



Original Article

Effect of Firing Temperature on some Mechanical Properties of Osun State Ceramic Tiles.

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Triaxial Blend,
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Ceramic Tiles.

The work evaluates the effect of firing temperature on the mechanical properties of ceramic tiles. This was with the view to determine the optimum processing condition for Osun State ceramic tiles. Ceramic raw materials collected from Osun State were batched using clay-feldspar-silica sand blending ratio of 5:4:1, 5:3:2, 5:2:3, 5:1:4, 6:3:1, 6:2:2, 6:1:3, 7:2:1, 7:1:2 and 8:1:1 by weight; and homogeneously mixed. Three replica samples were moulded by the method of dry forming, fired at 1200, 1300 and 1400 °C and subjected to breaking and flexural strength tests using the Universal Testing Machine while the hardness test was carried out on a Moh's scale. The results showed that breaking strength, flexural strength and Moh's hardness fell within the range 199.43 to 325 N, 11.97 to 19.50 N/mm² and 2.5 to 4 MH respectively, while Figures revealed that samples with 60% clay, 10% feldspar and 30% silica sand fired at 1320 °C will process the best mechanical properties. In conclusion, ceramic raw materials collected from Osun State are viable for ceramic tile production.

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INTRODUCTION

Ceramic tiles are highly vitrified materials produced from a triaxial blend of kaolin ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), quartz (SiO_2) and feldspar ($\text{K, Na, Ca})_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$) (Iraor et al., 2014; Toe et al., 2016; El Nouhy, 2013; Martín-Márquez et al., 2008; Misra et al., 2013; Soni et al., 2015). It is primarily a hygiene product made of the porous body with a coating of white or coloured glaze extensively used in the living room, bathrooms, kitchens, medical centres, laboratories, schools, public conveniences, and shopping malls (El Nouhy, 2013). Ceramics can also be inorganic, non-metallic crystalline materials comprising metal, nonmetal or metalloid atoms primarily held by ionic and covalent bonds (European Commission, 2007; Adindu et al., 2014; Solanki & Shah, 2016). Ceramic products are brittle, hard, strong in compression, weak in shearing and tension.

Amount, size and distribution of porosity are among the important factors which affect the physical and mechanical properties of ceramic tiles (Ozturk & Ay, 2014). Mechanical properties common to ceramic tiles are breaking strength and flexural strength, Impact resistance, Resistance to deep abrasion, Hardness, Coefficient of friction and Tensile strength.

The flexural strength also known as modulus of rupture or bending strength or transverse rupture is the stress in a material just before it yields in a flexure test (Ashby, 2005) or an indication of product breaking strength (Alege & Alege, 2013). Flexural strength of ceramics is an important property for all the applications involving impact loading conditions (El Nouhy, 2013; Iseri et al., 2010; Belenky & Rittel, 2012; Mojiska et al., 2016), with tiles falling into that category due to various machining operations carried out on it before and

after installation. The limits of breaking strength and flexural strength of any specimen are determined by both water absorption and thickness of the specimen and it depends on the body composition, dimensions, and morphology of the flaws (El Nouhy, 2013). The flexural strength represents the highest stress experienced within the material at the point of failure. Ceramic systems usually exhibit a low modulus of rupture (Ashby, 2005). Hardness, meanwhile, is the resistance of a material to plastic deformation, usually by scratch, indentation rebound (Chinn, 2009).

The mechanical properties of any ceramic body are dependent on the properties of the raw material and firing temperature. According to El Nouhy (2013) and Soni *et al.* (2015), the mechanical properties of ceramics depend on the amount of mullite formation which is a factor of the type, properties and proportion of the clay used as well as the firing temperature. Meanwhile, several researchers have reported different triaxial blends as well as diverse optimum firing temperatures for ceramic tiles production. Braganca and Bargmann (2004) reported 1340 °C; in the works of Amoros *et al.* (2007) and Idowu, (2014), between 1190 and 1220 °C was reported; for El-fadaly (2015) it was between 1190 and 1230 °C; The American Ceramic Society (2005) stated 1400 °C as the maximum firing temperature; while Mathew & Fatile (2014) reported 1218 °C as suitable firing temperature. Consequently, the work seeks to determine the effect of firing temperature on the mechanical properties of ceramic tiles.

