

# Mechanical and Physicochemical Evaluation of Alkaleri Fireclay for Refractory Application

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**Abstract.** The mechanical, chemical and physical property of Alkaleri fireclay was investigated for its appropriateness for refractory application. The chemical composition was performed and analyzed using the X-ray fluorescence (XRF) spectrometer Bench top XRF analyzer technique. Chemical property result indicated that the clay contained 67.4 % silica (SiO<sub>2</sub>), 30.06 % aluminum (Al<sub>2</sub>O<sub>3</sub>), and other impurities. The clay was subjected to mechanical activation through sintering process at varied sintering temperatures of 900 °C, 1000 °C, 1100 °C and 1200 °C. At the best sintering temperature of 1200 °C, the cold crushing strength (CCS) was 17.82 MPa, in the physical properties; apparent porosity was 22.8 %, bulk density was 1.8 g/cm<sup>3</sup>, and firing shrinkage was 8.9 %. The Alkaleri clay belongs to alumino-silicate fireclay group and therefore, suitable materials for refractory application of ladle, kiln dryer, boilers, cook stoves, furnace lining and bricks.

**Keywords:** Fireclay; sintering temperatures; refractory; mechanical; strength.

## Introduction

Clay as mineral that consist of silica (SiO<sub>2</sub>), Alumina (Al<sub>2</sub>O<sub>3</sub>), water (H<sub>2</sub>O), and other impurities are alumino-silicate which mostly is responsible for its thermal property of refractoriness that finds its application in the manufacture of refractory products [1, 2]. Clay is a mineral material that possesses grain size of less than 2 micrometers and is used for structural features [3]. Clay is earthen and soil although with intricate inorganic blend, whose structure diverges generally depending on the ecological and geographical position. It is a natural material occurring in unlimited richness in nature; it is formed as a result of rock weathering on the earth's surface [3, 4].

Mechanical method of materials treatment through sintering process was performed in order to attain particular desired parameters and distinctiveness of Alkaleri clay properties was performed at varied sintering temperatures [5, 6]. The application of clay as a refractory material

depends severally on its thermal property of refractoriness, chemical composition, mechanical and physical properties [7]. These properties are responsible for its numerous structural engineering materials in the arena of advanced ceramics and refractory materials [8, 9, 10]. The county Nigeria is endowed with vast land, lucrative solid minerals with rich and plentiful clays. It is available in commercial quantity but they remained untapped and under-utilized in the country [11]. Nearly all the refractory bricks used in the metallurgical and ceramic manufacturing industry in Nigeria are imported [12].

## Materials and Methods

Alkaleri fireclay was used in this study. It was collected from Alkaleri local government area of Bauchi state, North-East geo-political zone of Nigeria. The collection and transportation of the clay sample was performed according to ASTM D4220/D4220M-14 [13].

*Chemical property analysis.* Chemical composition (XRF) of Alkaleri fireclay was determined using x-ray fluorescence spectrometer Bench top XRF analyzer, model X-Supreme 8000 branded; Oxford instrument.

*Sample preparation.* The clay sample was ball milled into a fine powder particles and the powder was compacted using a metal mould. It was press the clay substance into pallets using caver hydraulic pressing machine. The force applied was 5 KN with a holding time of 60 seconds.

*Sintering process.* The clay sample was subjected to mechanical activation by sintering process. The compacted samples were transferred into the furnace and sintered for 8 hours with a heating rate of 2.5 °C/min, and a soaking time of 2 hours at varied sintering temperatures of 900 °C, 1000 °C, 1100 °C and 1200 °C

*Physical property. Apparent porosity.* Apparent porosity Alkaleri fireclay was determined using 5 pieces of prepared test sample of size 30 mm diameter by 4 mm thickness. The compacted test sample was put in the furnace and sintered as specified in the test samples preparation above. After cooling, it was brought out of the furnace, weighed and recorded as weight  $D$ . The clay sample was suspended in distil water for 30 minutes and weighed as weight  $W$  and recorded. The clay sample was removed from water and the water on the surface of the specimen was cleaned off using filter paper and weighed as soaked weight  $S$  and recorded. The apparent porosity experimental procedure was achieved according to ASTM C20-2000 [14]. Apparent porosity ( $AP$ ) was calculated from Equation (1):

$$AP = \frac{W - D}{W - S} \times 100, \quad (1)$$

where  $AP$  – Apparent porosity, %;

$W$  – Suspended weight, g;

$D$  – Dry weight, g;

$S$  – Soaked weight, g.

