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Research Article

Characterization of Some Local Clay Minerals and Fabrication into Ceramic Floor-Tiles

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Abstract

Clay minerals deposits, which include, kaolin, ball-clay, fireclay and zircon, were investigated and used in fabrication of ceramic floor tiles. X-ray diffractometer (XRD-6100, Japan), was used to examine the various compositions and structures of the clay samples. 2000 grams of 0.005 mm grain size of each sample were homogeneously mixed into dough, compressed in metallic mould of 12 mm × 36 mm dimension, and allowed to dry for about 5 hours. Kiln draught oven, at 1200 ℃ was set for glazing and firing of the moulded tiles, to ensure glossy appearance, smoothness and improved strength. XRD test showed higher content of alumina (Al_2O_3) and Silicon (iv) oxide (SiO_2) in all the clay samples. The percentage composition of Critobalite, Diphosphorus trioxide (P_2O_3), Potassium Oxide (K_2O) and Sodium Oxide (Na_2O), were reasons for their excellent workability, improved mechanical and rheological properties of ceramics floor-tiles. Water absorption, chemical resistance, shrinkage properties of the different ceramic tiles were compare to some commercial products. This investigation had shown that locally available clay minerals could be fully integrated into ceramic industries to reduce cost of ceramic importation and create job opportunities for youths.

Keywords

Ceramics, Clay Deposits, Floor-Tiles, Properties, Compositions

1. Introduction

Ceramics, which include materials from silicates and their derivatives may be of inorganic, organic or non-metallic compounds, nitrides or carbides materials. They consist basically of elements, such as carbon, oxygen and silicon. Ceramic materials could be used in a wide area of life, ranging from domestic wares, automobile parts, dentistry, etc. They are also used in military aircraft, sensors, rocket nozzles, weapons, missile guidance parts, etc.

Recently, [6] scientists have discovered the unique properties of ceramics in relation to their ease of production, low material cost, durability, beauty, availability of raw materials, fire resistance, and non-toxicity, bulk density, water absorption, and flexural strength, according to [5, 7]. The components of ceramics are chemically held together by either covalent or ionic bonds. The weak bonding power could cause ceramics to brittle when they are under an increased stress or impact. It had been observed that the composition and degree of homogeneity of ceramic can affect their mechanical prop-

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erties, cross-sectional features and surface attractiveness. Reports had shown, that ceramic materials are one of the causes of environmental wastes, because of their non-degradable properties, [19]. They discovered that incorporation of nano-particle size of waste ceramic dusts into soil used for highway construction, improved its stability, sustainability, thickness, more economical, thereby, reducing the ceramic waste menace to the surroundings. Studies has shown that waste ceramic materials could be processed or recycled to reduce their wastes from the environment, when they are no longer in use [20].

However, the commonest challenge in ceramics production and utilization is their extreme brittleness and crack. This properties had been reported to be improved by adjusting their designs and composition. Other nano-materials, such as carbon-nano tubes and graphene, which are allotropes of carbon, could be incorporated to ceramic matrices to improve their thermal resistance, electrical conductivity and strength [7, 21].

Innovations in ceramic technology had resulted into a wider application of ceramics in building constructions, electrical insulators, heat conductors, spark plugs, artificial bone joints, fiber optics, body amours, brake plates, etc, according [5]. Research reports had also shown that, silica ceramic cores are very important part in the manufacture of hollow blades, due to their excellent chemical stability and moderate high-temperature mechanical properties, [16]. They fabricated ceramics-tiles and used transmission electron microscopic analysis to discover the microstructure of carbon atoms in the tiles. The properties of ceramics generally depend on their grain-size, shape, density, hardness, mechanical strength and optical properties, etc. Ceramics could structurally be poly-crystalline, which are made up of multiple grain crystals joined together during processing. They could be mono-crystalline, which are made up of single three-dimensional crystal. Clay could also be mixed with plastics to form polymer composites, in order to improve their fracture toughness, chemical inertness, moisture resistance, hardness properties, [12, 13]. The role of ceramics tiles in kitchens, swimming pools, flooring, walls, etc had really ensured beauty, attraction, ease of cleaning, and reduction of growth of microorganism on surfaces, according to [16]. We have observed with great disappointment that most ceramic materials used in our localities are imported, not minding the huge deposit of various types of clays suitable for any type of ceramic materials.

