EVALUATION OF REFRACTORY CHARACTERISTICS OF BADEGGI CLAY WITH VARIED RATIOS OF GRAPHITE AND ASBESTOS ADDITIVES

OLUSOLA, Emmanuel Omowumi.[1], AJAO, Majeed Opeyemi[2]

[1]Senior Lecturer, Mechanical Engineering, Olusegun Agagu University of Science and Technology, Okitipupa, Ondo State
[2]PG Student, Federal University of Technology Minna, Niger State, Nigeria

Abstract: Refractory materials are crucial in industries operating at high temperatures, such as metallurgy, ceramics, and glass manufacturing. This study evaluates the refractory properties of Badeggi clay from Niger State, Nigeria, when mixed with varying proportions of graphite and asbestos additives. Badeggi clay, primarily composed of silica and alumina, has demonstrated potential as a refractory material. Enhancing its properties through additives intends to improve its thermal stability, mechanical strength, and resistance to corrosive agents. In this investigation, Badeggi clay samples were mixed with graphite and asbestos at 20%, 30%, and 40% ratios. The effects on refractoriness, thermal shock resistance, apparent porosity, bulk density, specific gravity, and firing shrinkage were systematically studied. Results indicated that the initial refractoriness of Badeggi clay, at 1400ºC, increased to 1700ºC with graphite and 1600ºC with asbestos additives. Firing shrinkage remained consistent across samples at 3.5%. Thermal shock resistance decreased with higher additive content, while apparent porosity and bulk density increased. Specifically, graphite and asbestos enhanced the refractory properties, with the optimal compositions found at 30% graphite and 40% asbestos for improved insulation and durability. The study concludes that Badeggi clay, with appropriate additive ratios, is suitable for high-temperature applications such as furnace linings and general-purpose bricks. Future research should explore additional additives like sawdust and rice husk to further enhance the clay's properties. These findings contribute to materials science, offering insights into optimizing local resources for industrial use.

1.0 INTRODUCTION

Refractory materials are indispensable in industries that operate at high temperatures, such as metallurgy, ceramics, and glass manufacturing. These materials must withstand extreme thermal, mechanical, and chemical stresses without degrading (Olusola, 2023). Clays are a common choice for refractory materials due to their wide availability, cost-effectiveness, and desirable properties. Refractory materials, including alumino-silicate silica, magnesite chrome, and carbon, serve critical roles in metallurgical industries for constructing and maintaining high-temperature equipment such as furnaces, kilns, and reactors (Akinbode, 1996; Olusola et al., 2023). Badeggi clay, discovered in Niger State, Nigeria, exhibits great potential as a refractory material (Ibitoye, 2015; Olusola, 2024).

Clay, a finely grained unconsolidated rock material, exhibits plasticity when moistened and hardens into a stony form upon heating (Olusola et al., 2024). It originates from natural processes involving complex weathering, sedimentation, and geological deposition. Composed primarily of silica (SiO_2) and alumina (Al_2O_3), alongside water (H_2O) and significant concentrations of iron oxides, alkali, and alkaline earth metals, clay also contains crystalline clay minerals such as quartz, feldspar, and mica (Chester, 1973; Olusola, 2006).

To enhance the refractory properties of clay, various additives are often incorporated. These additives can improve thermal stability, mechanical strength, and resistance to corrosive agents, making the material more suitable for high-temperature applications (Mokobane et al., 2019; Olusola, 2024). The focus of this study is to assess the refractory properties of Badeggi clay when mixed with different ratios of additives. By varying the proportions of these additives, the goal is to identify the optimal composition that enhances the clay's performance under extreme conditions.

Previous studies have shown that additives like graphite and asbestos can significantly influence the properties of refractory clays. Graphite is known for its excellent thermal conductivity and lubricating properties, which can help in reducing wear and thermal stress (Kim & Kim, 2018). Asbestos, although less commonly used now due to health concerns, has been historically valued for its thermal resistance and strength (Gianfagna & Oberti, 2017). Abolarin, Olugboji, & Ugwuoke, (2004) demonstrated that Badeggi clay exhibits favorable refractory properties comparable to firebricks, suggesting room for enhancement through additives or modifications.

Moreover, Aderibigbe and Chukwuogo (1984) explored the use of Badeggi clay in producing insulating bricks, highlighting challenges in balancing insulation with operational temperature due to modest refractoriness levels (1,400°C). Subsequent studies proposed improvements in various properties such as refractoriness, porosity, thermal shock resistance, bulk density, and linear shrinkage through additives like sawdust, asbestos, graphite, and rice husk binders.

