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## THE EFFECT OF FIRING TEMPERATURE ON SOME PHYSICAL PROPERTIES OF OSUN STATE CERAMIC TILES

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**Abstract:** The work evaluates the effect of firing temperature on the physical 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 molded by dry forming and fired at 1200, 1300 and 1400°C and subjected to water absorption, apparent porosity, apparent relative density and bulk density tests in line with the ISO 10545–3 (1996) standard. The results showed that water absorption, apparent porosity, apparent relative density, and bulk density were within the range 10.43 to 15.02%, 22.77 to 30.20%, 2.26 to 3.09 and 1.34 to 1.45 g/cm<sup>3</sup> respectively, while the Figures revealed that sample with 60% clay, 30% feldspar and 10% silica sand fired at 1320°C will exhibit the best physical properties. In conclusion, ceramic raw materials collected from Osun State are viable for ceramic tile production.

**Keywords:** clay feldspar, silica sand, triaxial blend, physical properties, ceramic tiles

### INTRODUCTION

Ceramic tiles are tiles made of a triaxial clay–based combination that are frequently used to cover walls and floors in buildings (Ilorin *et al.*, 2014; El Nouhy, 2013; Martin–Marquez *et al.*, 2008; Iqbal and Lee, 2000; Abiola *et al.*, 2021). It is essentially a hygienic item with a porous body and a thick layer of white or colorful glaze that is widely used in living rooms, bathrooms, kitchens, hospitals, labs, schools, public restrooms, and shopping centers (El Nouhy, 2013). According to Iyasara *et al.* (2014) The three silicate clay minerals clay, silica sand, and feldspar are combined to create ceramics, which are inorganic compounds that react with one another at the right high temperature. They can survive extremely high temperatures as well as chemical erosion, which many other materials are susceptible to (Carter and Norton, 2007; European Commission, 2007; Jung, 2008).

Ceramic tiles are characterized by low water absorption, usually between 3.3% and 11.1% (Amoros *et al.*, 2007; Griese, 2007; Bryne, 2008; ISO 10545, 1996). Water absorption is commonly referred to as an indicator of porosity for wall and floor tiles (Chukwudi *et al.*, 2012). Amount, size and distribution of porosity are among the important factors which affect the physical and mechanical properties of ceramic tiles. Tiles with the lowest apparent porosity have the lowest water absorption, high bulk density and high compressive strength (Soni *et al.*, 2015). The porosity is connected to the liquid phase during firing and is affected by the transformations that occur during sintering (Ozturk and Ay, 2014).

Meanwhile, the properties of any ceramic body are dependent on the properties of the raw material and firing temperature (Choudhury *et al.*, 2012). 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; Amoros *et al.* (2007) and Idowu, (2014), between 1190 and 1220°C; 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 and Fatile (2014) reported 1218°C as suitable firing temperature.

According to ISO standard 10545 (1996), the physical properties of any ceramic tile (water absorption, apparent porosity, apparent relative density and bulk density) depend mostly on the amount, size and distribution of particles; material composition; and firing temperature (Ozturk and Ay, 2014; Lin and Lan, 2013). Since studies have provided several processing methods for ceramic tiles, it has become imperative to study the effect of material blending ratio and firing temperature on the physical properties of ceramic tiles produced from raw materials collected from Osun State.

### MATERIALS AND METHODS

The kaolinite clay utilized in this study was gathered from Ipetumodu, the administrative center of the Ife North Local Government region of Osun State, Nigeria (Oke and Omidiji, 2016). Silica sand was gathered at the Isasa River in Osun State, Nigeria, which divides the local governments of Ayedaade and Ife North. Feldspar was gotten at Osogbo, the capital of Nigeria’s Osun State. The three raw materials

collected were beneficiated separately as specified by Abiola *et al.*, (2019).

— Preparation of materials

The ceramic raw materials were prepared as described by Abiola *et al.* (2021). Clay, silica sand, and feldspar were thoroughly combined to create a more chemically and physically homogenous substance for producing tiles. Due to many discrepancies regarding the ideal mixing ratio for ceramic materials, the mixing ratios for the raw materials were varied (Braganca and Bargmann, 2004; Amoros *et al.*, 2007; El-Fadaly 2015; Martín-Márquez *et al.*, 2010; Idowu, 2014).

