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Assessment of the Mineralogical and Elemental Compositions of the Clays of Otukpo, Benue State, Nigeria as Potential Raw Materials for Manufacturing of Bricks, Refractories and Geopolymers

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Abstract
More than ever, the present age has witnessed a global rise in the cost of building and construction materials especially cement and unprecedented high-rising emission of greenhouse gases like CO_2. In the present century of fast-growing global building and construction industries, it has become highly essential to find alternatives to cement mortar and concrete, through green chemistry research initiatives. Therefore, the mineralogical characterization and elemental assessment of Otukpo clays was performed by a combination of different instrumental analysis. This study utilized X-ray fluorescence (XRF), flame atomic absorption spectroscopy for the compositions of the oxides of the elements of the clay, XRD for mineralogical analysis and SEM for crystal structural analysis. The results showed that the clay contains the elements: Boron aluminide-Bal (95.719%) > Fe (2.468%) > K (0.973%) > Ti (0.588%) > Ca (0.169) and other trace elements including: Zr = 0.021%; V = 0.015%; Cr = 0.013%; Sr = 0.011%; Bi = 0.010; Rb = 0.008% and Nb = 0.004%. XRD showed that the clay contains the minerals: Al_2O_3 = (197368 ppm); SiO_2 = (97200 ppm); Fe_2O_3 = (63921ppm); CaO = (28040 ppm); MgO = (20150 ppm); TiO_2 = (11211 ppm); K_2O = (2008.30ppm); Na_2O (674ppm). These results reveal a high content of Al_2O_3 and SiO_2, which are suitable requirements for production of cement and other geopolymers. And therefore, the clays could be explored and utilized to meet the housing deficit of the nation, and also as a rich source of minerals and metals that can be extracted, for foreign exchange earnings.

Keywords: Cement, Concrete, Building, Construction, Mortar

Introduction

Before now, the study of mineralogical composition of naturally occurring minerals like clays, and metal ores was an area long been exclusively within the domain of geology and mining, but with the advent of X-ray diffraction (XRD), X-ray fluorescence (XRF), flame atomic absorption spectroscopy (ASS), scanning electron microscope (SEM) couple with EDS equipment, compositional mineralogical analysis of both natural and synthetic mineral substances is becoming increasingly relevance to inorganic, industrial and even material chemists.

As we continue to navigate through this 21st century with emerging and soaring high cost of living, high cost of building, construction, educational materials, and high cost of electricity and access to healthcare facilities which can be very challenging, it is essential to recognize that such challenges can come with all profound ramifications including inability to afford good and comfortable houses for living [1]. Therefore, emerging modern scientific and technological approaches to produce affordable building and construction materials that are not only eco-friendly but also help in sustaining our natural green environment is being encouraged.

Meanwhile, it seems more correct to say that, of all the naturally occurring mineral substances within man's environment particularly the Earth crust, clays seem to be the most abundant in varieties than other Earth's mineral substances. It is equally necessary to understand, in first instance that, clay soils are abundantly found in various regions across the globe including Nigeria in Africa [2] and of all the various types of soil, clays have continued to occupy a special place in the human history due to their early usage in the construction of huts and bricks during the pre-civilization era.

Notably, clay is essentially a type of soil which is made up of very fine mineral particles with relatively low quantity of organic materials. Interestingly also, it is imperative to note that, as soils, clays are rich in minerals and plant nutrients and they have continued to play the role of providing the environment with essential minerals for almost all plants' growth and thus, for nearly all life of plants on the Earth's surface [3]. The work of Adams and co-workers [4] has established that most clays are formed as a result of weathering processes including ceramic clays, clay shales, mudstones, glacial clays with great volumes of detrital and transported clays, and deep-sea clays (red clay, blue clay, and blue mud) and these are all characterized by the presence of one or more clay minerals, together with varying amounts of organic and detrital materials, among which quartz is predominant [5].