This paper presents further investigations on Badeggi clay as a refractory material, focusing on mixtures incorporating graphite powder and asbestos at proportions of 20%, 30%, and 40%. The effects of these variations are systematically studied and analyzed. This research aims to fill the gap in understanding how different additive ratios impact the refractory properties of Badeggi clay. By systematically analyzing these effects, the study will provide insights into optimizing Badeggi clay for industrial applications where high-temperature performance is crucial. This knowledge can lead to the development of more efficient and durable refractory materials, leveraging local resources effectively.

The findings from this study are anticipated to contribute significantly to materials science and engineering, particularly in regions where Badeggi clay is readily available. Furthermore, the insights gained can inform the broader application of additive-enhanced clays in various high-temperature industrial processes.

2.0 Materials and Methods

2.1 Materials

The investigation utilized Badeggi clay and additives, specifically graphite and asbestos. Badeggi clay samples were collected from a designated area in Niger State, Nigeria.

2.2 Methods

2.2.1 Preparation of Samples

The clay samples collected were prepared by crushing, sizing, and grading through sieving to eliminate extraneous materials like leaves and other impurities. Each experimental sample consisted of 200g of clay, with graphite and asbestos additives added in varying percentages of 20%, 30%, and 40%. This resulted in three distinct sample compositions for evaluation. The mixtures were blended thoroughly and shaped using a die and hydraulic press, followed by oven drying and firing at 1100°C for subsequent testing.

2.3 Analysis of the Properties of the Prepared Samples

2.3.1 Chemical Analysis of the Clay Samples

Chemical composition analysis employed X-ray methods,

2.3.2 Analysis of the Impact of the Additives on the Refractory Properties of Badeggi Clays

The impact of graphite and asbestos additives at different ratios on the properties of the Badeggi clay sample was investigated through several tests. These included refractoriness, thermal shock resistance, apparent porosity, bulk density, specific gravity, and firing shrinkage.

2.3.2.1 Refractoriness

Refractoriness is the ability of a material to withstand high temperatures without melting or softening. It is commonly measured using the pyrometric cone equivalent (PCE). PCE involves comparing the material's softening temperature with that of standard cones made from known refractory materials. A higher refractoriness indicates better performance in high-temperature environments, which is critical for applications like furnaces and kilns (Taylor & Bullock, 2009).

The refractoriness of the samples was tested using the Pyrometric Cone Equivalent (PCE) method (ASTM, 2020). Prepared mixture of clay and additive sample and standard pyrometric cones were placed in a furnace, which was then heated at a controlled rate. Observations were made to identify the temperature at which the samples deform similarly to the standard cones, indicating its softening point. The recorded temperature was compared with the known softening points of the standard cones to determine the refractoriness of the samples. The results were documented and analyzed for reporting.

2.3.2.2 Thermal shock resistance.

Thermal shock resistance is the ability of a material to withstand rapid temperature changes without cracking or losing structural integrity. This property is typically assessed by subjecting the material to rapid heating and cooling cycles and observing any resultant damage. This property is crucial for materials exposed to fluctuating temperatures, such as in blast furnaces or thermal reactors (Richerson & Lee, 2005).

To assess the thermal shock resistance of clay materials with various additives, prepared specimens, each 50mm in diameter and length, were subjected to repeated heating in an electric furnace at 900°C for one hour. Subsequently, the samples underwent repeated cycles: heating in the furnace at 900°C for 10 minutes followed by rapid cooling in a stream of air. After multiple cycles, surface cracking and minor spilling were observed, indicating the thermal shock resistance of each of the samples (ASTM, 2013).

2.3.2.3 Apparent Porosity

Apparent porosity measures the percentage of the volume of open pores in the clay material. High apparent porosity indicates a more porous structure, affecting the material's strength and thermal insulation properties.

The capability to absorb water is critical, particularly during the rainy season, to prevent condensed water vapor on internal brick surfaces. Test pieces, initially fired at 1100°C for 30 minutes in an electric oven, were subsequently cooled in boiling water and submerged. Their weights were recorded fully immersed in water and when suspended in water. To calculate their apparent porosity the formula shown below was used.

$$\text{Apparent porosity} = \frac{ws - wd}{Ws - wp} x100 \qquad (2.1)$$

Where; Ws= soaked weight; W_d= Dry weight ; W_p= Suspended weight.

2.3.2.4 Average Bulk

Bulk density is the weight per unit volume of the refractory including the volume of open pore space. The principle of the determination was to find the volume of a suitable liquid displaced on placing in it a weighed amount of finely ground material sample.

