



## Engineering Characterization of Agbani Clay Deposit as Refractory Material for Furnace Lining

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*The engineering characterization of Agbani clay deposits has been investigated with a view of finding its usefulness in the engineering industry and home. The chemical analysis was carried out using X-ray fluorescence (XRF). Physical properties tests such as apparent density, apparent porosity, water absorption, shrinkage, modulus of rupture, bulk density, moisture content, conductivity, thermal shock resistance and specific gravity were all carried out using international accepted standard techniques. The result of the chemical analyzing showed that the clay has Al<sub>2</sub>O<sub>3</sub> (25.20%), SiO<sub>2</sub> (53.41%), K<sub>2</sub>O (2.06%), CaO (1.20%), TiO<sub>2</sub> (0.06%), V<sub>2</sub>O<sub>3</sub> (0.15%), MnO (0.43%) Fe<sub>2</sub>O<sub>3</sub> (2.10%) and CuO (0.01%), HgO (0.05%) as trace. The results of the physical properties tests conducted at a firing temperature of 900°C, 950°C, 1000°C, 1050°C and 1100°C respectively revealed that the clay has: apparent density (2.50g/cm<sup>3</sup>), apparent porosity (25.30%), water absorption (9.05%), shrinkage (15.76%), modulus of rupture (29.40kg/cm<sup>3</sup>) and bulk density (g/cm<sup>3</sup>) at firing temperature of 1100°C with estimated refractories of 1659°C. The clay deposits can be used in different industries for the production of heat treatment furnaces, tiles, ceramic wares, refractory bricks.*

←  
ABSTRACT

**Keywords:** Engine Characterization, Clay Deposit, Ceramic Wares, Tiles Production, Furnace Lining

## Introduction

Refractory materials are materials capable of withstanding high temperatures, and that high quality refractory materials resist high temperature fluctuations between 1000°C and 1500°C and are also good thermal and electrical insulators (Ameh and Obasi, 2012). Clay is a type of fine-grained natural soil material containing clay minerals. Clays develop plasticity when wet due to a molecular film of water surrounding the clay particles, but become hard, brittle and non-plastic upon drying or firing. Refractories are composed of thermally stable mineral aggregate, which are inorganic and nonmetallic (Hassan et al., 2019). Refractories belong to the category of ceramic materials, that are utilized for prime temperature typically higher than 1100°C (Hassan, S. B. 2005). Most refractories are made from naturally occurring high melting point oxides such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, MgO, Cr<sub>2</sub>O<sub>3</sub>, ZrO (Jock, et al., 2013).

Refractories are mostly made up of inorganic compounds which are usually derived from clay. It is characterized by the ability to withstand physical, chemical and corrosive attack without deterioration at elevated temperatures (Musa et al, 2012). These materials are mostly used in Metallurgical industries for the linings of internal furnaces well, melting holding and transportation of metal and slag.

In Nigeria abundant deposits of clay have been reported across major geological zone (Musa & Alyu, 2011). But it is surprising that the growth of refractory industries in the country is still at its infancy stage. The present economic meltdown in the nation uncertainty facing the Petrochemical, Metallurgical and other allied industries has necessitated the need to source for local raw materials to nurture the growth of these industries. This will lead to the production of high-quality commercial refractory which will eventually substitute for importation and help to save the much-needed foreign exchange (Musa et al, 2012).

Agbajelola et al (2019) investigated on the evaluation of refractory properties of selected Sokoto state, North West in Nigeria and discovered that the clay samples investigated have quality characteristics for their potential applications in the production of floor tiles, fire refractory brick, paint, chalk and earthenware manufacturing.

