



THE STUDY OF INSULATING PROPERTIES OF REFRACTORY BRICKS PRODUCED USING MELON SEED HUSKS AS PORE FORMER

Eugenia O. Obidiegwu^{1*}, Henry E. Mgbemere¹ and Arhuere O. Akporehe¹

¹ Department of metallurgical and Materials Engineering, Faculty of Engineering, University of Lagos, Lagos, Akoka, Nigeria.

***Corresponding author email: eobidiegwu@unilag.edu.ng**

<https://doi.org/10.30572/2018/KJE/150205>

ABSTRACT

Recently, the price of energy has escalated. To minimize the use of energy and to reduce its leakage to the surroundings, composite materials called insulating refractories are very important. This study investigated physical, mechanical and thermal characteristics of porous refractory bricks produced from Nigerian clay mixed with melon seed husks. The aim is to reduce the cost of production which arises from importation as a result of absence of superior quality local refractory bricks for insulation in most industries in Nigeria. The test samples were produced by blending fire-clay and melon seed husk having grain sizes of 212 - 300 μm . The test samples were shaped using steel moulds of internal dimensions (76.2 $\times$ 76.2 $\times$ 76.2) mm, (96.2 $\times$ 20 $\times$ 20) mm and (60 $\times$ 60 $\times$ 20) mm, for various tests. The samples were oven dried and fired at temperatures 950°C to 1150°C at 50°C intervals. Physical, mechanical, thermal tests, chemical compositions, Mineralogical and Microstructural analysis were conducted. The results showed that, clay with 25 and 30 weight percentage (wt.%) melon seed husks possessed the required refractory properties with cold crushing strength above the recommended ASTM Standard of 1000 kN/m².

KEYWORDS: Clay; Melon seed husk; Bulk density; Refractory bricks; pores



1. INTRODUCTION

Refractories are materials that can withstand physical wear, abrasion, impact, thermal shock, chemical attack, and high temperatures. With these refractories, reduction in heat loss from the reheating furnaces can be realized (Komori et al., 2023). Most of the industries in Nigeria depend heavily on the importation of refractory products for the linings and insulation of their furnaces working at a very high temperature. For example, in foundry, furnaces used for melting and for heat treatment of metals, use imported refractories which affect the cost of production and final selling prices of their products (Esezobor, et al., 2014). There is a high demand of cheap and superior porous refractories in industries operating at high temperature, this brings about the necessity for researching for new raw materials and additives to serve as pore forming agents for the domestic production of insulating refractory bricks. Many researches have been carried out in this regard. The study on the impact of firing temperature and percentage of additives on the behaviour of porous bricks was carried out. The bricks were produced using; kaolin, sawdust and rice husk (Mgbemere, et al., 2020). The result revealed that the brick samples fired at 1200°C had improved properties when compared to those fired at 1100°C. It was also, observed that the brick samples contain required porosity for insulation. Nevertheless, when the amount of rice husk was increased above 30 wt.%, the pores begin to collapse. Angel et al., (2017), studied the impact of adding the husks from the seed of sunflower and the straw of wheat as additive to form pores in clay bricks. The results indicate that as the quantity of organic matter increases, the porosity also increases. The study on the effect of temperature of firing on the behaviour of porous bricks produced using mixtures of fire-clay and sawdust was carried out by (Folorunso, et al., 2015). Several tests were carried out on the samples. The results revealed that the bricks did not disintegrate at 1500°C, even at low percentage of alumina contents. Nevertheless, the samples with more than 20% composition of sawdust collapsed at higher temperatures. Obidiegwu et al., (2020) evaluated the thermal and mechanical behaviour of porous refractory bricks made from local fire-clay combined with the particulates of the shells of gmelina seeds. The results from the research revealed that, bricks containing 25 to 30 wt% of shells of Gmelina seeds acquired the recommended thermal conductivity (0.12 – 0.28 W/mK) and crushing strength more than 1000 kN/m² which is the standard range recommended by ASTM. Hassan, et al., (2014), researched on the effect of adding rice husk and sawdust on the behaviour of local fire-clay. From the results, there was reduction in refractoriness of the bricks from 1300°C to 1200°C. Increase in porosity was also observed in the bricks produced with sawdust and rice husk when compared with the samples produced without them. In this research, the review of literature made indicates that, many seed shells have been used as

additive for pore forming, but husks of melon seed has not been researched for the application as an additive, or as a pore former for producing porous refractory bricks for insulation of furnace. Hence, the aim of the current work is to study the insulating behaviour of the refractory bricks produced from melon seed husks. This will help to discover a new and an alternative raw material for domestic production of insulating refractory bricks, thus, reducing importation and cost of production of refractory bricks.