Moreso, structural evidence has established that, clay minerals have crystalline sheet-like structures, that consist of hydrous alumino-silicates and metallic ions [6]. Additionally, there is also increasing structural information that the common clay minerals include, Kaolinite, Montmorillonite, Halloysite, Smectite, Illite, Chrysotile, Palygorskite, Sepolite allophane, chlorite group and Imogolite [4]. In the same study, it has been well noted that, clay minerals are either amorphous or crystalline with some being two-layered type (Kaolinite), three-layered type (Montmorillonite), mixed-layered type (chlorite group) and chain-structured type (Palygorskite) [6].

At this point, it is instructive to consider that, clay in general embraces exceptional unique set of properties that profoundly influence it usage for planting of crops, building and construction works, and for other environmental considerations. Some insights into these properties are provided by different reporters. Notably, Mukherjee [4] has asserted that, clays generally have low strength, high compressibility and high volumetric changes. That, as a result of clay's high plasticity, permeability, bearing capacity and settlement characteristics, it is a material that has been studied and is still being studied in geotechnical engineering. Furthermore, additional insights into the properties of clays have been provided in review by Firoozi and co-workers [6] and hence, it is necessary to understand that, clay soils are generally stiff when they are in dry state but when become wet and saturated with water, they lose their stiffness. It should be noted that, clays show plasticity when mixed with appropriate amount of moisture and become hard when dried or fired [7].

Unarguably, clay materials according to the report of [3] have a wide variety of uses in engineering. Accordingly, Earth dams are made impermeable to water by adding suitable clay materials to porous soil and water loss in canals may be reduced by adding clay. In addition, it has been recognized that, the essential raw materials of Portland cement are limestone and clays, which are commonly impure, but after acid treatment, clays have been used as water softeners as the clay removes calcium and magnesium from the solution and substitutes sodium [3].

More importantly, it has also been widely reported that another major use of clay is as drilling mud where heavy suspension consisting of chemical additives and weighting materials, along with clays are employed in rotary drilling [3]. Meanwhile, available reports in literature on elemental and mineralogical compositions of clays include that of Mukasa-Tebandeke *et al.* [8]. Mukasa-Tebandeke and co-workers have reported the results on the elemental and mineralogical compositions of clays from Central Uganda which differed from those from the volcanic sediments of the Mt. Elgon in Eastern Uganda and the utilization of the two types of clays was recommended to be strictly based on their structural differences and whereas the elemental, mineralogical, DTA, IR, XRD and pH data on selected clays from different study locations indicated that they were kaolinites.

In another recent report, Anumata *et al.* [9] reported on five clay samples from Iyinwaogba, Umuosode, Alike, Ikenanzizi and Obowo in Southeastern Nigeria. The characterization was based on their physicochemical, mineralogy and elemental analysis using Scanning Electron Microscopy (SEM-EDS) and it was concluded that the clays were good candidates for support in the manufacture of industrial catalysts for petroleum catalytic cracking operation and can also be used to produce good refractory materials for high temperature applications.

In general, the purpose of this study is to have insight about the elemental and mineralogical compositions of clays abundantly available in

Otukpo, in Benue State in order to ascertain whether they possess the perequisite properties and mineralogical compositions that could enhance their usefulness for processing valuable geopolymer materials like cement.

Materials and Methods

Sample Location

The clay samples' sites were carefully selected at different locations in Otukpo Metropolis, where abundant clays exist and the locations were Otada (Latitude 7.2167°N and Longitude 8.1500°E), GRA (Latitude 7.1930°N and Longitude 8.1464°E) and Enugu roundabout (Latitude 7.1982°N and Longitude: 8.1393°E). At each location, the clay temperature was taken and thereafter, the clays were labelled as C/OT I, C/GRA II and C/ER III and were transported to the laboratory for preparation for analysis.

Figure 1: Map of Benue State Showing the Location of Otukpo: Adapted from OpenStreet Map: https://www.openstreetmap.org/copyright).