To predict the mixing ratio of ceramic ingredients, Norsker and Danisch’s (1993) 10–step tri–axial blending chart was used. Only the ceramic blends with clay–feldspar–silica sand ratios 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 and designated as blends A, B, C, up to J were chosen from the chart’s potential sixty–six combinations. Due of the fact that ceramic is referred to as a triaxial blend product, thirty blends that contain fewer than three materials were disregarded (Teo *et al.*, 2016; Idowu, 2014; Irabor *et al.*, 2014; Iyasara *et al.*, 2014; Soni *et al.*, 2015; El Fadaly, 2015). Since clay is the main component used in the creation of ceramic tiles, 26 additional blends with less than 50% clay percentage were also disregarded (Solanki and Shah, 2016; Soni *et al.*, 2015; Irabor *et al.*, 2014; Adindu *et al.*, 2014; El Ouahabi *et al.*, 2014; El Nouhy, 2013; Misra *et al.*, 2013; Manfredini and Hanuskova, 2012; Murray 1999).

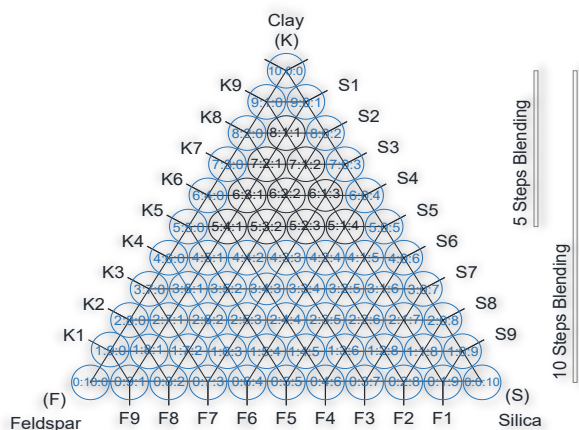


Figure 1: 10 steps triaxial blending chart

Each blend of the ceramic materials, A through J, was compacted to the recommended size for the physical property test sample, 50 x 15 x 15 mm, according to the instructions provided by Bresciani *et al.* (2004). 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. Since water should evaporate from a ceramic combination below 100°C, the air around the samples was held at 95°C to remove any water content. The materials were maintained in this condition for 20 hours using the Br.MSME–DI technique (2011). It is anticipated that this drying procedure will

stop the final product’s eventual differential shrinkage, warping, cracking, and deformation.

According to Br.MSME–DI (2011) recommendation, the ceramic samples were heat–treated in the laboratory drying oven at 300°C before firing in order to provide additional drying, vaporize or decompose organic additives and other impurities, as well as remove leftover, crystalline, and chemically bound water. According to the plan put forth by Br.MSME–DI (2011), the ceramic samples were kept in the kiln for 20 hours at a constant temperature of 300°C. The ceramic samples were removed from the kiln after this time, allowed to naturally cool at room temperature, then fired in a furnace (model XD–1700M manufactured by Zhengzhou Brother Furnace Company, China). The fire temperatures employed for the experiment were 1200, 1300, and 1400°C. These temperatures were used because Martín–Márquez *et al.* (2008) claimed that firing ceramic tile above the vitrification range will result in a drastic fall in the physical properties due to the forced expulsion of trapped gases, resulting in bloating and blisters and Abiola and Oke (2017) claimed that Ipetumodu clay begins to disintegrate at 1450°C. Additionally, as the American Ceramic Society (2005) states that the highest sintering temperature for the manufacture of ceramic tiles is 1400°C, which value was chosen as the maximum firing temperature as well. To assess the behavior of the local tiles made below the recommended maximum firing temperature for ceramic products, lower temperatures (1200°C and 1300°C) were necessary. This was because many researchers had suggested sintering temperatures ranging from 1190 to 1340°C. (Braganca and Bargmann 2004; Amoros *et al.*, 2007; Idowu, 2014; Mathew and Fatile, 2014; El–fadaly, 2015). To guarantee that the samples’ cross–sections were heated to the same temperature, the samples were kept at the appropriate firing temperatures for around an hour (Abeid and Park, 2018; Ashby, 2005). Following that, the samples were held in the kiln for a cycle of at least 18 hours where it was cooled, in line with Br. MSME–DI (2011).

— Physical properties test

The ceramic tiles were removed from the kiln after cooling and tested for water absorption, apparent porosity, and apparent relative density in accordance with the recommendations of Abiola *et al.* (2021) and ISO 10545–3 (1996).