*Physical property. Bulk density.* The Alkaleri fireclay bulk density was determined using 5 pieces of prepared test sample which was initially made into standard brick of size 100 mm x 50 mm x 20 mm. The compacted test sample was put in the furnace and sintered as mentioned in test sam-

ples preparation above. After cooling, the sintered standard brick was further cut into millimeter cubic sizes of 20 × 20 × 20 using metal hacksaw blade. The sample was weighed on a digital balance and the weights were recorded as dried weight  $W_1$  (g). Mercury was weighed 1000 g in a beaker and the volume taken as  $V_1$  (cm<sup>3</sup>). A rope was used to tie clay sample at the upper arm of the tecramics densometer and suspended the clay sample above the mercury. The pointer attached to the densometer pushed the suspended specimen to be submerged and soaked 5 minutes in the mercury by displacing the mercury and weight was taken as  $V_2$  (cm<sup>3</sup>). The experimental procedure was according to ASTM C20-2000 [14].

The bulk density was calculated from the Equation (2):

$$BD = \frac{W_1}{V_1 - V_2}, \quad (2)$$

where  $BD$  – bulk density, g/cm<sup>3</sup>

$W_1$  – Dried weight, g;

$V_1$  – Soaked weight, cm<sup>3</sup>;

$V_2$  – Suspended weight, cm<sup>3</sup>.

*Physical property. Firing shrinkage.* In the firing shrinkage preparation, 5 pieces of test sample of dimension 100 mm × 20 mm × 50 mm was produced to standard brick size as required by the test. The length of the standard brick unfired (green) was measured and taken as dried green length ( $L_1$ ). The sintered Alkaleri fireclay brick's length was measures using vainer caliper and recorded as fired length ( $L_2$ ). The experiment was determined according to ASTM C179-14 [14].

The firing shrinkage was calculated as percentage of original green length as indicated in the Equation (3):

$$FS = \frac{L_1 - L_2}{L_1} \times 100, \quad (3)$$

where  $FS$  – Firing shrinkage, %;

$L_1$  – Dried green length, cm;

$L_2$  – Fired length, cm.

**Mechanical Property.** The Cold crushing strength (CCS) of Alkaleri fireclay was performed by preparing 5 pieces of the test samples to standard brick of size 20 mm × 50 mm × 100 mm. The sintered specimen was further cut and reduced to sizes of 20 mm × 20 mm × 20 mm. These cubic shaped sizes were used to test for cold crushing strength using the universal strength testing machine. The failure of compressive strength of the specimen is suggestive of its mechanical property and its behavior likely under load. The application of a uniform load on it was followed. The CCS was determined according to ASTM C133-97 [15]. The weight pressure at which crack was discovered on the clay sample was detected and the cold crushing strength was calculated as in Equation (4):

$$CCS = \frac{LD}{SSA}, \quad (4)$$

where *CCS* – Cold crushing strength, KN/m<sup>2</sup>;  
*LD* – Load to fracture, KN;  
*SSA* – Sample Surface Area, m<sup>2</sup>.

**Results and Discussion**

**Chemical composition.** In the chemical composition result as presented in Table 1 showed that the dominant oxides in the clay are SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> with 2.56 % of impurities and can be categorized as alumino-silicate fireclay brick. The Alkaleri clay is in the range of chemical composition of 65–80 % SiO<sub>2</sub> and 18–30 % Al<sub>2</sub>O<sub>3</sub> which are classified as high duty (Siliceous) refractory fireclay brick [2, 6, 12]. The Gombe clay is suitable for production of furnace linings (Table 1).