2. METHODOLOGY

2.1. Materials and Equipment Used

The clay for this work was collected from Osiele in Ogun State, South West of Nigeria. Melon seed husk (Combustible material) as shown in Fig. 1, was sourced from a farm in Nasarawa state (North Central Nigeria). The equipment used for this study include; X-ray Fluorescence analyser, X-ray Diffractometer, electric oven, electric furnace, gas-fired kiln, electric water heater, pulverizer, ball mill, Scanning Electron Microscope and compression testing machine.

Other apparatus used are mesh sieves, digital weighing balance, spring balance, vernier calliper, thermometer, steel moulds, basin, and containers.



Fig. 1a. Melon seed husks before grinding.



Fig. 1b. Ground melon seed husk and mesh sieve.

2.2. Materials Preparation

Refractory insulating bricks were made from clay and melon seed husk. The melon seed husk was sundried to remove moisture present and crushed. Clay was milled and immersed in water for three days to form a slurry. Dirt and foreign substances were removed from the filtrate and decanted after three days. The remaining clay material was dried and ground into finer particles. The raw materials were ball-milled to powder and sieved using mesh sieves (212 μ m to 300 μ m). The prepared materials were measured and mixed with 50 ml of water (until a uniform consistency was achieved) in a bowl. The clay samples were blended with melon seed husk at

various proportions of 20%, 25%, 30%, 35% and 40% by weight [Table 1](#). The test samples were shaped by compressing in a steel mould of internal dimensions (76.2 × 76.2 × 76.2) mm, (96.2 × 20 × 20) mm and (60 × 60 × 20) mm were used for the production of different test samples. The mould surface was cleaned properly and lubricated with oil. The aggregate mixture was poured into the mould and rammed to fill the shape of the mould. The mould was then removed leaving behind the insulation brick in its green state. The dimensions and weight of the samples were measured to obtain their values.

The bricks were dried under sun for twenty-four (24) hours and the values of the dimensions were taken before they were placed in an electric oven for drying at 110°C for 24 hours. The dimensions were taken and recorded. After oven drying, the samples of the insulating brick were placed in a furnace, then fired at different temperatures (950°C, 1000°C, 1050°C, 1100°C and 1150°C) at the rate of 2.5°C/min and held for 2 hours. The fired length and weight of each respective sample was measured and recorded for various tests to be carried out.

Table 1. Composition of clay mixed with melon seed husk brick samples.

S/N	Wt. % of Clay	Wt. % of melon seed husk
M	100	0
M1	80	20
M2	75	25
M3	70	30
M4	65	35
M5	60	40

2.2.1. Apparent porosity

Characterization of the test samples were carried out in conformity with ASTM C373-88 (2006) standard, for bulk density and apparent porosity.

The samples were dried in oven for twenty-four (24) hours at 110°C and heated to the various sintering temperatures. The digital weighing balance was used to obtain weights of the dried samples (W_1) of the sintered sample bricks. Then the sample bricks were immersed in boiling water for two (2) hours, and afterwards allowed to cool to room temperature while still immersed in water. The test samples were suspended in water and their weights (S) were taken with the aid of a spring balance. The samples were removed from water, cleaned lightly and weighed in air to obtain the saturated weight (W_2). The apparent porosity can be calculated using

equation (1).

$$\text{Apparent porosity} = \frac{W_2 - W_1}{W_2 - S} \times 100 \% \quad (1)$$

Where: W_1 = Sample dried weight, S = Weight of the sample suspended in water,
 W_2 = Saturated weight (weight of samples Soaked in water and suspended in air)

2.2.2. Bulk density

It is the weight per unit volume of pore spaces present in the material. The bulk density was calculated using equation (2).

$$\text{Bulk density} = \frac{W_1 \times \rho_w}{W_2 - S} \quad (2)$$

Where: W_1 = Weight of dried sample, S = Weight of sample suspended in water

W_2 = Weight of soaked sample suspended in air, ρ_w = density of water

2.2.3. Cold crushing strength (CCS):

This is the capability of a material to withstand failure under compressive load at room temperature. The Cold crushing strength was determined by steadily increasing a compressive load on the test piece via a compressive strength testing machine until failure occurs on the sample. Fig. 2 shows the sintered test samples positioned between two plates of the compressive strength testing machine. A uniform load was applied on the cube-shaped ($76.2 \times 76.2 \times 76.2$) mm samples. The load or force at which a crack appears on the brick sample (maximum load) was noted. The CCS was obtained from the formula in equation (3).

$$\text{Cold crushing strength} = \frac{\text{Maximum Load (KN)}}{\text{Cross sectional Area (m}^2\text{)}} \quad (3)$$



Fig. 2. Compression strength testing machine.

