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Influence of Grog Size on the Performance of NSU Clay-Based Dense Refractory Bricks

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Abstract The suitability of using local kaolin (Nsu clay) and Nsu clay grog to enhance efficiency (reduce shrinkage, improve abrasion and reduce porosity) in the production of dense refractory bricks was studied. The chemical analysis, crystal structure examination and microstructural analysis were determined using the atomic absorption spectrophotometer (AAS), x-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively. The physical properties in terms of refractoriness, linear shrinkage, bulk density and apparent porosity as well as mechanical properties in terms of cold crushing strength (CCS) were carried out using American Society for Testing and Material (ASTM) stipulated standard methods. Test specimens (sample A = 20, sample B = 30 and sample C = 40 % grog sizes) were prepared and tested using the standard methods. The overall chemical and structural analysis of the raw Nsu clay showed that it is rich in SiO_2 (59.20 wt. %) and Al_2O_3 (26.30 wt. %) with trace amounts of MgO, Fe_2O_3 and K_2O, hence an alumino-silicate clay. The refractory properties measured showed acceptable and efficient results. Maximum apparent porosity (20.22 %) and CCS (61.77 MPa) were obtained at sample B = 30 % grog size.

Keywords: *Kaolin, Grog, Dense refractory, X-ray diffraction, Alumino-silicate*

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1. Introduction

Clay is a plastic material composed mainly of fine-grained minerals which hardens by losing the contained water during drying or firing [1,2]. It is also defined as a complex alumina-silicate compound containing attached water molecules [3]. Clay is widely used for the production of refractories and a large variety of items such as tiles, sewage pipes, sanitary wares, and porcelain.

Refractories (refractory ceramics) are very important in modern industries due to their capacity to withstand high temperatures, ability to react with the environment without melting, inert, possession of reversible thermal expansion and resistance to thermal shocks [4]. Refractories are non-metallic materials typically composed of silicon and aluminium oxides with the functional ability to withstand both physical and chemical wear, possess high melting temperatures and maintain their structural properties at very high temperatures (> 1000°C) [5,6,7,8]. They are employed in the metallurgical, glassmaking, cement and ceramic industries where they are used for interior linings for furnaces, kilns, reactors and other devices that process materials at very high temperatures [9,10].

The linings of high temperature furnace or kilns are referred to as refractory bricks or simply refractories [11]. The inner linings are known as dense refractory bricks and are exposed to the highest temperature because they are in direct contact with the contents of the furnace or kiln. These contents consist of molten metal, slag, corrosive (high velocity gases) and fluidized particles. In view of this, grog or calcined clay is added to the refractory brick mix to enhance drying performance, reduce shrinkage, improve fired abrasion resistance, reduce density and provide stability in applications [12,13]. Grog has particle sizes ranging from fine to coarse.

This research is aimed at studying the effect of grog on the performance of dense refractory bricks using Nsu clay as a source of kaolin. Nsu clay is named after a community, Agbahara Nsu in Ehime-Mbano local government area of Imo state, Nigeria. The Nsu clay is presumed to be a kaolinite clay deposited in Nsu community in commercial quantity.

Kaolinite (kaolin in pure form) is a white clay mineral classified as 1:1 type layer silicates. The structural classification is due to its upper layer (gibbsite layer) been linked to the lower layer [14,15]. The upper layer is the octahedral sheet which is composed of alumina (Al, O, OH) while the lower layer is the tetrahedral sheet composed of silica (Si, O, OH). Diagrammatic structure of kaolinite is represented in Figure 1. It is a non-expanding mineral, and therefore is unable to absorb water into the interlayer position. This makes kaolinite to swell on wetting and shrinks on drying [15]. In pure form, kaolinite has a melting point of 1770°C, and a melting point between 1200 and 1450°C in a clay form due to the presence of highly fluxed feldspar [14]. Kaolinite has numerous industrial uses as shown in Table 1.

