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Study of the Mechanical and Thermal Properties of Refractory Mortars from Kaolin and Bentonite

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Abstract

In this paper, Mortar was prepared from medium alumina refractory grog, bricks crashed as a mean material to a particular size, and Iraqi raw (kaolin or bentonite) as binding materials. Refractory bricks were crushed, milled, then sieved to three particle sizes: fine as (1.18 >fine> 0) mm, medium as (2.36 > medium > 1.18) mm, crushed as (400 > coarse > 2.36) mm. Then these particle sizes were mixed with Iraqi raw kaolin or bentonite with selected ratios (10,15,20,30 and 40) %. Specimens were formed by the wetting method, then drying it at laboratory temperature for one day, followed by firing it at 1200 ℃. Results showed that the porosity of specimens decreases when increasing the clay ratio from 3-4% (kaolin or bentonite), and the bond strength between grog and clay increases when increasing the clay ratio from 2-3% (kaolin or bentonite). Also, the diametrical strength increases when increasing the clay ratio from 4-7% (kaolin or bentonite). The thermal shock results showed that K-mortar is better than B-mortar, depending on the results we obtained through the effect of temperature and diametrical strength. The SEM results showed that mortar structure was produced by adding 40% bentonite with small irregularly shaped. The mortar was produced by adding 40% of kaolin which possesses regular mullite crystals. Finally, the results of the test EDS that K-mortar were revealed in showed that there is no adsorption of carbon while B-mortar showed that there is adsorption of carbon atoms.

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1. Introduction

Ceramic materials are inorganic materials used in many applications such as thermal applications, electrical applications, and biological applications [1-3]. Refractory products are inorganic and non-metallic materials that withstand high temperatures without chemical and physical changes [4]. Crude clay is the oldest heat-resistant material used to produce refractories used as oxides after extracting them from raw materials and entering the industry [5]. Raw ceramic materials were used to prepare refractory materials used in various applications [6, 7]. Refractories can be used in broad applications such as furnace linings and thermal boilers, smelting and pouring various minerals, glass industry, steel processing, petrochemical industry, cement industry, military applications, and advanced space applications [8, 9]. The main factor that controls the production of refractories and their characteristics is the application they are used for, operating conditions in terms of operating temperature, ambient air (gases, vapors, and liquids), the mechanical load, and the extent of the change in temperature [10]. Thermal mortars are irregularly shaped ceramic materials used to connect the thermal bricks in the lining of the ovens and are usually used in a full layer to cover the ovens from the inside. The most important factors that must be considered in the manufacture of the thermal mortar are the thermal properties and that it should be close to the bricks which are used to connect the thermal mortar.

Thermometers are divided into two types, general and special. The special type is prepared for a special type of refractory bricks, which is prepared from the same bricks with the material that helps connect them. The general type is prepared in general, and its properties are studied, and the properties that fit the bricks used for bonding should be considered [11-13]. In 2014, K. Andreev *et al*. measured refractory mortar's compressive stress-strain performance at temperatures up to 1400°C [14]. They used calcium aluminate cement, betonies clay, mono aluminum phosphate, and water glass as binders [14]. The results explained that the failure in the mortar occurred due to the formation of micro cracks [14]. Crack propagation took the shortest way through the larger pores and was affected by the cohesion and interlocking of grains [14]. Also, clay mortar showed the highest compressibility [14]. In 2015, D.Slika studied the mechanical properties of mortar prepared with flint, black limestone, and granite at elevated temperature [15]. The specimen was also tested with dilatomtetry and TGA/DSC test. The results showed that the strength degradation was different for the three types of mortar at high temperature due to the mineral structure of the additives [15]. In 2015, E. Muhi *et al*. carried out a physical and mineralogical test on the Iraqi bauxite mineral to assess its suitability for the production of refractory bricks [16]. Two types of additives were used, Iraqi white kaolin as a binding material in ratio 10%, and (micro and nano) zirconia as additives, synthetic zirconia in ratios of (5, 10, 15, 20) % to improve the general characteristics of the Iraqi bauxite bricks. The bricks were formed by semi-dry pressing, with added potassium silicate in10% as binder material. The formed samples were of cylindrical shape (50×50) mm for diameter and height. The drying process of the samples was carried out at 110˚C for 24h, then fired at 1400˚C. In addition, physical properties such as microstructure analysis for the bricks test were carried out [16].

