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PHYSICO-MECHANICAL PROPERTIES OF REFRACTORY BRICKS OF ILARO CLAY WITH BLEND OF MELON SHELL AND RICE HUSK ASHES

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ABSTRACT

Refractory bricks (RB) made from clay (kaolin) are widely utilized as linings in high thermal system where intense heat is desired. Melon Shell Ash (MSA) and Rice Husk Ash (RHA) have the potential to increase and stabilize the SiO_2 and Al_2O_3 contents in kaolin due to appreciable percentages. This study examined the physico-mechanical properties of refractory bricks produced from Ilaro kaolin reinforced with MSA and RHA. Kaolin deposits from three locations in Ilaro, South West Nigeria were harvested. Taguchi Orthogonal array was adopted for the experimental design batch mix of kaolin, MSA, and RHA which were: 100:0:0 (ED_{m0}); 75:12.5:12.5 (ED_{m1}); 50:25:25 (ED_{m2}); 25:37.5:37.5 (ED_{m3}); 75:17.5:7.5 (ED_{m4}); 75:7.5:17.5 (ED_{m5}); 50:35:15 (ED_{m6}); 50:15:35(ED_{m7}); 25:50:25 (ED_{m8}) and 25:25:50 (ED_{m9}) weight percentage compositions. The samples were then used to produce ten batches of RB. Key physico-mechanical and thermal properties including: Density (D), Porosity (P), Cold Crushing Strength (CCS), Drying Shrinkage (DS), Firing Shrinkage (FS), Thermal Shock Resistance (TSR), and refractoriness (R) on the RB were determined following standard procedures and codes. Results for D, P, and CCS of the RB were: 1.65±0.1 – 1.85±0.2 g/cm^3, 28.2±0.1 – 50.4± 0.3%, and 14.45±0.1 – 22.5±0.2 MPa, respectively. Results for DS, FS, TSR, and R of the RB were: 0.87±0.1 – 2.23±0.2%, 0.45±0.1 – 1.83±0.3%, 22±0.1 - 29±0.2 cycles, and 1515±0.1 – 1551±0.3^{o}C, respectively. Sample ED_{m4} with DS, FS, TSR and R values of 0.87%, 0.45%, 29 cycles and 1551^{o}C respectively, had the best properties. The study found that Ilaro clay enhanced with MSA and RHA at an optimum ratio of 75:17.5:7.5 can be used for production of fireclay bricks for furnace linings.

Keywords: Refractory; clay; physico-mechanical; additives; Taguchi-Orthogonal.

INTRODUCTION

Clay is a naturally occurring fine-grain substance that becomes malleable when combined with a small amount of water (Aliyu *et al.*, 2021). As a raw material, clay varies widely in terms of its distribution of particle sizes, workability, structure, and mineralogical makeup. Clay is categorised on these basis into ball clays, plastic clays, fireclays, kaolin clays and flint clays (Andrew *et al*, 2020). They are divided and categorized as kaolinite, Illite, and Montmorillonite in the refractory industries (Momade and Gawu, 2019). Clay materials used in refractories are typically able to withstand extremely high temperatures without softening, deforming, or changing structurally. The kind of refractory needed for a particular function dictates the type of refractories. Refractories in these category are basic, neutral and acid refractories (ASTM-C27-98, 2020). Traditionally, clay materials contain $Al_2O_3.SiO_2.H_2O$, which results in refractory fireclay bricks made of alumino-silicates. They are further classified by temperature. At 1500–1700°C, it is referred to as refractory fireclay. For example, chromite, is classified as high refractory when it reaches 1700–2000 °C, while for temperatures over 2000 °C, (example zircon), it is referred to as super refractory. (ASTM-C27-98, 2020).

