of Nigeria. This study therefore, examined the physicochemical characteristics of clay mineral from Ikpeshi in Akoko-Edo Local Government Area, Edo State, Nigeria using various standard techniques. Results obtained reveal that the sample was kaolinite with SiO_2 45.116wt%, and Al_2O 20.39wt% as the most predominant elements. Wavenumbers of 909.47043cm^{-1} to 998.92654cm^{-1} with bold peaks revealed the presence of SiO_4^{-4}. The study revealed that the clay from Ikpeshi has kaolinite characteristics and strong thermal stability suitable for lining furnace kilns.

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Clay is a byproduct of the earth surface being weathered by geological processes. Clay is made up of extremely small particles. Due to the presence of aluminum (Al_2O_3), silica (SiO_2), and water (H_2O), pure clay is also known as hydrous aluminum silicate and has the chemical formula $Al_2O_3.2SiO_2.2H_2O$. Any ceramic or pottery bodies must contain clay as a basic component. It is the hydrated aluminum silicate [$Al_2O_3.2SiO_2.2H_2O$]. A common, affordable, and widely-present mineral in nature is clay (Kovo et al. 2005). It can be broken up, combined with water, and formed into many shapes and forms. Clay is a material that feels chilly to the touch, is soft, flexible, and impressionable. Natural inorganic materials known as clay minerals have well-known structural adsorption, rheological, and thermal characteristics. These materials were initially hydrophilic due to the hydroxyl (-OH) groups on their surfaces, which may attach water molecules quickly (Aramide et al., 2014; Orodu, 2017). Clay minerals are part of a category of minerals that are incredibly important because of particular characteristics like plasticity, hydration, and catalytic capabilities. They are used in pharmaceutical, material and agricultural industries. For a long time, clay materials have been utilized for the adsorption of anions like nitrates, phosphates, and sulfates, heavy metals, color molecules, herbicides, and gases like SO_2 (Mokwa et al., 2019). Clay becomes hard, thick, rock-like, long-lasting, and permanent when it is burnt. The necessity for excellent handling skill and the prevention of improper handling are crucial when creating anything out of clay. Commercially available catalysts include clays or clay-modified catalysts (Moshoeshoe et al. 2017). Their surface characteristics make them particularly important minerals, and their reactivity makes them suitable for industrial

applications and environmental management. Because of their acidity, high surface area, and cation exchange capacity (CEC), clay minerals are useful as adsorbents, supports, and catalysts for hazardous compounds (Olaremu, 2021). Clays have a wide variety of uses due to their swelling, adsorption, ion exchange, and enormous surface areas (Maciver et al. 2020). Clays have been used as catalysts ever since the petrochemical and refining industries first emerged. Acid-modified clays were used in the first hydrocracking process, which was developed more than eighty years ago. Zeolites and other aluminosilicate clays which are some of the acid modified clays are still in use today (Perego & Carati, 2008; Zimmermann & Haranczyk, 2016). These clays are known to have large surface areas (Peng et al.,2020). Clay deposits are widespread in Nigeria and have numerous domestic uses, the physicochemical properties for these uses have not been well studied. Clay is utilized in Nigeria for different purposes, including geophagy, modern pottery, construction, and water filtration (Mamudu et at. 2020). There are many different clay deposits, and each one has unique properties that can be tailored to a particular industrial use. Edo state is very rich in solid mineral resources including kaolinic clay (Mokwa et al., 2019). Therefore, this study evaluates the physicochemical characteristics of clay mineral from Ikpeshi in Akpoko-Edo Local Government Area, Edo State, Nigeria.

MATERIALS AND METHODS

Clay sample (Figure 1) was collected from Ikpeshi community in Akoko-Edo Local Government Area of Edo State. The sampling location lies between longitude 6° 06' 0" E and latitude 7° 17' 0" N. The clay sample was collected from a spot at about 20cm deep. They were crushed and sundried to remove loose moisture and sieved with a 100µm mesh to remove unwanted materials such as plant roots. The sieved sample was thus kept in a sealed container until further use.