Clay refractories are mainly produced from clay that has alumina (Al<sub>2</sub>CO<sub>3</sub>) and silica (SiO<sub>2</sub>) contents between 18 – 44% and 50 – 70% respectively (Musa and Aliyu, 2011) stability at high temperature both physical and chemical is the primary requirements for refractory materials. The ability of a refractory to withstand high temperature in service is known as refractoriness and its degree in any particular refractory depend on the amount of alumina (Al<sub>2</sub>O<sub>3</sub>) present, which is usually high for alumina content (Agbo, et al., 2015). The percentage of the minerals (Fe<sub>2</sub>O<sub>3</sub>, MgO, CaO, Na<sub>2</sub>O etc) in the clay ultimately determine the area of application of the clay such as a in bricks, floor tiles, cement etc (Idenyi, and Nwajagu, 2013).

## Materials and Method

The local raw material (clay) used in this research work was collected from Agbani in Nkanu West Local Government Area in Enugu State of Nigeria. The clay sample was collected from four different locations of the deposits and mixed together by pounding process. The chemical analysis was carried out using X-ray florescence (XRF). The physical analysis was carried out using international accepted standard.

### Sample Preparation for Physical Analysis

The samples (clay) used for the analysis was sieved, measured and soaked in a calculated amount of water, then stirred vigorously in order to bring it to plastic state. It was allowed to dry for 40 mins for the suspended particles decoupled. The sample was allowed to dewater for 4 days after which it was oven dried for 5hours for proper processing.

**Cold Crushing Strength (C.C.S.):** Test pieces measuring 50 x 30 x 30mm were prepared and air-dried for 48 hours after which they were transferred to a furnace and heated for a period of 5 hours and at a temperature of 1100°C. After the heating process, samples were removed and allowed to cool at room temperature and each piece was placed in a crusher. During test, the pressure adding surface was adequately aligned to the centre of the spherical seat of the equipment. Load was applied axially and continually until the test piece fractured. The procedure was repeated for other test pieces. The respective loads at which each test piece fractured were recorded. The cold crushing strength (CSS) was calculated from the equation.

$$CCS = \frac{F}{A} \dots \dots \dots (1)$$

Where A = Area of test specimen

F = Applied load

**Plasticity and Plasticity Ratio**

Five (5) specimens were produced using cylindrical mould and a plastomer was used in deforming them.

$$\text{Modulus of plasticity} = \frac{h_1}{y_1}$$

$$\text{Plasticity ratio} = \frac{h_2}{y_2}$$

Where  $h_1$  = original height of deforming load

$y_1$  = deformed height of load

$h_2$  = original height of sample

$y_2$  = deformed height of sample

Moisture content: Five cylindrical specimens (1.5cm dia. x 10cm length) were weighed green and their weight noted. They were then dried in air for two weeks and later oven dried at a temperature of 110°C for twenty four hours. The dry weight of the specimens was measured and recorded.

$$\text{Moisture content} = \frac{W_w - d_w}{W_w} \times \frac{100}{1}$$

Where  $W_w$  = wet weight

$d_w$  = dry weight

Green, Dry and Fired Strength; five rectangular specimens (1.5 x 3.5cm) were taken and recorded. They were oven dried for two days and a rupture testing machine was used to test for the strength at given, dry and fired state. The fired strength was tested after firing the specimens to temperatures of 900°C, 950°C, 1000°C, 1050°C and 1100°C respectively.

$$\text{modulus of repture: } \frac{3pl}{2bh} \text{ or } \frac{8pl}{\pi d^2}$$

- Where: p = applied load (kg)
- l = distance between supports constant (cm)
- b = width of the specimen at the point of rupture (cm)
- h = height of the specimen at the point of rupture
- d = diameter of the cylindrical specimen

Apparent porosity, water absorption, apparent and bulk density tests. Rectangular specimens (5cm x 7cm) were prepared and their weight recorded. They were dried for two weeks, oven dried and their new weight recorded. They were fired to temperatures of 900°C, 950°C, 1000°C, 1050°C and 1100°C respectively. Their weights at each stage of firing were recorded. The fired specimens were soaked in water for twenty-four hours and the weight taken and recorded. They were calculated as follows:

$$\text{Apparent porosity} = \frac{S_w - F_w}{S_w - S_{sw}} \times \frac{100}{1}$$