Our target therefore, is to source and characterize some unutilized clay samples, fabricate them into ceramic tiles. Ceramic materials having wide applications in a varieties of life, there will not be a limit into investigating other available and sustainable raw materials for its fabrication.

2. Materials and Methods

2.1. Clay Sample Collections

The samples of clay used in this research were gotten from different locations as listed below:

(1) Fire clay:

This is a hydrous silicates of aluminum ($Al_2O_3.2SiO_2.2H_2O$), commonly referred to as refractory clay, was gotten from Obollo-afor, Nsukka, at Enugu State.

(2) Borehole (Ball Clay):

A mineral plastic clay with very fine grain was gotten from bore-hole mining site at Rock Land, by Ngozika Housing Estate, Awka, Anambra State.

(3) Zircon or Zirconium Silicate ($ZrSiO_4$):

A nesosilicates (orthosilicate, SiO_4) was gotten from Lafia, Nasarawa State.

(4) Kaolin or China Clay ($Al_2Si_2O_5$ $(OH)_4$)

It is the hydrated aluminum Silicate derived from the mineral kaolinite, and was gotten from Amagu, Nsugbe in Anambra State.

2.2. Equipment/Apparatus

Some the equipment and apparatus used includes: Ohaus Weighing balance with high sensitivity; a metallic sieve of 0.005 mesh, for uniform grain size of the clay. A Flat wooden board was used for mixing. 12 mm × 36 mm metallic mould was used for formation of specific dimension of the floor tiles. Stirrer, a device used for mixing the clay to a homogeneous dough. Filter, was used to separate the suspended particulate materials present in the hydrated clay and water. Hammer was used to break hard lumps of clay to increase the surface area.

Kiln draught: Was used for glazing the fabricated clay tiles and bricks to ensure glossy appearance, smoothness and improved strength.

X-ray diffractometer (XRD-6100, Japan): was used to examine the composition, and structure of the different clay samples.

2.3. Preparation of Sample Floor-Tiles

The floor tiles from the different clay samples were fabricated as shown below:

i. Borehole or Ball Clay Floor Tiles:

1500 g of bore-hole clay was mixed with 2000 cm^3 of water to form a dough texture. This was kneaded on the flat wooden board to a smooth texture, stuffed, compressed and pounded in the metallic mould, flattened using a slab roller. This was left to dry in a room temperature to reduce the water content. Glazing was carried out before subjecting the sample to firing at 1200 ℃ (degree - Celsius), in the kiln draught.

ii. Zircon or Zirconium Silicate ($ZrSiO_4$), Ceramic Floor

Tiles

This sample was prepared, glazed as in (i) above, glazed, fired and kept for further tests. The x-ray diffraction of the prepared tiles was carried out at different phases before heat treatment and to identify the composition of ($ZrSiO_4$).

iii. Preparation of Kaolin Floor Tiles

Sample of kaolin tiles was prepared using the process in (i) above.

