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Influence of Agro-Wastes Additive on the Microstructural View, Bulk Density, Apparent Porosity and Firing Shrinkage Properties of Locally Produced Refractory Bricks

Adeyemi Gbenga Joshua, Akomolede Adeyemo Moyosiore, Oni Taiwo Oluwasesan and Stephen Joseph Temitope*

Department of Mechanical Engineering, Ekiti State University, Ado-Ekiti, Nigeria

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ABSTRACT

The increasing need for refractory bricks has advanced research into the utilization of readily available agricultural by-products. This work examined the influence of cassava peel ash (CPA) and palm kernel ash (PKA) as admixtures on the physical properties of locally produced refractory bricks. Samples coded CTR (Control), clay/30CPA, clay/30PKA, and clay/30CPA-PKA were prepared, moulded by machine, fired at elevated temperature, and tested for bulk density, apparent porosity, and firing shrinkage to assess the influence of addition of agro-wastes. The objective of the study is to determine the effects of addition of CPA, PKA, and its hybrid on mechanical properties of refractory bricks. The result showed that the control sample has the highest density of 2.3 gcm^{-3} while clay/30CPA, clay/30PKA and clay/30CPA-PKA samples have 2.0, 1.8 and 1.8 gcm^{-3} respectively. The percent porosities are 19.8, 20.63, 26.51 and 25.5% for CTR, clay/30CPA, clay/30PKA and clay/30CPA-PKA, respectively. Both CTR and clay/30CPA shared similar percent shrinkage of 4.3%, while clay/30PKA and clay/30CPA-PKA had percent shrinkage of 2.17%. The results showed that the addition of the agro-waste additives did not affect the physical integrity of the refractory bricks but showed that clay/30CPA-PKA followed by clay/30PKA are most suitable for the production of insulating refractory bricks, and also fall within the standard values required for fire clay refractory bricks.

Keywords: Refractory bricks; cassava peel ash; palm kernel ash; Porosity; Bulk Density

INTRODUCTION

In most formative manufacturing or metallurgical industries, a furnace is predominantly required for smelting or refining ores and recycling metal scraps to form a molten metal (liquid), which is processed into the desired product. A furnace is an enclosed chamber where the chemical energy of a solid, liquid, or gaseous fuel is converted into heat energy (Frey et al., 2003). The heat energy due to temperature difference therein is transferred either by conduction, convection, radiation, or some combination. The temperature in the furnace is relatively high, often above the ambient temperature. Consequently, there is a need for lining the furnace with brick composed of inorganic material, which can withstand high temperatures (usually above 1500^oC) under the physical and chemical action of molten metal, slag, and gases to prevent heat loss during the melting process. This lining material is called a refractory or fire brick (Agha, 1998).

Refractory bricks are generally materials that can withstand high temperatures together with abrasion, impact, thermal shock, chemical attack, and high-level loads at elevated temperatures between 325 - 3500^oC (Sengupta, 2020). Including some other characteristics such as resistance to thermal shock, porosity, density and shrinkage of the materials that play major roles in its performance. However, the refractory bricks are principally used in furnace construction, and to thermally insulate structural chambers for accommodating excessive temperature atmosphere (Chandra et al, 2019). The refractory bricks produced locally are used in lining the different types of metallurgical furnaces, such as blast furnaces, open-hearth furnaces, soaking pits, heat-treatment furnaces, for the conservation of energy within the minimum and affordable cost of production (Bhatia, 2005).

Table 1 provides the composition and some key properties of standard refractories, highlighting reported values for important properties of the most commonly used refractories (Yami, 2007).