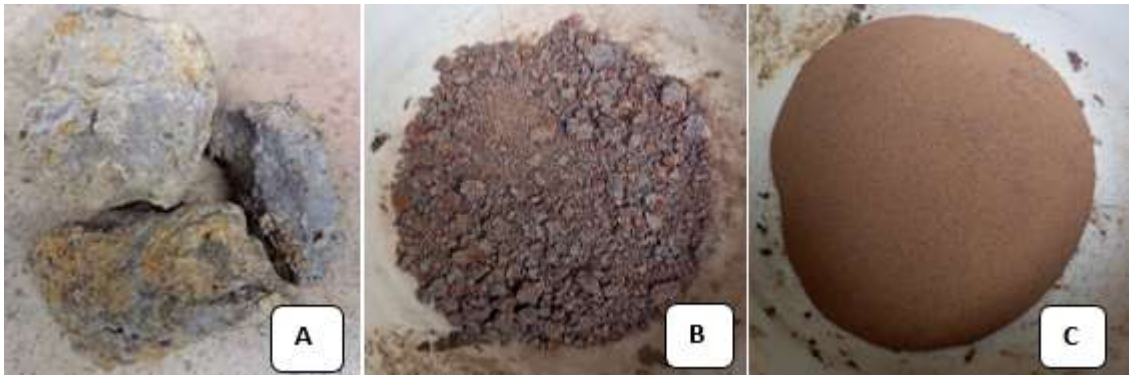

Fig 1 Clay sample preparation process. A) Raw clay sample. B) Crushed clay sample C) Sieved clay sample.

Beneficiation of Clay: Clay sample was beneficiated by soaking in distilled water for three days in a 2.0L conical flask, intermittently agitating with an overhead stirrer/homogenizer, and daily decanting of the suspended impurities. After passing through a 100-micron sieve, the mixture was allowed to settle for 24 hours (Kayode et al. 2019). To remove the more reactive phases of the sample, the beneficiated clay was centrifuged, air dried for about 48 hours, and then calcined for 6 hours at a temperature of about 600°C to activate and ionize the clay sample for easy mineral and chemical composition and identification.

Cation Exchange Capacity (CEC): Approximately 25.0g of soil was added to a 500 ml Erlenmeyer flask, 125 ml of 1M NH_4OAc was then added and mixed thoroughly then allowed to stand for 24 hours after which it was filtered to remove dirt using moist filter paper and repeated until filtrate became clear. The sample was then gently rinsed with NH_4OAc four times in increments of 25ml, allowing each addition to filter through while guarding against the soil drying out or cracking. The leachate was discarded after determining the exchangeable cations. The soil was then washed with large amount of 95% ethanol to remove extra saturating solution. The adsorbed NH_4 was removed by slowly leaching with 50ml of 1.0M KCl solution. The leachate was added to a clean 250ml volumetric flask and diluted to mark. This was emptied into a 500ml conical flask and titrated with 0.1M HCl to measure the concentration of NH_4-N with the use of methyl red indicator.

$$CEC\left(^{cmolc}/_{kg}\right) = (NH_4N_{in\ extract} - NH_4N_{in\ blank})/14$$

Where NH_4-N is reported in mg NH_4/L

*Determination of Moisture Content***:** The method reported by Okop and Ekpo (2012) was used to determine the moisture content. 5g of soil was weighed in a crucible using the electronic mass balance. The crucible containing the soil was placed in an oven at 110ºC. At time intervals of 20 minutes, the crucible was taken out and weighed and the weights recorded. The process was continued until constant weight of the soil was obtained. The moisture content was calculated using the following equation;

$$\%Moisture = \frac{W_m - W_d}{W_m} \times 100$$

W_m is weight of moist sample. W_d is weight of dry sample

Soil-pH and Electrical Conductivity (EC) (USDA, 2003)**:** Using a desktop pH meter, the hydrogen concentration of the soil sample was monitored. In a soil to water extract ratio of 1:10, soil pH was measured. Each soil sample weighed five grams (5.0 g), and 50 ml of distilled water was added to each sample cell. The dirt lump was mixed to create a homogeneous slurry, and then pH (Jenway 3015 model) and EC (Jenway 4010 model) probes were inserted into the sample, respectively. Once the sample had stabilized at 30ºC, the pH and EC were measured.