The water absorption was calculated from equation (1):

$$A_w = \frac{m_2 - m_1}{m_1} \times 100\% \quad (1)$$

where  $A_w$  is water absorption (%);  $m_1$  is the average mass of the dry samples in gram (g); and  $m_2$  is the average mass of the wet samples in gram (g) (ISO 10545–3, 1996).

Apparent porosity was calculated from equation (2) as:

$$P_a = \frac{m_2 - m_1}{m_2 - m_3} \times 100\% \quad (2)$$

where  $P_a$  is the apparent porosity (%);  $m_1$  is the average mass of the dry samples in gram (g);  $m_2$  is the average mass

of the wet samples in gram (g); and  $m_3$  is the average mass of the suspended samples impregnated by boiling water in gram (g) (ISO 10545–3, 1996).

Meanwhile, apparent relative density was calculated using equation (3) (ISO 10545–3, 1996) as:

$$RD_a = \frac{m_1}{m_1 - m_3} \quad (3)$$

where  $RD_a$  is the apparent relative density;  $m_1$  and  $m_3$  are the same as in equation (2) (ISO 10545–3, 1996).

Bulk density was determined using equation (4) as:

$$BD = \frac{m_1}{V} \quad (4)$$

where  $BD$  is the bulk density, in  $g/cm^3$ ;  $m_1$  is the average mass of the dry samples in gram (g); and  $V$  is the exterior volume of the sample, in  $cm^3$  (ISO 10545–3, 1996).

### — Experimental design

The experiment was created using Design Expert 6.0.8 Portable using the surface response approach. In order to determine the impact of firing temperatures (1200 oC, 1300 oC, and 1400 oC) and triaxial blend ratios (5:4:1, 5:3:2, 6:2:2, 6:1:3, 7:2:1, 7:1:2, and 8:1:1) on the physical properties (water absorption, apparent porosity, apparent relative density, and bulk density) of ceramic tiles, a two-factor design matrix linear model was used. Design matrix of sixty (60) experiments design for two factors were used on each combination of firing temperature and blending ratio for physical properties.

## RESULTS AND DISCUSSION

### — Water absorption

The results of the physical properties tests are as seen in Figure 2. Water absorption decreased with increased temperature and later reduced with continued increase in temperature.

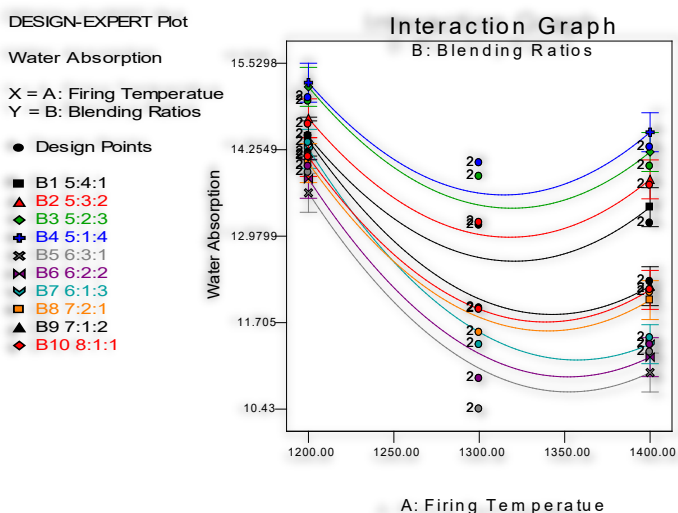


Figure 2: Water absorption of the ceramic tile samples fired at different temperatures. The reduced water absorption with increased temperature could be due to the liquid phase formation at high temperature and densification as the sample cools to room temperature (Alves *et al.*, 2015; Kimambo *et al.*, 2014). The liquid phase that is formed fills the pores and decreased the porosity (Viruthagiri *et al.*, 2009). Thus, increased temperature

reduces or eliminates pores within the ceramic article, thereby, reducing the article's porosity and its tendency to absorb water. (Soni *et al.*, 2015).

The general increase in water absorption for all samples as firing temperature rises from 1300°C to 1400°C may be due to bloating which takes place as a result of the expansion of gases enclosed in the pores; this causes the increase in porosity (Kimambo *et al.*, 2014; Hettiarachchil *et al.*, 2014). In addition, the high firing temperature can cause mullite crystals to become coarse and decrease densification and hence increase water absorption (Kimambo *et al.*, 2014).