2.2.4. Thermal conductivity (T.C.)

It is the capability of a material to transfer heat by conduction. The T.C. of the various test samples was determined using the formula in equation (4).

$$T.C. = \frac{2.303WHD[\text{Log}(\varphi_1/\varphi_2)]}{A \times \tau} \quad (4)$$

Where *T.C.* is the thermal conductivity of the sample (W/m°C), *T₃* is temperature of steam (°C), *T₁* is the lowest temperature of water (°C), *T₂* is the last temperature of water (°C), *τ* is Time (s), *A* is cross sectional area of the sample (m²), *W* is the mass of water (kg), *H* is the specific heat capacity of water (J/kg°C), *D* is samples' thickness (m), $\varphi_1 = T_3 - T_1$ and $\varphi_2 = T_3 - T_2$.

The microstructural examination of the brick samples was obtained using Scanning Electron Microscope (SEM).

The mineralogy of the fired bricks was determined using X-ray diffractometer (XRD). Each test sample was passed through the Rigaku XtaLAB mini 11 X-ray diffractometer.

3. RESULTS AND DISCUSSIONS

3.1. Chemical analysis

The chemical composition of the raw materials was carried out using X-ray Fluorescence Analyser. The result of the chemical analysis of the melon seed husk and clay using XRF analyser is shown in Table 2. The clay is composed mainly of 46.24% SiO₂ and 37.10% Al₂O₃ which indicates that it belongs to the family of aluminosilicate and semi-acidic refractory (Apeh et al., 2011). Other impurities such as MgO, MnO, K₂O, Fe₂O₃ and Na₂O are present in smaller proportion between 0.04% and 1.33%. However, the melon seed husk has a lower alumina content (Al₂O₃) of 3.83% and a higher loss on ignition of 26.5 % which indicates the presence of volatile compounds that will enhance pore formation.

Table 2. Chemical composition of raw materials using X-ray fluorescence.

Materials	Parameters (%)									
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	H ₂ O	L.O.I
Clay	46.24	37.1	1.33	0.06	0.86	0.5	1.04	0.04	0.05	1.24
Melon seed husk	51.013	3.234	3.27	10.21	0.537	-	5.014	0.221	-	26.5

L.O.I means Loss on ignition

3.2. Porosity

The apparent porosity of the samples as a function of firing temperature is shown in Fig. 3. The samples with the legend M, M1, M2, M3, M4 and M5 represent additives in weight percentage (wt. %) 0, 20, 25, 30, 35 and 40 wt. % of melon seed husks respectively. The results showed that the porosity increased from 21.1% to 55.4% with an increase in the composition of melon seed husk at 950°C. This is due to the fact that the melon seed husks escaped during firing and create pores in the brick. The porosity also decreases from 55.4% to 37.5% as the temperature increases from 950°C to 1150°C. This can be attributed to the bonding of the particles that took place at high temperatures as the pores tends to shrink.

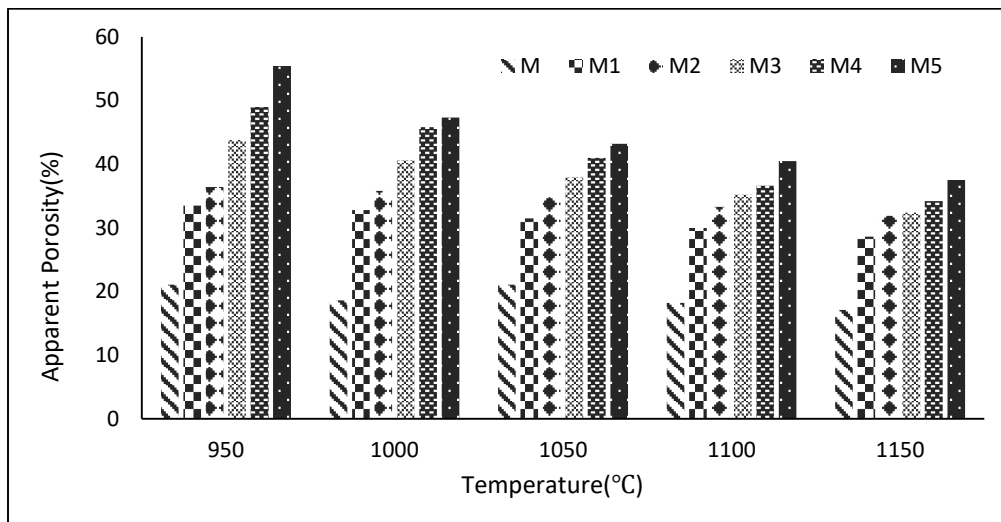


Fig. 3. Effect of firing temperature on porosity of melon seed husk brick.

