



Determination of the Suitability of Clay Samples from Ebonyi State, Nigeria for Ceramics Production

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Abstract

The characterization of clay samples from four locations in Ebonyi State (Ezamgbo, Abakaliki, Ikwo and Abaomege) and the determination of their potential for ceramics and other industrial applications were studied. The physicochemical characteristics as well as the thermal degradation pattern of the clay minerals were determined following American Society for Testing and Materials (ASTM) standard methods of analysis. X-ray fluorescence (XRF) and thermo-gravimetric analyzer (TGA) were used for mineralogical and thermal degradation analyses, respectively. The results of physical characteristics of the clay revealed that moisture ranged from 22.63% to 27.36%. Other results are apparent porosity, 25.73%-31.26%; linear shrinkage, 2.60%-8.00%; cold crushing strength 1440 KN/m² - 1555 KN/m²; apparent density; 1.95-2.60 g/cm; bulk density; 1.34-1.84 g/cm³; plasticity; 22.60-46.00% and loss on ignition; 8.80% - 12.60%. Silica (SiO₂) has the highest composition, with a range of 69.24%-62.29%. Other results of chemical analysis are Alumina (Al₂O₃) (18.49%-22.58%), Fe₂O₃ (5.49%-7.39%), ZnO (3.00%-5.00%), TiO₂ (0.023%-2.95%). The thermal behaviour showed weight loss similar to kaolinite degradation. The results revealed that the clay contains two major minerals: kaolinite and quartz, montmorillonite and other alkali and basic oxides in small amounts. When compared with standards, the results revealed that some of the physical and chemical parameters are within acceptable limits, which makes the clay from the four locations suitable as raw material for production of ceramics and with potential in various industrial applications. These potentials therefore qualify the exploration of these huge clay deposits for industrial applications.

Keywords: clay minerals, characterization, ceramics, physicochemical parameters, industrial applications

Introduction

Solid mineral resources' usefulness has been of great importance to man right from existence; clay minerals affect life on earth in far-reaching

ways. Clay can be defined as a natural material that is composed of fine-grained minerals with layered structures that acquire plasticity when

mixed with a certain quantity of water but harden when fired [1].

Clay does not occur in a pure state; the difference in its nature of occurrence depends on the organic matter, inorganic impurities, and the minerals inherent in them. It occurs mostly in solid sediments, sedimentary rock, and hydrothermal deposits [2]. They are minerals containing a complex aluminosilicate compound with attached water molecules having a general molecular formula of $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ [3]. Clay's origin is from rocks that have been disintegrated through chemical and mechanical processes. The formation of different types of clay minerals is influenced by the nature of the starting material, the physicochemical conditions of the immediate weathering environment, and other environmental factors [4]. Their composition, physical characteristics, and structure determines their applications. Clay products are widely known in various industries to serve different purposes, such as in ceramics, refractory bricks, glasses, porcelain, and floor tiles [5]. The percentage of the mineral oxides inherent in clay determines its usefulness in the production of bricks, ceramics, floor tiles etc. Whereas alkali metal oxides indicate their suitability for ceramic products production [6]

One of the world's top consumers of building materials made from clay is Nigeria. The rate of importation of clayey based ceramics into Nigerian market is far higher than the demand for different clay products. Every region across the

Nigeria has enormous deposits of clay, with different characteristics ranging from one location to another location based on the site's geology. Ebonyi State in Southeastern Nigeria has significant clay deposits. The clay mineralogical and chemical compositions (particularly the silica and alumina contents) make the clay suitable for the production of different industrial products [7]. The sourcing of local materials to meet the increased demand for manufacturing of products is necessitated by the present economic situation of the country. Apart from clay, carbonate rock is another raw material resource used for ceramic production. A type of sedimentary rock that is composed of primarily of sedimentary rocks is called carbonate rocks. The two major types are limestone, which is composed of calcite and dolomite.

