



## Evaluation of Thermo-Mechanical Properties of Insulating Refractory Bricks Made from Indigenous Clay Mixed with Gmelina Seed Shells Particulates

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**Abstract:** Due to lack of good insulating refractory materials in commercial quantity, most of the manufacturing and processing industries in developing countries such as Nigeria depend mainly on imported insulating refractory bricks to line their furnaces. This is to improve energy efficiency and reduction in energy loss. Despite the abundant resources of fireclay in the country for the production of insulating refractory bricks, these industries depend mainly on the imported refractory bricks. This leads to the high cost of the final product. This work studied the properties of insulating refractory bricks produced from Nigerian clay blended with Gmelina seed shell particulate. The chemical compositions of the raw materials were analysed using Atomic Absorption Spectrometer. The samples were obtained by mixing both clay and Gmelina seed shells such that the composition of the Gmelina seed were 0 %, 20%, 25 %, and 30 %. The compressed bricks were dried and sintered at a heating rate of 2.5°C/min to temperatures ranging from 950°C to 1100°C at 50°C intervals. The results of the physical, thermal and mechanical properties show that when Gmelina content were between 25-30 wt% at temperatures between 1000°C to 1100°C, the porosity increased from 26.1 – 72.4%, bulk density decreased from 3.4 – 1.5 g/cm<sup>3</sup>, Cold crushing strength increased from 126 – 1162 kN/m<sup>2</sup> and thermal conductivity decreased from 0.28 – 0.12 W/mK. These results indicate that Gmelina shell particles helped to produce bricks with enhanced insulating properties. In conclusion, this work discovered a new refractory material (Gmelina seed shells which are in abundance in Nigeria). This can increase the physical, thermal and mechanical characteristics of a refractory brick. Also, this work could benefit the manufacturing and pottery industries, and in the production of refractory materials.

**Keywords:** Refractory, bricks, insulation, Gmelina seed.

### 1. INTRODUCTION

Refractory bricks play a very important role in manufacturing industries mainly in the lining of furnaces, kilns, fireboxes and fireplaces [1]. Insulating refractory brick also known as porous refractory brick is usually light in weight, low in thermal conductivity and resistant to temperature. It is used on the hot side of the furnace wall, where low thermal conductivity and low heat content are required. The low heat content is responsible for saving fuel and time on heating up. It allows rapid changes in temperature and permits rapid cooling.

These bricks are usually characterized by large amount of porosity, most of which are closed. The existence of high porosity in the bricks decreases the thermal conductivity of the refractory bricks [2].

Depending on the working environment, refractories need to be resistant to thermal shock and also be chemically inert. Good fireclay refractories should always have 24-26% plasticity [3].

The increasing demand of low cost and high-quality insulating refractory materials, in high temperature industries has brought about the need of studying new raw and additive materials for the local production of insulating refractory bricks. Numerous pore-forming agents have been investigated in the literature.

The investigation on the effect of sintering temperature and amount of pore formers on the properties of insulation bricks was carried out [4]. Kaolin ball clay, rice husk and sawdust were used for the production. It was observed that the samples that were sintered at 1200°C had slightly better properties compared to those sintered at 1100°C. Also, the structure of the samples contained adequate pores for insulation. However, the pores begin to collapse when the amount of rice husk was more than 30 wt.%.

In a paper [5], the characterization of Baruten fireclays as a suitable refractory material was studied. The clays are found to be good materials for refractory applications and could suitably replace imported clays in refractory application.

The effect of wheat straw and sunflower seeds husks as pore forming agents on the properties of porous clay bricks was studied [6]. There is a significant positive correlation between the increasing quantity of organic matter and the porosity.

This study shows that 5 mass % of wheat straw and 3 mass % of sunflower seeds husks can be used as pore forming agents in bricks production to improve their thermal conductivity and keep acceptable compressive strength.

The influence of particle size of ball clay blended with kaolin and sawdust on thermal diffusivity of ceramic bricks was studied by [7]. The research revealed that the coefficient of thermal diffusivity increases with decrease in particle size of kaolin, ball clay and sawdust.

The insulating bricks made from rice husk blended with two clay deposits from Ekiti State in Nigeria was characterized by [8]. The results indicate that they all had enhanced refractoriness and insulating characteristics.

The use of sawdust in the production of insulating bricks was studied by [9]. The influence of adding this sawdust on the properties of the brick was determined by testing the physical, thermal and mechanical properties. These tests showed that the produced bricks were appropriate for use.