The fact that raw clay resources for modern bricks are readily available everywhere is primarily responsible for their widespread use. The mechanical, physico-thermal and fundamental mineralogical phases of various clay bricks are the main criteria implored to evaluate their quality and durability (Yagya *et al*., 2020). According to Karhu *et al*., (2019) and Vijayaragavan (2021), the quality of ceramic bodies is also allegedly dependent on the type of clay used in their production as well as the firing temperature conditions. These factors are important because they modulate the physico-mechanical characteristics of the various ceramic bodies (Karaman and Ersahin, 2023). Natural clay materials undergo a number of mineralogical phase transformations when fired. Al_2O_3 and SiO_2 impart the plasticity,refractoriness and shrinkage properties of the bricks, which differ depending on the kind of clay (Bordia and Canacho-Montes, 2018). Conversely, a range of feldspar types and oxides of titanium,potassium, magnessium, sodium, iron and calcium are dependable fluxing materials; their viscosity decrease as the clay brick products are formed (Njoya *et al*., 2020). The presence of low-melting fluxing oxides in clay minerals play significant roles in the production of all types of clay bricks. All varieties of clay bricks are produced in large part by the presence of low-melting fluxing oxides in clay minerals. At high temperatures, the fluxing oxides, primarily potassium and sodium oxides, react with the silica and alumina of the clay minerals to generate a liquid phase that aids in the densification process of the brick bodies (Sokola *et al*., 2020). They exhibit an inverse relationship with the apparent porosity or water absorption capacity of the fired brick bodies and a direct relationship with the bulk density characteristics (Sokolar and Vodova, 2019).

The development of environmentally-friendly clay bricks, enhanced with addition of trace amounts of agricultural wastes are recent areas of interests in the material technology world (Maraveas 2020; Kazmi *et al,* 2022, Abbas *et al,* 2023 and Bilgin *et al*, 2018). These waste products are typically abundant sources of fluxing oxides:, SiO_2, and Al_2O_3. In addition to minimizing the pollution overburden brought on by their deposition in environments, the waste materials demonstrated to have a good effect on the mechanical, chemical, and physical strength of the clay bricks at low production costs, resulting in more environmentally-friendly building. The various qualities of clay bricks were improved by these waste components, resulting in more affordable and environmentally-friendly construction (Yagya *et al*., 2020).

The mechanical characteristics of the fired-ceramic body diminishes, as the pore distribution, porosity and pore size increases (Aduda and Nyongesa, 2019). As the firing temperature rises, the total porosity of the brick samples falls and the distribution of pore sizes shifts towards larger pore sizes (Grubesa *et al*., 2020). Based on earlier researches, it can be said that brick specimens with high porosity and a large proportion of pores with a diameter of less than 2µm suffer from serious mechanical damages. Extremely porous clay bodies have huge capacity for water absorbtion, low density, and tendency to be less resilient with lesser stress resistance qualities (Coletti *et al*., 2018).

The mineralogical composition and physico-mechanical characteristics of bricks determine how good they are. Despite this, not enough research has been done on the physico-mechanical properties of commercial clay bricks in the Ilaro region. The technical feasibility of the commercial bricks is demonstrated by experiments conducted on a laboratory scale, making it an extremely feasible choice (Yagya *et al*., 2020). This research is aimed at determining the key physico-mechanical properties of Ilaro refractory bricks blended with melon shell ash and rice husk ash for use as linners in ovens.

MATERIALS AND METHODOLOGY

The clay sample used in this study was harvested from Ilaro in Yewa South Local Government Area of Ogun State. The clay samples deposit site were dug 2 m deep into the earth, using digger and shovel; 50 kg of the clay sample were packed into a polyethlene bag. Melon shell and rice husk additives of 20 kg each were purchased from a local market in Ilaro. The collection and transportation of the clay sample and additives were done afterwords (ASTM-D4220, 2020).

Sample Preparation

The as-mined clay sample was crushed, sieved using 750 µm sieve and soaked in water for one week, after which the water was decanted, and the slurry was sun dried for two weeks. The dried clay was then crushed and oven dried/pre-calcined at an average temperature of 110°C, and sieved into powder using a sieve with a mesh size of 75 µm in accordance with ISO 565. To create a homogeneous material mix, the clay was next combined with Melon shell ash (MSA) and later with Rice Husk Ash (RHA) additives, using Taguchi orthogonal array formulations (Table 1). The base materials were mixed manually. The homogeneous material mix was then supplemented with 15% by mass of Ordinary Portland Cement (OPC) of grade 42.5N as a chemical stabilizer until the proper mixing was achieved. Refractory bricks produced were subjected to physical and mechanical properties tests (Sahabi, 2017).

Chemical characterization of Clay Sample

The chemical composition of the clay was determined using X-ray Fluorescence (XRF) at the Federal University of Agriculture, Abeokuta, Center of Excellence in Agricultural Development and Sustenance Environment (CEADESE) Laboratory.