3.3. Bulk density

The bulk density values of the samples fired at various temperatures is shown in Fig. 4. it was discovered that the bulk density decreases from 1.86 g/cm³ to 0.9 g/cm³ as the composition of melon seed husk increases from 0% to 40% at 1100°C. This can be attributed to an increase in the amount of pore spaces created inside the bricks during sintering (Ewais, et al., 2017) which led to a reduction in the packing efficiency and reduced matter contents of the samples. The bulk density also reduced from 1.86 g/cm³ to 0.81 g/cm³ as the sintering temperature increases.

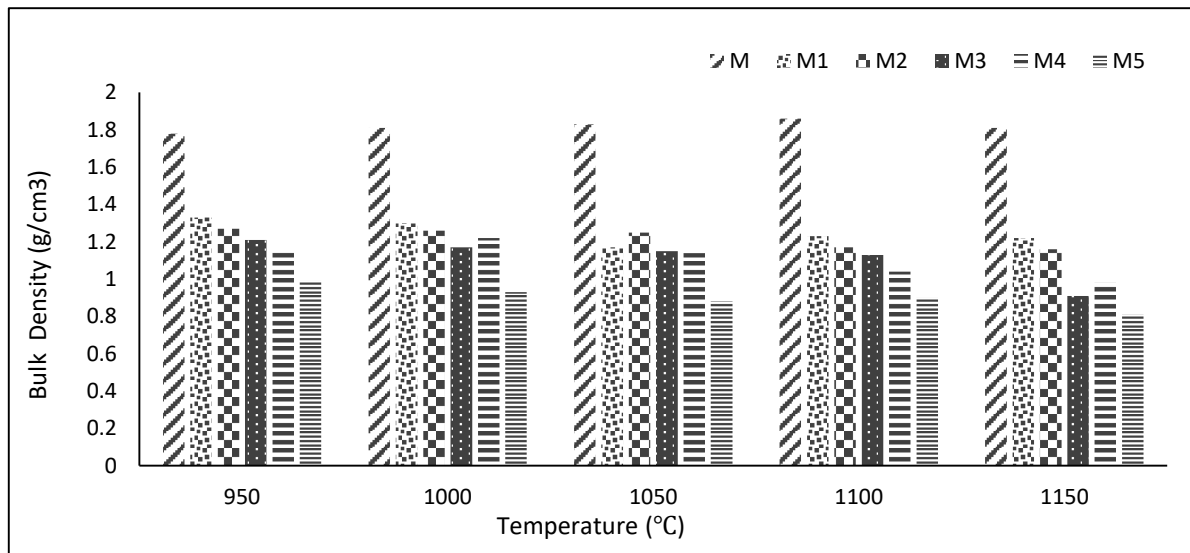


Fig. 4. Impact of firing temperature on Bulk density of melon seed husk bricks.

3.4. Cold Crushing strength (CCS)

Fig. 5 shows the impact of composition and sintering temperature on the cold crushing strength (CCS) of melon seed husk brick samples. It was observed from the results that the cold crushing strength reduced from 3448.48 KN/m² to 517.24 KN/m² with increase in melon seed husk addition from 0 - 40 wt.% (sample M to M5), when the samples were sintered at 950°C. At 1150°C, the cold crushing strength decreased from 4310.34 KN/m² to 517.24 KN/m² with the increase of melon shell husk composition. This is in agreement with the research carried out by (Ogbonlaiye et al., 2023). This can be attributed to the increase in porosity at that temperature which led to the decrease in load bearing capacity resulting to a decrease in the CCS. However, it was also observed that the cold crushing strength increased from 3448.48 KN/m² to 5172.41KN/m² with increase in sintering temperature from 950°C to 1150°C with 0 wt. % melon seed husk and from 948.28 KN/m² at 1000 °C to 1724.14 KN/m² at 1150°C with 30% melon seed husk addition. This could also be attributed to the formation of more bond in the body as fusion takes place within the sample upon increase in temperature. Also, increasing sintering temperature aided the completion of the crystallization process, closing of the open pores, which results in ceramic bond formation and improved CCS (Ewais, et al., 2017).