Sample Collection

The clay samples were collected in sterilized polyethene bags and were taken to Federal University of Health Sciences, Otukpo (FUHSO) laboratory, and were stored under regulated temperature of 35°C for 48 hours. Thereafter, the clay samples were spread on dry pan vessels and debris, sticks and rock were removed. The samples were then sun-dried for 24 hours before being stored in a refrigerated for preparation for analysis.

Sample Preparation

A 5.0 kg portion of each dry clay sample was sieved before they were ground using a jaw (10-300TPH concrete crusher, China) and roller crushers (2PG series, 350 x 350Jpeq, Japan). The samples were then riffled to obtain representative samples by using a Jones Riffler equipment and then followed by homogenization and thereafter, each sample was divided into three equal portions and were stored for analysis.

Determination of Physicochemical Parameters

Temperature measurement

The temperature of the clay samples was taken *in-situ*, at the point of collection of each sample. The temperature of each clay sample was taken in degree Celsius but was converted to Kelvin using the relationship; T ^oC = (273 + t ^oC)K.

Particle size measurement

Particle size distribution measurement may be carried out by either using sieve analysis method or hydrometer analysis. However, in this study the clay particle size was determined using sieve analysis method, where each of the clay samples freed from debris and stones was sieved using a mesh of known hole size (75 – 300 µm).

Electrical conductivity measurement

Electrical conductivity of the samples was determined using a sensitive electrical conductivity measuring meter, DDS meter (DDS 307) with ID number 023883. The accuracy Degree of the instrument was 0.001 with the measuring range of 0.000$\mu S/cm$ to 199.90 $\mu S/cm$ and the measurement was carried out in FUHSO Chemistry/Biochemistry laboratory.

pH Tests

The clay samples (100. 0 g) were dissolved in distilled de-ionized water and the resulting solution was first tested with litmus papers and then with pH meter equipment (Digital pH meter). With litmus papers, the solution turned a blue litmus paper to red but there was no observable effect or changed with red litmus paper. However, with the digital pH meter (Lab Tech Model, USA), the pH reading was obtained at a standard temperature of 25^oC or 298^oK.

Specific Gravity Measurement

This measurement was performed using relative density glass bottle (50ml/20 ^oC) in order to determine the specific gravity of the clay samples and the results were further confirmed using Archimedes' principle methodology.

Mineralogical and Chemical Analysis

For each powdered clay sample, 40.0 g was analyzed for structural arrangement of particles in the clay using Scanning Electron Microscope (SEM) and for optical mineralogical properties. For the XRF analysis, 50.0 g of fine powder clay samples were mixed with a binding aid and pressed to produce homogeneous sample pellets and thereafter the samples were subjected to XRF analysis using Thermo Fisher Scientific Machine (Ma 01876, USA; XL3-98293) model.

Thermal Properties Tests

A 100.0 g portion of each clay sample obtained in Otukpo was homogenized to form composite sample. The thermogravimetric test of the clay samples was carried out using a muffle electric furnace. For the test, 100.0 g of the composite clay sample was heated to high temperatures in the muffle electric furnace (SX-5-12; PC:22070222/2000°C) and the weight loss behaviour of the samples were carefully monitored at various temperatures.

All the experiments were carried out in triplicates.

Results and Discussion

Table 1: Mean Physico-Chemical Properties of Otukpo Clays in Benue State, Nigeria, from **Three Study Sites**

Property	**Clay soil of Otada**	**Clay soil of GRA**	**Clay soil of Enugu roundabout**
Temperature (°C)	55- 1320	55-1320	55-1320
Conductivity (mS/cm)	2.13 x 10^2	2.14 x 10^2	2.12 x 10^2
pH	6.15	6.14	6.16
Colour	Grey	Grey	Grey
Particle size (mm)	1.60	1.61	1.59
Pore volume (mL g^{-1})	0.32	0.32	0.32
Specific surface area (m^2g^{-1})	153.50	153	153
Refractive index	1.55	1.55	1.55
Specific gravity	3.90	3.80	3.70
Harness	2.61	2.61	2.61

The results of the physico-chemical properties of the clays in Table 1 show that, the clays have good conductivity, small particle sizes within the range of 1.59 -1.61mm and high refractive index of 1.55. Typically, of clays, the pH of the samples was slightly acidic between the range of 6.14 - 6.16. The simplest explanation of the data is that the clays have moderate hardness and specific gravity in agreement with those reported in literature. Clearly, the high conductivity of the clays suggests availability of mobile ions while the slightly acidic pH has defensibly suggested that the clays could be suitable for production of geopolymer products like cement. The charge-balancing due to mobile ions like cations is very vital in determining the structural integrity and fragility of geopolymers [10, 11].