Table 1: Standardized International Values for Physical Properties of Refractories

Type	Density (g/cm^3)	Porosity (%)	Permeability	Dimensional shrinkage (%)	Thermal Shock Resistance (cycles)	Cold Crushing Strength (Kg/cm^3)	Water Absorption (%)	Refractoriness (ºC)
Fire clay	1.71-2.1	20-30	25-50	7-10	25-30	15000	< 22	1500-1700

Over the years, refractory materials for industrial furnaces have been imported despite the abundance of clay deposits and other raw materials in Nigeria. The over-reliance on imported refractory materials has led to its high cost and makes it unaffordable to several indigenous industries using furnaces. This high cost of imported refractory materials coupled with their increasing demand has necessitated attempts and actions to locally produce refractory products by harnessing the vast abundant clay deposits in Nigeria. In a recent study, Osarenmwinda et al, (2014) produced refractory locally bricks from some local clay deposits in Delta State, Nigeria. The brick produced was found suitable for medium thermal application in furnaces, due to improved insulating properties after tested for shrinkage, bulk density, Loss on ignition, cold compression strength, apparent porosity, and thermal shock resistance.

Due to industrial growth in Nigeria, huge volumes of agro-waste are generated annually. The disposal of these wastes poses a serious threat on socio-economic and health within the societies. The application of the waste as addition to clay to improve its properties was narrated by a researcher (Barnabas et al., 2023) and on who to produce insulating refractory bricks with it. The addition of various agro-waste, such as chemicals (enzymes), biomass, and agricultural waste, among others, with clays was deeply explored (Barnabas et al., 2023).

However, this has motivated some researchers to investigate the effects of agro-waste on the performances of locally produced refectory brick. Such as Esezobor et al (2014), investigated the influence of Agro-forestry wastes additives on the insulating properties of Osiele clay for the production of insulating refractory bricks a good performance of the refractory bricks was recorded. Eugenia et al. (2020) evaluate the thermo-mechanical properties of insulating refractory bricks made from indigenous clay mixed with gmelina seed shells particulates. The results of the physical, thermal and mechanical properties indicated that Gmelina shell particles can enhance the insulating properties of refractory bricks made from the indigenous clay (Osiele clay deposit, Nigeria). Also, Ojukwu et al (2022), studied the impacts of groundnut shell as grog additives on the refractory properties of Isiagu Clay. The findings showed that the clay containing groundnut shell is suitable for insulating applications. Their findings yield positively to the influence of clay as lining furnace during metal melting processing.

In this regard, this present study investigates the influence of agro-wastes on the bulk density, apparent porosity and firing shrinkage of locally produced refractory bricks.

MATERIALS AND METHODS

Materials Selection

The materials used for the production of the agro-wastes-based refractory bricks include kaolin, ball clay, cassava peels and palm kernel shaft. Both kaolin and ball clay were sourced from a clay deposit at Ikere-Ekiti in Ekiti State, Nigeria, while both cassava peels and palm kernel shaft were sourced from cassava flour and palm oil processing plants, respectively, at Ado-Ekiti, also in Ekiti State, Nigeria. The as-received clays (kaolin and ball clay) were initially ground separately using a crusher, then soaked in water for 72 hours, while organic matters and impurities were removed during intermittent stirring. Afterwards, the slurry clay was then wet-sieved through a 75 µm size sieve into a Plaster of Paris (P.O.P) mould for ambient drying for 96 hours to obtain plastic clay. The plastic clays were then oven-dried at 110 ºC in an oven to obtain dried clay. The dried clays were further pulverized to obtain the clays in processed powdered form.

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The as-received agro-wastes (cassava peel and palm kernel shaft) were first rinsed under running water to remove sands and dirt attached to them. After this, they were sun-dried for 48 hours, followed by oven drying at 110 ^oC for 7 hours. The dried agro-wastes were then poured separately on flat refractory bat and placed inside a gas-fired kiln. They were then heated to a temperature of 850 °C to obtain cassava peel ash (CPA) and palm kernel shaft ash (PKA), respectively. Each of the CPA and PKA was then sieved using 75 µm sieve to obtain fine powdered ash.

Sample Preparation

The clay bricks were prepared by mixing the processed clay in powder form with each additive (cassava peel ash and palm kernel ash). The clay was mixed with various weights (wt. %) proportion of each additive, and the mixing proportion of the clay and the additives are shown in Table 2.