2. Theoretical Part

2.1. Apparent Porosity %

The Archimedes method is used to measure the specimen's apparent porosity, according to ASTM (C-773). Using the following equation:

$$(\mathbf{A.P})\% = \frac{\mathbf{ws - wd}}{\mathbf{ws - wi}} \times \mathbf{100} \quad \dots\dots\dots (\mathbf{1})$$

Where: W_d: The mass of the dried specimen (g), W_s: Mass of specimen immersed in water (g), W_i: Mass of specimen immersed in water (g).

2.2. Bond Strength and Diametrical Strength

Bond strength ASTM (C-198), diametrical strength ASTM (C-496). Modulus of rupture is calculated by using the following equation:

$$\mathbf{MOR = 3PL / 2bd^2} \quad \dots\dots\dots (2)$$

Where: MOR: Modulus of Rupture (MPa), P: Maximum applied load before failed (N), L: Span between supports (mm), b: Width of specimen (mm), d: depth of specimens (mm). Diametrical strength is calculated by using the following equation:

$$\sigma_{D.S} = 2F/\pi t\, D \quad \dots\dots\dots (3)$$

Where: $\sigma_{D.S}$: Diametrical strength (MPa), **F**: Force (N), **D**: Diameter of specimens (mm), **t**: thickness of specimens (mm).

2.3. Thermal Shock

Resistance Thermal shock resistance is measured by cooling from high temperatures and by measuring strength declining (specimens were compared with others at room temperature). The specimens were divided into five groups, each group heated with a different temperature ranging (200-1000) °C for 60 minutes and then rapidly put down in cold water. Diametrical strength was measured in compression strength test apparatus according to ASTM (C1525).

2.4. Energy Dispersive Spectroscopy (EDS)

It is a result of the electron interaction of the scanning electron microscope with the surface of the specimen. The manufactured mortar and the organic material that produces carbon dioxide are placed in a furnace and burned for 15 cycles. It appeared in bentonite peck as a result of mortar exposure in the presence of organic materials that give work, the oven's temperature changes from room temperature to 1000 ℃. Here we used EDS to compare between K-mortar and B-mortar.

2.5. Scanning Electron Microscope (SEM)

Scanning Electron Microscope was used to investigate microstructure for firing specimens, a suitable surface from the optimum specimen chosen and prepared by cutting in a longitudinal cross-section to be characterized with an electron microscope.

3. Experimental Procedure

Medium alumina bricks were used to prepare the refractory mortar for the interior of the furnaces; it was crushed, milled, and sieved in different sizes. Then, the binder was added, which was kaolin or bentonite in different ratios (10, 15, 20, 30, and 40) %. When water is added to the mixture, the specimens are formed and then dried for 24 hours. After that, the formed specimen is fired at 1200 °C for 5 hours, as shown in Figure 1.

Figure 1: Specimen after firing at 1200 ℃.

Kaolin is a rock that was collected from the west desert (Dwekhla). Iraqi bentonite raw materials were collected from (Falluja /Al-Anbar) by the "National Geological Survey and Mining Company. Raw rocks were crushed and sieved to produce fine powder 0.075 mm. chemical compositions for kaolin and bentonite are shown in Table 1.

Table 1: Chemical compositions for kaolin and bentonite.