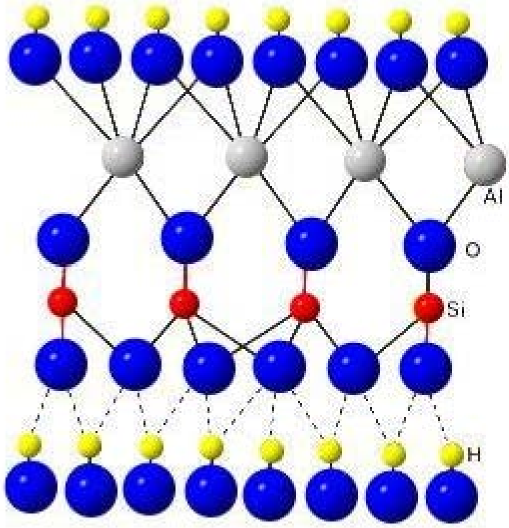

Figure 1. Diagrammatic representation of kaolinite structure [14]

Table 1. Industrial applications of kaolin [16,17]

Industry	Level of application (%)
Paper	45
Refractories and ceramics	31
Fiber glass	6
Cement	6
Rubber and plastic	5
Paint	3
Others	4

The X-ray diffraction (XRD) pattern of kaolin (Figure 2) shows that kaolin powder consists basically of kaolinite, quartz and a trace amount of illite phase [18].

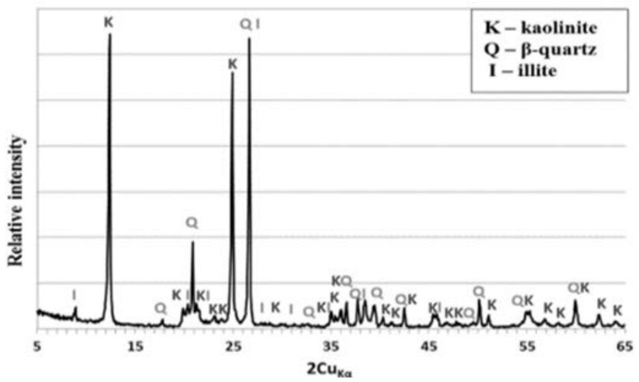

Figure 2. The XRD pattern of kaolin powder [18]

2. Experimental Work

2.1. Raw Materials and Sample Preparation

The materials used in this work are Nsu clay (as a source of kaolin), Nsu grog and water. The grog was produced by firing some quantity of the Nsu clay at 1000°C for 1 h. The process is called calcination. The calcined clay was allowed to cool, thereafter ground using a pan mill and sieved with mesh 100 (< 150 μm) to the required size.

The different batch mixture of Nsu clay and grog (as shown in Table 2) with ~ 100 ml of water was allowed to age for 24 hours (to achieve a workable mix), and then manually pressed in a wooden brick mould of dimension, 13 x 7 x 5 cm. The green pressed bricks were dried for 4 days and 3 days at 110°C in air and in electric drying cabinet respectively. After the drying process, the bricks were sintered in the kiln at 1250°C for 2 hours and allowed to cool.

Table 2. Batch Composition of Nsu Clay and Grog. (Total weight = 2000 g)

Batch	Nsu clay		Grog	
	%	G	%	g
A	80	1600	20	400
B	70	1400	30	600
C	60	1200	40	800

The chemical analysis of the raw Nsu clay was determined using atomic absorption spectrophotometer, AAS (AA320N) at research centre, Caritas University, Enugu, Nigeria.

2.2. X-ray Diffraction (XRD)

The crystal structure or mineralogical phase of Nsu clay was analyzed. In this work, the XRD of Nsu clay (same as the one used in this work) as reported by Chukwudi and Uche [19] was adopted.

2.3. Scanning Electron Microscopy (SEM)

The grain morphology and microstructure of Nsu clay were determined using electron scanning microscope (JEOL JSM-3SC SEM) operated at 15 KV. The analyzed result was adopted from previous literature [20].