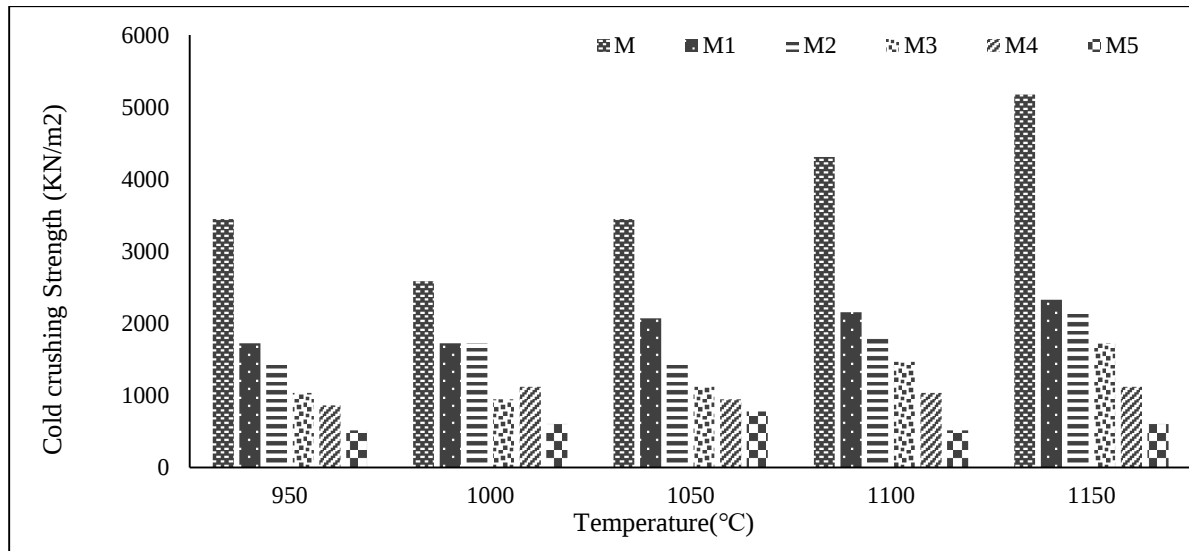


Fig. 5. Impact of firing temperature on CCS of Melon seed husk bricks.

3.5. Thermal Conductivity (T.C.)

The T.C. values of the bricks containing melon seed husk are presented in Fig. 6. The T.C. value reduces from 0.33 W/mK at 0 wt% melon seed husk (M) to 0.14 W/mK at 40 wt% melon seed husk content (M5) at 950°C. The T.C. value also decreases from 0.28 W/mK at 0 wt% (M) to 0.08 W/mK at 40 wt% melon seed husks (M5) at 1100°C. This could be as a result of the pores created by the melon seed husks on the bricks during sintering. These pore spaces or voids present in the samples hinders thermal flow which results in low T.C. of the bricks. The value decreases as the content of the melon shell increases. It was also observed that increasing the sintering temperature decreases the T.C. possibly due to the removal of volatile organic matter which create pores in the system.

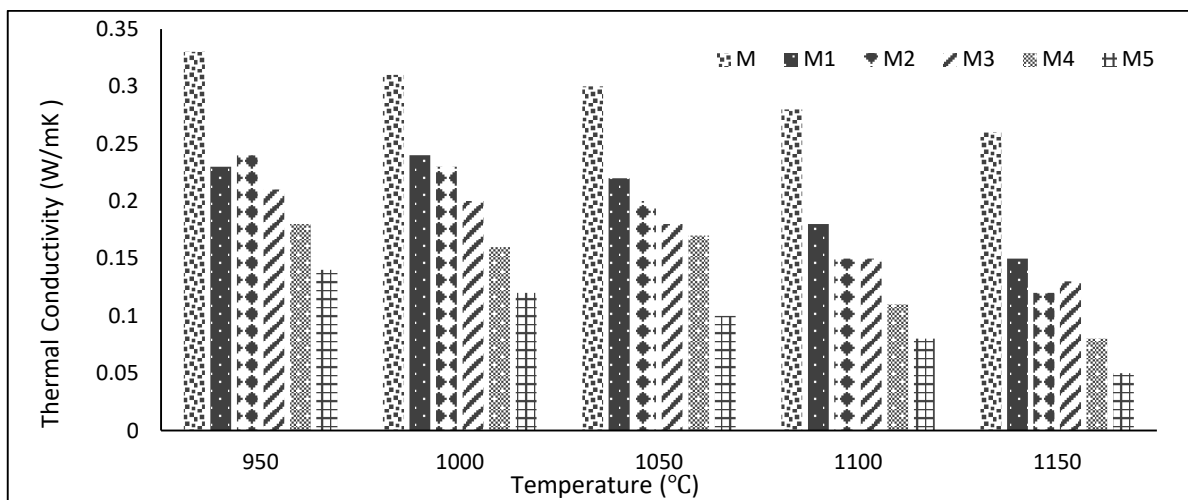


Fig. 6. Impact of composition and firing temperature on the T.C. of melon seed husk bricks.

