Adaptation of Odolewu Clay for Use as Refractory Material

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ABSTRACT - This study investigated the refractory properties of Odolewu clay, varying the composition with dolomite. Spectrometric analysis was carried out on the clay and dolomite to determine their chemical compositions. The clay was then crushed, sieved and blended with dolomite in proportions ranging from 0-30 wt.%. Tests for thermal shock resistance, cold crushing strength, percentage linear shrinkage, bulk density, apparent porosity, permeability and refractoriness were carried out on each composition. The results obtained showed that the cold crushing strength, bulk density, permeability and refractoriness decreased while the apparent porosity of the samples increased with increase in dolomite content. Also, the linear shrinkage for clay-dolomite mixtures with 5 wt.% dolomite and 10 wt.% dolomite, thermal shock resistance for 10 wt.% dolomite and 15 wt.% dolomite, apparent porosity for samples with 10-30 wt.% dolomite, and refractoriness for all the samples are within recommended values for fireclays.It was inferred that the refractory properties of Odolewu clay especially thermal shock resistance and permeability can be improved with the addition of appropriate proportion of dolomite. This study also revealed that optimum refractory properties were obtained for clay-dolomite mixture with 10 wt.% dolomite.

(Keywords: Refractoriness; Permeability; Porosity; Fireclay; Dolomite)

1 INTRODUCTION

In Nigeria, clay is widely distributed though not always found in sufficient quantity or suitable quality for modern industrial purposes. It occurs both as residual and sedimentary clay. More than eighty clay deposits have been reported from all parts of the country [1]. The occurrence and importance of clay have been recognized since ancient times, when it was used for primitive applications like pottery, construction of mud houses, bricks, etc. [2]. While the geology of many places where clay deposits occur have been studied, relatively little efforts have been made to assess these deposits in order to determine their usability.

Refractory can be defined as any material which does not react significantly with its environment and has useful level of strength for useful period of time at high temperature [3]. Some studies have shown that some clays in Nigeria are good for bricks based on their natural water content, the natural swelling and the cold crushing strength [4], [5], [6]. Other studies have found clays in some other parts of Nigeria useful for furnace construction as a result of their higher thermal shock resistance, crushing strength, bulk density and refractoriness values [7], [8] and ceramic making [9].

The demand for refractory materials for furnace building as well as other related high temperature processes is enormous. Hassan and Adewara [5] reported that over 80 percent of the refractory materials are used by the metallurgical industries for the construction and up-keep of furnaces, reactor vessels, kilns and boilers. This is beside the fact that refractory materials are also used in non-metallurgical industries such as cement, glass and hardware industries.

The economic situation in the country has made it imperative for inward sourcing of raw materials. Some local clay deposits have been investigated with good results. Studies on the Otukpo clay in Benue State by Nnuka and Agbo [10] showed that the clay is Kaolinitic and belongs to the medium duty fire clay class of aluminium silicates. Aiyedun *et al.*[11] investigated the strength characteristics of Osiele clay, and reported that the total shrinkage, density and cold crushing strength increased as the firing temperature increased. This in an indication that Osiele clay can be effective as a refractory material with little or no additive. Also, Dukku clay deposit have been reported to be a good substitute for imported refractory materials used in our metallurgical industries for processing refractory bricks needed for lining the walls of furnaces, soaking pits, ovens, ladles, crucibles and kilns [12]. The chemical composition of a given refractory depends on the composition of the starting materials, and the physical properties of refractory bricks are a function of the physical properties of the original raw material [13].

Despite having extensive clay mineral deposits in Nigeria, Nigeria continues to depend on external sources of refractory materials for many of its industries [14]. For example, Nigeria imported about 27 million metric tonnes of refractory materials in 1987 [15]. In view of reasons above, this study investigates the refractory properties of Odolewu clay blended with varying dolomite contents to reveal its potentials for refractory industry.