In the study by [10] the influence of sintering temperatures on the properties of insulating bricks made from a mixture of hydro-metallurgically processed clay and sawdust was investigated. The samples were subjected to several refractory tests and it was observed that the bricks did not crumble at lower alumina contents, even at 1500°C. However, the samples containing more than 20% alumina crumbled at elevated temperatures,

The influence of adding rice husk and sawdust on the properties of indigenous fire-clay was studied by [11]. The results showed that the refractoriness of the bricks reduced from 1300°C to 1200°C. It was also discovered that the porosity of the samples containing sawdust and rice husk increased compared with the samples made without additives.

The use of coconut shell to improve the insulating properties of selected Nigerian refractory fire clays was investigated [12]. Their results indicated that clays containing 25 wt. % - 30wt% coconut shell with particle sizes of 212-300  $\mu\text{m}$  sintered at 1150°C - 1200°C possessed required Porosity, cold crushing strength and thermal conductivity. It was therefore concluded that refractory bricks with high quality and improved thermal properties could be made from Kankara, Osiele and Ukpok, clays mixed with coconut shell particulates.

The investigation conducted on the Obe clay deposit for Refractory Production showed that its service properties have favourable results in comparison to standard fireclay refractory materials [13].

The literature review conducted in this research revealed that there are other shells which their uses as pore former have been studied, but to the best of the authors' knowledge, Gmelina seed shells have not been studied or being used as a pore former in the production of insulating refractory bricks. Therefore, the aim of this study is to utilize the Gmelina seed shell as a pore former for the production of low cost, high quality refractory bricks. This can provide an alternative to the imported refractory insulating bricks in the country, thereby reducing the cost of production.

## 2. METHODOLOGY

### 2.1 Materials used

In this study, the materials used are Gmelina seed shells, Clay and Lubricant (engine oil). Gmelina arborea is a medium-sized deciduous tree up to 40 m tall and 140 cm in diameter. It is a fast-growing tree frequently planted in plantations to produce wood for light construction, crafts, decorative veneers, pulp, fuel, and charcoal [14]. The fruit is up to 2.5 cm long, smooth, dark green, turning yellow when ripe and has a fruity smell.

The Gmelina tree and seeds are shown in Figure 1. The seed shells would be used in this study as a pore former.

**2.1.1. Method:** The Gmelina seed shells which were obtained along the Lagos-Ibadan express road were crushed and ground to powder and were sieved to obtain  $\leq 250$  microns particle sizes.



Figure 1a: Typical images of Gmelina tree [15]



Figure 1b: Gmelina seeds [15]



Figure 1c: Sample of the raw Shells



Figure 1d: Sample of the Ground Gmelina Seed Shells

The clay which was also obtained in lump forms from the Osiele clay deposit was also jaw crushed and ball milled into powder form. The jaw crushed and ball milling machine is shown in Figure 2a and 2b



Figure 2(a): Jaw Crushing Machine



2(b): Milling Machine

The clay bricks were prepared by manual mixing of 100, 80, 75, and 70 wt% of clay with 0, 20, 25 and 30wt% of ground gmelina seed shell respectively. The mixture was carried out by adding a little quantity of water to improve plasticity and ensure homogeneity of the Gmelina seed shell powder and clay. The brick samples were produced by manual shaping and compressing in a stainless steel mould using manual ramming. To ensure that the same compression force is applied, the ramming was done 50 times for each moulding. Figure 3 shows different moulds and ram used.



Figure 3(a): (76.2 mm cubic mould and ram)



3(b) 60 x 60 x 15 mm mould

After moulding, the weight and dimensions of the cross section were measured to obtain its values. The obtained samples were then sun-dried for 24 hrs after which the dimensions were measured again. After undergoing sun drying, the samples were then dried in an oven for about 24 hours after which the cross section was retaken. Lastly, the brick samples were put inside a furnace and sintered at varying temperatures (950, 1000, 1050, 1100 & 1150 °C) at the rate of 25°C/min. Various physical, mechanical and thermal tests were carried on the sintered samples.

## 2.2. Chemical Composition Analysis

The chemical analysis of materials was carried out using Atomic Absorption Spectrometer (AAS) at the Laboratory of the Department of Chemistry, University of Lagos. Figure 4 shows the picture of AAS.