3.6. Mineralogical examination

The X-ray diffraction study of the refractory bricks containing 0 wt. % and 20 wt.% of the melon seed husks and sintered at 1100°C are shown in Fig. 7 and Fig. 8 respectively. The XRD of fired clay brick without additive reveals the presence of quartz (SiO_2), Kaolin (kaolinite) ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), Mica ($\text{KAl}_2[\text{AlSi}_3\text{O}_{10}](\text{OH})_2$), feldspar (KAlSi_3O_8), illite ($\text{K}_2(\text{Al})_2(\text{Si}_3\text{AlO}_{10})(\text{OH})_2$), and Smectite ($\text{Al}_2\text{Si}_4\text{O}_{10}(\text{OH})_2$) with quartz having the highest peak intensity Fig. 7. Furthermore, the XRD result of the clay brick with melon seed husk additive also reveals the presence of quartz, mica, feldspar, illite and kaoline (kaolinite). These minerals such as kaolinite, quartz, feldspar and illite enhances the formation of liquid phases based on the temperature and percentage of silica and alumina content, aluminosilicate clays form different combinations of alumina, cristobalite, mullite, and liquid when fired (Obidiegwu et al., 2015). More so, at temperatures above 1100°C, the percentage of newly formed phases are inversely connected to the quantity of amorphous phase (glassy phase formed from the melting of feldspars and illite) in the sintered bricks. This conforms with the research, ‘The use of illitic clays in the production of stoneware tile ceramics’ by (Ferrari and Gualtieri, 2006).

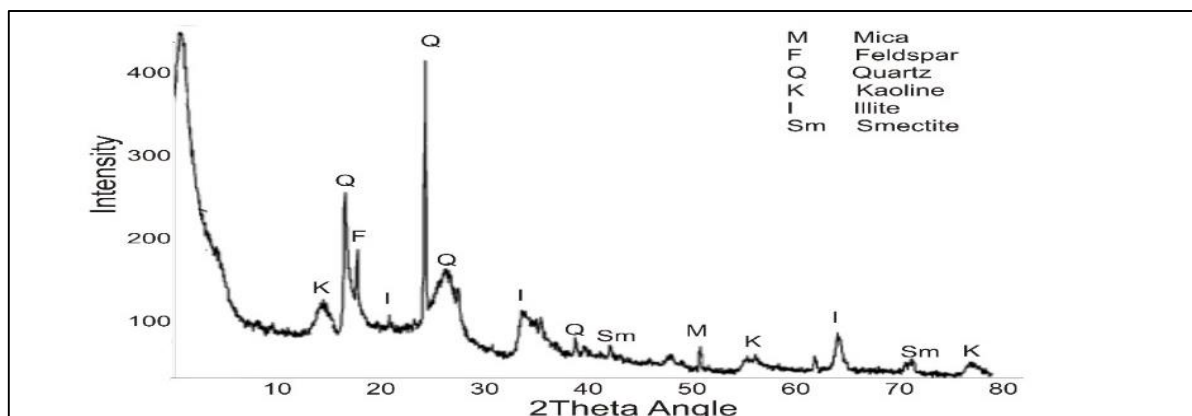


Fig. 7. XRD pattern of clay brick (0% additive) fired at 1100°C.

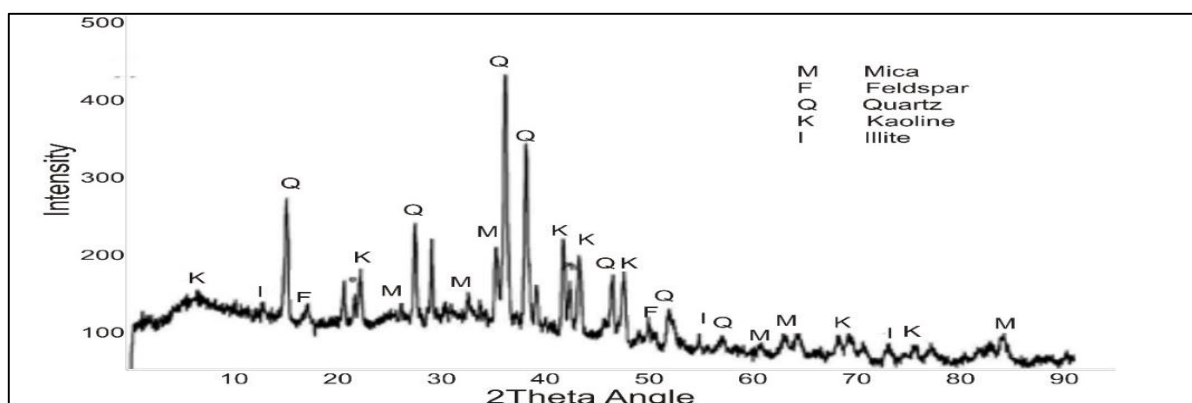


Fig. 8. XRD of brick (20% melon seed husk) fired at 1100°C.