2 MATERIALS AND METHODS

2.1 Preparation on Clay Sample

The clay samples were collected from Odolewu in Ogun State. Water was added to the collected samples in a container, and was thoroughly mixed by stirring using mechanical stirrer. It was then left to settle for some days. A slurry mixture of the clay sample which was left on top was collected and carefully sieved by wet sieving through a 100 μm (1 mm) sieve. The slurry mix was then left to settle leaving the clay beneath and water on top. The water was siphoned and the clay that was left was allowed to dry and collected.

The pure clay sample obtained was blended with dolomite in varying proportions ranging from 0-30 wt.% dolomite. Test pieces for various properties were then prepared according to the American foundry mans society standard. The test pieces were dried in an oven at 110 °C for about 24 hrs. They were then soaked at 1400 °C for 8 hours and furnace-cooled before the determination of the properties.

2.2 Chemical Analysis

The chemical analysis of the processed clay and dolomite was carried out using the Atomic Absorption Spectrometry (AAS).

2.3 Permeability to Air

Test samples were prepared to specifications of 50.8 mm diameter and 50.8 mm height from a standard rammer. The test pieces were air-dried for 24 hrs and then dried at 110 °C for 12 hrs in an oven. The samples were completely sealed in the sides and the lower surface was exposed to an orifice. The cylinder was filled with 2000 cm^3 of water and the bell jar was put in place. The orifice was opened and a manometer measured the time taken for 2000 cm^3 of water to displace equal volume of air through the surface. Permeability was calculated using equation (1):

$$P_A = \frac{Vh}{Apt} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where:

P_A = Permeability number

h = height of specimen (cm)

p = pressure of air in cm of water

V = volume of air (cm^3)

A = cross-sectional area of specimen (cm^2)

t = time (in minutes)

2.4 Thermal Shock Resistance

Test pieces measuring 50 mm x 75 mm were inserted in a furnace preset at 900 °C for 10 minutes. The specimens were then cooled outside the furnace for 10 minutes. The process was continued until cracks were observed in the samples. The number of heating and cooling (cycles) before cracking occurred was recorded.

2.5 Moisture Content

The test pieces were weighed and recorded as wet weight. Thereafter, they were transferred and dried in an oven set at a temperature of 110 °C for 24 hrs, removed re-weighed and their weights recorded. The drying and weighing continued until constant weight was obtained. The moisture content was calculated using equation (2):

$$\%\, moisture\ content = \frac{loss\ in\ weight}{original\ weight} \times 100 \ldots.(2)$$

2.6 Refractoriness

The test pieces were mounted on a refractory plague along with some standard cone whose melting point is slightly below that expected of the test cone. The plague was then put inside the furnace and the temperature was raised at a rate of 10 °C per minute. The temperature increase continued until a temperature is reached when the top of the test cone bent over level with the base. Thereafter, the plague bearing the test piece was removed, cooled to the room temperature and the test cone examined under the microscope.

2.7 Cold Crushing Strength

Test pieces of clay samples were prepared to a standard size of 76.2 mm cube on flat surfaces. The test pieces were fired in a furnace at 1100 °C and the temperature maintained for 6 hrs. The samples were then cooled to room temperature. The specimen was placed on compressive tester and load was applied axially by turning the hand wheel at a uniform rate till failure occurred. The manometer readings were recorded. Cold crushing strength (CCS) was calculated from the equation (3):

$$CCS = \frac{maximum\ load}{cross\ sectional\ area}\ \left(\frac{kg}{cm^2}\right) \ \ldots\ldots (3)$$

2.8 Linear Shrinkage

Test pieces measuring 60 mm × 60 mm × 15 mm were marked along a line in order to maintain the position after heat treatment. The distance between the two ends of the slab was measured with Vernier caliper. The samples were air dried for 24 hrs and oven dried at 110 °C for another 24 hrs. They were then fired at 1100 °C for 6 hrs. The test pieces were cooled to room temperature and measurements taken. The fired linear shrinkage was then calculated by equation (4):

$$Fired\ shrinkage = \frac{100\%\,(D_L - F_L)}{D_L} \quad \ldots.\ (4)$$

Where:

D_L = Dried length

F_L = Fired length

2.9 Densities and Porosity

Representative samples of each of the prepared clay-dolomite mixes were made into test specimens measuring 60 mm x 60 mm x 15 mm. The test pieces were air dried for 24 hrs, and then oven dried at 110 °C for another 24 hrs. They were then cooled in a desiccator and weighed to the accuracy of 0.001 (dry weight). The weight obtained was recorded as dried weight (D) after which it was transferred to a beaker and heated at 100 °C for 30 minutes to assist in releasing trapped air. It was then cooled to ambient temperature, soaked in water for 20 minutes and the soaked weight (W) taken. The specimen was then suspended in water using a beaker placed on a weight balance and the suspended weight(S) measured. The bulk density and apparent porosity were then calculated using equations (5) and (6) respectively.

$$Bulk\ Density = \frac{D\rho}{W-S} g/cm^3 \quad …….. (5)$$

$$Apparent\ Porosity = \frac{W-D}{W-S} \times 100\% \quad …. (6)$$

Where $\rho = Density\ of\ water$ and s = suspended weight

2.10 Loss on Ignition(LOI)

50g of each sample (clay and dolomite) was dried at 110 °C and cooled in a desiccator. Porcelain crucible was cleaned, dried and weighed (M_1) to the nearest 0.001g. The dried sample was introduced into the crucible, and the crucible together with the sample weighed (M_2) to an accuracy of 0.001g. The crucible containing the sample was placed in a muffle furnace and heated to a temperature of 900 °C for three hours. The crucible and its contents were cooled in a desiccator and then weighed to the nearest 0.001g (M_3). The LOI was calculated with the aid of equation (7):

$$LOI = \left(\frac{M_2-M_3}{M_2-M_1}\right) \times 100\%…………....(7)$$

3 RESULTS AND DISCUSSION

Table 3: The physical properties of the prepared clay–dolomite mixes

Dolomite content (wt.%)	0	5	10	15	20	25	30
Thermal Shock Resistance (cycles)	20	24	28	30	32	16	10
Cold Crushing strength (kg/cm^2)	495	415	378	342	328	320	312
Percentage Linear Shrinkage (%)	2.8	7.2	9.0	12.0	6.0	4.0	4.0
Bulk Density (g/cm^3)	1.98	1.95	1.92	1.84	1.84	1.80	1.73
Apparent Porosity (%)	14.05	16.68	20.72	21.53	23.80	25.48	26.53
Permeability	90	86	70	60	46	35	37
Refractoriness (°C)	1740	1710	1690	1670	1650	1600	1560

The results of chemical analysis of the clay and dolomite are presented in Tables 1 and 2 respectively while the determined physical properties are presented in Table 3. The changes in various refractory properties of the prepared clay samples with clay contents are presented in Figures 1-7.

Table 1: Chemical Composition Analysis of Odolewu Clay Deposit

Constituent	Percentage
SiO_2	47.11
Al_2O_3	37.24
Fe_2O_3	0.56
TiO_2	0.96
CaO	0.07
MgO	0.08
Na_2O	0.04
K_2O	0.30
LOI	13.60

Table 2: Chemical Composition of Dolomite

Constituent	Percentage
SiO_2	4.07
Al_2O_3	0.20
Fe_2O_3	0.26
MgO	20.76
CaO	31.02
$CaCO_3$ equivalent	55.4
$MgCO_3$ equivalent	41.70
LOI	43.45

3.1 Thermal Shock Resistance

The thermal shock resistance of the clay samples increases with increase in the dolomite content with a peak value at 20 wt.% dolomite (Fig. 1). Refractory materials with low thermal coefficient of expansion and coarse textures have increased resistance to sudden changes in temperature [16]. It has also been reported that high alkali content favours high thermal expansion which in turn leads to low thermal shock resistance [17].

The chemical compositions of the clay and the dolomite (Table 2 and 3) shows that unlike the clay, the dolomite has no potassium and sodium oxide contents. Therefore, increase in the dolomite content in the mixture will lead to decrease in the alkali content of the clay-dolomite mixture, which will in turn lead to increase in the thermal shock resistance. This accounts for the increase in thermal shock resistance of the samples as dolomite content increases.