Production of the Fireclay Bricks

In order to eliminate trapped air that could cause the moulded brick materials to crack, the evenly mixed materials (clay, additives and binder) was carefully cast into a mould measuring 228.6 x 114.3 x 76.2 mm. It was thereafter extensively pressed, using hydraulic pressing equipment with a 12 tonnes capacity. To prevent cracking due to shrinkage, the created bricks were sun-dried for five days. Samples of the sun-dried bricks were oven dried for eight hours at 110^{0}C. The sample was left to cool and kept for physico-mechanical test.

Table 1: Percentage Proportion of Materials Mixes

Experimental Run	Kaolin (wt %)	MSA (wt%)	RHA (wt%)	Sample Identity
	100	0	0	ED_{m0}
	75	12.5	12.5	ED_{m1}
	50	25	25	ED_{m2}
	25	37.5	37.5	ED_{m3}
	75	17.5	7.5	ED_{m4}
	75	7.5	17.5	ED_{m5}
	50	35	15	ED_{m6}
	50	15	35	ED_{m7}
	25	50	25	ED_{m8}
	25	25	50	ED_{m9}

Physico-Mechanical Properties of the Brick Sample

Various laboratory analysis were carried out on the produced brick samples to investigate the physico-mechanical properties of the bricks using the specified American Society for Testing and Materials (ASTM) standard appropriately. The properties of the bricks samples were examined in the Material and Foundry workshop of the Department of Mechanical Engineering, The Federal Polytechnic Ilaro, Ogun State. ASTM standards testing procedure ASTM, (2021); ASTM-C20, (2020); ASTM-C1100-88, (2017); ASTM-C326-15, (2022); and ASTM-17 (2020) were employed to examine the key physico-mechanical properties. The details of the methods for the analysis procedure are as earlier reported (Shuaib-Babata *et al,* 2019; Momade and Gawu, 2019). The Physico-mechanical properties examined were: Density, Porosity, Cold crushing strength, Drying shrinkage, Firing shrinkage, refractoriness, and thermal shock resistance.

Density

Density is defined as the mass to volume ratio. It makes an estimate of how much clay there is in the volume of a brick. Mass, length, breadth, and height of the brick were measured using a mechanical balance and a Vernier caliper with sensitivity of 0.1 g and 0.01 mm, respectively, to determine the brick density. Three bricks from each series were measured, and the average densities were determined using Equation (1) (Christopher, 2020).

$$D = \frac{\text{Mass (m)}}{\text{Volume (v)}} \qquad (1)$$

Porosity (P)

The porosity of the produced fireclay bricks was calculated in accordance with Aliyu *et al*, (2021):

$$P = \frac{W-D}{W-S} \times 100 \qquad (2)$$

where:

W = Air weight of the sample, including moisture in its open pores.

D = Dry sample constant weight

S = Weight of the suspended sample in water

Cold Crushing Strength (CCS)

To get the CCS, the produced bricks were dried and heated to 1100°C in a furnace, for 8 hours and cooled in the furnace for 48 hours. To reach room temperature, the brick samples were cooled. The brick sample was installed on a compressive tester, and the cold crushing strength (CCS) was calculated according to Andrew *et al* (2020) using Equation (3): the crushing load applied axially was recorded.

$$\text{CCS} = \frac{\text{Maximun Load (kg)}}{\text{Cross Section Area (cm}^2\text{)}} \qquad (3)$$

Drying Shrinkage (DS)

The percentage difference between the initial dimensions of the brick materials and the dimensions obtained after drying was used to calculate the shrinkage of the brick samples. Equation (4) was used (Christopher, 2020).

$$\text{DS} = \frac{\text{WL} - \text{DL}}{\text{WL}} \times 100\% \qquad (4)$$

Where:

DS = Dry shrinkage, WL = Wet length, DL = Dry length

Firing Shrinkage (FS)

The dry bricks were heated to approximately 1100°C in a crucible furnace, then immersed for 8 hours at that temperature before being cooled for 48 hours in the furnace. Equation (5) was be used to evaluate shrinkage values (Christopher, 2020).

$$FS = \frac{DL - FL}{DL} \times 100\% \quad (5)$$

where: FS = Firing Shrinkage, DL = Dry Length, and FL = Firing Length

Refractoriness (R)

The refractoriness of any material refers to its capacity to withstand high fired temperatures without experiencing significant changes to its chemical, physical, or mechanical properties. Shuen's formula Ameh and Obasi (2019) - Equation (6), was hypothetically used to evaluate the refractoriness of each refractory brick.

$$R^{o}C = \frac{Al_2O_3 - Ro + 360}{0.228} \quad (6)$$

where: 0.228 and 360 are constants, Al_2O_3 = alumina % in the clay, R_O = sum of all other oxides besides silica.