3.7. Microstructural Examination

Figs. 9 and 10 show the scanning electron micrographs of clay sample M (without additive) and M1 (20% additive) respectively, sintered at 1100°C. The SEM micrograph shows the formation of small number of pores within the fired microstructure of the insulation brick sample M Fig. 9 while the size and number of pores within the fired microstructure of the insulation brick sample M1 (20% melon seed husk) increases with clear grain boundaries and contours (Mocciaroet. Al., 2023) Fig. 10. The increase in porosity as observed in Fig. 10 reduces the cold crushing strength of the refractory brick. This can be attributed to the fact that high strength varies inversely with high porosity. The greater and more numerous the pores, the thinner the walls of the solid material and the lower the strength (Jonker, 2006; Esezabor *et al.*, 2014). In addition, the thermal conductivity also decreases with increased porosity as the rate of heat flow in solid material reduces with increasing amount of pores. The EDX of the fired brick samples reveals the presence of Ca, Si, Al, Fe, N, O and C. This is in agreement with the chemical composition of the clay and melon seed husk used in the production of the insulating brick samples.

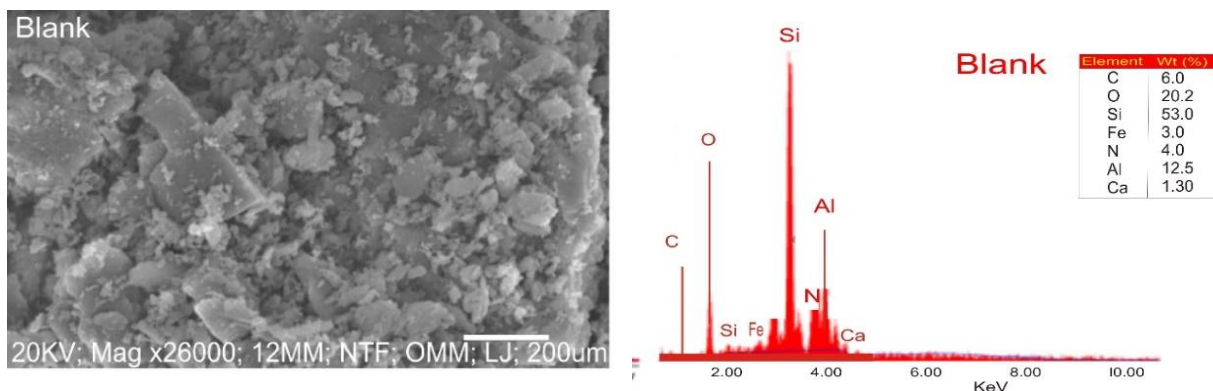


Fig. 9. SEM/EDX of Clay sample M (0 % wt additives) fired at 1100°C.

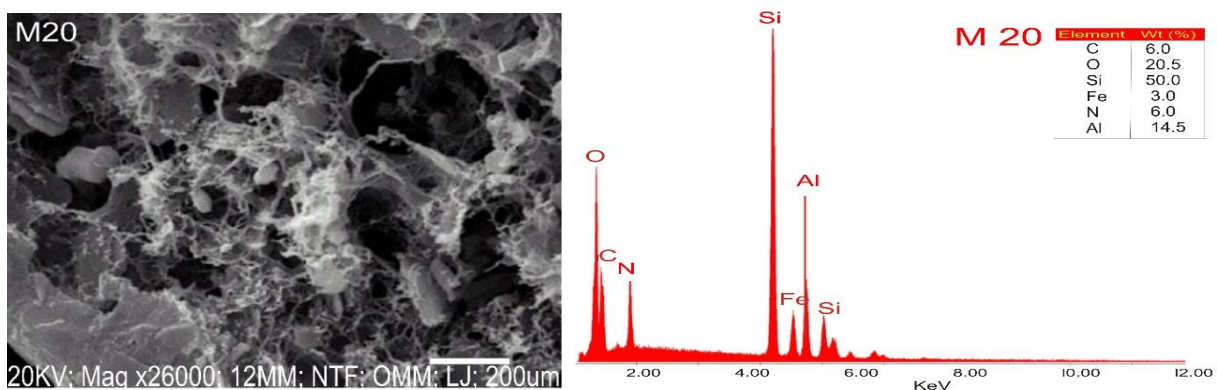


Fig. 10. SEM/EDX of brick sample M1 (20% melon seed husk) fired at 1100°C.