3.2 Cold Crushing Strength

This is the load at which cracks appear in the specimen. The cold crushing strength of the samples decreases with increase in the dolomite content (Fig. 2). This property is affected by factors such as composition, ramming pressure, firing temperature, particle size, sintering characteristics and the amount of water content [12], [18]. Also, high silica content together with alkali metal presence results in glassy fusion which in turn results in high compressive strength [17]. As the dolomite content increases in the clay-dolomite mixture, the silica as well as the potassium and sodium oxide contents decrease. Therefore, the decrease in the cold crushing strength of the samples with increase in dolomite content is as a result of the decrease in silica and alkali contents as the dolomite content increases.
However, the minimum requirement of cold crushing strength for refractory brick is 15000 kg/cm^2 [7] while the values obtained for all the clay samples are within the range of 312 and 495 kg/cm^2.

3.3 Percentage Linear Shrinkage

The average linear shrinkage of the clay samples increases with increase in the dolomite content with a peak value at about 15wt.% dolomite. Further increase in the dolomite content causes a decrease in the average linear shrinkage (Fig. 3). However, since the recommended value of linear shrinkage for fireclay is 7-10%, as reported by Chester [19], the average linear shrinkage for samples with 5 wt.% and 10 wt.% dolomite are within the recommended values. Higher shrinkage values may result in warping and cracking of the brick and this may cause loss of heat in the furnace.

3.4 Bulk Density

The results of the bulk density of the samples are shown in Figure 4. The bulk density of the samples decreases as the dolomite content of the clay-dolomite mixture increases. This property is affected by factors such as nature of the materials in the clay sample, particle size and treatment during manufacturing [17]. Tables 2 and 3 reveal that as the dolomite content in the mixture increases, the loss on ignition of the samples would increase. Since bulk density of refractory clay decreases as the amount of organic matter present increases, the decrease in the bulk density values observed may be due to increase in organic matter content in the clay-dolomite mixture as the dolomite content increases.

3.5 Apparent Porosity

The presence of pores in clay affects the strength by reducing the cross-sectional area exposed to an applied load. They also act as stress raiser in brittle clay [20]. Factors affecting the porosity of refractory materials include clay composition, size and shapes of particles, ramming pressure, and reaction occurring on firing [17]. The results show that the apparent porosity of the clay-dolomite mixture increases as the dolomite content increases (Fig. 5).

The loss on ignition of the dolomite is much higher than that of the pure clay sample (Tables 2 and 3). This implies that the dolomite consists of higher amount of combustible materials which indicates higher porosity [12]. This might have accounted for the observed increase in apparent porosity of the clay-dolomite mixes as the dolomite content increased.
However, the values of apparent porosity for most of the clay samples fall within the standard values of 20-30% according to Chester [19].

3.6 Permeability to Air

The permeability values for the clay samples decrease as the dolomite content increases from 0–25wt.%. For 30wt.% dolomite content, there is a slight increase in the permeability (Fig. 6). However, permeability number of all the samples are within the internationally accepted range of 25-90 [19]. Refractories under the influence of gases and liquids should be impervious, since this will help to eliminate leakage of gases and penetration of liquids through the walls of the furnace [12].

3.7 Refractoriness

The variation of the refractoriness of the clay samples with dolomite contents is shown in Fig. 7. A hundred weight percent (100 wt.%) clay sample possesses the highest refractoriness (1740 °C) while 70 wt.% clay sample possesses the lowest (1560 °C). This is because the higher the proportion of alumina, the higher the temperature (i.e. higher refractoriness) necessary to form the glassy ceramic bonding material which characterizes ceramic products [21]. The chemical composition analysis showed that the alumina content of the Odolewu clay is high while that of the dolomite in very low. Therefore, as revealed by Fig. 5, the refractoriness of the samples decreases as the dolomite content increases. The results obtained give an indication of good refractoriness which fall within the range of 1580-1750°C for fireclays according to Grimshaw [18]. The presence of alkali metals in clay usually lowers its fusion temperature [17]. Therefore, the high refractoriness of the clay is probably due to its high alumina content and low potassium and sodium oxide contents.