Ceramic tiles, characterized by low water absorption between 3.3% and 11.1% is the most important physical property for ceramic tiles mostly used as floor tiles (Amoros *et al.*, 2007; Bryne, 2008; ISO 10545, 1996). According to technical standards, the tiles with water absorption higher than 10% can be used as wall tiles and the majority of standard wall tiles have glazed porous bodies with water absorption between 10% and 20% (Kimambo *et al.*, 2014). Thus, the tiles produced in this study (with water absorption in the range 15.02–10.43%) are suitable wall tiles.

Figure 2 shows that only the ceramic samples “E” and “F” fired at temperature 1300°C have water absorption 10.43% and 10.88% respectively which falls within the ISO standard and 15.02% been the maximum recorded for sample D fired at 1200°C. Meanwhile Idowu (2014) recorded a much higher water absorption of 20% with ceramic tiles produced from clay, silica sand and feldspar collected from Ifon, Igbokoda and Ijero respectively while Mathew and Fatile (2014) recorded a much better water absorption of between 0.2% to 0.38% with material collected from Ijero, Ajaokuta and Okpella. Ogundare *et al.*, (2015) alighted a similar result (water absorption: 0.9%– 3.9%) with porcelain tiles produced materials collected from Ijero and Okpella. The water absorption of 5.61% to 17.12% was recorded in the study of Chukwudi *et al.*, (2012) while Soni *et al.*, (2015) recorded 0.9% to 5.9% in its study. El-Nouhy (2013) collected 14 different ceramic tile samples produced in Egypt and found all the samples to have water absorption ranging from 8.5% to 16.0%.

### — Apparent porosity

The results in Figure 3 show that the behaviour of apparent porosity is similar to water absorption (Soni *et al.*, 2015) as they both generally decreased with increased temperature and later increased with continued increase in temperature. The reduced apparent porosity with increased temperature could also be due to the liquid phase formation at high temperature and densification as the sample cools to room temperature (Alves *et al.*, 2015; Kimambo *et al.*, 2014) as explained for water absorption.

Figure 3 shows the minimum and maximum apparent porosity recorded as 22.77% for sample E fired at 1300°C and 30.20% for sample D fired at 1200°C respectively. The result is similar to the findings of Chukwudi *et al.*, (2012); Ogundare *et al.*, (2015); and Soni *et al.*, (2015) which recording apparent

porosity of 11.29% to 31.32%; 11% to 16%; and 5% to 26% for samples fired at 1200°C; 1250°C; and 1350°C respectively. The apparent porosity established by El-Nouhy (2013) in his study ranged from 17.5% to 27.5%.

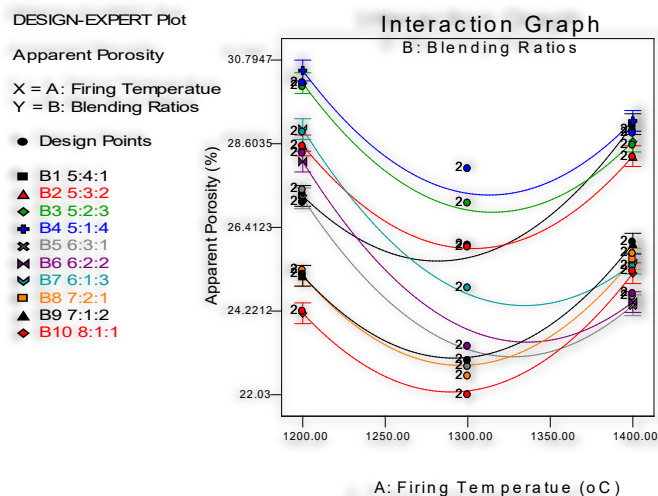


Figure 3: Apparent porosity of the ceramic tile samples fired at different temperatures. The values of the apparent porosity of all the ceramic samples were found to decrease with an increase in firing temperature and later increase with further increase in temperature as shown in Figure 3. The increase in apparent porosity at higher firing temperature is believed to be caused by bloating which takes place as gas is expelled from the matrixes, thereby resulting in increased apparent porosity (Kimambo *et al.*, 2014; Matin-Marquez *et al.*, 2008). The increase in the apparent porosity with further increase in firing temperature from 1300°C to 1400°C may be due to increase in fluxing oxides, Na<sub>2</sub>O and K<sub>2</sub>O and feldspar content in the tiles (Kimambo *et al.*, 2014; El Nouhy, 2013; Matin-Marquez *et al.*, 2008).