Method: Test pieces were prepared from the clay sample and dried in an electric oven at 110°C for 24 hours. The dried weight (Wd) of each specimen was recorded. The specimens were then fired in an electric furnace at 1100°C. Post-firing, the specimens were placed in a beaker of water inside a vacuum desiccator and evacuated until bubbling ceased, indicating that the air in the samples had been displaced by water. The soaked weight (W_S) was then recorded. Each specimen was subsequently suspended in a beaker of water, and the suspended weight (Wp) was recorded. The average bulk densities were calculated using the expression given below.

$$\text{Bulk Density} = \frac{Wd}{Ws - Wp}\, \rho w \qquad (2.2)$$

Where ρw = Density water at room temperature (T°C)
Wd = dried weight of each specimen
Ws = soaked weight

Wp = suspended weight

2.3.2.5 Specific Gravity

Specific gravity, or true density, measures the density of the clay material excluding the pore spaces. It provides insight into the material's true solid content. The principle of this determination is to find the volume of a suitable liquid displaced on placing in it a weighed amount of finely ground material/sample.

Method:-

Crushed clay samples were passed through a 100 mesh B.S.S. sieve and then introduced into a previously weighed specific gravity (SG) bottle (Wa) using a glass funnel. The bottle containing the sample was weighed (Wb). Distilled water was added to the powder under vacuum, and the bottle was suspended in water at a known temperature for approximately 15 minutes to ensure uniform liberation of air bubbles, and then weighed again (Wc). The bottle was then washed, filled with distilled water, allowed to equilibrate for another 15 minutes, and weighed once more (Wd). The specific gravity was calculated using the following expression.

$$\text{Specific gravity} = \frac{Wb - Wa}{(Wd - Wa)\ (Wc - Wb)} \times \rho w \qquad (2.3)$$

Where: S. G. = Specific gravity; Wa = weight of S.G. bottle; Wb = Wt of S.G bottle + sample

Wc = wt of S. G bottle + sample + Distilled water ; Wd = wt of S.G. bottle + distilled water;

ρw = Density water at room temperature (T^{o}C)

2.3.2.6 Firing Shrinkage Test of the Clay Sample

This is an important characteristic especially when articles made from it are required to be of definite size.

Method:- 500g of the clay sample was passed through a BS 180 µm sieve, and sufficient water was added to create a creamy consistency. This mixture was dewatered on a plaster bat. The plastic clay was then carefully kneaded to remove any air bubbles. Five slabs were formed from the plastic clay. Two fine lines, each 5 cm long and 2 cm apart, were marked on the slabs using a pair of dividers. The slabs were allowed to dry for 3 days. The wet to dry shrinkage was measured using the specified formula.

$$\%\ \text{wet shrinkage} = \frac{\text{change in length}}{\text{original length}} \times 100 \qquad (2.4)$$

$$\text{Fired shrinkage (F.S)} = \frac{\text{change in length}}{\text{original length}} \times 100 \qquad (2.5)$$

3.0 Results and Discussions

3.1. Results

The chemical analysis summary presented in Table 1 indicates a predominant presence of silica sands in the Badeggi clay sample. This classification categorizes the clay as siliceous firebricks, consistent with established standards (Abolarin, Olugboji, & Ugwuoke, 2004). The chemical compositions of Badeggi clay, detailed in Tables 3. 1 and 3.2, align closely with previous studies (Aderibigbe & Chukwuogo, 1984; Abolarin, Olugboji, & Ugwuoke, 2004). Therefore, the discussion based on these findings will focus on determining the optimal levels of each property through varied proportions of additives, reinforcing the clay's suitability as a refractory material, as demonstrated in prior research.

Table 3. 1: Chemical Composition of Badeggi Clay (Abolarin ,Olugboji and Ugwuoke , 2004)

Composition	Percentages (%)
SiO_2	60.5

Al_2O_3	25
K_2O	1.75
Fe_2O_3	5.90
TiO_3	2.07
CaO	0.47
MgO	0.16
Na_2O	0.12

Table3.2: Properties of Badeggi clay (Abolarin, Olugboji and Ugwuoke , 2004)

S/N	PROPERTY	VALUES
1.	Refractoriness (°C)	1,400
2.	Thermal Shock Resistance (cycles)	15^+
3	Porosity (%)	22.83
4.	Bulk Density (g/cm^3)	2.19
5.	Specific Gravity	2.54g
6.	Firing Shrinkage (%)	3.5%
7	**Apparent Porosity**	22.83

Tables 3.3 and 3.4 outline the properties under investigation. Table 3.3 specifies the properties of Badeggi clay with varying additions of graphite powder (20%, 30%, and 40%), while Table 3.4 details the properties with asbestos additives in the same percentage range.