Figure 4(a): Atomic Absorption Spectrophotometer (AAS) Perkin Elmer Analyst 200 and (b)DR/ 2000 Direct Reading Spectrophotometer

The clay (50 g) was mixed with flux (sodium carbonate) using manual roller, in the proportion of 1:5. The mixed sample was transferred to a silica evaporating dish and evaporated to dryness on a steam bath. The dish was then placed on a muffle furnace and heated to 400-500°C until the sample became a white ash. For sodium determination, the sample was heated to 600°C. The left-over ash or residue was dissolved in undiluted HNO<sub>3</sub> and warm water. It was then filtered to make up the volume of 100 ml in a volumetric flask, with final HNO<sub>3</sub> concentration of 1%. The collected filtrate was tested for various parameters using Atomic Absorption Spectrometer (AAS) and the result of each parameter was expressed as the percentage of the total filtrate for better understanding. The result of chemical analysis of the raw materials is presented in Table 1.

Table 1: Chemical analysis of the raw materials

Materials	Parameters (%)									
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	H <sub>2</sub> O	L.O. I
Osiele Clay	46.24	37.10	1.33	0.06	0.86	0.50	1.04	0.04	0.05	1.24
Gmelina seed Shell	0.017	0.009	0.027	0.05	0.02	0.02	0.001	0.009	0.09	99.45

Note: L.O. I means Loss on Ignition

### 2.3. Bulk Density

This is the weight per unit volume of the refractory material, this include the volume of open pore space. Bulk density and apparent porosity were determined in accordance with ASTM C373-88 2006 standard. The weights (D) of the fired brick samples were measured. Then the bricks were soaked in hot (boiled) water for 3 hours, the soaked weights (W) were measured. The bricks were suspended in water thereafter and the weights (S) were measured. The bulk density of the bricks was calculated using Equation (1).

$$\text{Bulk Density} = \frac{\rho \times D}{W - S} \quad [16] \quad (1)$$

$\rho$ , D, S and W represent; density of water, dried, soaked, and suspended weights of the samples respectively.

### 2.4. Porosity

Porosity refers to the percentage change in volume of voids over the total volume of the sample. This can be calculated using Equation 2.

$$\text{Porosity} = \frac{(W - D)}{(W - S)} \times 100 \quad [16] \quad (2)$$

### 2.5. Linear Shrinkage

This is the reduction in size of ceramic sample when it is sintered due to particle rearrangement through diffusion. The shapes undergo change on heating or after reheating under a given set of conditions. Even, if a brick is capable of withstanding normal loading in operation it can cause serious problems due to permanent shrinkage. The linear shrinkage of the brick samples was determined by measuring the dimension of the bricks before and after sintering. The average linear shrinkage was evaluated using Equation 3

$$\text{Linear shrinkage} = \frac{(L_0 - L_1)}{L_0} \times 100\% \quad [16] \quad (3)$$

Where, L<sub>0</sub> and L<sub>1</sub> are the original and final length in (mm) of the samples before and after sintering respectively.



**2.6. Cold Crushing Strength (CCS)**

It is the ability of a material to withstand abrasion and loading without damaging or crumbling into powdered form. A Universal testing machine (Testometric M-500-25kN) was used to determine the maximum load. The cold crushing strength was calculated using Equation 4 in accordance with ASTM C133-97(2008) E1.

$$CCS = \frac{\text{Maximum Load}}{\text{Cross Sectional Area}} [16] \tag{4}$$

**2.7. Thermal Conductivity**

The brick samples were tested for thermal conductivity after determining the parameters in Equation 5, which is the formula for thermal conductivity.

$$K = \frac{2.303MC\delta[\log(\frac{\theta_1}{\theta_2})]}{A \times \tau} [17] \tag{5}$$

K represents the thermal conductivity of the specimen, the unit is W/m°C. T<sub>s</sub> is temperature of steam (°C), T<sub>1</sub> is the initial temperature of water (°C), T<sub>2</sub> is Final temperature of water (°C), τ is Time (s), A is the area of the Specimen in m<sup>2</sup>, M is the mass of water (kg), C is specific heat capacity of water (J/kg°C), δ is thickness of specimen (m),  
θ<sub>1</sub> = T<sub>s</sub> – T and θ<sub>2</sub> = T<sub>s</sub> – T<sub>2</sub>.

**3. RESULTS AND DISCUSSION**

The results of the chemical composition analysis in Table 1 indicate that the main constituents in Osiele clay are Silica (SiO<sub>2</sub>) and Alumina (Al<sub>2</sub>O<sub>3</sub>) so, the clay can be grouped into alumino-silicate family.