They are usually formed near (or at) the earth's surface by accumulation and lithification of fragments of pre-existing rocks or remains of organisms or through precipitation from solution at surface temperatures. The production of ceramic tiles, bricks, and cement can come from limestone clay. The aftermath of limestone firing will lead to mass loss (35-45%) and may cause ceramic products to sinter, leading to problems like brick and tile formation. The dissociated magnesium or calcium carbonate, which forms carbon dioxide (CO_2), forms calcium oxide. Cracks in materials can be formed by expansion of calcium oxide when in contact with water. Researchers have studied the procedures for

setting the correct firing temperature and inhibiting the action of limestone on clays [8].

The main constituents of carbonate sedimentary rocks are calcium carbonate and magnesium carbonate. Research has shown that calcite exhibits bleaching action on burnt products whenever it is added to a formulated mass of clays. This can occur in proportions above 5% and < 30%. They may not react completely if they are presented as large grains (> 125 μm), and the resulting oxides may rehydrate, which can lead to expansion by humidity with consequent cracking [8]. One major attribute that makes clay most suitable for the production of ceramics products is its possession of mechanical resistance; as a result, it must have sufficient plasticity when forming by pressing.

The aim of this study is to examine the characteristics of clay samples from Ebonyi State and their potentials as low-cost raw materials in ceramics production.

Materials and Methods

Mapping of sites

Ebonyi State is one of the states in Southeastern Nigeria. It is located at latitude $6^{\circ} 19' 39''$ N and longitude $8^{\circ} 06' 46''$ E, in the tropical rain forest in Nigeria. It is located in the Benue Trough and Afikpo Syncline. The geology of the lower Benue trough in the Ebonyi area is made up of slightly

deformed Cretaceous sedimentary rocks (thick sequences (500 m) made up of essentially Albian shells and subordinate siltstones of the Asu River group. The study by Ike et al. on the suitability of clay using XRD shows that Manu and Nkporo formations in the Anambra Basin in southeastern Nigeria are basically made up of kaolinites, with smaller amounts of mixed layers, illite, and montmorillonite. According to the Nigerian National Population Commission in 2012, Ebonyi State has an estimated population of about three million (3,000,000), with large markets located in its various cities. The map of Ebonyi State showing the four sampled locations is shown in Figure 1.

Sample collection and preparation

The samples were collected from four different locations (Ikwo, Abakaliki, Abaomege, and Ezamgbo) in Ebonyi State at a depth of about 30 cm using a clean container free from contamination. An average weight of about 1 kg of the clay samples was used as a good representative of the total clay composition from Ebonyi State for this study. The clay samples were dried in open air and sieved with 0.40 mm mesh before oven drying at 110°C to a constant weight, which made them suitable for further analyses.