Oxide / Raw Material	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	Loss on ignition %
Kaolin	47.34	36.37	0.63	2.2	0.12	0.08	0.31	0.53	12.42
Bentonite	55.31	14.62	5.71	1.17	3.43	3.31	1.11	0.72	14.62

4. Results and Discussion

The apparent porosity test is shown in Figure 3. The apparent porosity decreases with the increase of clay (kaolin, bentonite) ratio. Since (kaolin, bentonite) have flux oxides (Cao, MgO, K2O), which form a liquid phase at the final stage of firing at high temperature, partially filling the porous in the ceramic body in the liquid state at the final stage of the firing process. Which consequently pulled the ceramic soled phase by surface tension at the solidification process when the temperature decreased, so the ceramic body became denser and had low porosity. Bentonite has fluxes oxides and silica more than kaolin, according to Tabel 1. Thus, it will produce a liquid phase more than kaolin, leading to less porosity at the same adding ratio [17].

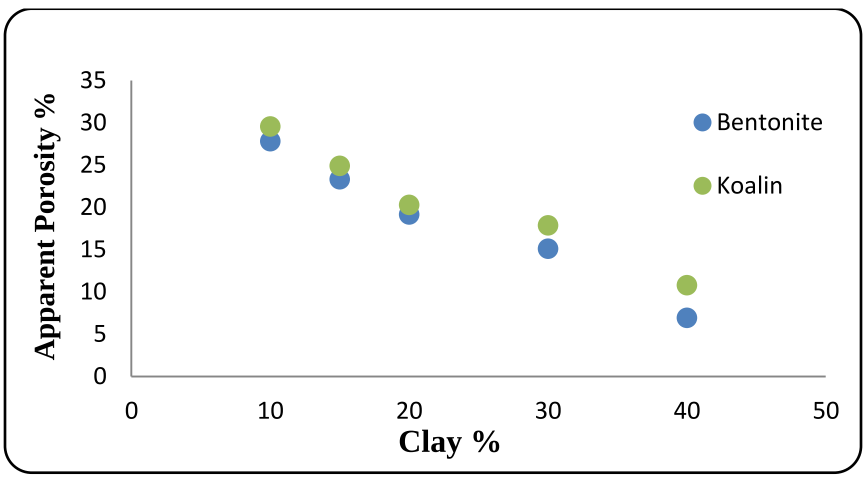

Figure 3: Apparent porosity for K-mortar and B-mortar.