2.4. Quality Control Tests on the Ceramic Floor-Tiles

i. Chemical Resistant Test (Indian Standard Testing 13830(10): 2006)

Soap solution was prepared using 1mol of sodium hydroxide and oleic acid at the proportion of 2.00 to 18.00 ml respectively. The weighed ceramic samples (glazed and unglazed) were immersed in the soap solution and allowed for 28 days to determine the chemical attack on their surfaces visually, at the interval of 7 days.

ii. Water Absorption Test

A piece of different ceramic research samples were cut, weighed and immersed in cold and boiling water inside a water-bath. The samples were reweighed after 2 hrs. and the increase in weight of the samples were expressed as a percentage weight increase. The water absorption was calculated accordingly.

iii. Action of Household Cleaning Solution Test

The household cleaning solution was prepared by mixing 100 ml of ammonium chloride {$NHCl_4$}; 3 g of sodium perorate $[Na^+]_2[B_2O_4(OH)_4]^{2-}$.; 1g sodium carbonate {$Na_2CO_3$}; 1.5 g sodium silicate (Na_2SiO_3); 2000 ml distilled water. A weighed sample the ceramics was immersed in a bowl containing the solution and allowed to stay for 48 hours. The color was compared to the original ceramic sample.

iv. Test for shrinkage according to [3]

This measurement was carried out to determine the water-loss, physical and chemical changes that can occur during drying or firing of the ceramics tiles. A definite size of the clays were cut and flattened, making sure the thickness were within 1.5 mm. A strip of 12 cm long was tied and a scratch line was made to the centre to identify the length. The temperature was set between 68 – 70 ℃. The reduction in length of the samples were recorded at interval of 2 hrs. The initial length were recorded. The samples were later placed in a drying oven at 100 – 110 ℃ and left for 24 hrs. After this the percentage shrinkage was calculated according to the formula:

$$Sd = \frac{Lp - Ld}{Lp} \times 100$$

Where: Sd=Linear drying shrinking (%); Lp=Length of test sample (cm); Ld=dry length of test sample (cm)

v. POROSITY

The percentage porosity of the ceramic tiles were determined by gravitational method. Known size and mass of ceramic samples were cut, immersed in a container of water, and left for 48 hours.

$$\text{Porosity (\%)} = \frac{W1 - W2}{W\,2} \times 100$$

Where: W1=weight before immersion; W2= weight after immersion; porosity= weight gain of ceramic samples.

vi. Test for ceramic scratch hardness

A rectangular size of the different ceramics were cut and placed on a firm support with the glazed surface facing upwards. A sharp object was used to scratch the surface uniformly. This was repeated with a quartz. The scratch was examined with naked eyes to determine the effect.

3. Results and Discussion

The results of the x-ray diffraction (XRD) analysis carried out on the clay samples to determine their compositions and different ceramics samples are stated on Tables 1, 2, and 3.

3.1. Results of Ceramic Tiles from Bore-hole or Ball Clay

It was observed that the borehole or ball clay has a high content of alumina (Al_2O_3) at 64.23 wt.% and Silicon (iv) oxide (SiO_2) at 31.98 wt.% respectively, as shown on Table 1. This XRD results are in agreement with that of [14]. They discovered from their XRD results that the fused silica, crystallizes into critobalite (mineral oxide) due to the presence of Al_2O_3. Aluminum oxide or alumina (Al_2O_3) is very important in ceramics, due to its stability to high temperature, hardness, abrasion, chemical and moisture resistance [17]. They later confirmed that presence of critobalite is the reason for the improved mechanical properties of silica-based ceramics, which is in conformity to our findings. Silicon (iv) oxide (SiO_2) could be used as surface passivation and semiconductors, when about 64.23 wt.% is embedded in the ball clay. This proved that our ceramic products could also be used as a semiconductor for electrical or heating appliances. Ball clay is confirmed as a good raw material in ceramic industries, due to their characteristic high plasticity, high unfired mechanical strength, high rheological properties, controlled residue, and excellent workability, according to [15]. XRD analysis had also provided us some information on the phases, structures, texture, grain sizes, etc, of our ceramic samples and the presence of some atoms or elements which enhanced the binding forces. Ball or borehole clay had been described as plastic clay and can fuse as cement without shrinking, sagging or lose their shapes when used in sanitary- ware.