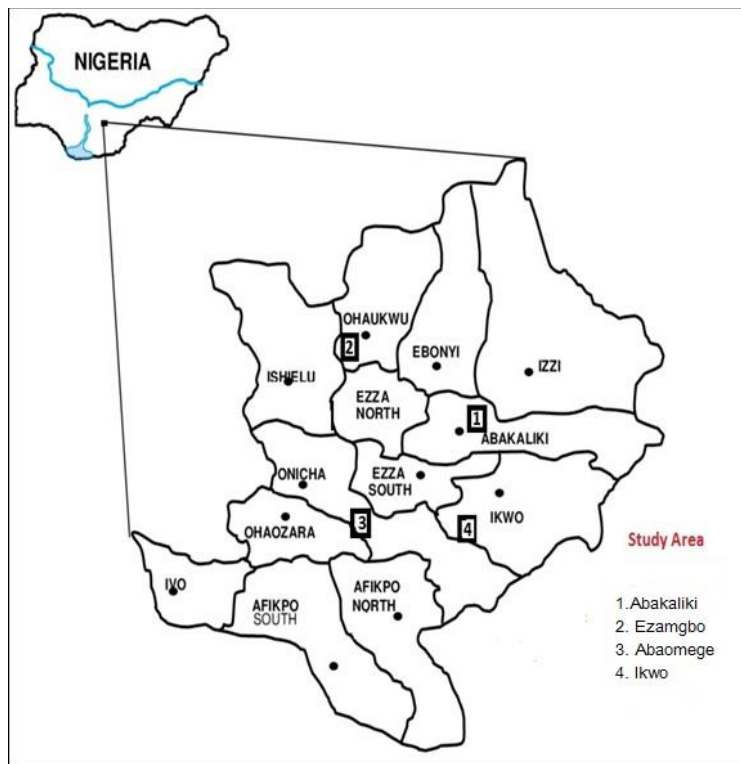


Figure 1: Map of Ebonyi State showing sampled locations (catalog.ihsn.org)

Determination of physical characteristics of clay

Parameters such as soil texture, apparent porosity, bulk density, making moisture, linear shrinkage, refractoriness, cold compressive strength, loss on ignition, and apparent density were determined following American Standard for Testing Materials [9] standard methods of analysis.

Making moisture determination was carried out by weighing the cylindrical test pieces immediately after moulding and recording it as the wet weight, W_o . The test pieces were air-dried for 24 hrs and then dried in an oven at 105°C until a constant weight was recorded. After drying, the test pieces were weighed, and the dried weight was recorded as W_i . The making moisture was calculated using equation 1.

$$\text{Water absorption (\%)} = \frac{(W_o - W_i)}{W_o} \times 100\% \quad 1$$

Where W_o = wet weight of the sample, W_i = weight after drying at 105°C for 24 hours

Porosity and density determination

A specimen of clay brick measuring $5 \times 5 \times 4$ cm was prepared by drying it in an oven at 110°C and heating it in the furnace (at heating rate of 100°C for every 1 min) until it reached a 900°C rise in temperature. The fired sample was put into a desiccator and dried to a weight (M_i) and recorded. It was transferred into 250 mL beaker containing water at room temperature so that it was totally dipped into water. The specimen was agitated and then soaked in boiled water for exactly 30 minutes. Then the new weight (soaked

weight) was recorded as M_2 . The specimen made from the sample was suspended in a beaker of water placed on a balance and the weight (M_3) was recorded, and the apparent porosity of the clay was calculated:

$$\text{Apparent porosity (\%)} = \frac{(W_2 - W_1)}{(W_2 - W_3)} \times 100\% \quad 2$$

where W_1 = weight after drying at 110°C and after being fired, W_2 = soaked weight, and W_3 = suspended weight

$$\text{Apparent Density (g/cm}^3\text{)} = \frac{W_2}{(W_3 - W_1)} \quad 3$$

where W_1 = weight after drying at 110°C and after being fired, W_2 = soaked weight,

W_3 = suspended weight

Bulk density test: The moulded brick (from the specimen) measuring $5 \times 5 \times 4$ cm was made and oven-dried at 110°C after drying it in air for 24 hours and put in a desiccator to cool and weighed (dry weight). The specimen was put into a beaker of water at room temperature and then heated for 30 minutes for air to be released. The soaked weight (W) recorded after cooling the specimen. A Beaker placed on the balance was used to suspend the specimen, and the weight was taken.

$$\text{Bulk density (g/cm}^3\text{)} = \frac{\rho W}{W - S} \quad 4$$

where ρW = the water density, W is the weight after drying for 24 hrs. and oven drying at 110°C , S is the weight after being heated for 30 mins.

Linear shrinkage test: The sample was wetted with water to the extent that it was wedging and cast into a fabricated wood box. The original/initial length of the clay bar was marked

(L_o), and the bar was heated for 3 hours at a temperature of 900°C . The length after firing (final length) was recorded (L_1) and the linear shrinkage was calculated using

$$\text{Linear shrinkage} = \frac{L_o - L_1}{L_o} \times 100\% \quad 5$$

Where L_o is the original length, and L_1 = the final length

Cold crushing strength test: A refractory brick was used to make a cube measuring 50 cm^3 on a flat surface. The specimen was fired at 900°C (in a furnace). The temperature was maintained for 5 hours. The specimen made from the sample was then put in a compressive tester after cooling to room temperature, and then, by turning the hand wheel, load was applied axially uniformly until failure occurred. The reading on the manometer was taken, and cold crushing strength (CCS) was estimated.