$$\text{Water absorption} = \frac{S_{sw} - F_w}{F_w} \times \frac{100}{1}$$

$$\text{Apparent density} = \frac{F_w}{F_w - S_{sw}} \times \frac{100}{1}$$

$$\text{Bulk density} = \frac{F_w - dw}{S_w - S_{sw}} \times \frac{100}{1}$$

Where  $S_w$  = soaked weight

$F_w$  = Fired weight

$S_{sw}$  = Suspended weight

$dw$  = Density of water

Linear dry and fired shrinkage: Rectangular specimen (5cm x 7cm) were produced and marked 5cm lengths. Temperatures of heating ranges was inscribed on the specimens to be fired. They were dried in air for two weeks and oven dried for forty hours. The change in the 5cm length mark was measured. They were then fired to temperatures of 900°C, 950°C, 1000°C, 1050°C and 1100°C respectively.

$$a. \text{ Dry Shrinkage (\%)} = \frac{wl - dl}{wl} \times \frac{100}{1}$$

$$b. \text{ Fired Shrinkage (\%)} = \frac{wl - fl}{dl} \times \frac{100}{1}$$

Where  $wl$  = wet length (cm)

$dl$  = dry length (cm)

$fl$  = fired length (cm)

**Permeability:** Five specimens were prepared using standard specifications of 5.08cm diameter and 5.08cm length/height. They were dried in air for two twenty hours and oven dried for ten hours. Permeability meter was filled with 2000cm<sup>3</sup> of water in a bel jar put in place. The orifice was opened and then taken for 2000cm<sup>3</sup> of water to displace equal volume of air through the specimen taken. The pressure difference was measured using manometer.

$$p = \frac{vxh}{pxAxt} \text{ or } \frac{vh}{pAt}$$

Where  $p$  = permeability meter

$V$  = volume or air passed through the specimen (cm<sup>2</sup>)

$h$  = height of specimen

$A$  = cross-sectional area of the specimen

$p$  = pressure head under which the air has passed

$t$  = time of flow in seconds

$$\text{or } p = \frac{30072}{pxt} \text{ or } \frac{30072}{pt}$$

**Thermal Shock Resistance:** The test was carried out using 50mm x 50mm specimens. They were inserted into a muffle furnace and heated to 900°C and held for 15 minutes. They were removed quickly from the furnace, and placed on fire bricks and allowed to cool for fifteen minutes, after the 15 minutes cooling, they were then returned to the furnace and the process repeated for 30 cycles. They were seen at the end of the 30 cycle not to have deformed.

**Refractoriness:** Pyrometric cone equivalent test was used to determine refractoriness specimen with 50mm pyramid height and 15mm rectangular base were used. They were put inside a refractory plague with two Seeger cone of 12 and 13 and oven dried at 110°C. The temperature equivalents for the two Seeger cones were 1340°C and 1348°C respectively. They were put in a furnace and temperature raised at the rate of 100°C per minute until the two Seeger cones bent over level with the base. Upon observation, it was noted that the specimen had not deformed or let alone melting. The firing was continued until temperature of 1400°C was attained, yet the specimen remained undeformed.

The refractoriness was estimated using Shuen's formular.

$$\text{refractoriness, } K(^{\circ}C) = \frac{360 + Al_2O_3 - RO}{0.228}$$

Where,  $Al_2O_3$  = % alumina in the clay

$RO$  = sum of all other oxides besides silica

360, 0.228 = constants.