Table 3.3: Properties of Badeggi Clay with Varied Percentages of Graphite Powder

S/N	Refractory Properties	Badeggi Clay + %Graphite		
		20%	30%	40%
1.	Refractoriness (°C)	1,700	1,700	1700
2.	T.S.R (cycles)	18	14	10
3.	Apparent Porosity (%)	25.61	29.86	-
4.	Bulk Density (g/cm^3)	2.62	2.71	2.82
5.	Specific Gravity	1.72	1.70	1.62
6.	Firing Shrinkage (%)	3.5	3.5	3.5

Table 3.4: Properties of Badeggi Clay with Varied Percentages of Asbestos

S/N	Refractory Properties	Badeggi Clay + %Graphite		
		20%	30%	40%
1.	Refractoriness (°C)	1,700	1,700	1700
2.	Thermal Shock Resistance T.S.R (cycles)	18	14	10
3.	Apparent Porosity (%)	25.61	29.86	-
4.	Bulk Density (g/cm^3)	2.62	2.71	2.82
5.	Specific Gravity	1.72	1.70	1.62
6.	Firing Shrinkage (%)	3.5	3.5	3.5

Table 3.5: Comparison of Pure Badeggi Refractory Value with Badeggi/Additive Values

S/N	Refractory Properties	Badeggi Clay + %Graphite			Badeggi Clay + % Asbestos			Badeggi Clay value
		20%	30%	40%	20%	30%	40%	
i.	Refractoriness (ºC)	1,700	1,700	1700	1,600	1.600	1,600	1,400
i.	Thermal Shock Resistance (cycles)	18	14	10	18	16	14	15+
i.	Apparent Porosity (%)	25.61	29.86	-	26.27	30.99	32.47	22.83
v.	Bulk Density (g/cm^3)	2.62	2.71	2.82	2.52	2.66	3.01	2.19
v.	Specific Gravity	1.72	1.70	1.62	1.84	1.80	2.0	2.54g
i.	Firing Shrinkage (%)	3.5	3.5	3.5	3.5	3.5	3.5	3.5%

3.2 Discussion

3.2.1 Firing Shrinkage

The firing shrinkage values remained consistent across all investigated samples, with a uniform 3.5% shrinkage observed, which falls within the allowable range specified by James (2010). This uniform firing shrinkage indicates efficient firing, as shown in the table 3.5.

3.2.2 Refractoriness

The average refractoriness of Badeggi clay refractory is 1,400ºC, which is lower compared to the typically quoted values for most dense refractory clays (over 1,500ºC, as reported by Woral, 1982; Ndaliman, 2000). This lower refractoriness may be attributed to its high silica content and other properties such as Fe_2O_3. However, the addition of additives has led to improved refractoriness values. Specifically, the refractoriness increased to 1,600ºC and 1,700ºC respectively for asbestos and graphite additives as shown in table 3.5. These achievements allow for the production of general-purpose bricks suitable for reheat furnaces, checkers, boilers, and ladles using these materials.

3.2.3 Bulk Density and Apparent Porosity

These parameters significantly impact both the strength and insulating properties of refractory materials. Porosity, in particular, dictates how resistant a material is to penetration by molten slags, metals, and flue gases. The apparent porosity of Badeggi clay measures at 22.83%. when graphite additives are introduced, the porosity of Badeggi clay increased across varying percentages. For instance, in Table 3.5, the porosity ranges from 25.61% to 29.85% with increasing graphite content, peaking at 30% before becoming non-porous.

Based on the findings in Tables 3.3 and 3.4, refractories containing 30% graphite or 40% asbestos show promise for insulation purposes due to their combined high refractoriness and manageable porosity.

Bulk densities across all categories align closely with standard firebrick values, with asbestos-mixed refractories registering the highest at 3.01 g/cm^3. Generally, bulk density trends upwards with increasing additive content.

3.2.4 Thermal Shock Resistance

The tests revealed that Badeggi clay resists spalling for up to 15 cycles. However, additives mixed into the clay showed a reduction in thermal shock resistance, decreasing from 18 to 10 cycles with increasing graphite content from 20% to 40%. Similarly, asbestos additives decreased thermal shock resistance from 18 to 14 cycles across the same additive range. Overall, the results indicate that the materials' resistance decreases as the percentage of additive increases.

Conclusions and Recommendations

Based on investigations into the Refractory Properties of Badeggi Clay with Varied Additive Proportions, significant enhancements were observed.

i. The initial refractoriness of Badeggi Clay, rated at 1400ºC, increased to 1700ºC with graphite and 1600ºC with asbestos additives, making it suitable for furnace linings.
ii. Despite increasing additive proportions, properties like firing shrinkage remained stable, while overall improvements were achieved due to the additives. Thermal shock resistance and specific gravity gradually decreased with higher additive content, whereas density and porosity showed an inverse relationship.
iii. Chemical analysis revealed a high proportion of silica sands, classifying the clay as siliceous firebricks. Based on these findings, specific additive compositions can be selected to achieve desired property compromises.
iv. The clay is recommended for applications such as ovens and furnaces operating between 1400ºC and 1700ºC, as well as general-purpose bricks for reheating furnaces and ladle linings.
v. Furthermore, exploring the effects of additional additives like sawdust and rice husk on clay properties is highly recommended for further enhancement."

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