MATERIALS AND METHOD**Materials and Sources**

The clay mineral used in this study was kaolinite clay (Oke & Omidiji, 2016), collected from Ipetumodu, headquarters of the Ife North Local

Government area of Osun State, Nigeria. Silica sand was collected from the Isasa River; the river serves as the boundary between the Ayedaade and Ife North Local Government areas of Osun State, Nigeria. Feldspar was collected from Osogbo, the capital of Osun State, Nigeria. The three raw materials collected were beneficiated separately as specified by Abiola *et al.*, (2019).

Preparation of Materials

The ceramic raw materials (clay, feldspar and silica sand) were homogeneously mixed, prepared into ten different blends of 5:4:1, 5:3:2, 5:2:3, 5:1:4, 6:3:1, 6:2:2, 6:1:3, 7:2:1, 7:1:2 and 8:1:1 ratio by weight; and designated as blend A, B, C, up to J respectively and fired as described by Abiola *et al.*, (2021). Three replicate ceramic samples for flexural strength (140 x 45 x 5 mm), breaking strength (140 x 45 x 5 mm) and hardness test (20 x 20 x 10 mm) were made from each blend, A - J of the ceramic materials by compacting them at 40 MPa pressure as specified by Bresciani *et al.* (2004) to recommended size for the different test.

After forming, the samples were dried by convection in an open laboratory drying oven (model DHG-9101-2A manufactured by Searchtech Instrument) where heated air was circulated around the ceramic samples. The air around the samples was kept at 95 °C to remove the water content since water is expected to evaporate from the ceramic mixture below 100 °C. The materials were kept under this condition for a period of 20 hours following the method of Br.MSME-DI (2011). This drying process is expected to prevent differential shrinkage, warping, cracking and distortion of the eventual product.

Prior to firing, the ceramic samples were heat-treated in the laboratory drying oven at a temperature of 300 °C in line with the recommendation of Br.MSME-DI (2011) to provide additional drying, vaporize or decompose organic additives and other impurities, as well as to remove

residual, crystalline, and chemically bound water. As proposed by Br.MSME-DI (2011), the ceramic samples were kept in the kiln for 20 hours while the temperature of 300 °C was held constant. After this period, the ceramic samples were brought out of the kiln and allowed to cool normally at ambient temperature and then fired in a furnace (model XD-1700M manufacturer by Zhengzhou Brother Furnace Company, China). The experimental firing temperatures of 1200, 1300 and 1400 °C were used. These temperatures were employed since according to Abiola & Oke (2017) Ipetumodu clay begin to disintegrate at 1450 °C. Also, 1400 °C was used as the maximum firing temperature, since according to the American Ceramic Society (2005), it is the maximum sintering temperature for ceramic tile production. The lower temperatures (1200 °C and 1300 °C) were required to evaluate the behaviour of the local tiles produced below the recommended maximum firing temperature for ceramic products. This was because diverse sintering temperatures between 1190 and 1340 °C have been recommended by various researchers (Braganca & Bergmann, 2004; Amoros *et al.*, 2007; Idowu, 2014; El-Fadaly., 2015). The samples were held at the respective firing temperatures for about 1 hour soaking time to ensure equalization of temperature throughout the cross-section of the samples (Ashby, 2005; Abeid & Park, 2018). Thereafter, the samples were kept in the kiln for a cycle of at least 18 hours where it was cooled, in line with Br.MSME-DI (2011).

Mechanical Property Test

After cooling, the ceramic tiles were off-loaded from the kiln and subjected to flexural strength and breaking strength test according to ISO 10545-3 (1996) while the hardness test was investigated using Moh's scale hardness test method in line with El Nouhy (2013).