Thermal Shock Resistance (TSR)

For this test, sixteen test bricks with dimensions of 228.6mm x 114.3mm x 76.2mm were used. Brick samples were placed in the muffle furnace, which was kept at a temperature of 1000°C to heat the bricks. The samples were allowed to soak for 60 minutes at the predetermined temperature, after which they were removed, cooled in still water for one minute, then air-cooled for five minutes, and then put back in the furnace to soak for 10 minutes at 1000°C before being placed in still water once more. This procedure was repeated until cracks were visible on the specimen. The number of full cycles that caused visible cracks on each specimen were recorded, and the average values for each batch were noted (Christopher, 2020).

RESULTS AND DISCUSSION

Characterization on Ilaro Clay deposit and Additives

The Ilaro Clay had Silica and Alumina contents of 44% and 48% respectively (Table 2). The Silica and Alumina contents of MSA and RHA additives were 74.8 %, 2.84 % and 78.35% and 0.18%, respectively (Table 2).

Table 2: Chemical Composition of Ilaro Clay and Additives Samples

Constituents	Clay	RHA	MSA
Al_2O_3	44.00	0.18	2.84
SiO_2	48.00	78.35	74.80
Fe_2O_3	2.20	0.23	1.13
CaO	0.05	2.72	2.31
MgO	-	-	0.40
K_2O	1.60	4.55	4.66
TiO_2	2.40	-	-
Na_2O	-	-	0.62
SO_3	-	-	0.72
P_2O_5	-	2.88	10.01
V_2O_5	0.50	-	0.01
MnO	0.14	-	0.37
Cr_2O_3	0.27	-	-
BaO	0.19	-	0.09
CuO	0.06	-	-
Cl	0.12	-	-
ZnO	-	-	0.49

Key Physico-mechanical Properties of the Produced Fireclay Bricks:

The key physico-mechanical properties of the produced fireclay bricks understudied in this research were: Bulk density, Porosity, Drying Shrinkage, Firing Shrinkage, Thermal Shock resistance, and Refractoriness.

Bulk Density

The values for bulk density of all samples were within the range of; 1.65±0.1 – 1.85±0.2 g/cm³ (Figure 1). These values compare well with standard density value of 1.7 – 1.9 g/cm³ for refractory brick (ASTM-C20-00, 2017). The density of the bricks reduced as the percentage composition of melon shell ash and rice husk ash increased. This is because the additives were calcined, before being employed for the brick production. The calcined structure of the additives resulted to increase in porosity, which leads to reduction in density values (Christopher (2020). Among all the brick samples, ED-m_8, with a density value of 1.65 g/cm^3 was closer in value to the standard brick.