Surface Morphology: Benchtop Confocal Microscope BC43 was used to determine the surface morphology of the clay sample. In scanning electron microscopy (SEM), a strong stream of high-energy electrons was used to make different signals on the surface of solid objects. Using the SEM data collected from the surface of the sample, a two-dimensional image was produced which revealed the change in properties like chemical composition, texture, and material orientation.

Functional Groups: Functional groups of compounds contained in the sample were determined using Fourier Transform Infrared (FTIR) Cary 630 Spectroscopy by Agilent Technologies, Scientific, CA, USA. The sample (1mg) and KBr (4mg) were combined to form a pellet which was subjected to infrared light with a wavelength range of 10,000 to 100 cm^{-1} by the FTIR instrument, some of which passed through and part of which was absorbed. The absorbed radiation was converted into rotational and/or vibrational energy by the sample molecules.

Surface Area, Pore Volume, and Pore Size: The N_2 gas adsorption and desorption isotherms were used to determine the porous characteristics with the aid of a Brunauer-Emmett-Teller (BET) NovaWin version 11.03, Quantachrome Instrument. Moisture content in the sample was first removed by degassing 200 mg of the sample at 200°C for two hours under nitrogen flow prior to conducting nitrogen adsorption tests. The specific surface area (SSA), the pore volume, and the pore size distribution were all determined using the BJH method using the maximum adsorption at a relative pressure. The surface area and volume of the micropores were calculated using the t-plot method.

Chemical Composition: Thermo Scientific's ARL QUANT'X EDXRF Spectrometer was used to determine the chemical and elemental components of the sample. Thermo Fisher Scientific standard reference material, Montana soil SRM 2710, was used for the EDXRF analysis, which was carried out according to procedure.

Structural morphology: The sample's crystalline phase was identified using the Shimadzu XRD-6000 powder diffractometer with Cu Kα-ray radiation (λ = 0.154nm) (40 kV, 40 mA). The Diffraction technique was conducted with a Panalytical X-Pert Pro diffractometer with Cu K monochromatic radiation, the kind of phases and the quantity of phases present in the sample were detected qualitatively and quantitatively.

Thermo-Gravimetry Analysis (TGA): TGA was conducted on a pre-warmed sample to remove adsorbed water and other volatile substances and heated to 600°C at 10°C/min and kept there isothermally for about 5 – 10 minutes until a constant mass was attained with the aid of a thermo-gravimetric (TG) TGA-55. Then, the furnace was cooled to 100°C and rebalanced.

RESULTS AND DISCUSSION

Physical and Chemical Properties: Physical characteristics of the clay sample (Table 1) revealed a low (2.79%) moisture content thus suitable for making refractory bricks. A brick is more resistant to damage from freezing if its water absorption is less than 3.25%. The electrical conductivity was 36 µS/cm (0.036 dS/m).

Table 1 Physical Properties of Cay Sample

Property (Unit)	Value
pH	6.35
Bulk Density (g/cm^3)	2.28
Porosity (%)	29.84
Moisture Content (%)	12.79
Water Absorption (%)	3.25
E. C. (µS/cm)	36
C. E. C. (cmolc/kg)	17.52

According to Agbai et al. (2022), this value coupled with the CEC (17.52 cmolc/kg) suggests the clay has low activity and can be highly weathered. Due to the

production of coarse particles that promote poor aggregation, heavily worn soils are more prone to erosion (Khitam et al. 2015; Kome et al.2019).

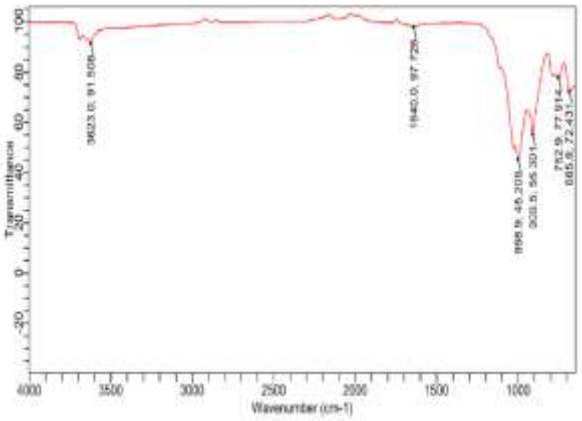

Fig 2: FTIR Spectrum of sample analysis

FTIR Analysis of Clay Sample: Results from the FTIR spectrum showed absorption between 3622.97 cm^{-1} and 685.83cm^{-1} (Figure 2). The IR spectra of silicate ions (SiO_4^{-4}) showed two distinct, long peaks between 998.926 cm^{-1} and 909.47 cm^{-1} (Table 2). The presence of (C - Cl) and (C - Br) groups attributed to aliphatic chloro compounds and aliphatic bromo compounds, respectively, in the clay samples was indicated by stretched peaks at 752.92cm^{-1} to 685.83cm^{-1}.