The breaking strength was calculated from equation (1) as:

$$S = \frac{FL}{b} \quad (1)$$

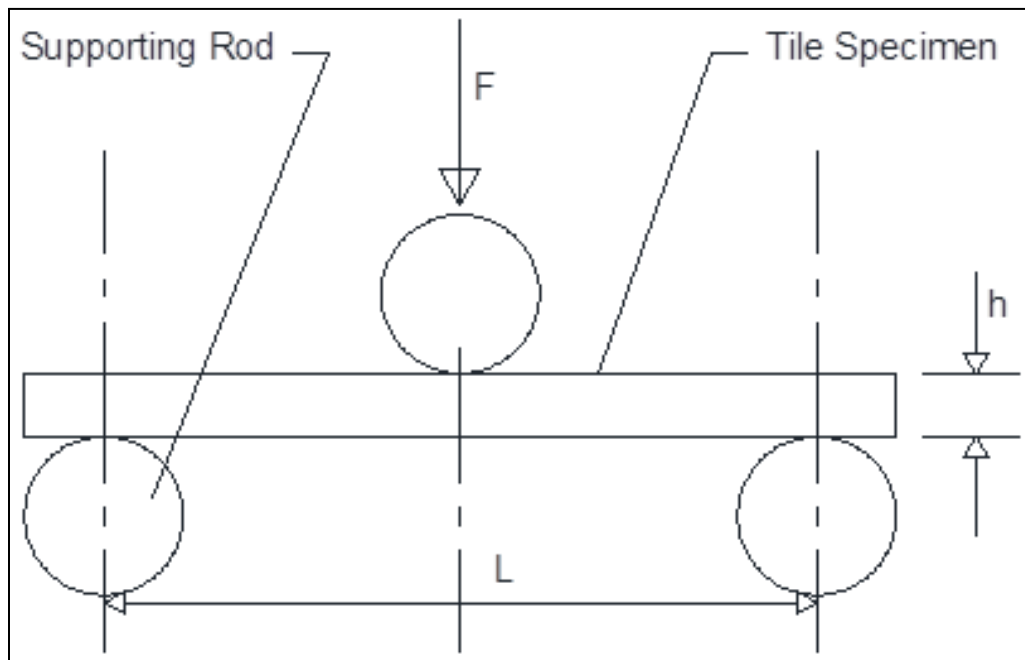
where, S is the breaking strength, in Newton; F is the breaking load, in Newton; L is the distance between the supporting rods, in millimetres (65 mm); b is the width of the test specimen, in millimetres as shown in Figure 1.

From the same test (Fig. 1), modulus of rupture also known as flexural strength was calculated for each sample of ceramic tiles using equation (2) (1996).

$$R = \frac{3S}{2h^2} \quad (2)$$

where R is the modulus of rupture, in N/mm^2 ; S is the breaking strength or force, in Newton; h is the minimum thickness of the test tile specimen, in millimetres.

Figure 1: Placement of specimen on Universal Strength Testing Machine for breaking strength and flexural strength test



The hardness test was investigated using Moh's scale hardness test method in line with El-Nouhy (2013). A minimum of three replicate tests were carried out on each specimen (20 x 20 x 10 mm). The constituents of the specimen with the highest Moh's hardness that resulted in no more than one scratch were recorded.

Experimental Design

Design Expert 6.0.8 Portable was used to design the experiment using the surface response method. Two-factor design matrix linear models were applied in designing the experiment to determine the effect of firing temperatures (1200 °C, 1300 °C

and 1400 °C) and triaxial blend ratios (5:4:1, 5:3:2, 5:2:3, 5:1:4, 6:3:1, 6:2:2, 6:1:3, 7:2:1, 7:1:2 and 8:1:1) on some mechanical properties (flexural strength, breaking strength and hardness) of ceramic tiles. Design matrix of sixty (60) experiments designed for two factors were used on each combination of firing temperature and blending ratio for mechanical properties.

RESULTS AND DISCUSSION

Breaking and Flexural Strength

The flexural strength of the samples which is a function of the breaking strength generally increases

as the temperature increased from 1200 °C to just above 1300 °C and then fell as the temperature further increased from just above 1300 °C to 1400 °C as shown in Figures 2 and 3.

