The Journal of Engineering Research (TJER), Vol. 16, No. 1 (2019) 53-62

DEVELOPMENT AND EVALUATION OF CERAMIC TILES USING WASTES AND SOLID MINERALS

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ABSTRACT: This paper focuses on the need for domesticating the production of building materials like tiles to address the problem of Nigeria over dependence on imported goods, despite adequate availability of mineral resources like clay, quartz and feldspar in the country. Clay, quartz and feldspar, and milled glass were respectively obtained from Ogijo in Ogun State, Okpila in Edo State and Oshodi, Lagos State of Nigeria were characterized using ASTM C71 as a guide. Samples of the ceramic tile were produced from varying mixtures of clay, quartz, feldspar and milled glass. The chemical and physio-mechanical properties of the samples were also determined. The results showed that the clay sample belong to Alumino-silicate group with 59.20% silica and 21.25% alumina. The samples' properties met the required standards; hence, the materials were found suitable for production of ceramic tiles of acceptable standards. Though, the porosity of the sample tiles increased as the percentage weight of the clay material in the mixture decreased the proportion of quartz, feldspar and milled glass increased. The sample containing 70% Clay and 30% Feldspar had better formulation properties than others.

Keywords: Apparent porosity; Bulk density; Ceramic; Tile; Milled glass; and Solid minerals.

انتاج وتقييم البلاط السيراميك الفخاري باستخدام النفايات والمعادن الصلبة

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الملخص: تركز الدراسة على الحاجة إلى إنتاج مواد البناء مثل البلاط محليا لحل مشكلة اعتماد نيجيريا الكبير على السلع المستوردة رغم توافر الموارد المعدنية الكافية مثل الطين والكوارتز وسيليكات الالومنيوم في البلاد. وقد حصلنا على الطين والكوارتز و سيليكات الالومنيوم والزجاج المطحون على التوالي منمنطقة أوجيو في ولاية اوغن و منطقة اوكيلا في ولاية ايدو ومنطقة اوشودي في ولاية لاغوس في نيجيريا باستخدام استيم 7 سي كدليل. و قمنا بإنتاج عينات من بلاط السيراميك من خليط من الطين و الكوارتز و سيليكات الالومنيوم والزجاج المطحون. كما قمنا بتحديد الخصائص الكيميائية والفيزيائية الميكانيكية للعينات. وقد أظهرت النتائج أن عينة الطين تنتمي إلى مجموعة ألومينو سيليكات بنسبة 59.20 سيليكا و 21.25٪ ألومينا. وقد استوفت خصائص العينات المعايير المطلوبة ؛ وبالتالي ، وجدنا ان المواد مناسبة لإنتاج بلاط السيراميك بالمعايير بالمقبولة . وعلى الرغم من ذلك ، فإن مسامية عينة البلاطات قد ازدادت مع انخفاض النسبة المئوية لوزن المادة الطينية في الخلطة المختلطة وزادت نسبة الكوارتز و سيليكات الالومنيوم والزجاج المطحون. وكان للعينة التي تحتوي على 70 ٪ من الطين و 30 ٪ من سيليكات الالومنيوم خصائص تكوين أفضل من غيرها.

الكلمات المفتاحية: المسامية الظاهرة ؛ الكثافة الرئيسة ؛ السيراميك ؛ البلاط؛ الزجاج المطحون ؛ المعادن الصلبة.

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DOI: 10.24200/tjer.vol16iss1pp53-62

1. INTRODUCTION

In Nigeria, natural mineral resources, such as clay, feldspar, quartz are available in large quantities Shuaib-Babata and Mudiare (2017), but the country is still highly being challenged with economic problems. Some studies have attributed the present Nigeria economic problem to over-dependence on foreign goods, oil and gas and to negligence of other sectors (Chete *et al*. 2017; Projectsxtra 2017; Shuaib-Babata *et al*. 2017a).

It is claimed that an identified veritable tool towards any nation development is Industrialization (Shuaib-Babata *et al*. 2017b; Shuaib-Babata *et al*. 2017c). Availability of mineral resources in Nigeria has been the backbone of the nation's economic and industrial development aspirations Foraminifera (2017). Presently, there are several calls for diversification of Nigeria's economy and significant reduction of its overdependence on oil and gas to come out permanently of the present economic recession Foraminifera (2017).

Production capacity from locally available raw materials used in development of technology in the transformation of the raw materials to finished products is a significant factor towards a successful industrialization (Shuaib-Babata *et al*. 2017a, Shuaib-Babata *et al*. 2017b). The identified bases for the development of any industrial sector include access to raw materials, labor force, funds and technology Hughes (2008). Effective industrialization could be significant to reduce overdependence of a nation on foreign goods and enhance export base Shuaib-Babata *et al*. (2017a).

Worldwide, production and consumption of ceramic tiles has recently increased all over the world as result of rapid development of real estate industry WIFI Ceramics (2017). The tiles used in the estate development are of different quality; it ranges from high to middle and low quality, which is imported to Nigeria, especially from China. With the present call for economic diversification in Nigeria, the need for local production of ceramic tiles with good quality is highly essential. This will be of help to proffer a solution to the problem and sustain the country economy through industrialization.