EDXRF Analysis of Clay Sample: The purpose of the XRF study was to determine the chemical composition of the clay and any potential chemical alterations introduced by treatment. The spectrum of the chemical analysis of the clay sample is shown in Figure 3 along with the peaks. While other oxides including magnesium oxide, calcium oxide, potassium oxide, zinc oxide, and titanium oxide are found in minor amounts in the clay, silica is present in significant number. The most abundant elements in the sample is observed to be silicon (45.116%), aluminum (20.39%), iron (3.2628%), and magnesium (2.61%).

Table 2: Results of FTIR analysis on clay sample

Peak Number	Wavenumber (cm^{-1})	Intensity	Functional Group	Comment
1	685.83016	72.43086	C – Br, stretch	Aliphatic bromo compounds,
2	752.92224	77.91423	C – Cl, stretch	Aliphatic chloro compounds
3	909.47043	55.30056	SiO_4^{-4}	Silicate ion
4	998.92654	45.20918	SiO_4^{-4}	Silicate ion
5	1640.02865	97.72610	- C = N -	Open-chain imino
6	3622.97238	91.50831	OH	Secondary alcohol

XRD Analysis of Clay Sample: Results of the clay sample mineral content identifications indicated the presence quartz, muscovite, albite and orthoclase. Figure 4 shows the samples XRD patterns in its spectrum. The clay sample displayed sharp reflection at about 28º, which is attributed to quartz at 77% presence with an intensity of about 950cps. Muscovite, which is a common impurity in kaolin-group minerals, is present in appreciable quantities in the clay sample, as seen by the presence of major peaks at 45Aº. Other peaks such as albite and orthoclase with presence at 7% and 9%. Figure 4 shows how the characteristic basal reflections of the clay material groups changed when they were treated with different chemicals and heat (Putzolu et al. 2018).