4. CONCLUSIONS

In this research work,

- Insulating refractory bricks were produced from locally available clay reinforced with melon seed husk particulates at different weight percentages and sintering temperatures.
- The Apparent porosity, bulk density, cold crushing strength, thermal conductivity tests as well as microstructural and mineralogical examination were carried out on the insulating refractory brick.
- As the amount of melon seed husk increases from 0% to 40%, the apparent porosity increases from 17.1% to 55.4% but decreases from 55.4% to 37.5% with increasing temperature which is within the standard range for insulating refractory bricks.
- The bulk density, cold crushing strength and thermal conductivity values generally decreased as the amount of melon seed husk increases.
- The SEM images showed that adequate pores for insulation were created in the sintered bricks with increasing weight percent of melon seed husk.
- Clay with 25% and 30% melon seed husk produced better refractory properties with cold crushing strength above the recommended ASTM Standard of 1000 kN/m², low thermal conductivity (0.12 to 0.24 W/mK) and bulk density (0.91g/cm³ to 1.27g/cm³) which falls within the standard values for refractory insulating bricks.

4. REFERENCE

Angel G., Albena Y. and Stoyan D. (2017). 'Effect of Wheat Straw and Sunflower Seeds Husks as Pore Forming Agents on the Properties of Porous Clay Bricks'. *Journal of Chemical Technology and Metallurgy*, 52(5), 885-891.

Apeh, F.I., Esezobor D.E., and Lawal G.I. (2011). 'Characterisation of Onibode and Owode-Ketu clays for use as refractory materials in foundry'. *Journal of Engineering Research*, 16(3), 69-77.

ASTM C 373-88 - Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products. West Conshohocken: ASTM International; 2006.

Esezobor D.E, Obidiegwu E.O. and Lawal G.I., (2014). 'The Influence of Agro-forestry waste additive on Thermal Insulating Properties of refractory bricks from Osiele Clay'. *Journal of Emerging Trends in Engineering and Applied Sciences*, 5(5), 305-311.

- Ewais, E.M., Elsaadany R. M., Ahmed A. A., Shalaby N. H, and Al-Anadouli B. E. H. (2017). 'Insulating Refractory Bricks from Water Treatment Sludge and Rice Husk Ash'. *Refractories and Industrial Ceramics*, 58(2), 136 – 144.
- Ferrari.S. and Gualtieri.A.F. (2006). 'The use of illitic clays in the production of stoneware tile ceramics'. *Applied Clay Science*, 32(1), 73–81.
- Folorunso, D.O., Aramide, F.O., Olubambi, P., and Borode, J.O. (2015). The Effects of Firing Temperature on the Performance of Insulating Firebricks Containing Different Proportion of Alumina and Sawdust, *Journal of Minerals and Materials Characterisation and Engineering*, 3(4), 309-317.
- Hassan, M., Yami. A., Raji., & Ngala, M. (2014). 'Effects of Sawdust and Rice Husk Additives on properties of Local Refractory clay'. *The International Journal of Engineering and Science*. 3(8), 40 – 44.
- Jonker A, (2006). 'Insulating Refractory Materials from inorganic Wastes Resources. PhD thesis submitted to the Department of Chemistry, Tshwane University of Technology, Pretoria, 11-47.
- Komori S., Komatsubara K., and Suetake S., (2023) 'Recent Technical Developments of Refractories and Ceramic Fiber Products for Reheating Furnace' *Reports*, 66(1), 1-11.
- Mgbemere H. E., Obidiegwu E.O., and Ubong A.U. (2020). 'The Effects of Sintering Temperature and Agro Wastes on the Properties of Insulation Bricks'. *Nigerian Journal of Technological Development (NJTD)*, 17(2), pp.113-119.
- Mocciaro A., Martinez J. M., Lopez P. V., Conconi M. S., and Rendtorff N. M. (2023). 'Mechanical and fracture behavior of insulating refractory bricks.' *Ceramics*, 69(1), 99-106.
- Obidiegwu E.O., Ochulor E.F. and Mgbemere H.E. (2020). 'Evaluation of Thermo-Mechanical Properties of Insulating Refractory Bricks Made from Indigenous Clay Mixed with Gmelina Seed Shells Particulates'. *ABUAD Journal of Engineering Research and Development (AJERD)*, 3(2), 19-26.
- Ogbonlaiye S. S., Erhuanga E. and Clement H. (2023) 'Development of High-Strength Insulating Refractory Bricks Using Locally Sourced Materials' *Journal of Materials Science Research and Reviews*, 6(4), 836-842.