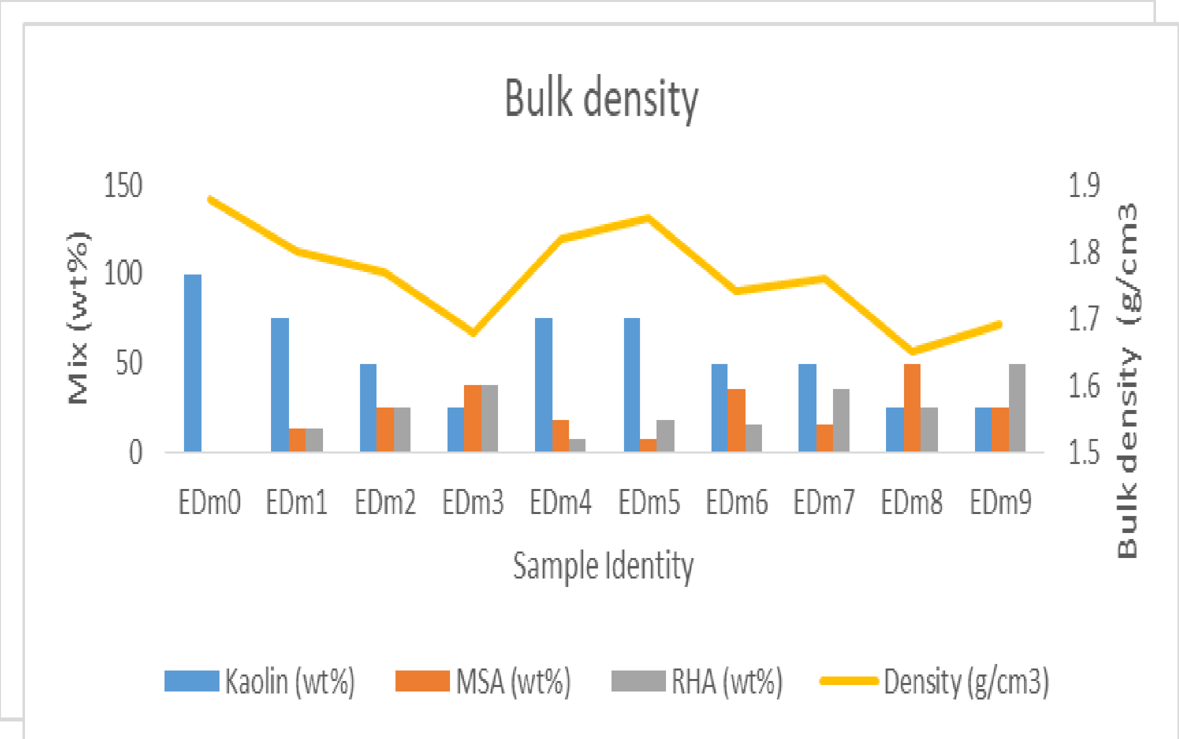

Figure 1: Bulk density values of the samples

Porosity

The porosity values of all samples were within the range of 28.2±0.1 – 50.4±0.3% (Table 3). These values fall within the range of standard bricks with values between 45 – 70% (ISTM, 2019). The results showed that an increase in the additive's percentage composition increased the porosity of the bricks. This is because more pores were created as the percentage of additives increased. This is in line with what was reported by Christopher, 2020 on porosity in this study.

Table 3: Porosity values of the samples

S/N	EDm$_0$	EDm$_1$	EDm$_2$	EDm$_3$	EDm$_4$	EDm$_5$	EDm$_6$	EDm$_7$	EDm$_8$	EDm$_9$ (%)
1	27.8	28.2	30.4	46.7	28.7	29.3	32.1	36.5	48.2	50.4

Cold Crushing Strength (CCS)

The CCS values of all the samples were within the range of: 14.45±0.1 – 22.5±0.2 MPa (Figure 2). Cold crushing strength increased with reduction in additives composition. This trend is attributed to high porosity caused by void created by the additives (Andrew *et al*, 2020). Among all the bricks produced, the brick with the highest cold crushing strength was EDm$_4$ with value of 22.5MPa. This may not be unconnected with the porosity value of EDm4. Research has shown that high porosity value affect cold crushing strength (Momade and Gawu, 2019). The brick with the least value of cold crushing strength was EDm$_0$ with value of 15.24MPa. The cold crushing strength of EDm$_0$ falls below the minimum standard value of 17.51MPa for refractory brick (ASTM-C27-98, 2020).