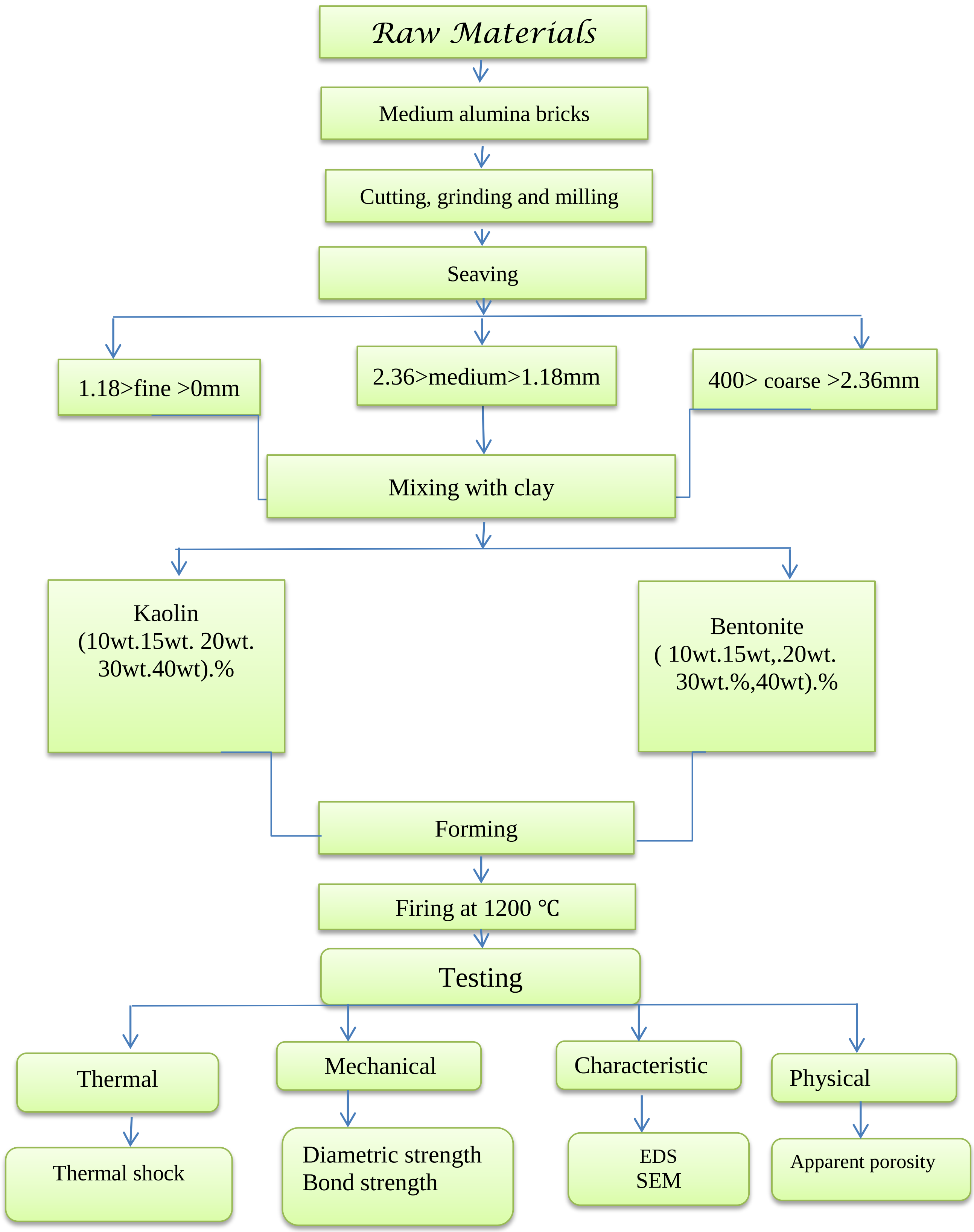

Figure 2: Chart of the experimental work.

Bond Strength and Diametrical Strength test are shown in Figures (4 and 5). Mechanical properties increased with the clay percentage added due to filling the pores and forming the vitreous bonding phase. When the added clay percentage decreased, the mullite phase will have better mechanical properties due to the higher formed mullite phase ratio and the lower glass phase. K-mortar has higher mechanical strength compared with B-mortar, so it has a higher ratio of mullite and cristobalite phases. The final ceramic body consists of three main phases:

1. Mullite phase: It improves the mechanical properties, which increases the resistance and the durability of the material. It consists of the union of Al2O3 with SiO2 at 900℃. It is produced from the chemical reaction of Al2O3 with SiO2 at (900-1400) ℃ in the raw materials. The cristobalite phase is a phase consisting of silica and a crystalline phase. It improves mechanical properties. The Mullite phase is part of the medium alumina bricks and consists of clay when Al2O3 reacts with SiO2 during firing.

2. Pore phase: on the contrary, it is classified from the model. The increase in the pores proportion leads to a decrease in the mechanical properties, decreasing resistance and durability.

3. Glass phase: This phase occurs in clay raw due to fluxes oxides and silica that help melt and silica. Thus, partially filling the pores at the last stages of the burning process.

The presence of SiO_2 and oxides assist fluxes on smelting in the raw and lead to the formation of the glass phase after the firing process, which acts as a binding phase that connects the rest of the crystals of the phases of the ceramic body while improving the mechanical properties, i.e., increasing the resistance and durability of the final ceramic body produced. We notice from the figures that k-mortar is higher than B-mortar because k-mortar kaolin contains smelting oxides and silica less than is in bentonite in B-mortar [17,18].