2.4. Refractory Tests

2.4.1. Refractoriness

Refractoriness is the ability of a refractory material to withstand high temperature in service. In view of this, the refractoriness of Nsu clay was determined using Shuen's formula [21].

$$K = \frac{360 + Al_2O_3 - RO}{0.228} \quad (1)$$

Where, K is the refractoriness (°C), Al_2O_3 is the amount of alumina in the clay (%), RO is the sum of other oxides in the clay except SiO_2 and Al_2O_3 (%), 360 and 0.228 are constants.

2.4.2. Linear Shrinkage Test

Shrinkage lines (10 cm length) were marked on the surfaces of the test specimens. The 10 cm length is noted as the original length. Test specimens from each batch were dried in the drying cabinet for 3 days at 110°C to ensure the total water loss. After drying, the test specimens were sintered at 1250°C for 2 hours and then allowed to cool. In other to minimize the errors involved, three different test specimens of the same batch composition were tested and the average values evaluated. The drying, firing and total shrinkage were calculated for each test specimen using standard formula [22].

$$\%\text{average drying shrinkage} = \frac{O_L - D_L}{O_L} \quad (2)$$

$$\%\text{average firing shrikage} = \frac{D_L - F_L}{F_L} \quad (3)$$

$$\%\text{average total shrinkage} = \frac{O_L - F_L}{O_L} \quad (4)$$

Where, O_L is the original length, D_L is the dry length and F_L is the fired length.

2.4.3. Bulk Density and Apparent Porosity Tests

The bulk density of a porous solid such as ceramics is defined in terms of its mass or weight relative to apparent volume, i.e.

$$\text{Bulk density}(BD) = \frac{\text{Weight}}{\text{Apparent volume}} \quad (5)$$

Where, the apparent volume is the envelope volume of the porous solid, and it includes the volume of the solid component, open and sealed pores.

BD can be determined using the following techniques [23]:

i. Physical measurement method
ii. Mercury displacement method
iii. Soaking-immersion method (Boiling method)

In this work, the physical measurement method was used to calculate the bulk density of the samples. The fired test specimen was weighed in air and the value recorded as W_1 (g). The apparent volume of the test sample was determined by measuring its dimensions (length, l, width, w and thickness, t) using Vernier callipers. BD (g/cm^3). Hence, it is evaluated using the following formula:

$$BD = \frac{W_1}{AV} \quad (6)$$

Where, AV is the apparent volume (l w t).

Apparent porosity, AP is based on the fact that a fired ceramic product will absorb more water when boiled than when soaked in a cold water. Therefore, the AP test used in this work is the boiling test method.

Test samples from each of the ceramic compositions were dried for 72 hours in a drying cabinet at 110°C. The dried samples were sintered at 1250°C for 2 hours and then allowed to cool. These samples were then immersed completely in water and boiled for 5 hours. After boiling, the samples were allowed to cool in the water, thereafter removed and cleaned using a clean damp cloth. The weight of the soaked sample was measured and recorded as W_2 (g). Finally, AP (%) was calculated using equation 7.

$$AP = \frac{W_2 - W_1}{W_1} * 100 \quad (7)$$

2.4.4. Cold Crushing Strength (CCS) Test

Refractories in service are required to withstand the load accruing from the furnace, its contents and the induced stress due to the temperature change. Therefore, the determination of CSS of refractories is very important. The test brick samples were dried and sintered as usual (110°C for 72 hours, and 1250°C for 2 hours, respectively). The sintered samples were mounted in turn on the saddle of a compressive strength testing machine (Seidner, 7940 Rieldling). A force was axially applied to the test sample at a uniform rate until the sample ruptures. The force at which the sample failed was noted. This represents the load required for determining the CSS of the test sample. The CSS was calculated using the following equation:

$$CSS = \frac{F}{A} \quad (8)$$

Where, F is the applied force (KN) and A is the cross sectional of the test sample (m^2)

3. Results and Discussion

3.1. Chemical Composition

Chemical composition of the Nsu clay in terms of oxides is shown in Table 3.