Table 1 – Chemical property of Alkaleri clay

| Oxides                         | %     | Oxides                        | %    |
|--------------------------------|-------|-------------------------------|------|
| Al <sub>2</sub> O <sub>3</sub> | 29.06 | K <sub>2</sub> O              | 0.14 |
| SiO <sub>2</sub>               | 66.02 | P <sub>2</sub> O <sub>5</sub> | 0.28 |
| Fe <sub>2</sub> O <sub>3</sub> | 1.02  | SO <sub>3</sub>               | 0.25 |
| TiO <sub>2</sub>               | 0.34  | CaO                           | 0.13 |
| MgO                            | 0.20  |                               |      |

**Particle size.** The Alkaleri fireclay has fine powder as shown in the particle size analysis result in Figure 1 in which the cumulative particle sizes falls within 0.03–12 µm.

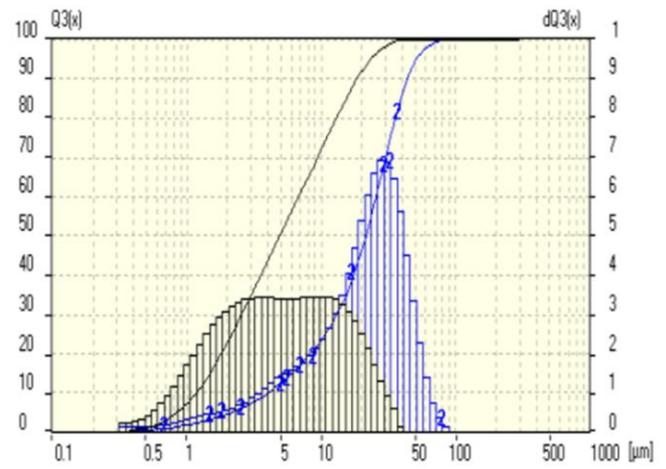


Figure 1 – Particle size of Alkaleri clay

The particle size percentile was 4.4 %. The particle size of Alkaleri clay contributed to the mechanical strength of the fireclay because the smaller the particle sizes the more the bonding of the structural clay [2].

**Apparent porosity.** The clay at the lowest sintering temperature of 900 °C had the highest porosity value of 42.77 % as compared with at the maximum sintering temperature of 1200 °C with the lowest value of 22.8 % as sketchily revealed in Figure 2.

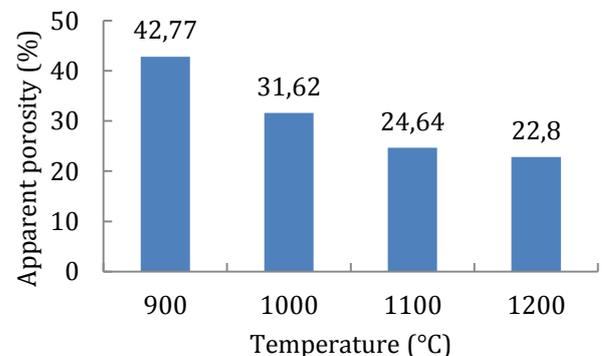


Figure 2 – Porosity of Alkaleri clay at varied sintering temperatures

It was observed that as the temperatures of sintering were raised the porosity of the clay reduced. The sintering temperature influenced the close up of the pore spaces and thereby increased strength of the fireclay brick. The Alkaleri clay apparent porosity fell within the standard value of 20-30 % porosity for fireclay refractory bricks [6, 7, 12].

Hence, the best sintering temperature was at 1200 °C. The result showed a typical characteristic of clay when exposed to extreme thermal treatment like the sintering process. The low porosity of the refractory fireclay brick make it suitable for its use for production of furnace linings [12].

**Bulk density.** The bulk density Alkaleri fireclay has a relationship with its particle sizes and sintering temperatures. The sample had the least bulk density value of 1.65 g/cm<sup>3</sup> at 900 °C sintering temperature as against the maximum sintering temperature at 1200 °C with highest value of 1.8 g/cm<sup>3</sup> as presented in Figure 3.