Researchers have proved that some clays rich in alumina could be used in variety of harsh processing objects, such as in floor or wall-tiles, or objects where dimensional stability, high temperature, chemical, and corrosion resistance are of paramount importance. Our sample ceramics are of high purity and could be used in fabricating chamber materials or interior surfaces, either as plasma etching, ion implant, etc., due to their strong dielectric properties and low heavy metallic content of Fe_2O_3, TiO_2, and ZnO, at 1.12%, 0.21%, and 0.03% respectively, as shown in Table 1.

Table 1. *Borehole (ball clay) result of analysis.*

Compositions	Content (wt.%)
SiO_2	64.23
Al_2O_3	31.98
Fe_2O_3	1.12
K_2O	0.28
Na_2O	0.36
CaO	1.17
MgO	0.26
TiO_2	0.21
MnO	0.35
ZnO	0.03
Raw color	Red

3.2. Results of Floor-Tile from Kaolin (China Clay)

The result of the x-ray diffraction analysis on Kaolin or China clay revealed that the percentage content of silica (SiO_2) and Alumina (Al_2O_3) are 62.29% and 25.40% respectively. Other components discovered are Diphosphorus trioxide (P_2O_3) 1.27%, Potassium Oxide (0.48%%), K_2O (0.48%) and Na_2O (0.046%). Kaolin is also a good raw material in filling and coating of paper materials, to improve their appearance, brightness, smoothness, printability, and opacity, according to [6]. Studies had shown the structural arrangement of kaolin as consisting of an alumina octahedral sheet and a silica tetrahedral sheet that share common plane of oxygen atoms and repeating layers of hydrogen atom bonded together, as the reasons for its resistance to weather and inertness to chemicals, according to [9]. They also concluded that Kaolin has an inert pH level, which makes it suitable for formulation of pharmaceutical and cosmetics storage containers. This property is a good quality for ceramics to be used in storage of food, drinks and drugs.

The presence of kaolin in our clay sample had been suspected as the reason for its fire extinguishing property during firing process, which was 8.99% of the mineral in 100% of water, as stated in Table 2. It simply means that ceramics formed with this clay can resist fire and heat at a high temperature, therefore could protect homes, offices or factories against fire, when used as walls or floors tiles.

Table 2. *Showing Results of Kaolin or White Clay Percentage Composition.*

Compositions	Content (wt.%)
SiO_2	62.29
Al_2O_3	25.40
P_2O_3	1.27
K_2O	0.48
Na_2O	0.046
CaO	-
MgO	0.032
TiO_2	0.83
Raw color	White
Fired color	White
Loss of ignition	8.99

3.3. Results on Ceramic-Tiles from Fire Clay

It was observed that fire clay from Obollo-Afor, has the highest Silicon Oxide and Aluminum Oxide content of 64 and 19.50% respectively, while, calcium oxide (CaO) content is the lowest at 0.07 w.%, as shown on Table 3 below. Other compositions of fire clay are titanium oxide (TiO_2) making of 2.3 wt.%, Sodium oxide with 0.27 wt.%, Iron (iii) oxide, etc. They are referred to as plastic clay and has very high resistance to water and heat. [8] studied on the properties of fire clay as a refractory raw material. They concluded that the clay could be used in bricks for glass-melting furnaces, chimney linings, pottery kilns, blast furnaces, etc, due to its high temperature tolerance. It also has high stress resistance, and is good for floor-tiles in conformation tour findings.