3.8 Loss on Ignition

The value obtained for the clay is within the range recommended for kaolinitic clay which have values in the range of 12-15% [22]. Dolomite has value higher than the recommended value and this may be due to high content of organic matters in the sample. Loss on ignition values are often required to be low [7]. This is because of its effect on the porosity of refractory bricks. The low value is an indication of low porosity value of the brick [12].

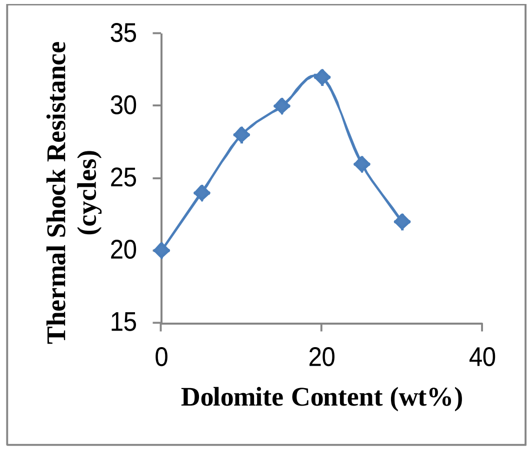

Figure 1: Variation of Thermal Shock Resistance with Dolomite Contents

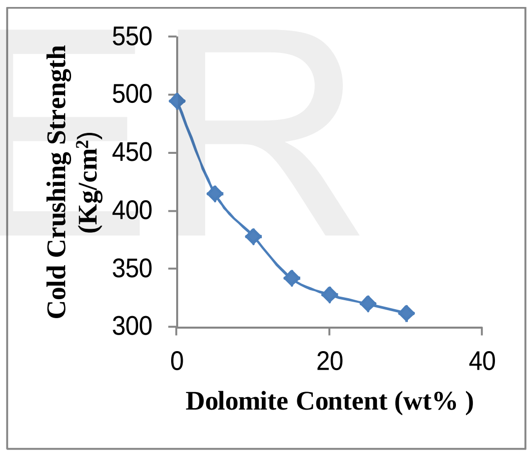

Figure 2: Variation of Cold Crushing Strength With Dolomite Contents

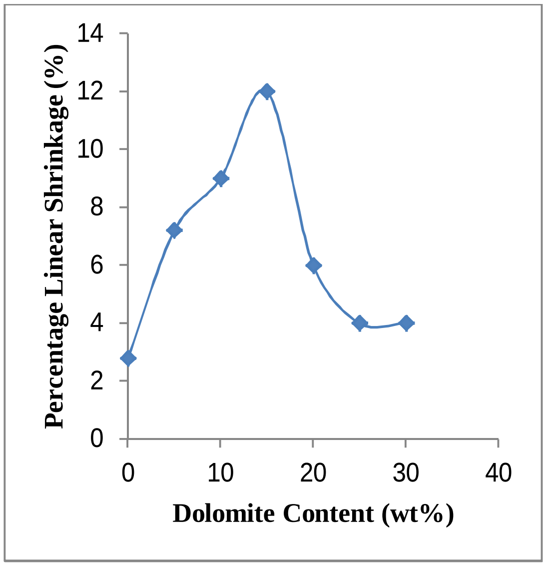

Figure 3: Variation of linear shrinkage with Dolomite Contents

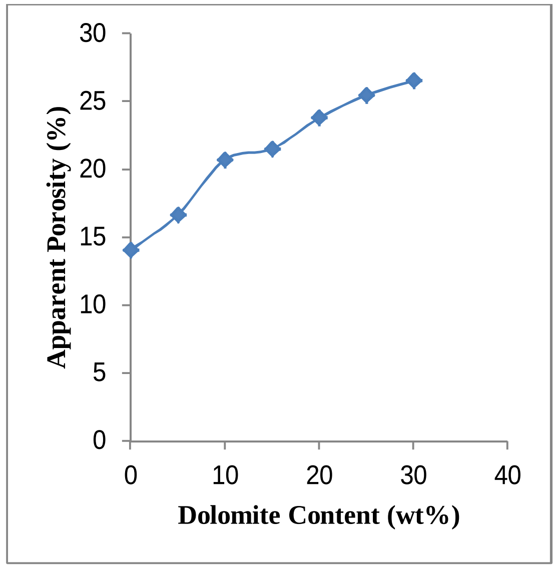

Figure 4: Variation of Apparent Porosity with with Dolomite Contents

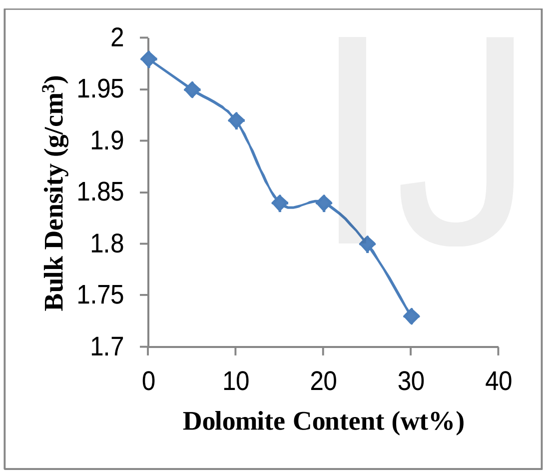

Figure 4: Variation of Bulk Density with dolomite contents