There have been numerous attempts to utilize either industrial or agricultural wastes in the manufacture of wall and floor ceramic tiles in the past last two decades (Elakhame *et. al*. 2016; Cesar *et al*. 2011; Juan, *et al*. 2010). Industrial wastes are considered to be an important part of the materials used. Cement kiln dust (CKD) as a great trouble maker waste, was utilized in ceramic tile bodies by Youssef (2002), to replace half the feldspar required for ceramic wall tile body with a double target of cost reduction and environment protection. In that study, verification parameters of the fired samples was measured and the water absorption was within both ISO and Egyptian standards limits of wall tiles. CKD in the mixture makes it less dense, more porous and lowers the compressive strength (Safiuddin *et al*. 2010; Yoshizawa *et al.* 2004; Central Pollution Control Board (CPCB), 2000; Torgal and Jalali 2010).

For better quality tiles, the basic raw materials employed for the production of ceramic tiles include quartz and feldspar (which serve as a source of silica and alumina to give the tiles the desired strength) and clay from a good source to serve as binder. In this study, milled glass and aluminum were considered as a primary source of alumina and silica.

This study aims at the development of standard composite ceramic tiles-based (CCT) using available mineral resources and wastes to aid local production of building materials in Nigeria. It also focuses on the use of cheaper and durable materials to fabricate the CCT to enhance its properties by variation of fillers to identify the best mechanical and physical properties of the produced tiles. In the end, this should assist in solving the problem of unemployment among youths. It should also help in diversification of economy and solve the problem of over dependence on the oil and gas sectors, which had led to economic recession in the country.

2. MATERIALS AND METHODS

2.1 Materials

In this study, the major materials used include clay (CY), quartz (QZ), Feldspar (FR) and milled glass (MG). The clay material and the milled glass bottle was sourced from Ogijo-Shagamu Area, Ogun State and Oshodi, Lagos Nigeria respectively. The quartz and feldspar were obtained at different deposits in Okpila, Edo State, Nigeria. Samples of the raw materials for the production of ceramic tiles in solid forms are shown in Fig 1.

*2.2 **Methods***

*2.2.1 **Chemical Analysis***

The chemical compositions of the representative samples of the clay, quartz, feldspar and milled glass were determined at Engineering Materials Development Institute (EMDI), Akure, Nigeria using Atomic Absorption Spectrometer (AAS) UNICAM 929. This is in line with the data reported by (Ryan 1978; Yoshizawa *et al*. 2004; and Safiuddin, *et al*. 2010).

Clay Feldspar

Quartz Glass Bottles

Figure 1. Samples of the raw materials used for the production of ceramic tiles.

2.2.2 ***Preparation of Materials***

2.2.2.1 ***Clay Materials***

To remove debris and other unwanted materials in the clay sample, the clay was soaked in water for five (5) days, sieved with a mesh of 350μm and dried at room temperature for one week (7 days). Subsequently, the dried sample was milled and sieved into 100 μm sizes in line with the practice of Elakhame *et al.* (2016b).

2.2.2.2 ***Quartz, Feldspar and Milled Bottle***.

The quartz and feldspar and the milled glass bottle (additive materials) were crushed using a Hammer Milling Machine (Model; 000T, PUISSANE; 1.5KV, S/N; 13634) and milled into fine size particles using ball-milling machine (Model; 87002…. Limoges-France, Type; A50---43), and then sieved with a Vibro-Sieve (Model; Fritsch GmbH, D-55743.1 Dar-Oberstein Germany) into 100 μm particles size.

2.3.2 ***Raw Materials Mixtures***

In forming the raw materials mixtures for the production of ceramic tiles, milled bottle, quartz and felspar were added to the clay materials at different proportion as presented in Table 1 (a & b).

Table 1a. Proportion of clay and other materials in forming the raw mixes for the production of ceramic tiles (Formulation A – C).

Samples	Sample Formulation A		Sample Formation B		Sample Formation C	
	CY (%)	**QZ (%)**	**CY (%)**	**FR (%)**	**CY (%)**	**MG (%)**
1	100	0	100	0	100	0
2	80	20	80	20	80	20
3	70	30	70	30	70	30
4	60	40	60	40	60	40
5	50	50	50	50	50	50
6	40	60	40	60	40	60
7	30	70	30	70	30	70
8	20	80	20	80	20	80

Table 1b. Proportion of clay and other materials in forming the raw mixes for the production of ceramic tiles (Formulation D.

FORMULATION SAMPLE D			
CY (%)	**QZ (%)**	**FR (%)**	**MG (%)**
50	0	40	10
50	10	30	10
50	40	0	10
50	5	5	40
0	35	35	30

Note: Clay = CY, Quartz = QZ, Feldspar = FR, and Milled glass = MG

2.3.3 ***Tiles Production***

In development of ceramic tile samples, various raw materials mixes were processed sequentially using the process technology flow chart presented in Fig. 2.