Table 3. Chemical composition of Nsu clay

Composition	Weight (%)
SiO_2	59.20
Al_2O_3	26.30
MgO	0.62
Fe_2O_3	2.01
K_2O	0.20
Loss on ignition (LOI)	12.71
Al_2O_3:SiO_2	0.44

As shown in Table 3, the Nsu clay has a high content of alumina (26.3 %) and silica (59.2 %), hence it can be classified as an alumino-silicate type of clay. It has been reported that the major refractory clay deposits in Nigeria are kaolinitic and fireclay in nature with less than 45 wt. % alumina [24,25]. Therefore, alumina content of Nsu clay agreed with this report.

3.2. XRD Patterns and SEM Images

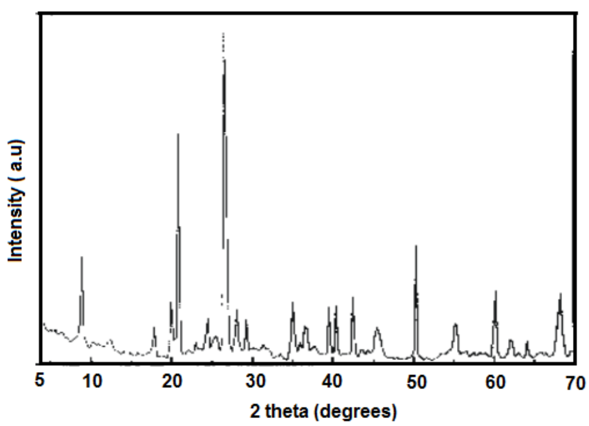

Figure 3. XRD pattern of Nsu Clay [19]

The XRD patterns of the Nsu clay is shown in Figure 3. The XRD pattern exhibits ordered, narrow and intense diffraction peaks with the first peak (100) at ~ 9^o (2θ). Grim [26] reported that in a poor crystalline clay, the first order spacing (d_{100}) occurs around 10^o (2θ). This confirms that the Nsu clay is a poor crystalline clay.

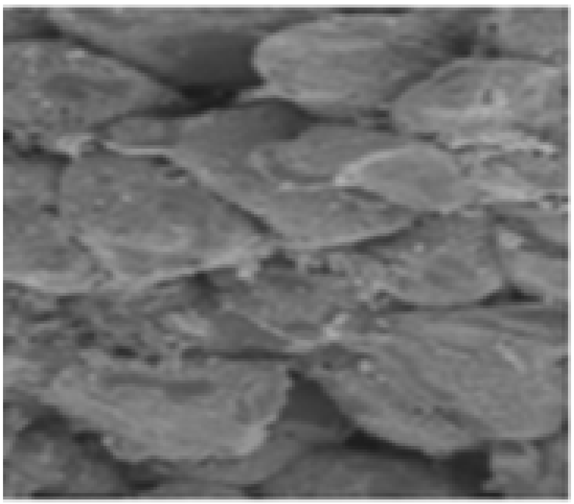

Figure 4. SEM Micrograph of Nsu Clay [20]

Figure 4 shows the SEM micrograph of the Nsu clay. The clay sample has a homogenous microstructure with small grains. The edges of the grains are irregular with flake (thin fragment) surface dimension (~3 μ). The grain size is within reported size in the literature [20,26].

3.3. Refractoriness

Table 4 represents the refractoriness of the Nsu clay sample. The refractoriness occurred at a high temperature of 1682°C which falls within the pyrometric Segar cone of 31. The high Al_2O_3 content (26.3%) of the Nsu clay is a contributor to its high refractoriness. This agrees with a reported literature [3] that the used temperature raises as the alumina content increases. The high refractoriness is also attributed in part to the absence of manganese oxide in the clay.