Table 2: Composition of samples

	Composition			
Samples	**Kaolin (wt. %)**	**Ball-clay (wt. %)**	**CPA (wt. %)**	**PKA (wt. %)**
Control (CTR)	90	10	0	0
Clay/30CPA	60	10	30	0
Clay/30PKA	60	10	0	30
Clay/30CPA-PKA	60	10	15	15

In order to improve plasticity and ensure homogeneous mixture of the clays with each additive (CPA and PKA), 8% of water was added to each sample mix (see Table 2). The clay-agro-wastes mix were then poured inside a metal mould box of dimension 23 x 10.3 x 8.3 cm after engine oil had been applied into the mould to facilitate easy removal of the brick after compaction. The samples were then compressed using hydraulic pressing machine at a force of 30 kN to obtain well-compacted rectangular-shaped as shown in Figure 1 below.

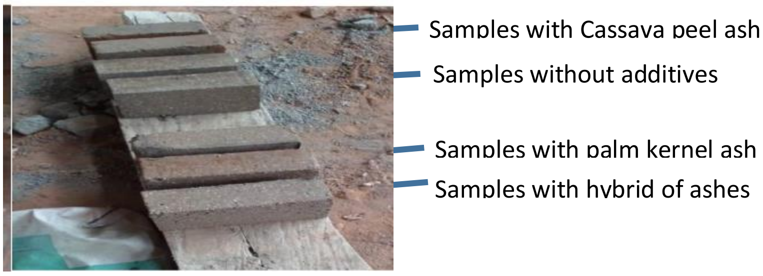

Figure 1: The refractory Bricks Produced

The test samples were left to air dry for seven days, oven-dried at 110°C, and fired at 1200°C. The samples were heated and held at the above mentioned temperatures for 1 h and allowed to cool to room temperature in the furnace. The samples were subsequently taken to laboratory for property tests.

Characterization of the Produced Refractory Bricks

i. Porosity Test

The porosity of the fired test samples was determined using the boiling method as described by Hassan (2005). Samples measuring 50 x 50 x 10 mm were cut from the fired specimens, dried in an oven at 110°C until a constant weight was achieved (denoted as ‘D’). Each sample was then submerged in water in a beaker, boiled for 2 hours, and allowed to cool to room temperature. The samples were re-weighed to determine the weight ‘S’ while suspended in water. Afterward, the samples were removed from the water, and their saturated weight (W) was recorded. The apparent porosity was calculated using the following formula:

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Apparent Porosity= $A.P = \frac{W-D}{W-S} X\ 100$ (1)

where W, D, and S are saturated weight, Dry weight, and suspended weight, respectively.

ii. Bulk Density Test

Similarly, the bulk density of the fired test samples was also determined by the boiling method as outlined by Hassan (2005). The same procedure was followed, with the samples measuring 50 x 50 x 10 mm, being dried in an oven at 110°C. The samples were then placed in a beaker of water and boiled for 2 hours to facilitate the release of any trapped air. Once soaked, the excess water was drained, and the saturated weight was recorded. The bulk density was calculated using the formula:

Bulk Density $= \frac{\rho_w\ X\ D}{W-S}$ (2)

where D, W, S, ρ_w are dry weight, saturated weight, suspended weight, and water density, respectively.

iii. Firing Shrinkage Analysis

The firing shrinkage of the pressed samples was determined by measuring the dimensional changes that occurred between the dried samples at 110°C and the fired samples at 1200°C, following the method outlined by Jock et al. (2013). The firing shrinkage was calculated using the equation:

Firing Shrinkage $= \frac{LD-LF}{LD}$ (3)

where LD is the dimension of the sample after drying at 110°C, and LF is the dimension of the sample after firing at 1200°C.

iv. Microstructural Analysis

The micro-structure of the refractory bricks with and without the additives (control sample (CTR), clay/30CPA, clay/30PKA and clay/30CPA-PKA) were observed using a Scanning Electron Microscope (SEM) at 80 **µm** magnifications. The sample for microstructure evaluation was initially sputtered with gold of about 5 nm and later clamped on the sample holder of the machine (SEM, JSM-6100 JEOL), and its surface was viewed at different magnifications at an acceleration voltage of 20 kV.