**Loss on Ignition:** 50g of the clay sample was oven dried 110°C and cooled in a desiccator. The dried sample was put inside the crucible and the weight of the crucible and the sample were recorded ( $m^2$ ). The crucible with its contents were cooled in a desicator and then re-weighed ( $m^3$ )

### Results and Discussion

The chemical analysis of Agbani Clay deposits as shown in the Table 3.1, was obtained using X-ray florescence (XRF). The chemical composition results as shown in Table 3.1, shows that Agbani clay deposits has 25-20%  $Al_2O_3$ , 53.41%  $SiO_2$ , 2.06%  $K_2O$ , 1.20%  $CaO$ , 2.10%  $Fe_2O_3$  as its predominant oxides with 0.01%  $HgO$  as minor oxides. It was clearly illustrated in the Table 3.1 from the chemical composition results, the clay has a characteristics composition of fire clay refractories with several composition standard range of 55 – 75%  $SiO_2$  and 25-44%  $Al_2O_3$  (Odo and Okorie 2001). The percentages of some other oxides of the clay fell within the specified standard (Ugwuoke and Amalu, 2017). The clay could therefore be used in some vital application such as in the production of tiles, ceramics ware, refractory bricks, heat treatment furnaces etc.

The estimated refractoriness of Agbani clay was found to be 1659°C using Shuen's formular (Nnuka and Enejor, 2001). The estimated refractories of the clay deposit fell within the acceptable standard range of 1500-1700°C for fire clay refractory materials (Agbo, et al., 2015). Alumina content of clay determines its refractoriness (Hussan et al, 1994) and the greater the percentage of alumina, in clay the higher the refractory property of the clay. The silica composition of the clay deposits was higher than the range recommended for the production of paper (45.0 – 45.8%) and paint (45.3 – 47.9%), but did not meet the requirements for the production of glass (80.90%) (Chester, 1973). High value of silica content and other oxide such as  $Fe_2O_3$  contribute to low refractoriness of any material.

The iron oxide ( $Fe_2O_3$ ) content of the clay deposit was higher than the refractory bricks (0.5 – 2.4%), glass (2 – 3%) and paper (0.3 – 0.6%) (Ezeofor, 2018), but met standard specification for the production of high melting clays which requires only 1-9% of  $Fe_2O_3$ .

The physical test results of the clay deposits were shown in Tables 3.2 – 3.3. The results indicated that the apparent density, apparent porosity, water absorption, dry shrinkage, total shrinkage, modulus of rupture (MOR) and Bulk density fell within the international standard organization specification for alumino-silicate refractories of fire clay (Eochvaron, 1977).

From Table 2, it was observed that some properties such as apparent density, total shrinkage and bulk density increased as the firing temperature increases. While the water absorption decreased as the firing temperature increases. It is clearly illustrated in the fig. 2.

The higher a clay material is fired, the more it loses its absorbed moisture and constitutional water, thereby leading to increased shrinkage, reduced porosity. Also at higher temperatures, the low melting point constituents of clay has tended to melt, oxidize and fuse to the highly refractory constituents thereby closing the pores and leading to the formation of a denser body and increased strength due to strong body formation. The more a clay body shrinks, the less porous it becomes (Harbisan, 1979).

The apparent density of the clay deposit was within the standard range of 2.3 – 3.5g/cm<sup>3</sup> (Rayn, 1962). From the Table 2, it was observed that the modulus of rupture increased from 20.18 – 29.40kg/cm<sup>3</sup> with increase in firing temperature from 900°C – 1100°C. This increase is as a result of increase in bulk density of the clay body as temperature increases.

**Table 1: Chemical Composition of Agbani Clay Deposit**

Compound	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	V <sub>2</sub> O <sub>5</sub>	MnO	Fe <sub>2</sub> O <sub>3</sub>
Con Unit (%)	25.20	53.41	2.06	1.20	0.06	0.15	0.43	2.10

Compound	CUO	Ga <sub>2</sub> O <sub>2</sub>	AgO	HgO
Con Unit (%)	0.01	0.014	0.86	0.05

**Table 1b: Chemical Composition of Refractory Clays by International Standard**

Constituent	Fired clay (%)	Refractory bricks (%)
SiO <sub>2</sub>	55 – 75	51 – 70
Al <sub>2</sub> O <sub>3</sub>	25 – 45	25 – 40
Fe <sub>2</sub> O <sub>3</sub>	0.5 – 2.0	0.5 – 2.4
K <sub>2</sub> O	< 2.0	
MgO	< 2.0	
LiO	12.15	