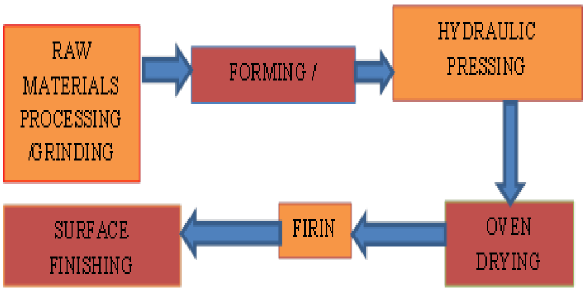

Figure 2. Process technology flow chart.

The milled powder clay, quartz, feldspar and glass bottles were individually measured appropriately as indicated in Table 1 using a 0.001mg sensitivity weighing scale. The samples were sieved with 100 µm mesh and mixed to homogeneity. Subsequently, the mixed material (granulated powder mixture) was poured into a prepared mould, 5cm × 5cm size (Fig. 3) to form shaped test specimens and then uniaxially compacted using hydraulic press (Weber- Hydraulic, AC-8800 Vibrorg, Denmark. Type; p-16H, Capacity; 16T, S/N; 29580) under a pressure of 50 MPa, in accordance with guidelines in mould Pressed Ceramic Tiles - Specification (First Revision of IS 15622) ICS91.100.23**.** The pressed specimens were held overnight and then dried at 90 - 100°C for 48 hours in an oven.

Dried specimens were fired in a laboratory type electric furnace (Nabertherm, more than heat 30-3000°C) at the rate of 5°C/ min. The technological parameters values were measured after firing steps. The firing procedure used involves heating the sample at a temperature of 1170°C, then soaked for 3 hours and naturally cooled Correlia *et al*. (2004). The procedures were repeated for all the formulations (A-

D). The produced samples of the ceramic tile specimens produced are shown in Fig. 4.

2.4 Determination of the Specimens' Properties

Some properties of the material mixes (moulded specimens) were tested for proper evaluation of their suitability. The properties include fired shrinkage, apparent porosity, bulk density, porosity and compressive strength, chemical composition and microstructural test. For the products to be in compliance with National and International Standards, these properties were evaluated using various national and international test procedures and standards as presented in Table 2.

2.4.1 Porosity of the Brick Sample

Porosity of the brick sample was determined using ASTM JISD4418-1996 guidelines by soaking the bricks in a bath of water at 100⁰C for 8 hours. The mass of each of the samples before and after soaking was determined (in mg) and recorded. The porosity of the samples were then determined using equation 1.

Figure 3a. The Mould.

Figure 3b. Samples of ceramic tile specimens.

Table 2. Various National and International test procedures and Standards used for evaluation of the samples.

S/N	Test procedures	Standards
1	Specific gravity	MS 474: Part 1: 2003
2	Compressive strain test method	MS ISO 6310:2003, Part 4
3	Determination of modulus of rupture and breaking strength	MS ISO (Part 6): 2006 (first revision)
4	Shear test procedure	MS ISO 6312:2003, Part 6
5	Determination of thermal Conductivity	ISO 7882: 1986
6	Porosity & Apparent porosity measurement	JIS D 4418 – 1996
7	Determination of linear thermal expansion	(Part 4): 2006 (first revision)
9	Determination of water absorption and bulk density.	(Part 2): 2006 (first revision)

DOC NO. CED 5(7888), Title: Pressed Ceramic Tiles – Specification (First Revision of IS 15622) ICS91.100.23.

$$\text{Porosity (p)} = \left(\frac{M_1 - M_2}{\rho V}\right) \times 100 \qquad (1)$$

Where M1 is the mass (g) of brick before soaking, M2 is the mass (g) of brick after soaking water, ρ is the density of the liquid and V is the volume of sample (cm^3).

2.4.2 Apparent Porosity and Bulk Density of the Brick Samples

The apparent porosity of each of the brick samples was also determined in accordance with JIS D 4418 – 1996. Brick samples (5 × 5 cm^2) dimension were used. The samples' dried weight in air (W_{sw}) and the saturated (W_{sa}) of each sample were determined. The apparent porosity (P_a) and bulk density (ρ_b) of each brick was determined using the relationships in equation 2 and 3 respectively.

$$\text{Apparent porosity } (P_a) = \frac{W_{sa} - W_{da}}{W_{sa} - W_{sw}} \times 100 \qquad (2)$$

$$\text{Bulk density } (\rho_b) = \frac{W_{da}}{W_{sa} - W_{sw}} \qquad (3)$$

2.4.3 The Compressive Property

The compressive property test was conducted on the brick samples using Testometeric Universal Testing Machine [TUF – C- 1000 KN (SI)] as specified by the MS ISO (part 6):2006 standard. Brick samples with diameter of 5 × 5 cm^2 were loaded gradually in compression until the brick failed to offer further resistance to deformation which was indicated by the bricks fracture. Various properties (stress, strain, extension and energy at different stages) of the samples were recorded from the display unit (monitor) attached to the machine.