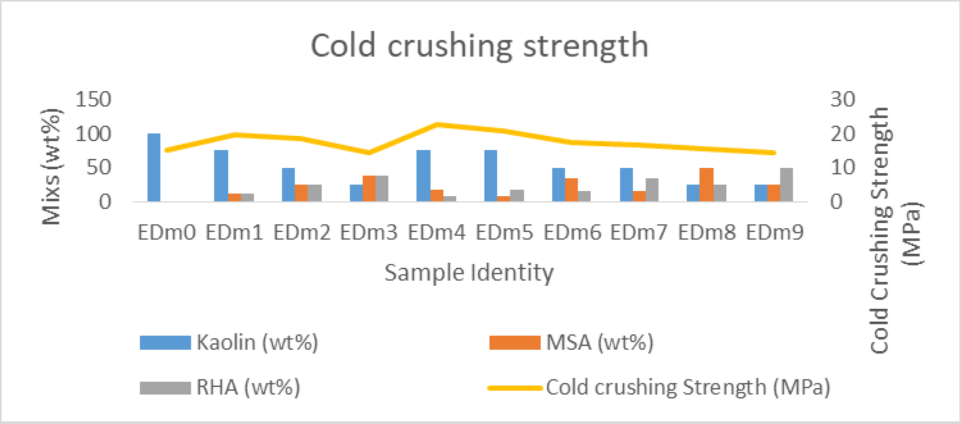

Figure 2: Cold crushing strength for the samples

Drying Shrinkage

The drying shrinkage results of all the samples were within the range of: 0.87±0.1 – 2.23±0.2% (Table 4). The percentage shrinkage increased with composition of additives. This is because bricks with high additives composition absorb more water, which subsequently shrink more and reduces water on drying. This trend falls in line with what Christopher (2020) reported on drying shrinkage. Among all the bricks produced, the brick with the highest drying shrinkage value was EDm$_8$ with value of 2.23%. The brick with the least value of drying shrinkage was EDm$_4$ with value of 0.87%. The results for drying shrinkage of the bricks compare well with standard value between 4 – 10% (Chima *et al,* 2017). The bricks also offer a 153% increase in drying shrinkage, when compared with that of the sample brick with no addiditive, with a value of 0.88%.

Table 4: Drying shrinkage values of the samples

S/N	EDm$_0$	EDm$_1$	EDm$_2$	EDm$_3$	EDm$_4$	EDm$_5$	EDm$_6$	EDm$_7$	EDm$_8$	EDm$_9$ (%)
1	0.88	0.96	1.14	1.26	0.87	1.01	1.14	1.04	2.23	2.01

Firing Shrinkage

The firing shrinkage values were within the range of; 0.45±0.1 – 1.83±0.2% (Figure 3). The firing shrinkage values increased with composition of additives. This is because bricks with high additives composition absorb more water, which subsequently shrink more and reduces water on firing. This trend falls in line with what Christopher (2020) reported on firing shrinkage. Among all the bricks produced, the brick with the highest firing shrinkage value was EDm_5 with value of 1.83%. The brick with the least value of firing shrinkage was EDm_4 with value of 0.45%. The results for firing shrinkage of the bricks compare well with standard value between 7 – 10% (ASTM-D4220, 2020). The results for firing shrinkage of the refractory bricks when compared with the control sample having value of 0.66%, the refractory bricks recorded 177% increase in firing shrinkage.

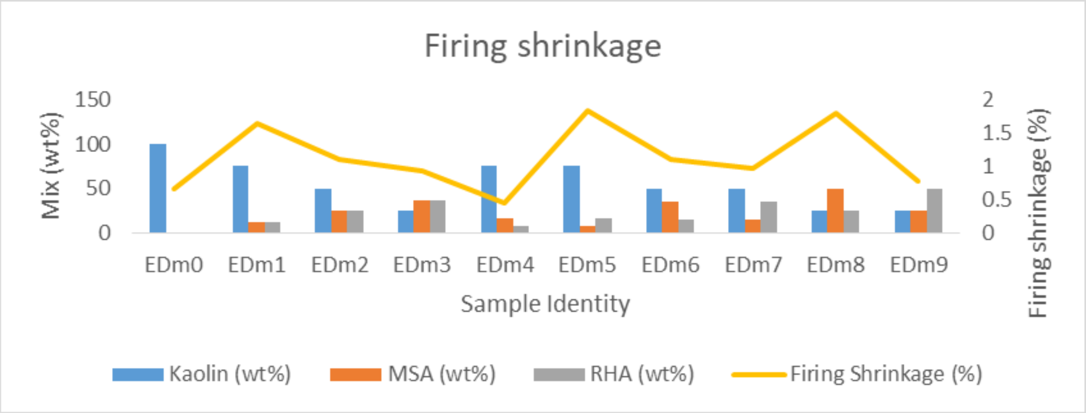

Figure 3: Firing shrinkage values of the samples

Thermal Shock Resistance

The values for thermal shock resistance of the samples were within the range of; 22±0.1 – 29±0.2 cycles (Figure 4). This essentially indicates that the bricks can resist minimum 22 cycles of heating and cooling and maximum 29 cycles of heating and cooling. The appreciable values of thermal shock resistance for the brick were not unconnected with the uniform distribution of pores in the bricks micro-structure thereby increasing its resistance to heating and cooling cycles. Among all the bricks produced the brick with the highest thermal shock resistance value is EDm_8 with value of 29 cycles. The brick with the least value of thermal shock resistance is EDm_5 with value of 22 cycles. The results for thermal shock resistance of the bricks compare well with standard value between 20–30 cycles (ASTM -D4220, 2020). The results for thermal shock resistance of the refractory bricks when compared with control sample having value of 23 cycles, the refractory bricks recorded 26% increase in thermal shock resistance.