The Gmelina seed shell has high value of loss on ignition (L.O.I) of 99.45 % which indicates that it is organic in nature and contain volatile compounds making it suitable for use as a pore former.

**3.1 Bulk Density**

A, B, C & D represent 0 wt%, 20 wt%, 25 wt%, and 30 wt% of gmelina seed shell additive respectively. The results in Figure 5, show that the bulk density of the bricks varies inversely with the percentage composition of Gmelina seed shells. The density decreased from 3.5 – 1.5 g/cm<sup>3</sup> as the composition of gmelina seed shell increased from 0 – 30 wt%. This could be attributed to the fact that the Gmelina seed shells particulates were burnt off during sintering leading to creation of pores which results in low packing factor of the particle content per unit volume thereby leading to decrease in the density. It was also observed that the bulk density decreased from 2.1 – 1.59 g/cm<sup>3</sup> as the sintering temperature increased from 950 – 1100°C. Nevertheless, the bulk density of most of the samples are still within the standard value of < 2.1g/cm<sup>3</sup> for refractory bricks.

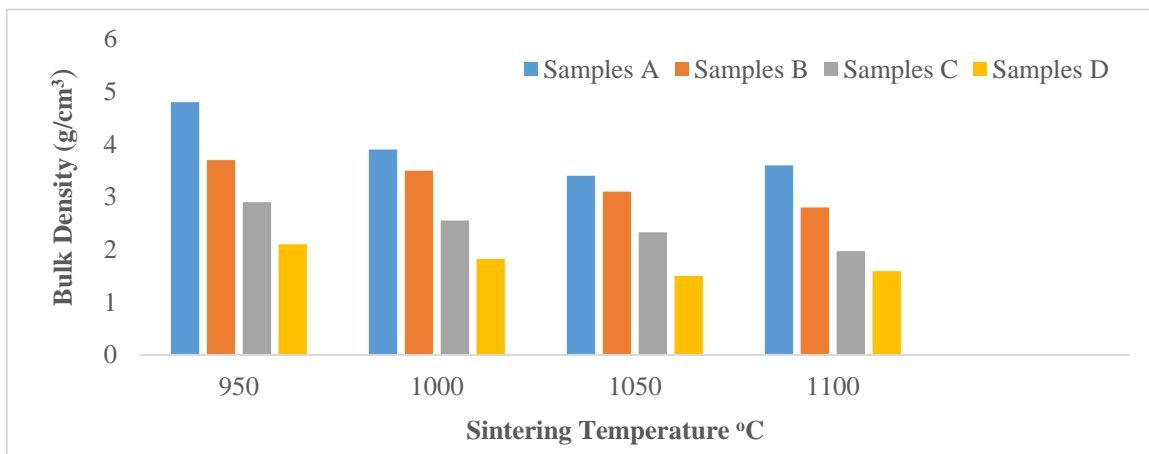


Figure 5: Effect of sintering temperature and composition on the bulk density of insulating brick samples

**3.2 Porosity**

From the result shown in Figure 6, it can be observed that the porosity increased from 26.1 – 82.5 % with an increase in the Gmelina seed shell composition. This is due to the amount of the organic matter that were burnt off during sintering. Nevertheless, as the sintering temperature increases from 950 - 1100°C, the apparent porosity decreases from 82.5 – 70 %.

The decrease in porosity is due to the fusion process that occurred at high temperature, which results to the formation of the particles into a coherent body with glassy phase. The process, tends to shrink the pores.

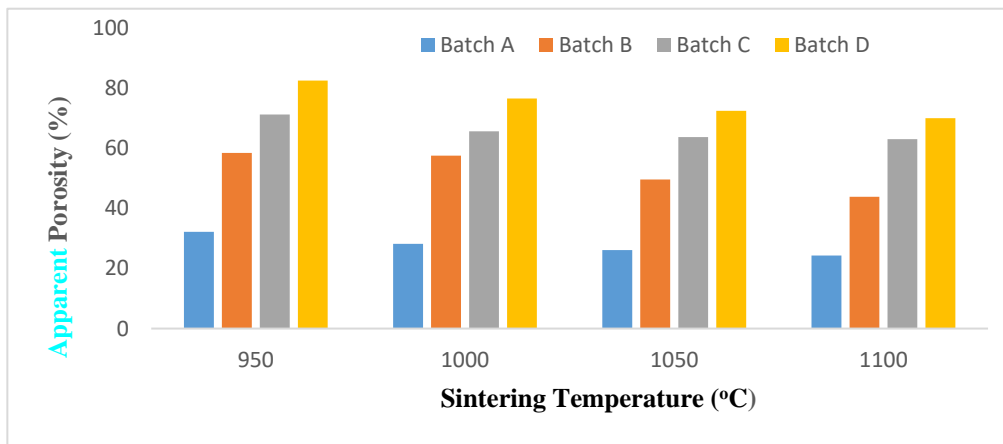


Figure 6: Influence of sintering temperature and % composition on the porosity of refractory brick