2.4.4 Measurement of Fired Shrinkage

The shrinkage properties of the bricks were determined by measuring both the green and fired dimensions of the 5 × 5 cm^2 brick samples, using Length Comparator with Digital Indicator in accordance with the guidelines in ASTM C227 / and as specified by the ISO 10545-2 standard. Both sides of the specimens were measured and the average linear shrinkage was calculated. The linear shrinkage of each side was calculated as a percentage of the original green dimension as expressed in equation 4.

$$\text{Linear Shrinkage (Ls)} = \frac{L_g - L_f}{L_g} \times 100 \qquad (4)$$

where L_g and L_f are green and fired length of bricks respectively.

3. RESULTS AND DISCUSSION

3.1 Materials Chemical Composition

The results of the chemical analysis in Table 3 show the major constituents of the clays, quartz and feldspar.

3.2 Porosity and Bulk Density

The porosity and bulk density values of the various produced moulded ceramic tiles samples are as shown in Fig. 4 and 5 respectively. The specimens' average porosity measurements ranged between 0.1 and 2.7%.

It is shown in the results (Table 2) that 80% and the above constituents of the clay and feldspar were silica (SiO_2) and Alumina (Al_2O_3). Clay possessed 59.2% silica and 21.25% alumina, while fledspar had 49.16% and 34.02% of silica and alumina respectively. Therefore, the clay and feldspar belong to Alumino-silicate group in line with the view of (Shuaib-Babata 2016; Abolarin *et al.* 2006; Hassan 1993; Encyclopedia Britannica (2018a; and Bastin 2018). Quartz also had silica as its major constituents with 99.96% value. Hobalt (2018) and Encyclopedia Britannica (2018b) affirmed that quartz primarily consists of silica.

It is observed from Fig. 4 that the fraction of the volume of the voids over the surface of the specimens produced were very closed-cell foam. However, the results revealed that specimens 6 and 8 in formulation C had the lowest porosity values of 0.1%. This might be the result of reduction in the clay percentage in the composition of the specimens. Materials with low

Table 3. The Chemical Compositions of all the raw solid minerals.

S/N	Parameters	Level of Detection (wt%)		
		Clay	Quartz	Feldspar
1	SiO_2	59.200	99.96	49.16
2	TiO_2	0.005	0.047	0.01
3	Al_2O_3	21.250	0.041	34.02
4	Fe_2O_3	15.700	0.006	0.74
5	CaO	1.920	0.100	2.87
6	MgO	0.880	0.007	0.08
7	Na_2O	0.050	0.000	2.63
8	K_2O	0.040	0.000	8.40
9	MnO	0.010	0.008	0.002
10	Moisture	0.002	0.001	0.005

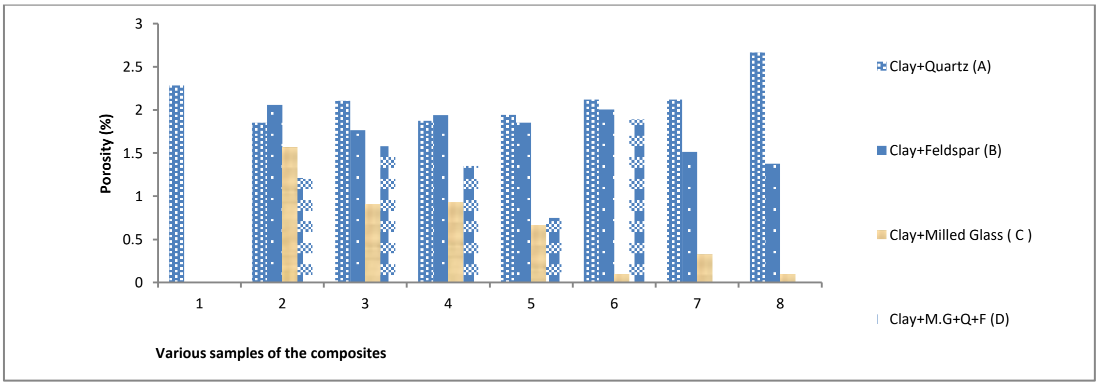

Figure 4. Average porosity values of different moulded ceramic tiles samples with varying Compositions.

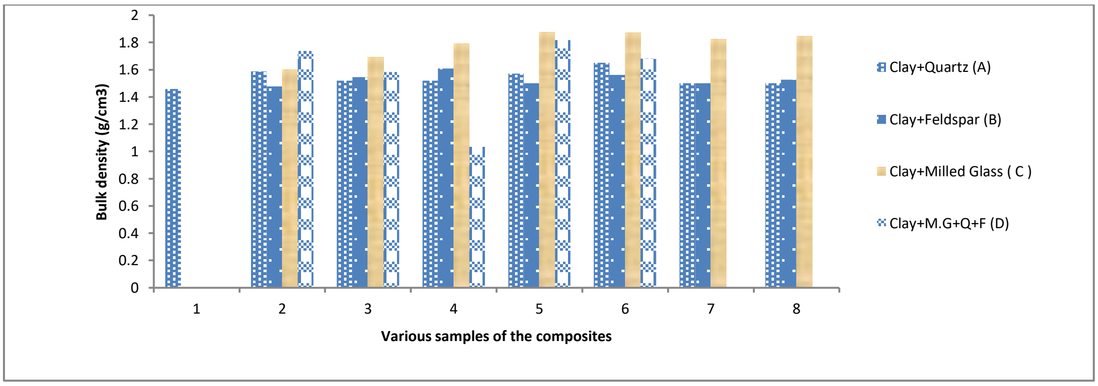

Figure 5. Average bulk density values of different moulded ceramic tiles samples with varying composition.