Table 4. Refractoriness of Nsu clay sample

Details	Sample
Segar Cone No (UK)	31
Temperature (°C)	1682

3.4. Grog size and the Refractory Properties relationship

The results of the refractory properties of the Nsu clay dense bricks are presented in Table 5.

Table 5. Refractory Properties of Nsu Clay Bricks

Sample	Grog size (%)	Linear Shrinkage (%)			Bulk Density (g/cm^3)	Apparent Porosity (%)	Cold Crushing Strength (MPa)
		Dry	Fired	Total			
A	20	4.00	8.33	12.00	1.87	18.66	49.24
B	30	2.00	9.18	11.00	1.80	20.22	61.77
C	40	2.00	9.18	11.00	1.76	19.64	55.15

3.4.1. Linear Shrinkage

The linear shrinkage results are shown in Table 5. Figure 5 presents the grog size dependence of the linear shrinkage. The results indicate that the dry and total linear shrinkage values initially decreased with the increase in grog size and finally remained constant with further increase of the grog size. This is attributed in part to the non-plastic nature of the grog. The linear fired shrinkage exhibited the reverse, where it increased and then remained constant with the increase in grog size.

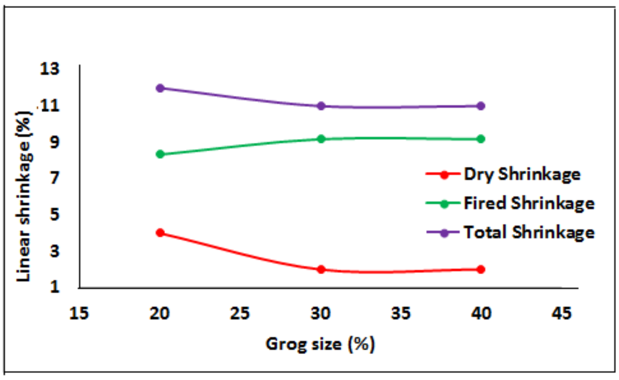

Figure 5. Grog size dependence of Linear Shrinkage

All values of the linear fired shrinkage (8.33 - 9.18 %) for the samples (A, B and C) fall within the recommended standard linear shrinkage range (7-10 %) for refractory bricks [27]. The linear drying shrinkage (2-4 %) and total linear shrinkage values (11-12 %) fell outside the recommended range of values. The obtained low linear drying shrinkage values are desirable since high shrinkage values cause warping and cracking of the bricks with a subsequent loss of heat in the furnace.

3.4.2. Bulk Density

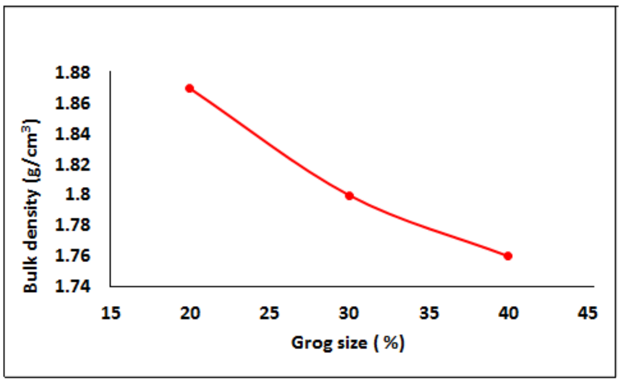

Figure 6. Grog size dependence of Bulk Density

The results of the bulk density are shown in Table 5, while Figure 6 shows the relationship between the grog size and bulk density of the refractory brick samples. As the size of grog increased, the bulk density decreased.

Sample A (20 % grog size) shows the highest bulk density (1.87 g/cm^3). This can be attributed to the lowest percentage apparent porosity shown by the sample.