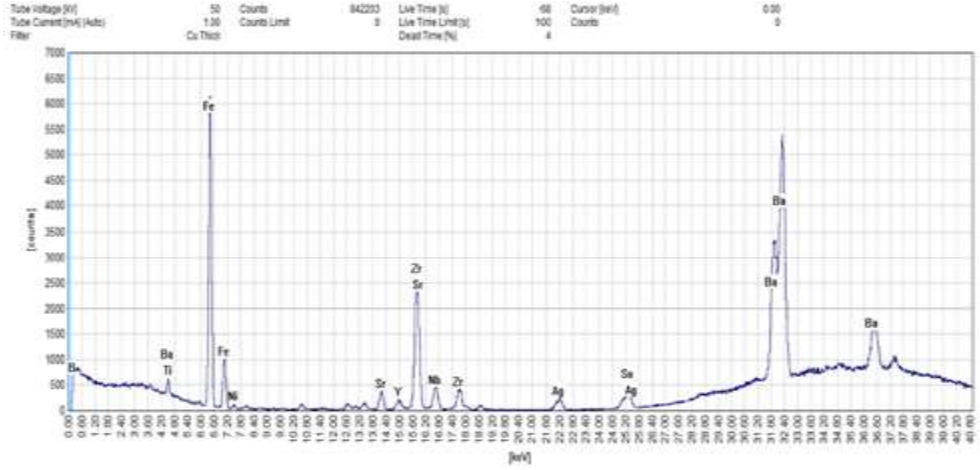

Fig 3 EXDRF analysis spectrum of clay sample

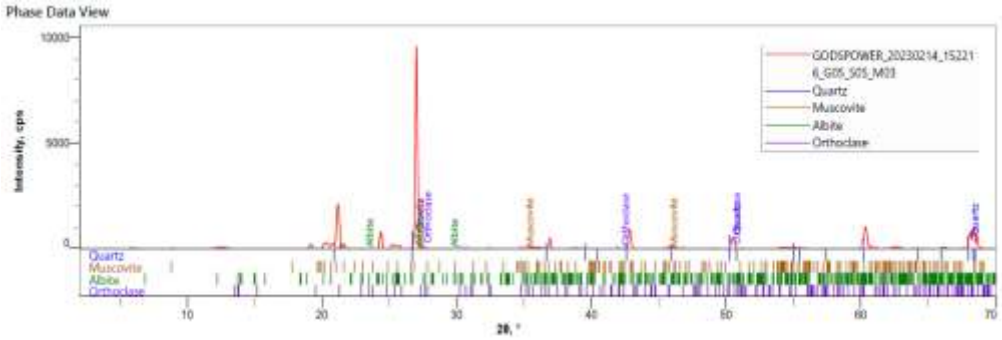

Fig 4 XRD pattern of clay sample

The impact of the beneficiation on structural alterations in the clay material was observed using the X-ray diffraction method. The clay displayed sharply defined reflections at 2 values of 120 and 250, which translate to the values of 7.154. Reflections of [001] can be seen at these summits.

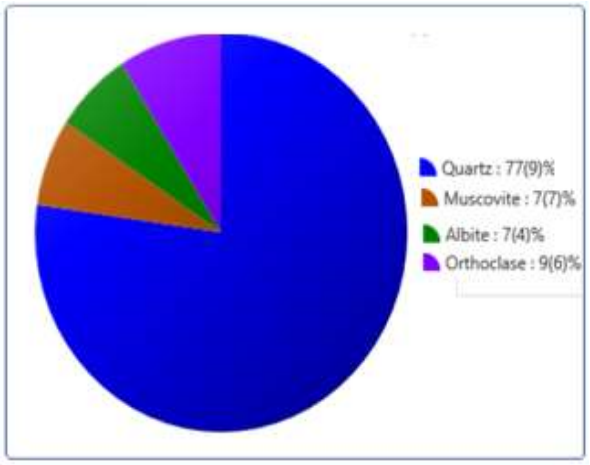

Fig 5 Plots of XRD results

Because of the silica to aluminum concentration ratio in the clay sample, Table 3 revealed that it can be used to synthesize a zeolite of the faujasite type (Mgbemere et al. 2017). Quartz makes up the majority of the clay's composition, with minor elements of Muscovite, Albite, and Orthoclase. The study found that quartz had the highest relative amount of minerals contained in the clay sample. The analysis findings indicate that the tested clay is ball clay.

Table 3 Quantitative Analysis Results

Phase name	Formula	Figure of Merit	Space Group
Quartz	SiO_2	1.688	154 : P3221
Muscovite	$K\ Al_2(Si_3Al)O\ 10(OH.F)_2$	3.199	15 : C12/c1
Albite	$Na\ Al\ Si_3\ O_8$	3.168	2 : P-1
Orthoclase	$Al_2O_3\ .\ K_2\ 6SiO_2$	3.211	12 : C12/m1

TGA Analysis of Clay Sample: In order to determine the carbonization conditions for the clay sample, the samples were analysed by thermogravimetric analysis (TGA). Volatilization of a liquid breakdown product or inert gas causes weight loss. The thermogram indicated decreasing mass plateaus as the breakdown progressed (Ye et al. 2019). Figure 6 depicted a close-up of the chemidesorption curve. It exhibited two weight loss zones which showed desorption from weakly acidic sites and highly acidic sites. Desorption from mildly acidic sites stopped at the TGA derivative minimum curve peak. The thermal stability of clay depended on the Si/Al ratio, cation type (La^{3+}, Ca^{2+}, or Na^{+}), and degree of ion exchange (Majchrzak-Kucba, 2013). DTA tested USY clay's hydrothermal thermal stability. The exothermic DTA peak was assumed to commence crystal collapse.