Gupta (2008)

**Table 2: Physical and Mechanical Properties of Agbani Clay Deposit**

Properties tested	Fired temperature °C				
	900	950	1000	1050	1100
Apparent density (%)	2.18	2.25	2.32	2.42	2.50
Apparent porosity (%)	30.45	26.48	23.40	20.12	18.60
Water absorption (%)	20.15	18.50	16.20	10.18	9.05
Dry shrinkage (%)	7.60	9.40	11.15	13.25	15.76
Fired shrinkage (%)	3.52	3.74	4.70	5.45	6.10
Total shrinkage (%)	11.12	13.14	15.85	18.70	21.86
Modulus of rupture (MOR)	20.18	23.22	25.64	7.50	29.40
Bulk density (g/cm <sup>3</sup> )	2.01	2.05	2.12	2.20	2.32

**Table 3: Other Physical Properties Tested**

	Property	Value
1.	Refractories °C	1659°C
2.	Moisture content (%)	3.21
3.	Conductivity	0.000677
4.	Thermal shock resistance	32 cycles
5.	Specific gravity (g/cm <sup>3</sup> )	2.53

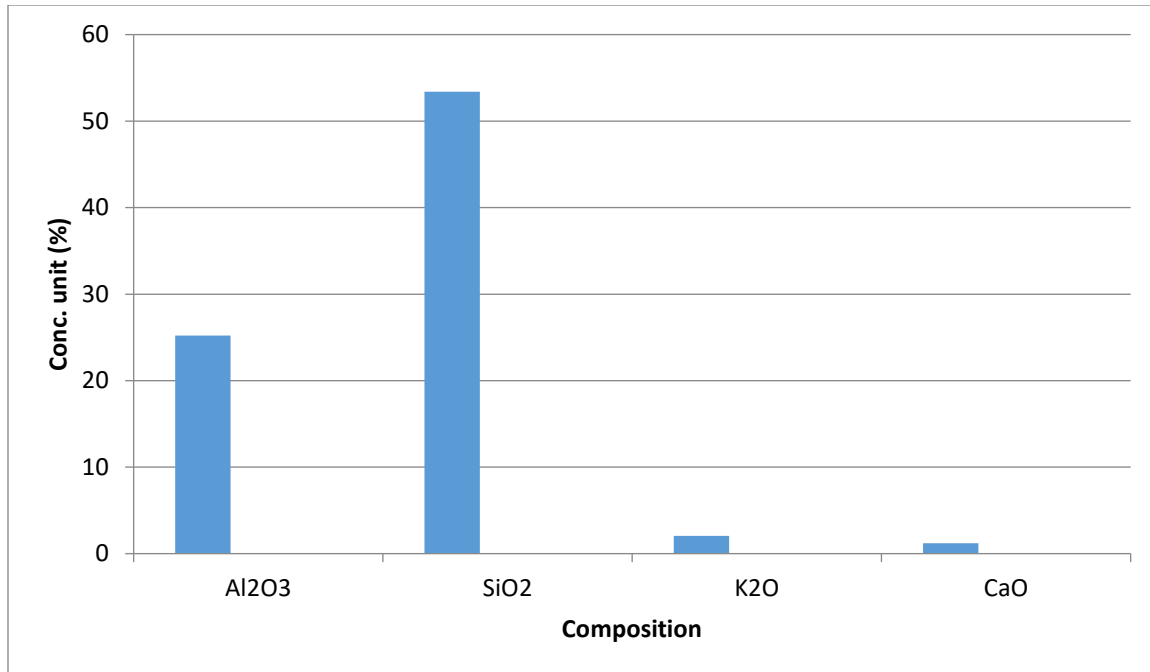


Fig. 1: Composition (oxide) Against the Unit Concentration

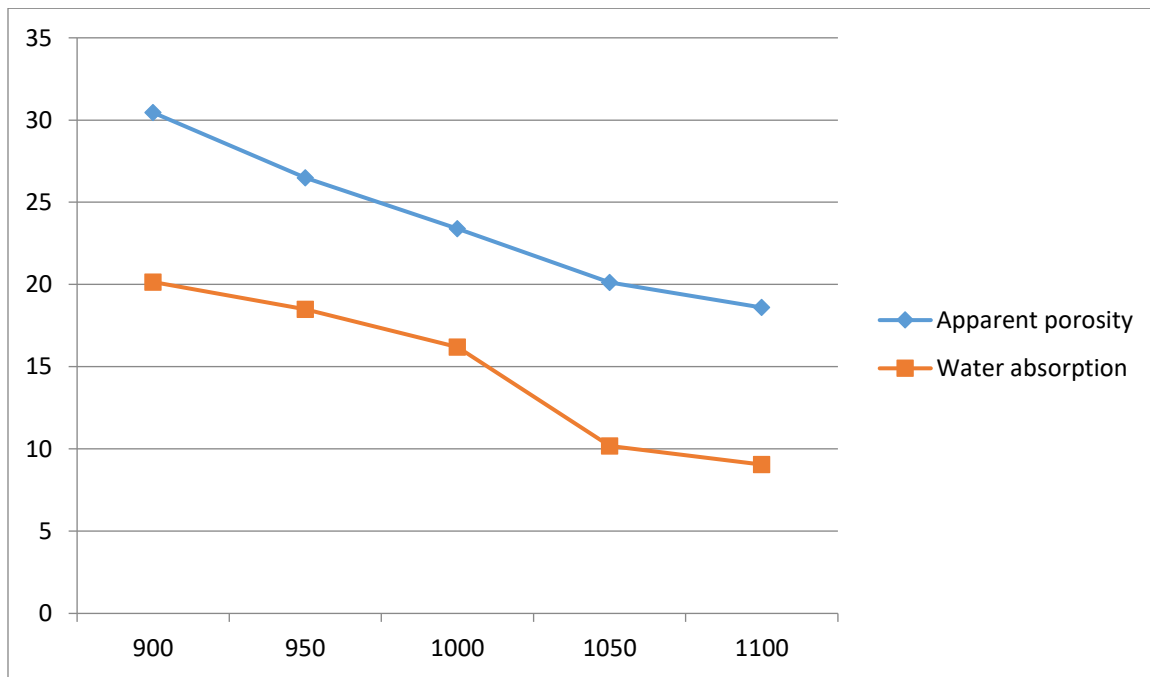


Fig.2: Relationship of Apparent Porosity and Water Absorption on Firing Temperature