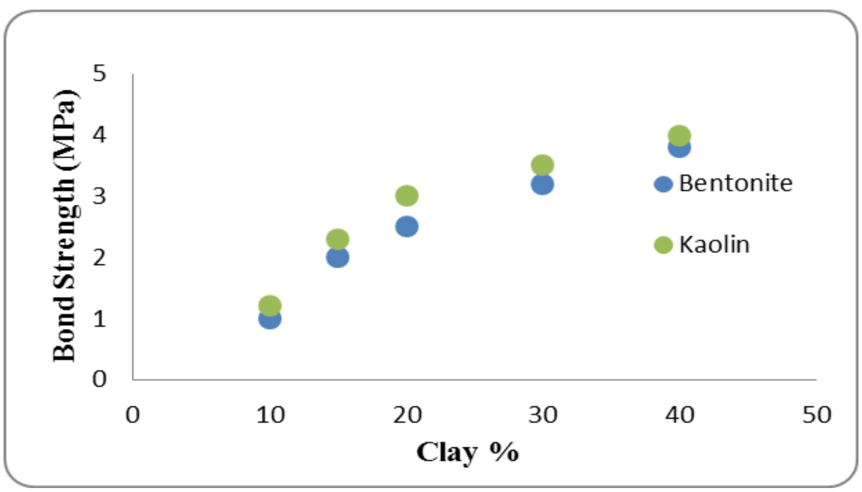

Figure 4: Bond strength for K-mortar and B-mortar.

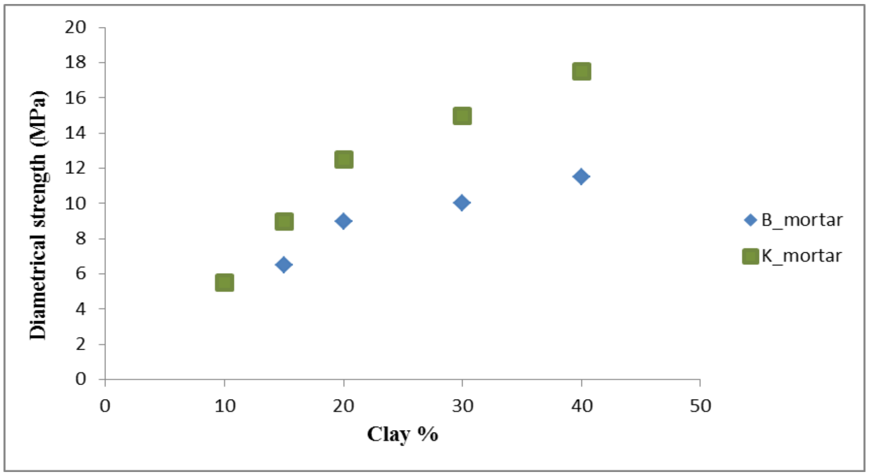

Figure 5: Diametric strength for K-mortar and B-mortar.

Thermal shock resistance is a necessary test to determine materials' thermal properties as it helps us understand mortar failure during service and extend its service life. Figures 6 and 7 show the change in the durability of the axial grinding after choosing the thermal shock with the temperature of the thermal shock, the decrease is gradual up to 400^{o}C, and this stage is called the phase of the flexible thermal behavior. The prepared refractory models consist of temperatures if it absorbs heat and stores it within the refractory ceramic. The mullite phase is formed at (900-1200) ℃ in raw is sufficient to generate the mullite phase in the additive raw (kaolin, bentonite) without breaking the first bonds and transforming from liquidity to solidity. It forms a glass phase that links the main ceramic phases together despite the low percentage of the pore phase, which disperses the heat, weak mechanical regions are formed. The main ceramic phases are (mullite and cristobalite) which give the ceramic body a high thermal shock resistance due to the high thermal capacity and the medium conductivity that disperses and stores heat. The thermal shock of mortar kaolin and mortar bentonite falls within (400 – 600) when the regression occurs.