All bulk density values (1.87, 1.80 and 1.76 g/cm^3) are within the recommended standard range (1.08-1.97 g/cm^3). Therefore, they are very suitable for siliceous fireclays [28] and/or fireclays [29].

3.4.3. Apparent Porosity

Apparent porosity results are shown in Table 5. Figure 7 illustrates the grog size dependence of the apparent porosity of the refractory brick samples. Sample B (30 % grog size) shows the highest porosity of 20.22 % against 19.64 % for sample C (40 % grog size) and 18.66 % for sample A (20 % grog size) , respectively. The lowest apparent porosity of sampl A is due to its highest bulk density (1.87 g/cm^3). The higher the porosity of refractory clay material, the higher and lower the insulating properties and thermal conductivity , respectively.

All samples show apparent porosity values that are within the suggested acceptable range (10-30 %) for refractory clays [27].

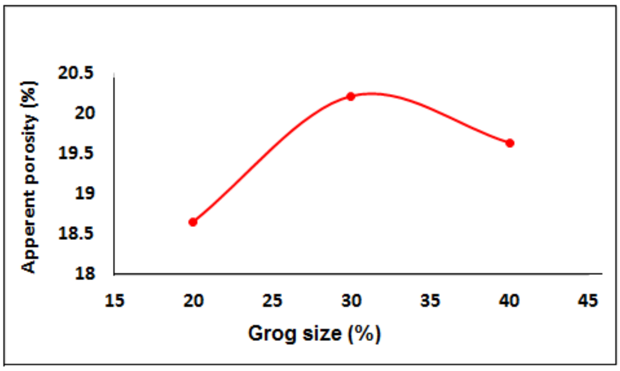

Figure 7. Grog size dependence of Apparent Porosity

3.4.4. Cold Crushing Strength (CCS)

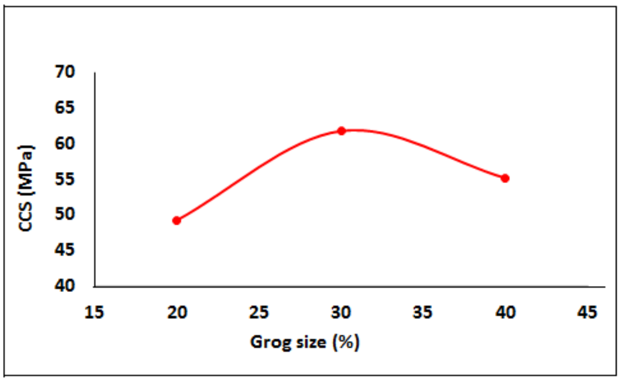

Figure 8. Grog size dependence of Cold Crushing Strength (CCS)

Results of the cold crushing strength are presented in Table 5. The relationship between the size of grog and the CCS is illustrated in Figure 8. The results showed that as the grog size increased, CCS initially increased and finally decreased with the increase in grog size. This trend is attributed in part to the initial increase and final decrease in the apparent porosity of the samples as the grog size increases. The highest CCS (61.77 MPa) is observed in sample B (30% grog size), followed by 55.15 and 49.24 MPa for samples C (40% grog size) and A (20 % grog size), respectively. The high compression strength shown by all samples indicates a load bearing capacity at low temperatures and an ability to withstand abrasion. The CCS values values obtained conform with the standard values of refractory clays as reported in the literature [27]

4. Conclusions

The effect of grog size on the performance of dense refractory bricks made from Nsu clay (kaolin) was carried out. As the percentage of grog increased, the linear shrinkage (dry and total) and the bulk density decreased. Furthermore, the optimal apparent porosity (20.22 %) and cold crushing strength (61.77 MPa) values were obtained in 30 % grog size (Sample B). A further increase of the grog size more than 30 % produced no change in the linear shrinkage but a reduction in bulk density, apparent porosity and CCS, respectively.

It is therefore suggested that in the production of refractory bricks using Nsu clay, 30 -40 % grog size should be applied for a high efficiency.

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