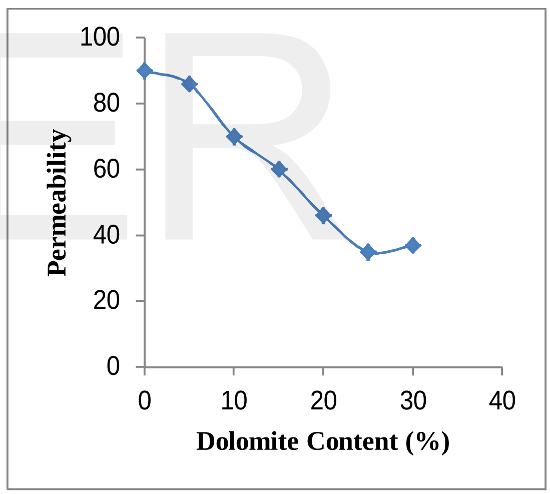

Figure 6: Variation of Permeability with dolomite contents

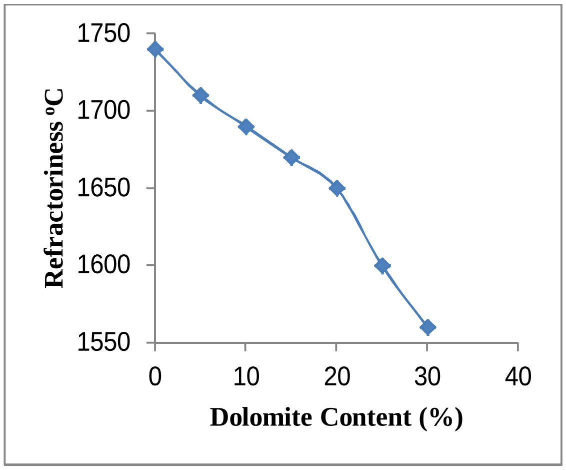

Figure 7: Variation of Refractoriness with dolomite contents

CONCLUSION

Linear shrinkage for clay-dolomite mixtures with 5 wt.% dolomite and 10 wt.% dolomite, thermal shock resistance for 10 wt.% dolomite and 15 wt.% dolomite, apparent porosity for samples with 10-30 wt.% dolomite, and refractoriness for all the samples are within recommended values for fireclays.

The thermal shock resistance and permeability of the Odolewu clay can be improved with addition of dolomite. Clay-dolomite mixture with 10 wt.% dolomite gave the optimum combination of properties required of a refractory.

The chemical analysis of the Odolewu clay revealed that alumina and silica are the major components. Therefore, the clay can be suitable as alumino-silicate refractory material.

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