RESULTS AND DISCUSSION

Bulk density of the produced refractory Bricks

The result of the bulk density of the refractory bricks is shown in Figure 3. The refractory bricks' bulk density is significantly influenced by the incorporation of agro-waste materials, as evidenced by the variations in bulk densities.

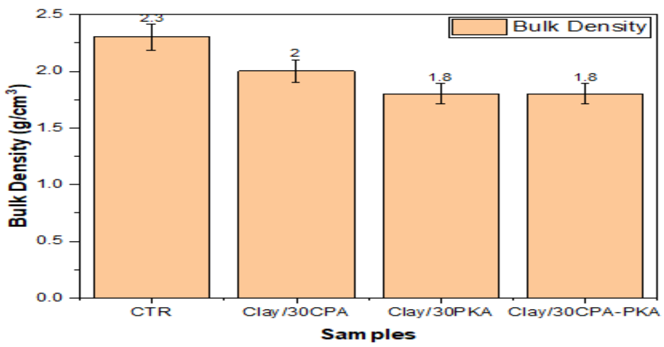

Figure 3. Bulk Density of the developed bricks

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The control sample exhibited the highest bulk density (2.3 g/cm³), indicative of a denser structure. In contrast, a decline in bulk density is observed for agro-waste-based refractory bricks. Sample Clay/30CPA displayed a reduced bulk density of 2.0 g/cm³, suggesting a lighter material with a more porous structure, potentially beneficial for thermal insulation applications. Both Clay/30PKA and clay/30CPA-PKA samples showed a similar further decrease in bulk density of 1.8 g/cm³, suggesting a characteristic of a high lightweight material, suitable for applications prioritizing thermal insulation and weight reduction. The reduction in bulk density can be attributed to the lower density of ashes of the agro-wastes used compared to the clay material. The combination of CPA/PKA in the hybrid sample does not show synergetic effect on the bulk density. The result is in agreement with several earlier reports (Abdel Hafez et al., 2022; Maafa et al., 2023; Onyia et al., 2023; Kazmi et al., 2016). Nonetheless, the bulk density of the agro-wastes based bricks fell within the standard value range between 1.71 – 2.1 g/cm^3 (Yami, 2007; Jock et al., 2013). The findings suggest that the control sample is optimal for applications requiring high density, low porosity and good durability, while the CPA sample offers a balance between density and porosity, suitable for moderate thermal insulation. In contrast, the PKA and CPA/PKA samples are ideal for lightweight, thermally insulating applications, but may be less suitable for high-stress environments due to reduced structural strength.

Apparent porosity of the produced Refractory Bricks

The result of the percent porosity of the refractory brick is shown in Figure 4.