#### — Apparent relative density

The apparent relative density for samples A, E, G, H, I and J shown in Figure 4 increased as firing temperature increases.

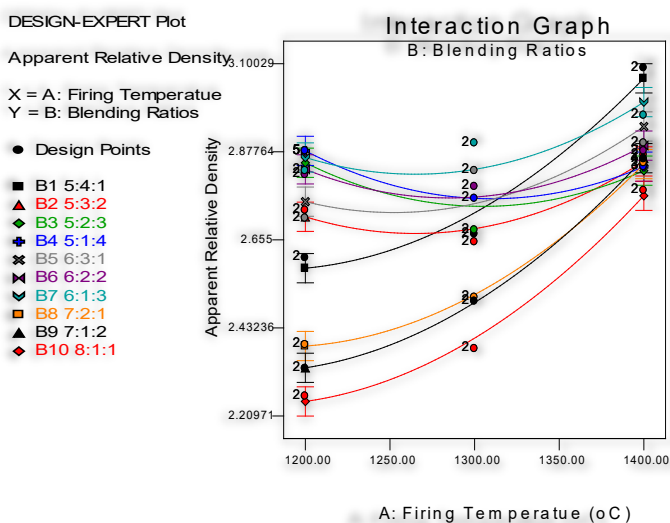


Figure 4: Apparent relative density of the ceramic tile samples fired at different temperatures

This is similar to the submission of Choudhury *et al.*, (2012) and Zabotto *et al.*, (2012). It could be seen with the samples

that feldspar has a direct relationship with the apparent relative density as increased feldspar result in increased apparent relative density. This is in line with the result of Chao *et al.*, (2010) and could be due to bloating in the matrixes. The increase in the apparent relative density of the ceramic samples with increased firing temperature could also be an indication that cation concentration is increased as increased cation within the matrix will cause increase apparent relative density (Zabotto *et al.*, 2012).

#### — Bulk density

The bulk density of ceramic samples reduced with increased firing temperature as shown in Figure 5. The lower bulk density at higher firing temperature may be attributed to bloating which takes place as gasses are expelled from the matrixes to cause the increase in porosity (Kimambo *et al.*, 2014). Sample D fired at 1400°C has the least bulk density when compared to the rest of the samples. This is because the bulk density of any ceramic article decreases with a decrease in the feldspar content. The feldspar which is a fluxing oxide helps in the production of liquid phases which fill the pores and increase the bulk density of the ceramic body (El Nouhy, 2013). The bulk density of the ceramic body attains its maximum level when the available liquid phase is enough to block the open pores (Kimambo *et al.*, 2014).

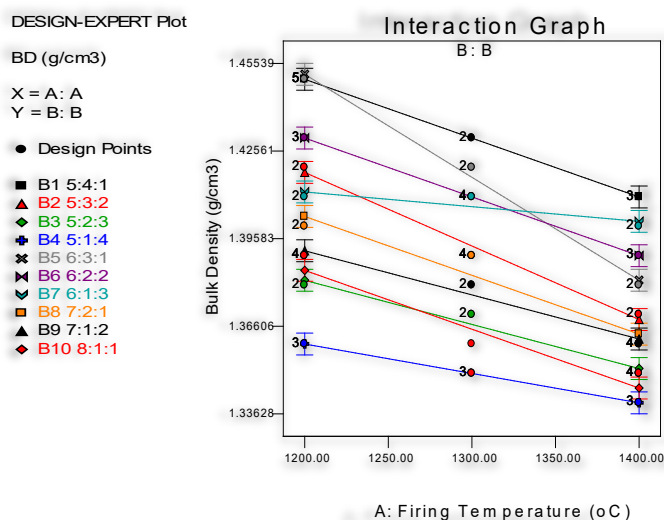


Figure 5: Bulk density of the ceramic tile samples fired at different temperatures

#### CONCLUSION

There is appreciation in the physical 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, the ceramic sample will attain the best physical properties at about 1320°C (as can be deduced from the Figures).

The ceramic sample produced from Osun State raw materials in this study will be suitable for wall tiles since the water absorption is between 10% and 15%.

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