Table 3. *Showing Results on Fire Clay.*

Compositions	Contents (wt.%)
SiO_2	64
Al_2O_3	19.50
Fe_2O_3	1.3
MgO	1.08
CaO	0.07
TiO_2	2.3
Na_2O	0.27
K_2O	2.01

Table 4. *Results of Ceramic tile from Zirconite Clay ($ZrSiO_4$).*

Compositions	Content (wt.%)
SiO_2	61.23
Al_2O_3	23.61
Fe_2O_3	11.79
MgO	0.91
CaO	1.13
TiO_2	1.5
Na_2O	0.16
K_2O	0.54

The results of composition of Zirconite clay as outline on Table 4, showed a high percentage of Silicon oxide at 61.23 wt.%, Alumina at 23.61 wt% Iron (iii) oxide at 11.79 wt.%, but low potassium and Sodium oxides at 0.16 wt.% and 0.54 wt.% respectively. Many investigations had shown that, shrinkages in ceramic materials could be due to the presence of some impurities. [2, 4] investigated on some properties of refractory clay and concluded that, they would perform better if blended with some composite materials, like fibers, wood dust, etc.

Researchers had shown that Zirconite clay is a good example of refractory clay which could be of both natural and synthetic materials, like, silicon carbide, magnetite, alumina, etc. It had been observed that at high temperature of 1150 ℃, $ZrSiO_4$ could be influenced by presence of some fusing impurities, such as, potassium (k), Iron (Fe), Calcium (Ca), Manganese (Mn) and other particles, according to [1]. The hardness, durability and chemical inertness, zircon persists in sedimentary deposits and is a common constituent of most sands.

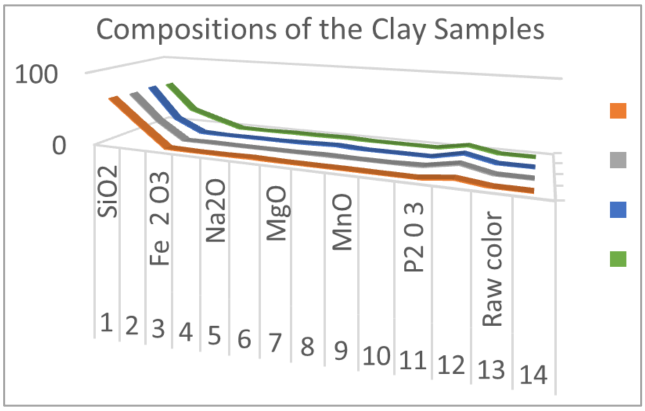

Figure 1. *Comparative composition of the clay samples.*

The various clay samples are composed of higher content of silicon oxide, (SiO_2) Aluminum oxide, (Al_2O_3), and Fe_2O_3 (Iron iii oxide). There is zero content of Manganese oxide and Zinc oxide in Kaolin, Fireclay and Zirconite clay as shown on Figure 1.

3.4. Result of Chemical Resistant of the Ceramic Samples

The result of the chemical resistance showed that the bore-hole (ball clay) ceramics cannot be used in the storage of chemical solutions, such as alkaline or acidic solutions. The glazed surface exhibited colour changes due to some reaction with solutions. Ceramic-tiles from fire clay exhibited resistance property to both the domestic soap solution, alkaline and acid solution, after a period of immersion. There was no visual cracks on the surface of the glazed ceramics, but cracks were observed on the unglazed ceramic tiles.

3.5. Water Absorption Test

The results on both cold (25 ℃) and hot water (100 ℃) are displayed on Figure 2. Water absorption values were higher in hot water than in cold water. It was observed that the ceramic tile from ball clay displayed the highest water uptake both in cold and hot water. According to the regulations of (National standard for water absorption), they stated that water absorption value allowable for wall tiles is within 10-20% for interior wall ceramic tiles; ≤0.5% for floor ceramic tiles, and ≤0.5% for porcelain ceramic tiles. However, ceramic tiles from ball clay is not suitable for bathtubs or bathroom tiles, because, the particles are more porous and can allow water permeability. Ceramic tiles from fire clay exhibited a suitable water absorption and could be used for both interior or out-door surfaces.