$$\text{Cold Crushing Strength} = \frac{\text{Maximum load}}{\text{cross sectional area}} \quad 6$$

Refractoriness test: Clay samples were made into the shape of simple cones of about 3.81 cm in height and 1.27 cm in base diameter. The specimens were heated at the steady rate of 5°C/min in a blast furnace till the test cone was compared with the standard cones.

Loss on ignition test: Fifty grams (50 g) of clay sample was dried at 110°C in an oven and put in desiccators to cool. A clean, dry crucible with known weight (W_1) was used to contain the clay, and the new weight was determined (W_2). The crucible that contained the sample was put in a muffle furnace and heated to 900°C for 3 hrs. It

was cooled and then weighed (W_3) and the loss on ignition was calculated using:

$$\text{Loss on ignition (\%)} = \frac{(W_2 - W_1)}{(W_2 - W_3)} \times 100\% \quad 7$$

Where the weight of crucible (g) = W_1 , the weight of the crucible + sample = W_2 , the weight of the crucible + sample after ignition at 1200°C = W_3

Relative plasticity determination: The vernier caliper was used to determine the original height (H_o) of the test pieces. A test piece was deformed with a manual plastometer, and the new height, (H_i) was recorded. The relative plasticity was then calculated using

$$\text{Plasticity} = \frac{H_o}{H_i} \quad 8$$

where H_o = original height, H_i = deformation height

Determination of chemical characteristics of clay samples

The X-ray fluorescence analyzer was used for the determination of the chemical composition of clay samples. The parameters determined include Cr_2O_3 , Mn_2O_3 , Fe_2O_3 , Na_2O , MgO , P_2O_5 , SrO ,

SO_3 , Cl , K_2O , CaO , Al_2O_3 , TiO_2 , SiO_2 , ZnO .. The oil in the sample was removed by rinsing with bleach, and carbonate was dissolved by immersing the sample in acetic acid. The clay sample was centrifuged. Then the sample was glycolated and scanned using XRF.

Thermogravimetric analysis (TGA)

The thermal stability of clay was determined using TGA with model number TGA 4000, Perkin Elmer. 11 mg of the clay sample was measured and put into the sample holder. The heating temperature was from 300°C to 700°C (at a heating rate of 10 minutes). Nitrogen gas flowed at the rate of 20 ml/minute at 3 bars. The TGA curve and generation of data were run with Pyris manager software.

Results and Discussion

Results of physical characterization of clay

The results obtained from the clay samples analysis for the four locations (Ezangbo, Abakaliki, Ikwo and Abaomege) in Ebonyi State are presented in Table 1.

Table 1: Physical characterization of clay sample compared with standard for industrial applications

Locations/parameter	Ezamgbo	Abakaliki	Ikwo	Abaomege	Standard		
					Ceramics	Refractory brick	Fire clay
Making moisture (%)	23.40±0.21	25.10±0.50	27.36±0.18	22.63±0.54	-	-	-
Apparent porosity (%)	28.97±0.42	29.36±0.43	25.73±0.46	31.26±0.49	10 – 30	10 – 30	20 – 30
Linear shrinkage (%)	2.60±0.35	6.60±0.42	8.00±1.09	4.00±0.30	-	-	4 - 10
Cold crushing strength (kN/m ²)	1530	1555	1440	14 90	15000	15000	15000
Apparent density (g/cm ³)	2.60± 0.27	2.57±0.25	2.59± 0.21	1.95 ±0.19	-	-	-
Bulk density (g/cm ³)	1.84 ±0.40	1.82 ±0.25	1.74 ±0.26	1.34±0.30	2.30	2.30	2.30
Plasticity (%)	46.00 ± 1.35	25.10 ± 1.4	27.40 ±2.00	22.60 ±1.80	-	-	-
Thermal shock resistance (cycles)					20 - 30	1430 - 1717	20 - 30
Loss on ignition (%)	8.80 ± 0.31	10.20 ±0.15	9.40 ± 0.25	12.60 ±0.31	-	-	-