Undoubtedly, it should also be noted that the clay has high specific surface area of (153 m^2g^{-1}) and according to Abdullah *et al.* [10], low surface area of clay limits the dissolution of kaolin/kaolinite mineral to provide Si^{4+} and Al^{4+} ions for further reaction, hence such clays are good materials for manufacturing of refractories and geopolymer materials. The result of thermal tests expounds that the clays experience weight loss from 55 °C and between 450 °C to 620 °C, a signification weight loss was observed as shown in (Figure 2). The high thermal property of the clays up 1320 °C as shown in (Figure 2) is an indication that the clays if utilize as refractory materials can withstand high temperature industrial applications.

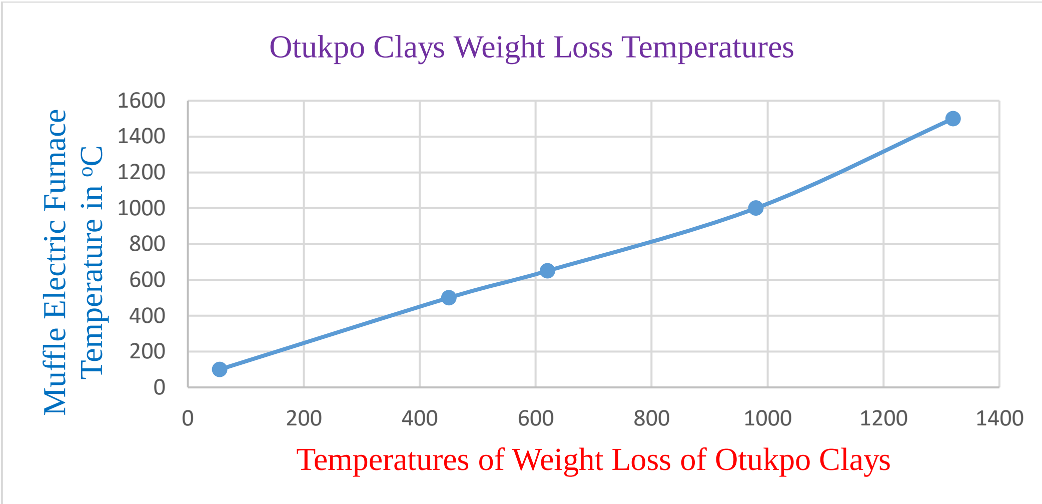

Figure 2: Thermogravimetric Graph of Weight Loss Temperatures of Otukpo Clays

Table 2: XRF Result of Elemental Composition of Otukpo Clays Produced by Thermo Fisher Scientific Machine, 2 Radcliff Road, Tewksbury, Ma 01876, USA; XL3-98293; Read No: 2330; 2024-09-01; 11:48.

Mineral	Result of % Composition	± Error
Bal	95.719	± 0.023
Nb	0.004	±0.001
Zr	0.021	±0.001
Sr	0.011	±0.001
Rb	0.008	±0.001
Bi	0.010	±0.005
Fe	2.468	±0.055
Cr	0.013	±0.001
V	0.015	±0.001
Ti	0.588	±0.012
Ca	0.169	±0.008
K	0.973	±0.020

The XRF result of the clay is shown in Table 2 and the first impression of these data is that there are significant number of elements including high percentage amount of boron aluminide (Bal) mineral. The elements and the mineral with significant percentage amount in the clay are in this order: Bal (95.719%) > Fe (2.468%) > K (0.973%) > Ti (0.588%) > Ca (0.169) and other trace elements are Zr = 0.021%; V = 0.015%; Cr = 0.013%; Sr = 0.011%; Bi = 0.010; Rb = 0.008% and Nb = 0.004%. Meanwhile, from this result there is some indication that a new discovery may have been made and this is of special interest.