### 3.3 Thermal Conductivity

The thermal conductivity values of the samples are shown in Figure 7. The thermal conductivity decreases from 0.28W/mK at 0 wt% (Sample A) composition of Gmelina seed shell particulates to 0.12W/mK at 30 wt% (Sample D) of Gmelina seed shell composition, these indicate that Gmelina seed shells that burnt out during sintering created plenty of pores in the bricks. The presence of pores in the bricks improves thermal insulating property of the bricks. The pores hinder heat transfer from one particle to another, which results in low thermal conductivity of the brick. Furthermore, as the sintering temperature increases, the thermal conductivity decreases as a result of volatile organic matter vaporizing thereby creating pores.

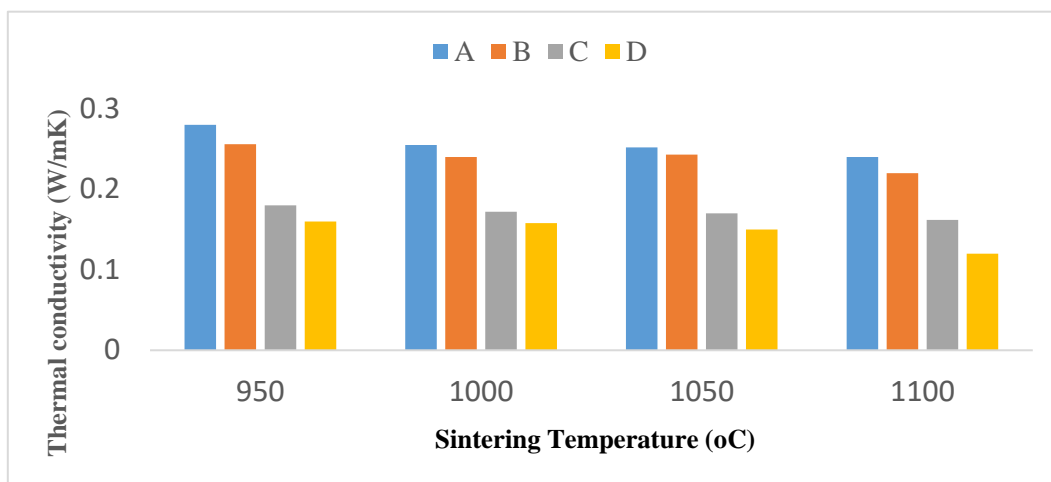


Figure 7: The effect of Sintering temperature and % composition on thermal conductivity of the bricks

### 3.4. Linear Shrinkage

The result in Figure 8 shows that the samples (A,B,C,D) sintered at 1100°C have the highest shrinkage value of 9.81%. Also, the increase in composition of the organic matter led to higher shrinkage which is a result of high percentage of Gmelina seed shells particulates that burnt off during sintering.

Generally, it was observed that linear shrinkage increases linearly with sintering temperature. This can be attributed to the loss in moisture and organic content in the brick. However, most of the Linear shrinkage obtained are within the ASTM standard value of less than 13 % for linear shrinkage of insulating refractory bricks.