The rising strength with increased firing temperature may be due to liquid or glass phase formation as a result of a high percentage of alkaline oxides (Na₂O and K₂O) present in the feldspar (Nouhy, 2013; Kimambo et al., 2014). As a result, a large amount of liquid phase form, fill and reduce pore spaces in the ceramic body. This results in increasing the flexural strength of the samples with increasing firing temperature (Hettiarachchi et al., 2014). The fall in the breaking strength and flexural strength as firing temperature increased from 1300 °C to 1400 °C may be due to bloating (Hettiarachchi

et al., 2014) which results in increased porosity within the same temperature range. (Kimambo et al., 2014; Hettiarachchi et al., 2014).

The result also shows that the strength of the samples increases as silica sand content increases and feldspar reduces. This may be due to a high percentage of alkaline earth oxides (CaO and MgO) contained in the silica sand even after beneficiation. Alkaline earth oxides can form high viscous liquid phases at a high temperature which enabled the formation of new crystals that enhance the densification and strength of the ceramic body (Hettiarachchi et al., 2014). Silica sand contributes higher strength to the ceramic body than what the glass phase reached in Na₂O and K₂O which are abundant in feldspar.

Figure 2: Breaking Strength of the ceramic tile samples fired at different temperatures

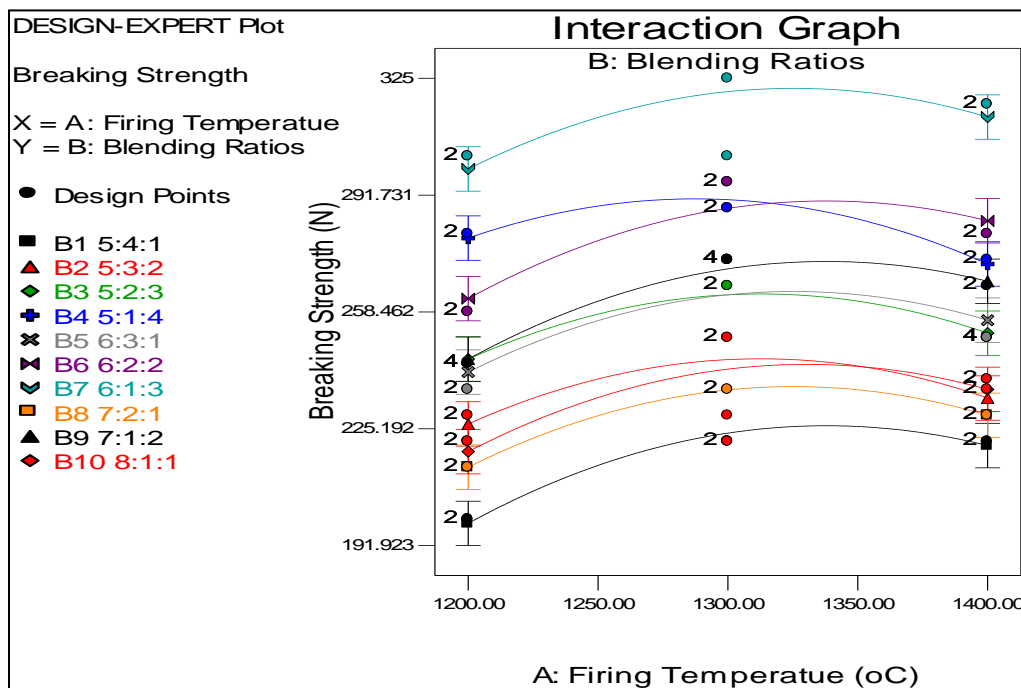
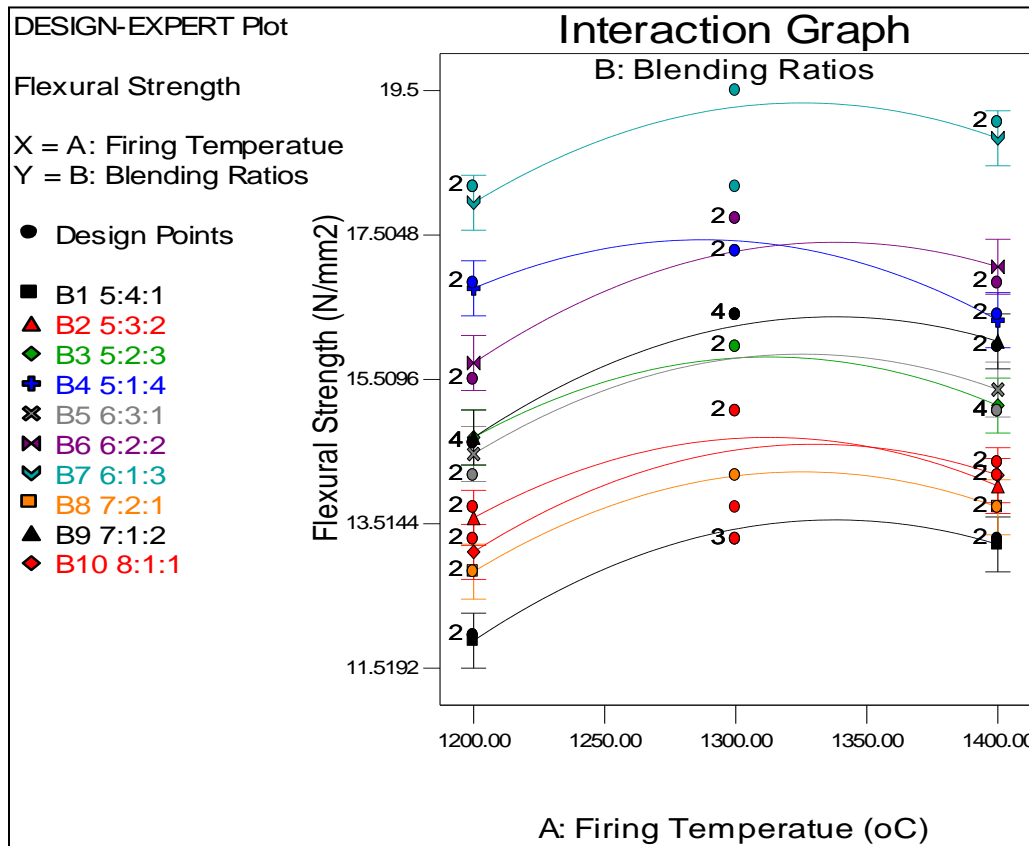