Making moisture: The mean results of percentage of moisture making ability of clay from the four locations sampled are presented in Table 1. The moisture content ranged from 22.63% at Abaomege to 27.36% at Ikwo. The water present in clay determines the plasticity and strength. Making Moisture is the moisture content corresponding to the plastic unit of clay according to [10]. The high moisture contents of the four samples are good for moulding.

Apparent porosity: The results of the apparent porosity of clay ranged from 25.73% at Ikwo to 31.26% at Abaomege (Table 1 and figure 2). The high percentage porosity at 900°C may discourage entrapment of gases, which can reduce cracking of products and also reduce pin-holing when used for

glaze materials. The high porosity means high thermal shock and can be used for refractory applications. This indicates that clay from Ebonyi State can be good for thermal insulation applications. The results range can also be good for ceramics manufacturing since the standard clay range for ceramic production is 10-30%, according to [11].

Apparent density and bulk density: The results of apparent density of clay samples from the four locations, as shown in Table 1 ranged from 1.95gcm⁻³ at Abaomege to 2.60gcm⁻³ at Ezamgbo. Apparent density is the mass divided by the apparent volume. Apparent density of clay at Abaomege (1.95 gcm⁻³) falls below standard for ceramics production while others are within the range of internationally accepted standard (2.3 – 3.5 g/cm³). Bulk density results ranged from 1.34gcm⁻³ at Abaomege to 1.84

gcm^{-3} at Ezamgbo (Table 1). All the results of bulk density except Abaomege (1.34 gcm^{-3}) fall within the internationally acceptable range of about $1.7 - 2.1 \text{ gcm}^{-3}$ for building and fireclays [12]. The high bulk density was probably due to increase in compaction and closure of pore spaces. With high temperature, compression of particles occurs since it can lead to the eviction of entrapped organic gases & liquids thereby creating pores.

Linear shrinkage: The linear shrinkage ranged from 2.60% at Ezamgbo to 8.00% at Ikwo. Only the result of the sample obtained from Ikwo falls within the internationally accepted range (7-10%) of linear shrinkage for refractory clay [13]. The low shrinkage value suggested that the muscovite content of the clay was high since it is a non-expansion material due to the silicon-oxygen (Si-O) bond. Results of samples from Abaomege and Abakaliki (Figure 5) are good for fireclay. The recommended range for fireclay was 4.00% to 10.00% [14].

Cold crushing strength (CCS): The results of cold crushing strength of clay for the four locations ranged from 1440 KN/M^2 at Ikwo to 1490 KN/M^2 at Abaomege. The results revealed that two locations (Ikwo and Abaomege) are below standards

(1500 KN/M^2) for refractory materials. The clay will be below the standard for thermal insulation, refractory, electrical, and other high-temperatures ceramics applications. Research has shown that CCS of materials is high; the resistance to clay attack will be high [15].

Loss on ignition: The loss on ignition of clay, as shown in Table 1, ranged from 8.80% at Ezamgbo to 12.60% at Abaomege. The loss on ignition was observed to be due to dehydroxylation reactions resulting in loss of water vapour in the samples. Decomposition of carbonates into carbon dioxide and other oxides causes loss on ignition. An increase in the loss on ignition can also be as a result of burning of the organic matter in the clay [16]. The results of LOI of samples from the four locations fall within the acceptable standard for the manufacturing of ceramics, refractory bricks (8-18%), ($>8.18\%$) and high melting clays (5-14%) according to [17].