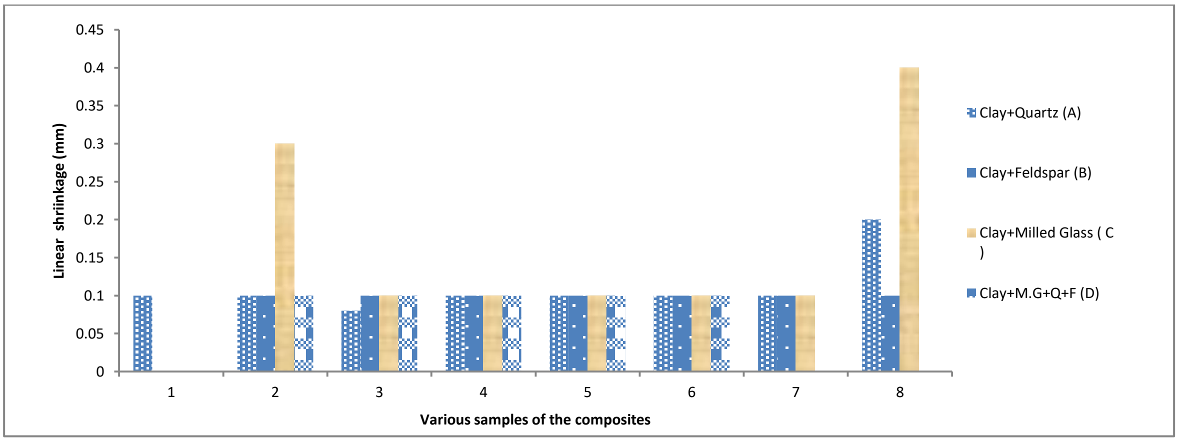

Figure 6. Average linear shrinkage values of different moulded ceramic tiles samples with varying Compositions.

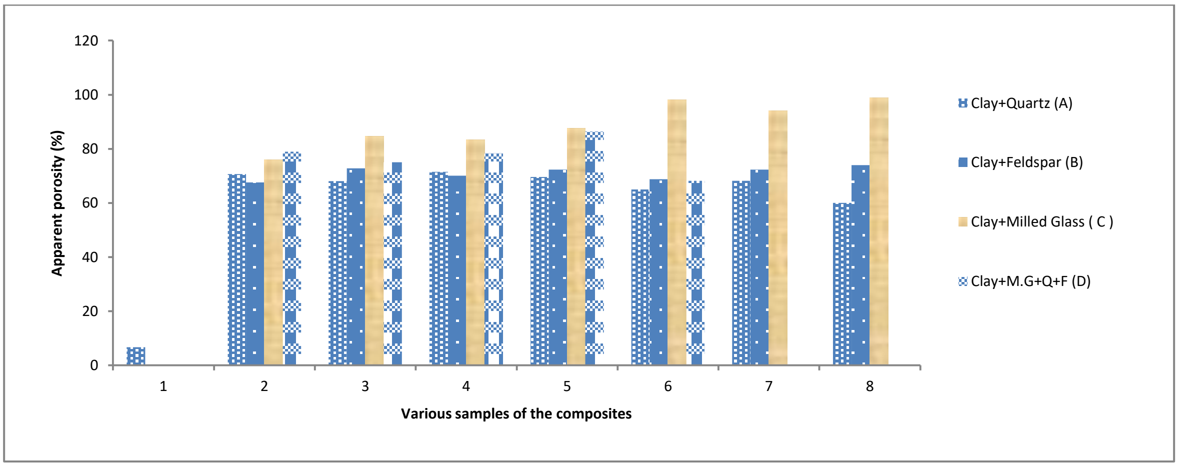

Figure 7. Apparent porosity values of different moulded ceramic tiles samples with varying Compositions.