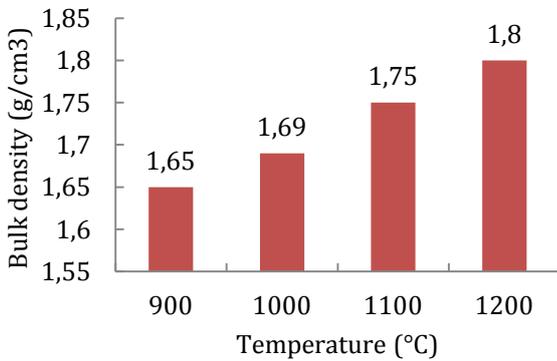


Figure 3 – Bulk density of Alkaleri fireclay at varied sintering temperatures

The bulk density increased as the sintering temperatures were increased. The extreme sintering temperature of 1200 °C was optimal and best. It is essential to note that bulk density as a clay property is a major criterion in material selection for refractory production. The bulk density of Alkaleri fireclay fell within the standard values of 1.7-2.3 g/cm<sup>3</sup> for fireclay refractory brick [6, 7, 12].

**Firing shrinkage.** Firing shrinkage of Alkaleri fireclay was 8.2 % at the lowest sintering temperature of 900 °C as compared with at 1200 °C the maximum sintering temperature with the highest shrinkage value of 8.9 % as revealed in Figure 4.

It can be concluded as showed in the result that as the temperatures of sintering were increased the shrinkage increased. The firing shrinkage of Alkaleri fireclay fell within the standard values of 7-10 % for fireclay refractory brick [6].

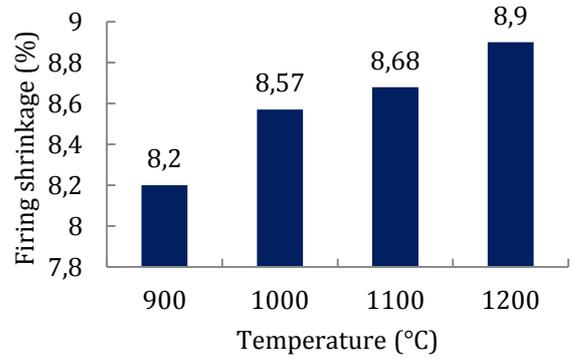


Figure 4 – Firing shrinkage for Alkaleri clay at varied sintering temperatures

**Cold crushing strength (CCS).** The Alkaleri fireclay at the minimum sintering temperature of 900 °C had the lowest cold crushing strength of 7.75 MPa as against the highest value of 17.82 MPa at the maximum sintering temperature of 1200 °C as graphically presented in Figure 5.

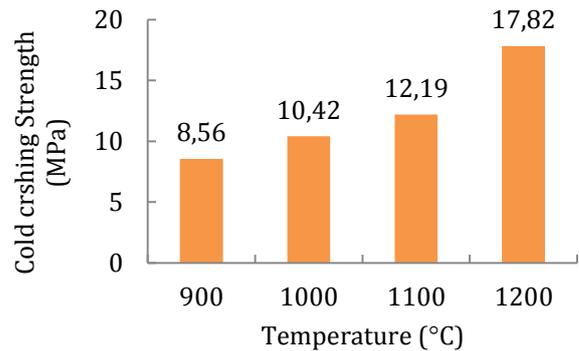


Figure 5 – Cold crushing strength of Alkaleri clay at varied sintering temperatures

The best sintering temperature was at 1200 °C. The effects of sintering on the clay indicated that as temperatures of sintering were increased the CCS increased. The increase in strength can be attributed to the amount of 36.4 % Al<sub>2</sub>O<sub>3</sub> in the Alkaleri fireclay. The strength of the clay fell within the standard values of 15–59 MPa for fireclay refractory brick and therefore, suitable for the production of fireclay refractory brick [6, 12].

**Conclusion**

The optimum sintering temperature was at 1200 °C; the optimum values of porosity was 24.52 %, the optimal value for CCS was

15.37 MPa, firing shrinkage was 8.9 % and 1.8 g/cm<sup>3</sup> was for bulk density. It was observed that the cold crushing strength, firing shrinkage, bulk density improved by the reason of mechanical activation through the sintering process and the varied sintering temperatures. There was reduction in percentage porosity as sintering temperatures were increased. This attribution is an indication that the sintering temperature influenced the final product development from the raw clay material.

Hence, the Alkalari clay is suitable for manufacture of fireclay for refractory application.

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