Results of chemical characterisation of clay

The results of the determination of percentage composition of the chemical parameters (P_2O_5 , SO_3 , Cl, K_2O , Na_2O , MgO , Al_2O_3 , SiO_2 , CaO , Fe_2O_3 , ZnO , SrO , TiO_2 , Cr_2O_3 , Mn_2O_3 ,) in clay samples with X-ray fluorescence spectra are shown in Table 2.

Table 2: Results of chemical characteristics of clay samples

Locations /parameters	Ezamgbo	Abakaliki	Ikwo	Abaomege	Standard	
					ceramics	Refractory brick
Na ₂ O	0.067	0.042	0.048	0.036	-	-
MgO	0.232	0.331	0.486	0.347	-	-
Al ₂ O ₃	18.493	21.393	22.584	20.684	26 - 50	25 – 44
SiO ₂	69.243	66.243	62.293	64.607	60-	51 – 73
P ₂ O ₅	0.438	0.478	0.349	0.349	-	-
SO ₃	0.754	0.764	0.367	0.387	-	-
Cl	0.042	0.042	0.000	0.000	-	-
K ₂ O	0.032	0.032	0.395	0.495	-	-
CaO	0.018	0.008	0.061	0.071	0.18 -3.0	0.10 – 20
TiO ₂	0.033	0.023	2.948	2.540	-	-
Cr ₂ O ₃	0.002	0.002	0.033	0.033	-	-
Mn ₂ O ₃	0.003	0.001	0.007	0.007	-	-
Fe ₂ O ₃	5.491	5.841	6.294	7.394	0.5 – 1.2	0.50 – 2.4
ZnO	5.000	4.900	4.006	3.006	-	-
SrO	0.004	0.004	0.027	0.027	-	-

Chemical characteristics of clay samples: The results for the chemical analysis of clay samples from four locations in Ebonyi States as shown in Table 2 revealed that Silica (SiO₂) and Alumina (Al₂O₃) form the major composition of the clay. It also has trace amounts of other minerals such as MgO, CaO, TiO₂, Fe₂O₃, ZnO etc. The results of silica (SiO₂) content ranged from 62.293% at Ikwo to 69.243% at Ezamgbo, 64.607% at Abaomege and 66.243% at Abakaliki. The high Silica content of the samples satisfies the requirements for the production of refractory bricks of ceramic origin (>51.7%), (>60.5%) and high-melting clays (53-70%) according to Chester (1973). However, the level of Silica was higher for paint (45.3 - 47.9%) and paper (45.0 - 45.8%) but lower than requirement for glass (80-90%) according to research by [18].

Silica can exist as compounds and clay in its free state (SiO₂). Due to the presence of silica in a mixture containing different elements such as Al₂O₃ forming kaolinite (Al₂(Si₂O₅)(OH)₄) etc according to Johari et al. (2001). The high content of alumina and silica is high in the clay samples indicates that they are kaolinite clay, which is often used for the manufacturing of floor ceramic tiles (Nwajuagu and Aneke, 2001).

Alumina: The alumina content of the sampled locations is presented in Table 2. Sample collected at Ikwo recorded the highest amount of % Al₂O₃ (22.58), followed by Abakaliki (21.39%), Abaomege (20.68%) and Ezamgbo (18.193%). The alumina compositions of the four sampled locations however not within the accepted level for production of paper (33.5-

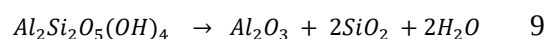
36.1%), refractory bricks (25-44%), ceramics (>26.5%), and production of paint (37.9-38.4%) but are good for glass (12-17%) and high melting clays (16-29%) according to [17]. The percentage composition of alumina in clay was a strong indicator for its refractoriness. The low percentage of alumina indicates that clay samples from four locations are likely to have moderate or low refractory properties.

Iron oxide: The iron content (Fe_2O_3) of the clay from the sampled locations ranged from 5.491% at Ezamgbo to 7.394% at Abomege. The high contents of iron in all the locations are above the standard required for glass (2-3%), refractory bricks (0.5-2.4%), and ceramics (0.5-1.2%) according to [6].