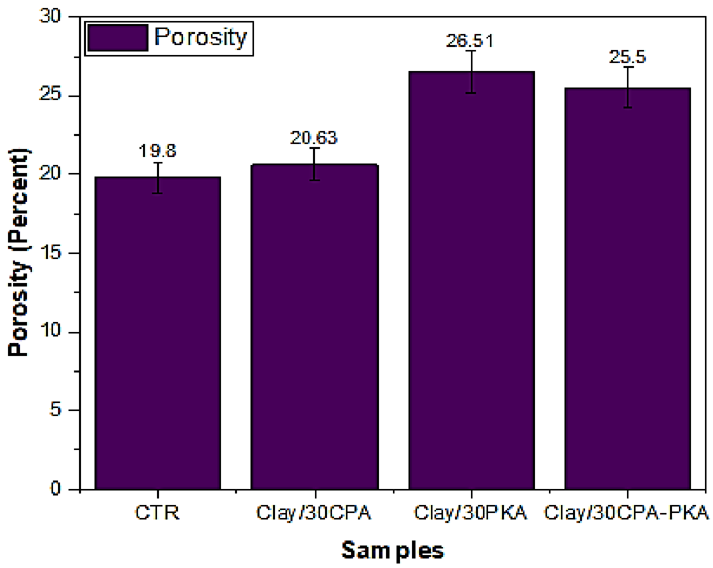

Figure 4. Percent Porosity of the developed bricks

The result shows that the percent porosity of the refractory bricks is significantly influenced by the incorporation of ashes of the agro-waste materials, as evidenced by the variations in percent porosity. The result also affirmed the relationship between bulk density and porosity. The control sample exhibited the lowest porosity (19.8%) as shown in Figure 4.3, indicative of a denser and less porous structure. In contrast, the clay/30CPA sample displayed increased porosity of 20.63%, clay/30PKA sample showed a further increase in porosity of 26.51%, and clay/30CPA-PKA has porosity of 25.5%. The increased in porosity observed for the agro-wastes-based refractory bricks might be attributed to burning off the ashes of the agro-wastes used leaving behind pores during the firing of the refractory bricks. The result agrees with several earlier reports (Abdel Hafez et al., 2022; Maafa et al., 2023; Onyia et al., 2023; Kazmi et al., 2016). The porosity of the agro-wastes-based bricks fell within the standard porosity value between 20 – 30% for typical fireclay refractory bricks (Yami, 2007; Jock et al., 2013).

Firing Shrinkage of the produced Refractory Bricks

Firing shrinkage is used as an indicator of the degree of vitrification as the bricks. Shrinkage is expected that as the bricks are fired to a higher temperature. However, the shrinkage must not be in excess to ensure suitability for furnace use. Figure 5 represents the percentage firing shrinkage of the developed refractory bricks.

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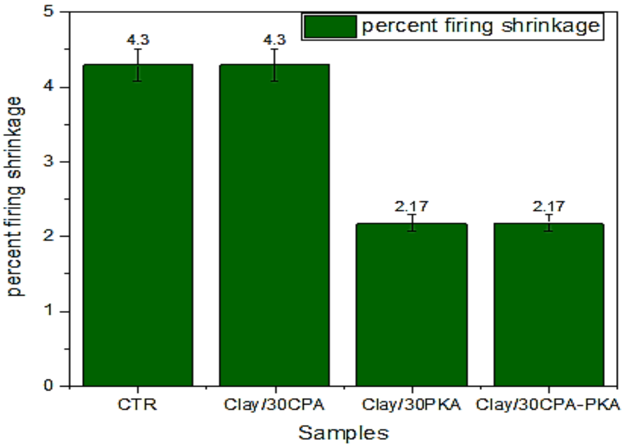

Figure 5: Firing Shrinkage of the developed bricks.

The results showed that both control sample and clay/30CPA samples showed identical percent firing shrinkage of 4.3% while samples clay/30PKA and clay/30CPA-PKA (hybrid) displayed similar shrinkage percentage of 2.17% respectively. The results showed incorporation of agro-wastes ashes did not allow excessive shrinkage to occur at higher firing temperature, suggesting their beneficial use in this work. The standard firing shrinkage percentage of fireclay bricks for furnace use has been suggested to be less than 8 % (Weng et al., 2003).

Micro-structural View of the produced Refractory Bricks

The results of the micro-structure observation conducted using a scanning electron microscope with attached energy dispersive spectroscopy (SEM-EDS) on the control sample (CTR), clay/30CPA, clay/30PKA and clay/30CPA-PKA are shown in Figures 6 (a – d).