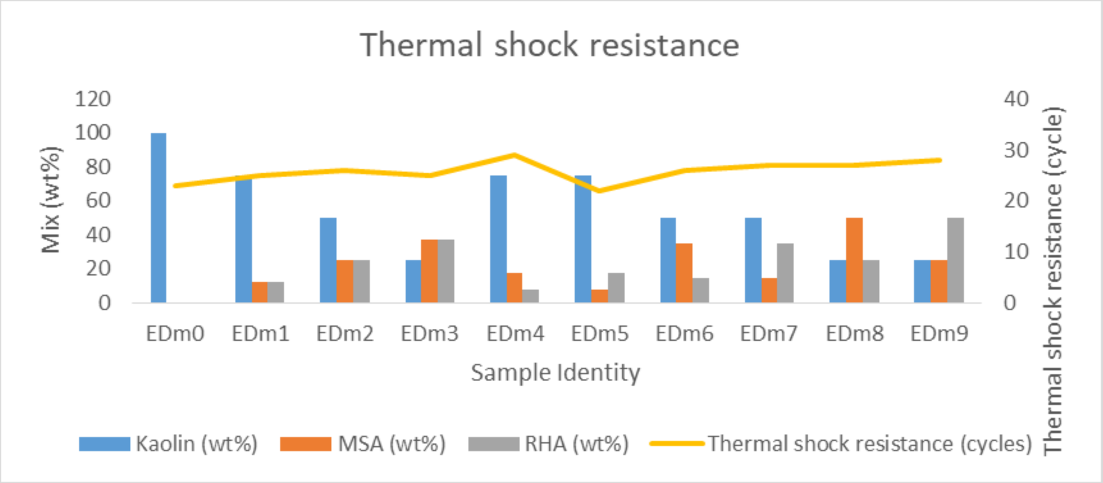

Figure 4: Thermal shock resistance of the samples

Refractoriness

The results for refractoriness of the samples were within the range of; 1515±0.1 - 1551±0.3°C (Table 5). The refractoriness values tend to increase with a corresponding increase in alumina content. This is because the alumina content in refractory bricks generally impact its refractoriness property (Christopher, 2020). Among all the bricks produced the brick with the highest refractoriness value is EDm$_4$ with value of 1551°C. The brick with the least value of refractoriness is EDm$_9$ with value of 1515°C. The results for refractoriness of the bricks compare well with standard value between 1500-1630°C (ASTM-D4220, 2020). The results for refractoriness of the refractory bricks when compared with the control sample having value of 1545°C, the refractory bricks recorded 0.4% increase in refractoriness

Table 5: Refractoriness Values of the Samples

S/N	EDm$_0$	EDm$_1$	EDm$_2$	EDm$_3$	EDm$_4$	EDm$_5$	EDm$_6$	EDm$_7$	EDm$_8$	EDm$_9$ (°C)
1.	1545	1538	1533	1525	1551	1537	1548	1526	1518	1515

CONCLUSION

The characterizations of the compositions were successfully determined for the clay deposit and additives and the materials were used to produce fireclay bricks.

Key physico-mechanical properties of Density, porosity cold crushing strength, drying shrinkage, firing shrinkage, refractoriness, and thermal shock resistance were determined.

The chemical compositions of the samples were within the range for a standard clay and additives employed in the study.

Sample EDm$_4$ have the best properties in Drying shrinkage, Firing shrinkage, Thermal shock resistance, and Refractoriness.

The study has shown that Ilaro clay enhanced with melon shell and rice husk ashes can be used for the production of fireclay bricks for oven construction.

RECOMMENDATION

Further study is necessary to determine:

- The expanded properties; physico-mechanical properties of Ilaro clay.
- The semi-conductive properties of Ilaro clay.
- The detailed refractory property of Ilaro clay for furnace application.

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