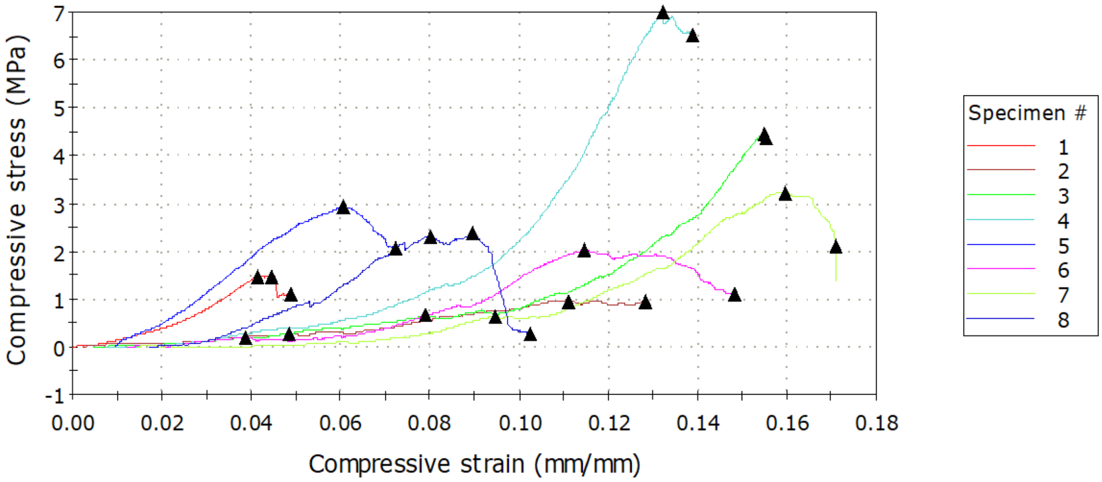

Figure 8. Stress – strain relationship in formulation A.

porosity value are more preferred for production of tiles Elakheme *et al.* (2016). Generally, the average porosity values were within the recommended value of 0.1- 3.5% for tiles (National Research Council of Italy, 2017).

The specimens' average bulk densities were within 1.033 and 1.875 g/cm^3 (Fig. 5), which fall within the recommended standard bulk density range of 1.0 to 2.5 g/cm^3 for tiles (Hassan, 2011; National Research Council of Italy, 2017) and 1.7 –2.1g/cm3 for dense ceramic materials Hassan (2001). Sample 4 in formulation D had the lowest bulk density value of 1.033 g/cm^3, while samples 5 & 6 in body formulation C recorded the highest value of 1.87 g/cm^3. Materials with low density would be better for production of tile.

3.3 Fired Shrinkage and Apparent Porosity

The samples' linear shrinkage ranged between 0.08 and 0.40 mm (Fig. 6), which were within the accepted range of 0.02 and 0.80 mm (National Research Council of Italy, 2017). The samples' linear shrinkage values at 1100°C were significantly low. This is very advantageous for wall and floor tiles composition, which may be due to the presence of appreciable SiO_2 in the composition. However, the magnitude of shrinkage increases with clay contents but the limit is reached for higher specific water contents Boivin *et al.* (2004).

The samples' calculated percentage apparent porosity ranged between 45.0 to 98.1%. These values were within the specified 30.0 to 60.0% values (National Research Council of Italy, 2017). Sample 8 in formulation A (80% and 20% quartz) had the lowest apparent porosity value of 45%, which is considered to be the best. This might be attributed to high proportion of SiO_2 in the composition. High shrinkage resulted to reduced apparent porosity in the closure of internal pores.

3.4 Compressive Strength Test

Figs. 8 to 11 show the stress-strain relation of the samples, which are better illustrated in Figs. 12 and 13 as an indicator for durability of the samples in service.

The variations of calculated apparent porosity values in various samples of the composite are as presented in Fig. 7.

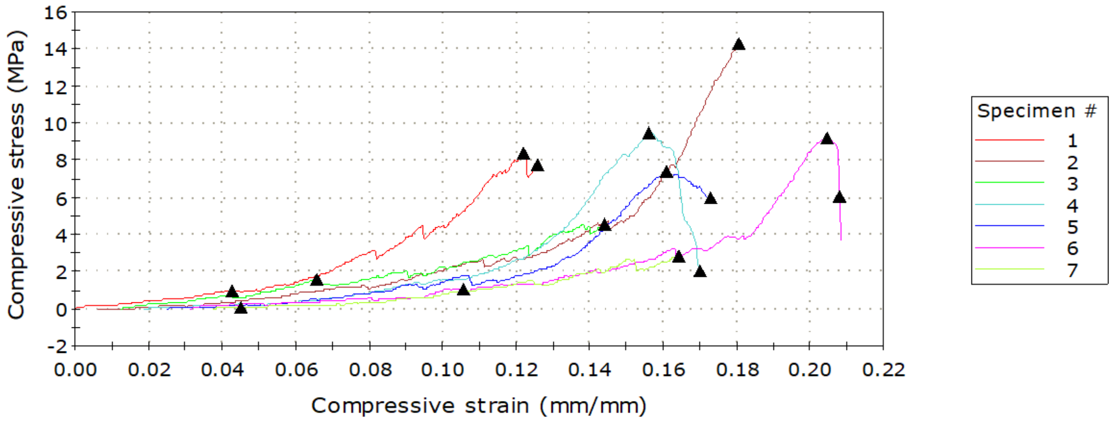

Figure 9. Stress–strain relationship in formulation B.