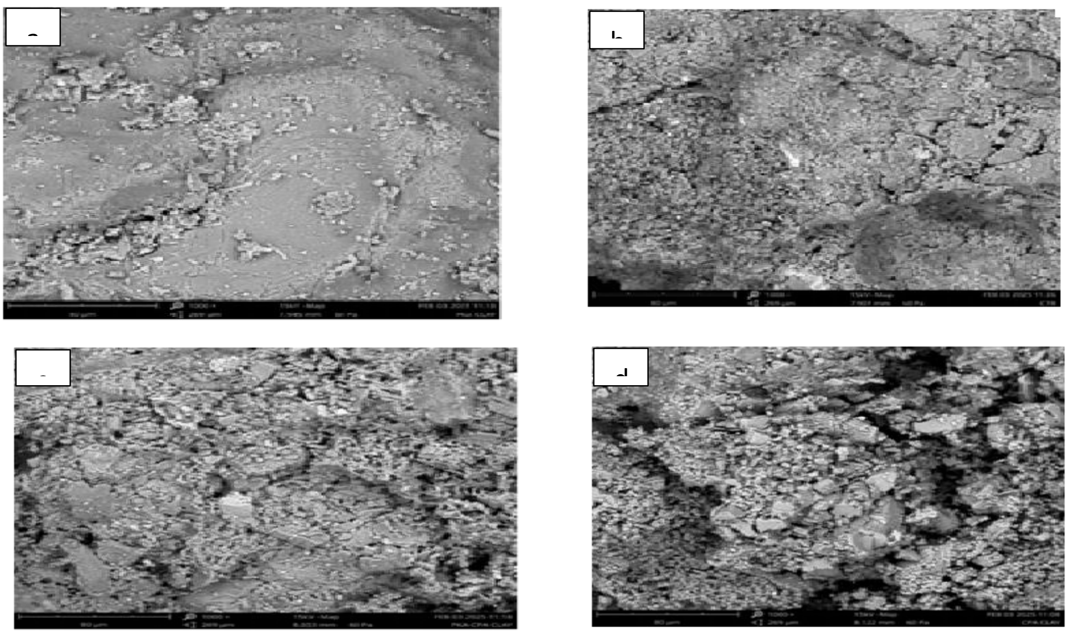

Figure 6: The Micro-structural Image of (a) control sample (b) clay/30CPA (c) clay/30PKA (d) clay/30CPA-PKA (× 80 µm magnifications).

The control sample exhibited a relatively dense matrix (Fig. 6a) with a somewhat rough and uneven surface texture. These suggest a matrix composed of fine particles that are reasonably well-packed without holes. This accounted for the better density observed for the sample. Pores or voids are also noticed within the matrix. The distribution of these pores is crucial for insulation. The sample, clay/30CPA, displayed open and porous structure which accounted for the reduction in the density observed for that sample (Fig 6b). There's also a noticeable presence of larger aggregates or lumps of particles, along with some flake-like or layered structures. This might be due to interaction of the CPA with the clay during firing. The brick containing PKA (clay/30PKA) exhibited a less densely packed structure compared to the control sample and CPA bricks, which accounted for its lower density (Fig. 6c). The hybrid sample containing a mixture of PKA and CPA (clay/30PKA-CPA) (Fig. 6d) showed characteristics that fall between the clay/30CPA and clay/30PKA bricks structure, with portions of agglomerated particles.

CONCLUSION

This work studied the influence of agro-wastes on the bulk density, apparent porosity and firing shrinkage of locally produced refractory bricks. The following conclusions were drawn within the limit of this research:

- The control sample possessed high density of 2.3 gcm^{-3} while samples clay/30CPA, clay/30PKA and clay/30CPA-PKA (hybrid) have densities 2.0, 1.8 and 1.8 gcm^{-3} respectively. Although they fall within the standard recommended density, samples containing agro-wastes are suitable for lightweight refractory brick.

- The porosity of the samples containing agro-wastes displayed higher porosity percentage of 20.63%, 26.51% and 25.5% for samples clay/30CPA, clay/30PKA and clay/30CPA-PKA (hybrid), respectively, compared to 19.8% for the control sample. This suggests the samples containing agro-wastes are suitable for insulation purposes.

- Samples containing agro-wastes clay/30PKA and clay/30CPA-PKA (hybrid) showed better shrinkage of 2.17% while control and sample clay/30CPA shared similar shrinkage of 4.3%.

- The results showed that the incorporation of the agro-wastes did not alter the required physical characteristics of the developed bricks. However, this research shows that high quality insulating refractory bricks can be produced with clay containing agro-wastes of 30%PKA, but the best is experienced with the clay of 30%CPA-PKA (hybrid) respectively. Also, the improvement of mechanical properties of the produced insulating bricks mentioned above are dependent on the combination of CPA and PKA the agro-waste used.

- Further study can be carried out to assess the effect of heating temperature on the property of the refractory bricks

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