Figure 3: Flexural strength of the ceramic tile samples fired at different temperatures



The main function of silica sand is to reduce the ceramic body's tendency to warp or distort when fired to high temperatures which result in the formation of relatively large amounts of molten glass in the body (El Nouhy, 2013). Hence, samples containing high silica sand generally show higher strength than those containing more feldspar.

The results further show that the minimum breaking strength (199.43 N) and flexural strength (11.97 N/mm²) were recorded for sample "A" fired at 1200 °C while the maximum breaking strength (325 N) and flexural strength (19.50 N/mm²) were recorded for sample G fired at 1300 °C fall way short of the ISO recommended standard of 600 N and 35 N/mm² respectively for tiles that are less than 7.5 mm thick. Meanwhile, Chukwudi *et al.* (2012) described 20 MPa to 35 MPa as the flexural strength for normal floor tiles irrespective of its thickness though it recorded a much higher flexural strength of between

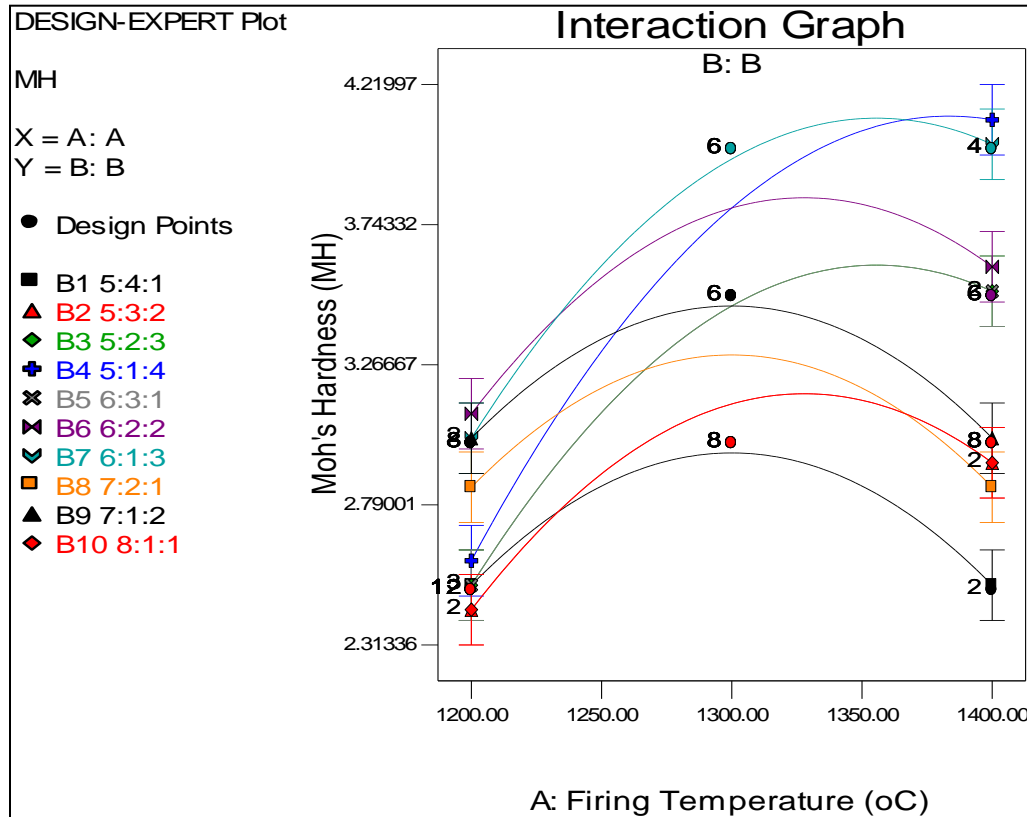
25 MPa to 59 MPa in its study after adding different percentages of steel slag to the mixture of clay, feldspar, and silica sand material. Ogundare *et al.* (2015) recorded a similarly high flexural strength ranging from 21 MPa to 54 MPa. El-Nouhy (2013) alighted between 13.6 N/mm² and 24.79 N/mm² as the flexural strength for the 14 different ceramic tile samples produced and collected from Egypt.

Moh's Hardness

The result showed that the hardness of the ceramic samples increases as the firing temperature rises and then began to drop with a further increase in temperature as shown in Figure 4. The increased Moh's hardness with an increase in the firing temperature from 1200 °C to 1300 °C may be as a result of the samples' pores being filled with the liquid phase of the molten feldspar (Nouhy, 2013; Kimambo *et al.*, 2014). However, the eventual

decrease in the hardness as temperature rises from 1300 °C to 1400 °C may be as a result of bloating which resulted from gas enclosed within the pores of the ceramic articles (Hettiarachchi et al., 2014).

Figure 4: Moh’s hardness of the ceramic tile samples fired at different temperatures



CONCLUSION

There is appreciation in the mechanical properties of ceramic tiles as firing temperature increases from 1200 °C to just above 1300 °C and these properties deteriorate with a further increase in temperature. Therefore, Figures 1 to 3 revealed that the ceramic sample will attain the best mechanical properties at about 1320 °C. The ceramic sample produced from Osun State raw materials in this study is good for wall tiles since the strength is just about the minimum (20 N/mm²) rather than the maximum (35 N/mm²). Meanwhile, ceramic sample ‘G’ with 60% clay, 10% feldspar and 30 % silica sand produced the sample with the best breaking strength (325.00 N), flexural strength (19.50 N/mm²), Moh’s hardness (4 MH). Also, samples with 60% clay make the sample with the best strength.

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