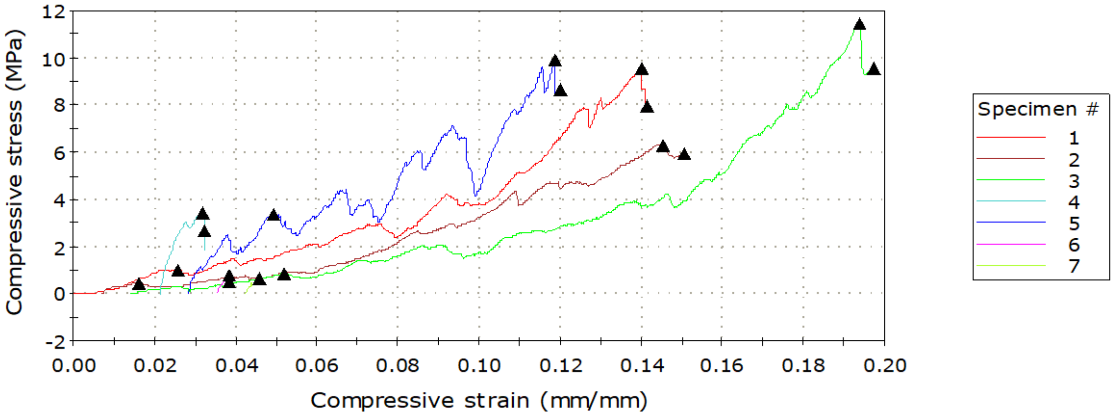

Figure 10. Stress–strain relationship in formulation C.

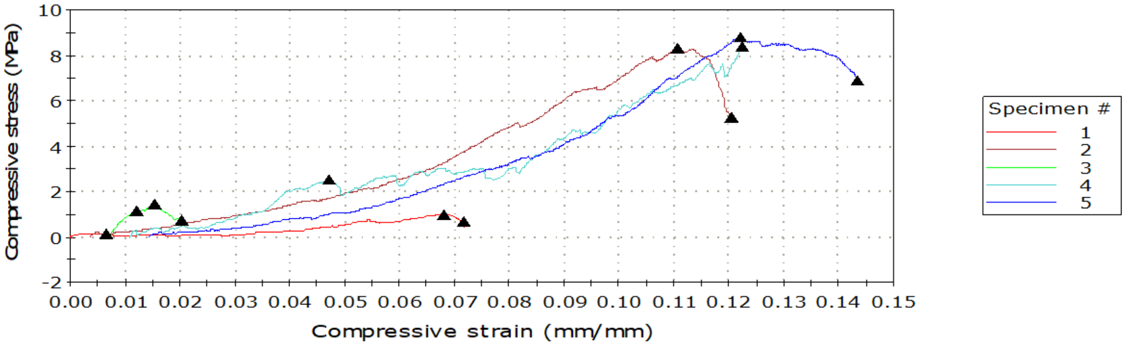

Figure 11. Stress–strain relationship in formulation D.

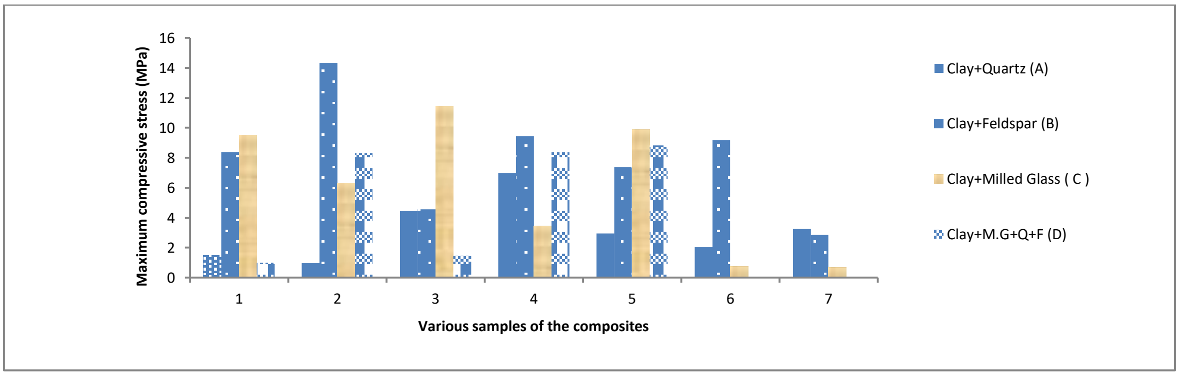

Figure 12. Average Compressive strength values of different moulded ceramic tiles samples with varying composition.