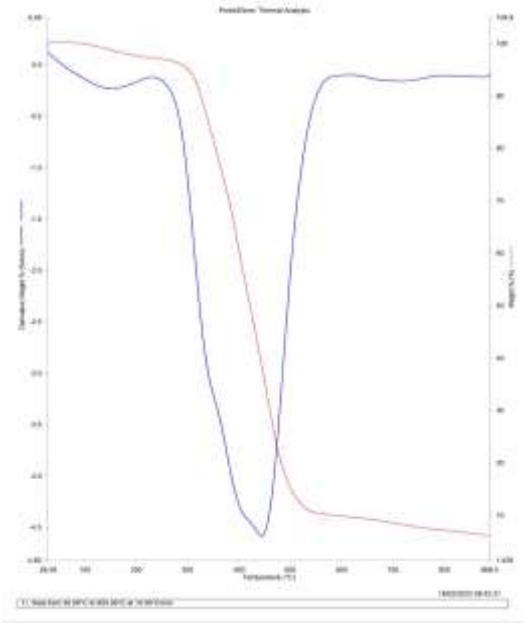

Fig 6 TGA/DTA curves of clay sample

SEM Analysis on Clay Sample: Figure 7 shows the SEM image of the clay sample at 1500x magnification. The sample revealed spherical particles in the 20-m

range that were largely homogenous. The morphologies of surfaces of these particles, which were rough and angular, made it easier to distinguish between the crystal and the amorphous phases.

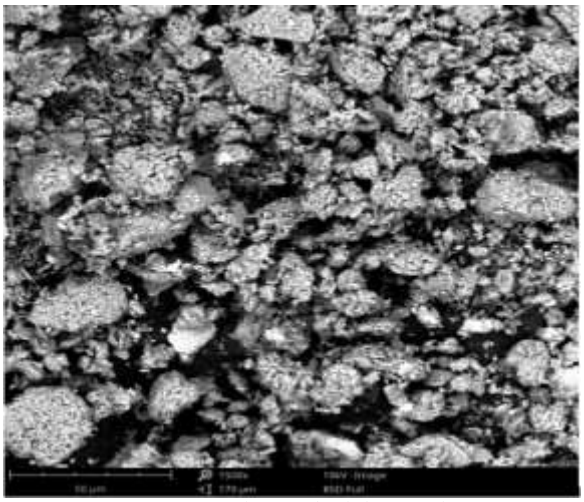

Fig 7 SEM micrograph of clay sample

BET Analysis on Clay Sample: Results of BET analysis revealed the pore sizes, pore volume and surface area of the particles of clay sample (Table 4). Results of multi-point BET analysis plot revealed the surface area of the sample was 287.252m^2/g while the DA method gave the pore diameter to be 214nm. The pore diameter according to BJH was 3.108nm with surface area as 290.370m^2/g and pore volume was 0.140cc/g.

Table 4 The BET Area-Volume summery

Surface Area Data	
Single Point BET	270.9 m^2/g
Multi Point BET	287.3 m^2/g
Langmuir Surface Area	483.7 m^2/g
BJH Cumulative Adsorption Surface Area	290.4 m^2/g
DH Cumulative Adsorption Surface Area	308.9 m^2/g
t-Micropore Area	287.3 m^2/g
DR Micropore Area	409.9 m^2/g
DFT Cumulative Surface Area	161.8 m^2/g
Pore Volume Data	
BJH Cumulative Adsorption Pore Volume	0.1404 cc/g
DH Cumulative Adsorption Pore Volume	0.1436 cc/g
DR Micropore Volume	0.1456 cc/g
HK Micropore Volume	0.1015 cc/g
SF Micropore Volume	0.0672 cc/g
DFT Cumulative Pore Volume	0.1276 cc/g
Pore Size Data	
BJH Adsorption Pore Diameter	2.108 nm
DH Adsorption Pore Diameter	2.108 nm
DR Micropore Pore Width	3.866 nm
DA Pore Diameter	2.140 nm
HK Pore Diameter	0.367 nm
DFT Pore Diameter	2.647 nm

Conclusion: The physical and chemical analysis of the clay sample showed that it consists of silica and alumina. From the result, it can be seen that the sample belonged to the category of kaolinite and ball clay. Chemical properties revealed a high silica concentration, making them suitable for the manufacture of zeolite X and Y. Two lengthy and distinct peaks in the IR spectra analysis also showed the presence of silicate ions. The samples revealed spherical particles which are mainly homogenous.

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