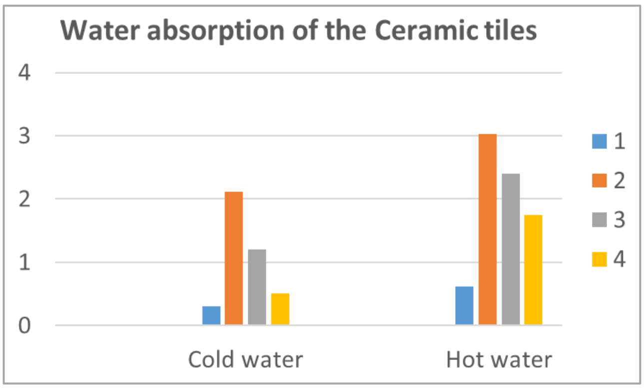

Figure 2. *Water absorption of the Ceramic- tiles.*

3.6. The Result of Free Shrinkage According to ASTM, (2009)

The result of the free shrinkage was considered acceptable because, the values compared well to the standard commercial ceramics. Our research samples free shrinkage values showed higher deformity, probably due to the unprofessional method of production and unfavourable improvised approaches. Good quality ceramics had been proved to exhibit shrinkage value within 6-8%. The majority of shrinkage occurred at bisque firing stage, which was accurately measured score lines on a test tile will indicate the amount of shrinking at these different stages [11]. Studies had shown that high value of shrinkage contributes to easy deformation of ceramic material [18]. Degree of shrinkage had been certified as property to determine the quality of ceramic materials [10]. They observed that ceramic shrinkage property could occur in three stages, the free shrinkage exhibited very little amount of water without crack while, the drying shrinkage is long lasting. Most water molecules are taken off resulting into tensile stress on the ceramic or significant cracking.

4. Conclusion

This investigation on some of the locally available clay deposits, such as borehole clay, kaolin, fireclay and zircon and their fabrication into ceramic materials had proved to a promising approach to minimizing over dependency on foreign raw materials. The results on some the properties of our research sample ceramics shows that fire clay will be suitable for wall tiles both at swimming pools and for interior usage. Ball clay exhibited reduced mechanical property probably due to the particle size of aggregate.

Zirconium silicate has been widely used in ceramic production because of its good chemical stability, so it is not affected by the burning atmosphere of ceramic, and can improve the binding performance of ceramic glaze significantly and improve the hardness of ceramic glaze. Zirconium silicate can also be further applied in the production of colour picture tube, emulsified glass and enamel glaze in the glass industry.

This research work using locally available raw material has great potential in reducing waste of fund in getting foreign materials, providing job opportunities for unemployed youths and reducing the high price of ceramic materials.

Table 5. *Comparative composition of the different clay soil samples investigated.*

S/N	Clay Composition (wt.%)	Borehole (Ball Clay)	Kaolin (white clay)	Fire Clay	Zirconite Clay
1.	SiO_2 (Silicon Oxide)	64.23	62.29	64.00	61.23
2.	Al_2O_3 (Aluminium Oxide)	31.98	25.40	19.50	23.61
3.	Fe_2O_3 (Iron iii Oxide)	1.12	—	1.30	11.79
4.	K_2O (Potassium Oxide)	0.28	0.48	—	0.54
5.	Na_2O (Sodium Oxide)	0.36	0.046	0.27	0.16
6.	CaO (Calcium Oxide)	1.17	—	0.07	1.13
7.	MgO (Magnesium Oxide)	0.26	0.032	1.08	0.91
8.	Ti_2O (Titanium Oxide)	0.21	0.83	2.30	1.50
9.	MnO (Manganese Oxide)	0.35	—	—	—
10.	ZnO	0.03	—	—	—
11.	P_2O_3	—	1.27	—	—
12.	Raw Colour	Red	White	White	Brownish
13.	Fire- colour	Ash	Greyish	White	Greyish
14.	Loss on ignition	5.02	8.31	6.7	7.64

Abbreviations

XRD	X-ray Diffractometer
ASTM	Standard Test Method

Conflicts of Interest

The authors declare no conflicts of interest.

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