The presence of high percentage of boron aluminide (Bal) is somewhat unexpected occurrence as boron aluminide in its original form is more of a synthetic alloy with almost no mention of its natural source in literature. Therefore, with this result it implies that the clays contain very high content of aluminum (Al) and (Fe), thereby making the clays to occupy special places in suitability for production of refractory bricks and other geopolymer products. Additionally, the presence of K in the clays suggests the presence of kaolinite mineral ($Al_4(Si_4O_{10})(OH)_8$) while the presence of Ti suggest the presence of TiO_2 which are very important clay minerals. Also, the presence of K and Ca are very essential for balancing the negative charge of Al^{3+} in IV-fold coordination; n=degree of polymerization for production of geopolymer materials [12].

Table 3: Results of Atomic Absorption Spectroscopic Analysis of Otukpo Clay; (JN: 356/2024)

Mineral	Result in Part Per Million (PPM)	% Composition
SiO_2	97200	9.72
Al_2O_3	197368	19.74
Fe_2O_3	63921	6.39
TiO_2	11211	1.12
CaO	28040	2.80
MgO	20150	2.02
Na_2O	674	0.067
K_2O	2008.30	0.201
Loss of Ignition (LOI)	146200	14.62

The result in Table 3 shows the oxides of the elements (ppm) in the raw clay, which are in the order: Al_2O_3 (197368 ppm) > SiO_2 (97200 ppm) > Fe_2O_3 (63921ppm) > CaO = (28040) > MgO (20150ppm) > TiO_2 (11211) > K_2O (2008.30ppm) > Na_2O (674ppm). A satisfactory interpretation of this result seems to be a clear testament to the fact that, Otukpo clays are very rich in alumina and silica and this appears to support the argument that, aluminosilicate materials like clays are rich in alumina and silica content [10, 13, 14].

This may point out that, silica (SiO_2) and Al_2O_3 were the main oxide minerals' components of the clay while Fe_2O_3, CaO, MgO and anatase (TiO_2) were also present in considerable amounts. Emphatically, another obvious information provided by this result is the ratio of Al_2O_3/SiO_2 in Otukpo clay, which is 1:2. However, it has been very recently explicated that the alumina-to-silica ratio for high kaolinite clay is 1:1 [15, 16]. Importantly, industrial relevance of Otukpo clays with high alumina to silica ratio of (2:1), is that the clays are suitable refractories materials for high temperature applications and for production of ceramics and fired bricks.

Clearly, it is of a special interest to agree that, the ratio of Alumina-to-silica in the clay has characteristically confirmed that Otukpo clay is high kaolinite ($2Al_2O_3.4SiO_2.4H_2O$) clay and the detection of K_2O in the clay further indicates the presence of illite, another essential clay mineral. Interestingly also, another good piece of evidence that further support the good quality of Otukpo clays as a material for production of refractories and geopolymers is the presence of anatase which confirms a considerable amount of TiO_2 in the clay as observed in XRF result. More often however, from the foregoing analysis, it can be safe to submit that, the high value of loss of ignition (LOI) as (146200 ppm), indicates a decrease in weight related to the loss of adsorbed water on the external surfaces of the raw clay due to dehydration reaction as the clay was subjected to spectroscopic analysis and the major weight losses occurred between the temperature range of 450−1320°C. Additionally, the thermal tests revealed the thermal behaviour of the clay as that of fireclays with first significant weight loss observed between the temperatures 450ºC and 620ºC, and this was probably due to loss of water of hydration by the kaolinite mineral [17] to form metakaolin as thus;

$$2Al_2O_3.4SiO_{2}.4H_2O_{(s)} \longrightarrow 2Al_2O_3.2SiO_{2(s)} + 4H_2O_{(g)}$$

Therefore, it is obvious that at this point it has to be said that the Otukpo clays in addition are likely fireclays with good thermal service property and fire resistance, that make them suitable to be fired into shaped refractories like bricks and shapeless castable or monolithic refractories or use directly to produce clay-based unfired bricks and ceramics.