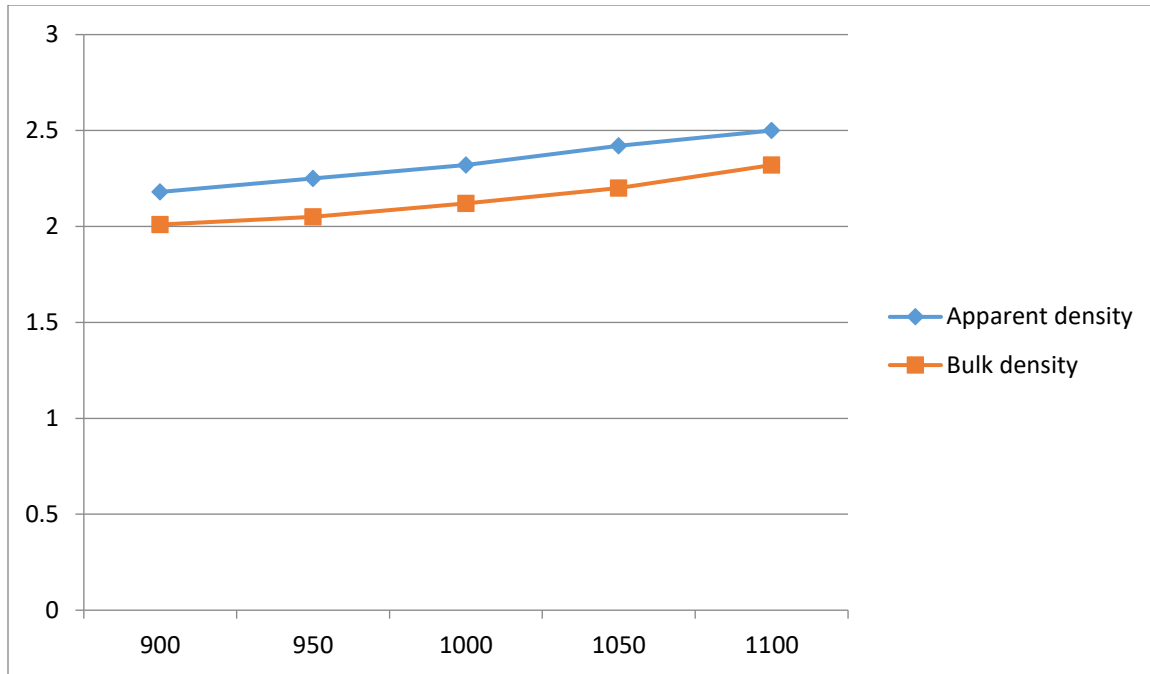


Fig. 3: Relationship of Apparent Density and Bulk Density on Firing Temperature

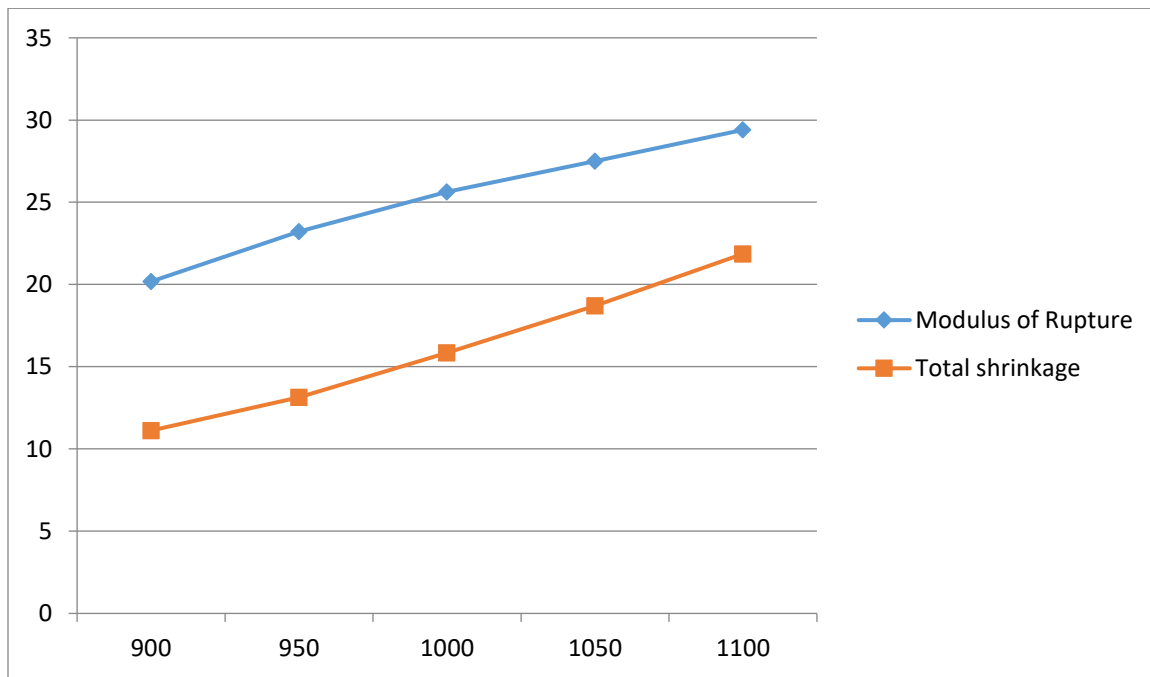


Fig.4: Relationship Modulus of Rupture and Shrinkage on Firing Temperature



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