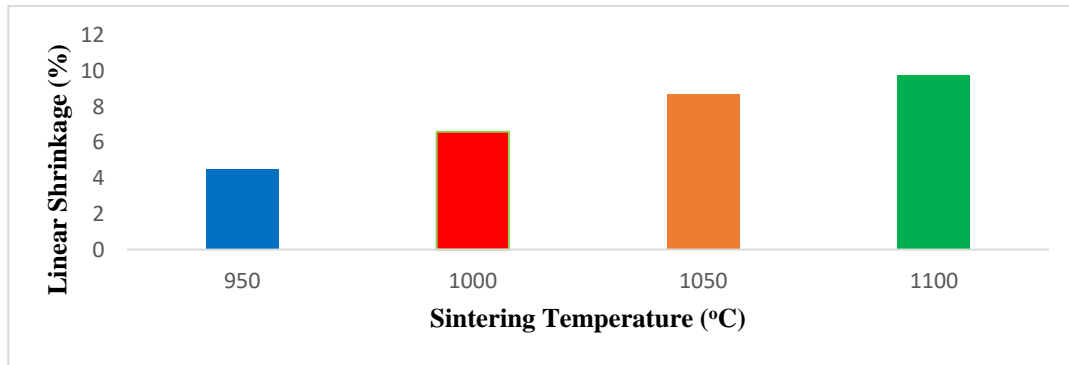


Figure 8: Effect of Sintering temperature on the Linear Shrinkage of the refractory brick samples

### 3.5. Cold Crushing Strength (CCS)

Figures The effect of sintering temperature and composition on the cold crushing strength of the brick is shown in Figure 9

It can be observed that there is an increase of CCS at temperature between 950°C to 1050°C. This indicates the range of temperature at which fusion of the particles took place. A coherent body is formed at this temperature leading to sharp increase in the CCS.

However, it was noted that there was a slight drop in the CCS between 1050°C and 1100°C. This is as a result of vitrification which significantly reduce strength of the bricks. Furthermore, CCS reduces with increase in the composition of Gmelina seed shell. Nevertheless, the values of CCS of the samples are within the standard value of not less than 1000 kN/m<sup>2</sup> recommended by [18] for porous refractory bricks.

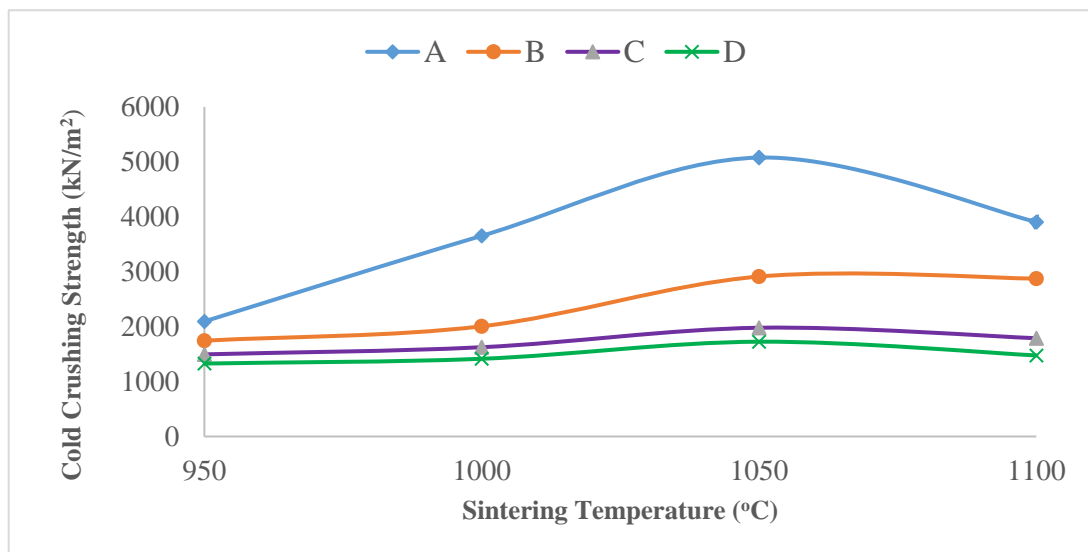


Figure 9: Effect of sintering temperature and composition on the cold crushing strength (CCS) of bricks samples

## 4. CONCLUSION

The effects of sintering temperature and the addition of Gmelina seed shell particulates in locally available clay to produce insulating refractory bricks has been studied in this research. The bulk density results show that as the sintering temperature increases from 950 – 1100°C, the bulk density decreases from 2.1 – 1.59 g/cm<sup>3</sup>. As the amount of Gmelina seed shell increases from 0 – 30 wt%, the bulk density decreases from 3.4 – 1.59 g/cm<sup>3</sup>.

Furthermore, from the results, bricks with 25 and 30 wt% of Gmelina seed shell particulates possess the required cold crushing strength above 1000 kN/m<sup>2</sup> which is within the recommended ASTM Standard. Also, thermal conductivity (0.28 – 0.12 W/mK), bulk density (< 2.1 g/cm<sup>3</sup>) and porosity (70 – 82.4 %) are all within the standard acceptable values. Therefore, Gmelina seed shell can be used as pore former for insulating refractory brick.

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