Meanwhile, there are several approaches for further explaining the mineralogical analysis results of these clays, however, it is instructive to consider in summary that, Otukpo clays are somewhat fireclays containing the required mineral compositions (Al_2O_3, SiO_2, Fe_2O_3, CaO, MgO, TiO_2) in appropriate ratios and with other essential properties that qualify the clays as suitable raw materials with composite minerals for manufacture of refractory bricks and geopolymer products. Emphatically, these explanations are consistent with the suggestions of other researchers elsewhere. It equally pertinent to express here that, the overall percentage abundance of minerals in the clay detected as shown in Table 3 is 56.678% and the remaining 43.322% undetected might be for the boron aluminide detected by XFR analysis.

Certainly, the result of the current status of the investigated clays is that, they contain very high percentage of boron aluminide (Bal), an intermetallic alloy of boron and aluminum with properties similar to both ceramic and metal and it is hard to dispute the existence of the compound [18] going by the percentage shown in (Figure 1). Hence, the presence of boron aluminide, a cheap, very dense, and compatible traditional propellant components, having heat of combustion close to that of boron and which can be obtained as nanosized particles by the means of plasma recondensation is thought to be a strong value-added component to the clays [18].

Interestingly, with the current high demand of boron aluminides, as aluminides have found applications in hydrogen storage technology, industrial manufacturing, and in coatings for furnaces and other high temperature applications [18], it is our utmost believe that, the Otukpo clays will be subjected to further studies as our meager information cannot establish the particular boron aluminide present in the clays, as whether it is aluminum diboride (AlB_2), or aluminum dodecaboride (AlB_{12}) or aluminum magnesium boride, or molybdenum aluminum boride or whether it is the newly synthesized titanium boron aluminide that in a recent series of hyper-gravity experiments, the European Space Agency (ESA) has created the unique alloy of whose light weight and durability is believed may prove critical to the aeronautical industry [18]. The percentage mineralogical and elemental composition of the of Otukpo clays are as shown in Figure 3.