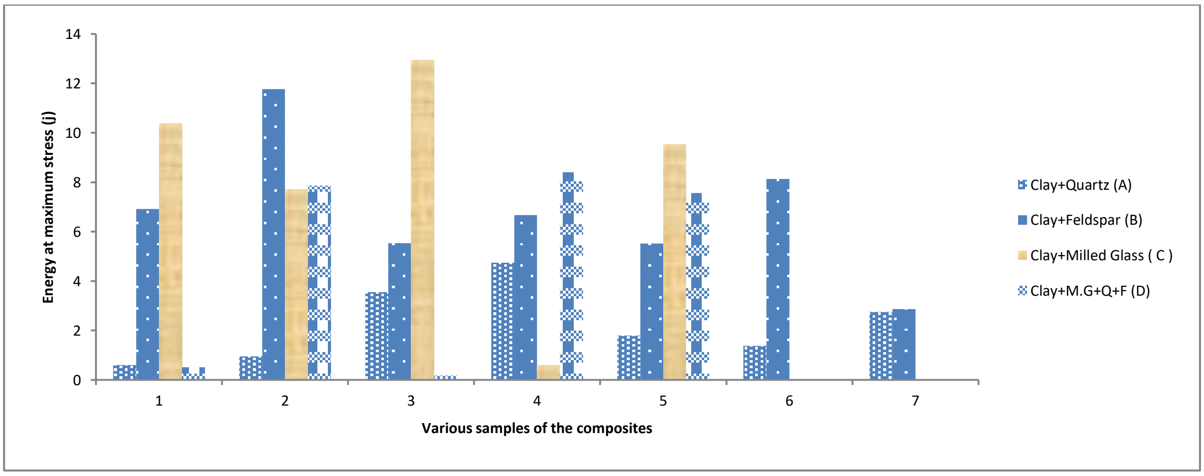

Figure 13. Average energy at maximum stress by different moulded ceramic tiles samples with varying composition.

Table 4. Summary of the sample's properties compared with the available information on the properties of tiles.

S/N	Properties	Tiles {CY + QZ & Dolomite} [National Research of Italy, 2017]	New Formulation Lab. Tiles {CY + QZ + MG & Alumina}
1	Compressive strength (MPa)	5.000 - 20.800	1.0117 - 14.331
2	Bulk density (g/cm3)	1.0-2.5	1.2 - 1.8
3	Porosity measurement (%)	0.1-3.5	0.1 - 2.7
4	Apparent Porosity %	30 – 60	45 – 98
5	Shrinkage measurement	0.02-0.80	0.08 - 0.4

Within the formulation A, B, C and D, sample 4, 2, 5 and 4 exhibited the highest compressive strength values of 6.98156 MPa, 14,33139 MPa, 11.45160 MPa and 8.83201 MPa respectively in each formulation group. These strength values were within the recommended range of 5.00 to 20.8 MPa for tiles Krivandin and Markiov (1980). However, other samples that had strength values within the recommended values include samples 1, 2, 4, 5 and 6 in formulation B, samples 1, 2, 3 and 4 in formulation B, and samples 2, 4 and 5 in formulation C (Fig. 12). From the obtained results, sample 2 in formulation B consisting of 80% clay and 20% feldspar exhibited the highest average compressive strength values (14,33139 MPa). This might be as a result of combination of high SiO_2 with Al_2O_3 from both clay and feldspar.

Samples 4, 2, 5 and 4 respectively in formulation A, B, C and D also had energy at maximum stress of 4.74391 J, 11.77584 J, 9.53256 J and 7.5682 J respectively, in each formulation group. Generally, the energy values exhibited by the samples ranged between 0.02818 and 11.77584 J. The majority of these energy values were considered to be adequate for sustainability of the tiles in service.

To determine the stability of the samples, it is essential to compare the properties exhibited by the samples with that of available information on the properties of ceramics tiles as presented in Table 4.

Thorough critical study of the data in Table 3 shows that various properties exhibited by sample 4 (formulation A), sample 2 (formulation B), sample 5 (formulation C) and sample 4 (formulation D) met the required standards for tiles. Therefore, the new formulation for production of ceramics tiles using locally available raw materials (clay, quartz, feldspar and milled bottles) proved suitable and adequate for local production of composite ceramic tiles in terms of strength and other properties. This will assist to address the nation’s economic problems and help towards job creation.

4. CONCLUSION

From this study, the following conclusions are drawn:

1. The clay and feldspar used in this study were found to belong to alumino-silicate group.

2. Suitable combinations of Nigerian natural resources and wastes such clay, feldspar, quartz and milled glasses were found suitable for the production of standard ceramic tiles. The suitable proportions of the materials in production of the tiles: 60: 40%, 80:20%, 50:50% and 60:5:5:30% of clay and quartz, clay and feldspar, clay, quartz and feldspar, quartz, feldspar and milled glasses respectively.

3. Clay, quartz, feldspar and milled glasses were characterized to ascertain for their potentials for ceramic tiles, refractory and others ceramics applications. The results obtained showed that they meet the criteria for use as refractory/ceramic raw materials in the entire characteristic investigated via chemical composition, fired shrinkage, porosity, apparent porosity, bulk density and compressive strength. The clay can be used as a fired refractory, while quartz, feldspar and milled bottles could be used as a source of silica and alumina for ceramic products.

4. Adequate utilization of available natural resources and wastes in Nigeria such as clay, quartz, feldspar and milled glasses will help in domesticating production of building materials like tiles.

CONFLICT OF INTEREST

The authors declare no conflicts of interest.

FUNDING

No funding was received for this project.

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