Alkali oxides: The results of alkali oxides (CaO , K_2O , and Na_2O) as shown in Table 2 indicated that they are present in low amount [16]. They (alkali oxide) usually react with the oxides of alumina and silica during firing to form eutectics and then reduce the temperature for vitrification and refractories of the clay [20]. Refractory materials retain their chemical and physical structure when heated to a high temperature. Research has shown that bentonites, ball clay, kaolin etc., are refractory materials, and the

presence of alkali oxides enhances plasticity characteristics of clay [21].

Thermogravimetric analysis of clay: Thermal degradation of clay samples from Ebonyi was studied using thermogravimetric analysis (TGA). Figure 2 shows the two weight loss regimes of the TGA. The first weight loss occurred at 26.75°C and 180.06°C which accounts for the weight loss of about 1.94%. The temperature (367.12°C) represents at which the sample of kaolin started changing into a mineral called kaolinite. According to [22], this process occurred slowly due to impurities in the sample in the form of mica and quartz (Table 2). Research has shown that the mass water loss through stoichiometric dehydroxylation of kaolin shows was calculated to be around 25%; this can be shown in equation 10.



There was no complete loss of hydrate water, which is in accordance to the work reported by Oluremu (2015). Complete loss of structural water normally occurs at high temperature ($600\text{--}750^\circ\text{C}$). At 420°C , the dehydroxylation of kaolinite took place. At 600°C , the high percentage content of kaolinite was stable.

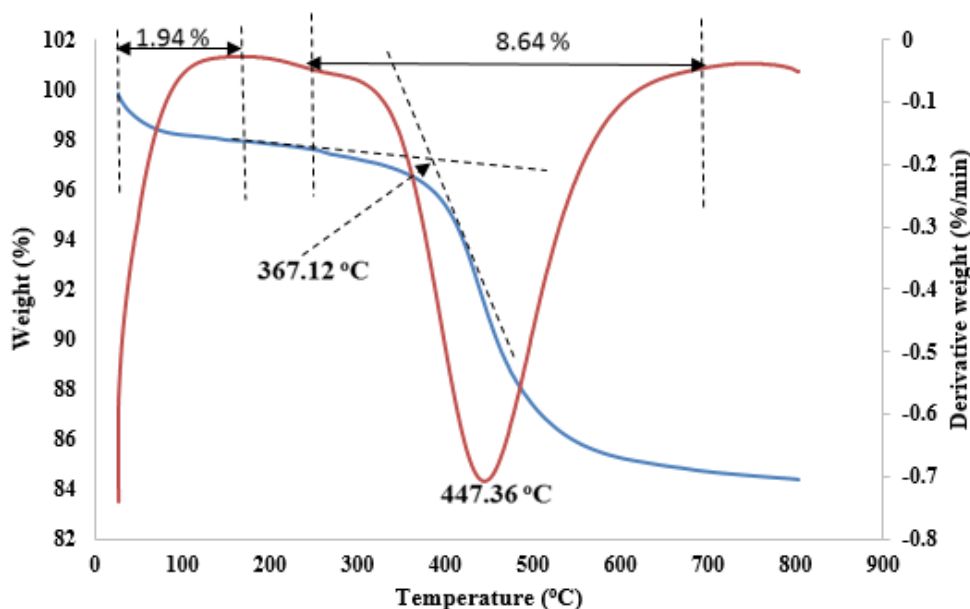


Figure 2: TGA of clay sample from Ebonyi State

Conclusion

The research revealed that clay sampled from locations contains three major minerals: Kaolinite, montmorillonite, and Quartz. The applications of clay samples from these locations are limited to only low-temperature ceramics, earthenware, and glazes because of the amount of silica and impurities.

The clay samples were suitable for porcelain, thermal insulation, electrical insulation etc., based on their fired behaviour but can be used for ceramics production after some beneficiation processes. These potentials are good and can encourage the exploration of this Ebonyi clay deposits for applications in the industries by the Government of Ebonyi State or private companies.

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Declaration of Interests

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

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