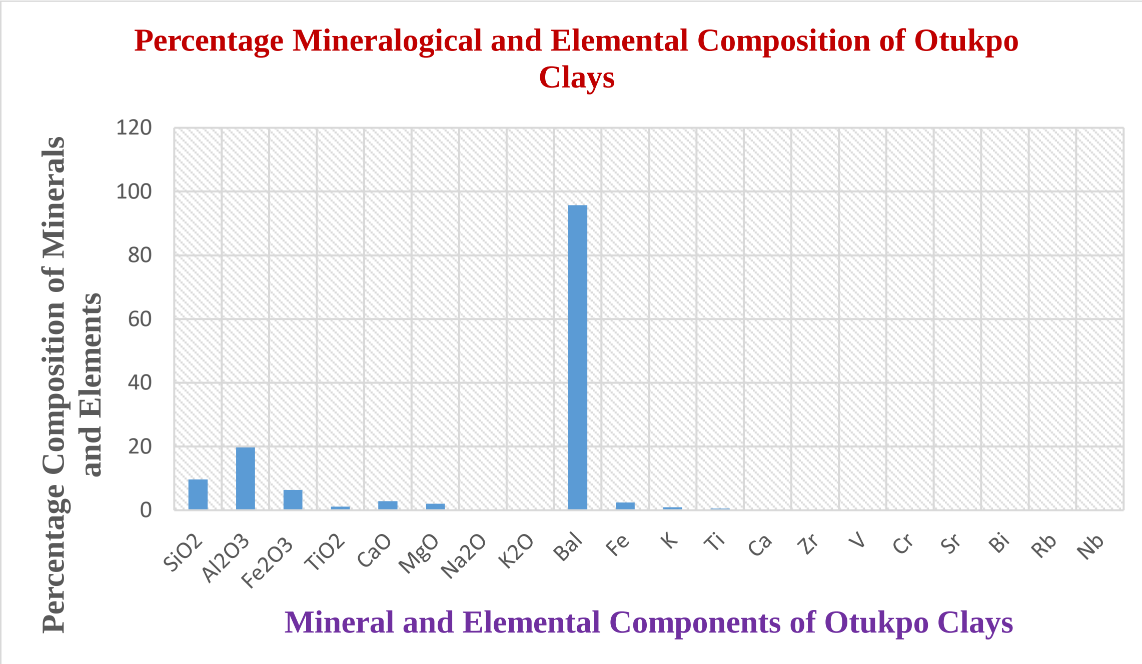

Figure 3: **Percentage Mineralogical and Elemental Composition of Otukpo Clays**

Figure 4: Scanning Electron Microscope (SEM) of GRA, Otukpo Clay Obtain by Thermo Fisher Scientific Machine, 2 Radcliff Road, Tewksbury, Ma 01876, USA; XL3-98293; Read No. 2330.

The result of SEM shown in Figure 4 agrees that the clays are made up of fine grains of clay minerals of relatively very small sizes. It is necessary to point out that a great deal of flexural strength information has been provided by the particle size and their arrangement within the clay structure as the flexural strength of clay-based geopolymer products depends on clay particle size and the smaller the particle size of clays, the decrease in probability of existence of crack-producing flaw along with a corresponding increase in the flexural strength of the materials. Also, the SEM result (Figure 4) of Otukpo clays shows that the clays irrespective of their locations, have similar particle size arrangements, are rich in mineral and elemental compositions and therefore are all suitable materials for production of geopolymer products.

Conclusion

Recently, scientific research is shifting from chemical chemistry to materials and green chemistry where attention is focusing on utilization of cheap raw materials like clays and commercial application of geopolymer products for sustainable development and maintenance of green environment. It has been shown that geopolymer concrete has good engineering properties [19], and moreover, it is a binder material which is produced from an alumino-silicate materials like clays and laterites through activation in a high alkali solution [20, 21, 22]. Therefore, geopolymers are badly needed to reduce the global CO_2 emission and this has continued to encourage researchers to persistently search for alternative sustainable building materials from those naturally available materials in localities that will lead to lower embodied energy and carbon dioxide emissions [23, 24, 25]. It is worthwhile after all to establish here in general conclusion that the valuation of inherent mineral composition of Otukpo clays reveals that the clays contain significant percentage quantity of silica (9.72 % of SiO_2), alumina (19.74 % of Al_2O_3), magnesia (2.02 % of MgO), titania (1.12 % of TiO_2) and lime (2.80 % of CaO) minerals which are the prerequisite minerals required in alumino-silicate materials for qualification as raw materials for production of refractory bricks, geopolymers and other ceramic products. Also, these inherent mineralogical compositions of the investigated clays (high alumina-to- silica ratio of 2:1) and their properties (high thermal property of 1320 ^oC, high electrical conductivity of 2.14 x 10^2 mS/cm and small particle sizes of between 1.59-1.61) have equally suggested that the clays are good fireclays containing appreciably amount of kaolinite and illite clay minerals.

Therefore, by implications, since alumina, silica, lime and magnesia are the common important materials used in the manufacturing of refractories and they are sufficiently present in Otukpo clays and in addition, fireclays are well-known for their extensive use in the manufacture of refractories, hence, we believe that with the results of this investigation, Otukpo clays are suitable materials for utilization for production of refractory bricks and other ceramic products. In addition, the presence of iron ore (hematite-Fe_2O_3) up to 6.39 % has equally added value to the clays which can be used as a rich source of iron for economical extraction of iron metal. We therefore call on government of Nigeria at all levels, industries and individual manufacturers to avail themselves with this information in order to explore the possible maximum exploitation of the natural abundance materials of the area for economic benefit of Nigeria.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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