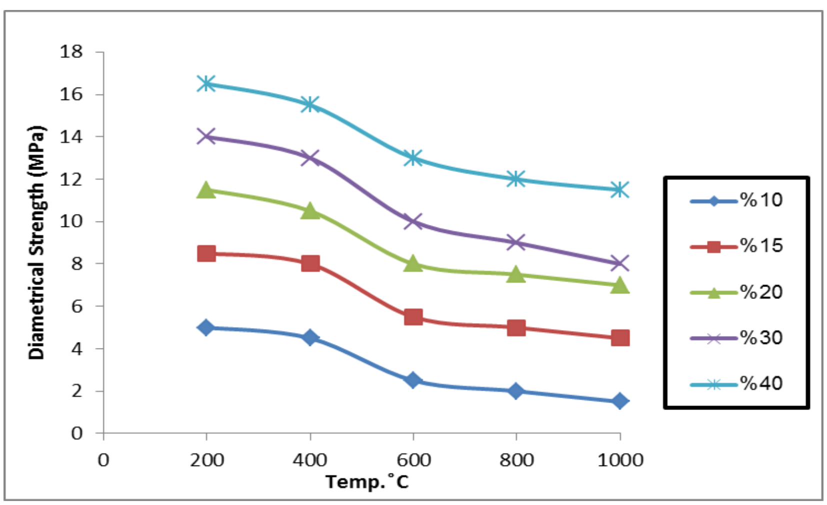

Figure 6: Diametrical Strength for kaolin mortar.

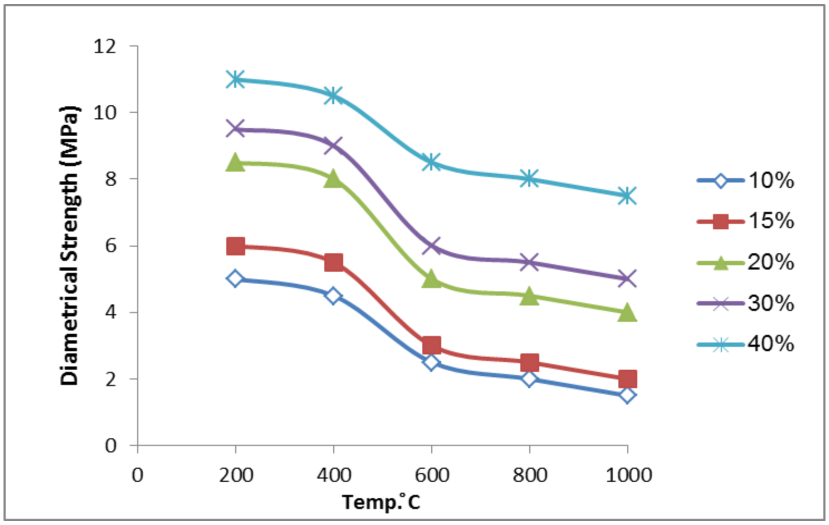

Figure 7: Diametrical Strength for bentonite mortar.

Energy Dispersive Spectroscopy (EDS):

EDS test was carried for Kaolin mortar and Bentonite mortar, and it was found that Al_2O_3 in K-mortar is more than B-mortar. Also, SiO_2 in K-Mortar is less than B-mortar, fluxes oxides (CaO, K_2O, TiO_2, NaO_2) in K-mortar are less than B-mortar. The finished ceramic body produced refractory mortar consists of three main phases:

- Mullite phase: It is the primary phase formed after burning clay raw. The nucleus begins at 900 ℃ from the reaction of alumina with silica, which is a crystalline phase with long crystals that improve mechanical properties. In K-mortar more than in B-mortar since it comes from the reaction Al2O3 with SiO2 at firing process at (900-1200℃).

- Cristobalite phase: The pores decrease with the increase in the amount of clay (kaolin, bentonite) added, which is one of the phases of the refractory mortar it is consistent with SiO2 tetragonal crystalline.

- Glass phase: This phase occurs in clay materials due to oxides that help smelting and silica. Partially fills the pores at the final stages of the firing process in K-mortar less than B-mortar since it comes from SiO2 and fluxes oxides at firing process.

- It holds the final ceramic body together and binds the crystalline phases together. For example, the cristobalite phase is a phase that consists of silica that improves the mechanical properties.

The prepared mortar was subjected to a heat treatment in the presence of organic materials and was treated with a temperature 1000˚C and for several times 15 cycles.

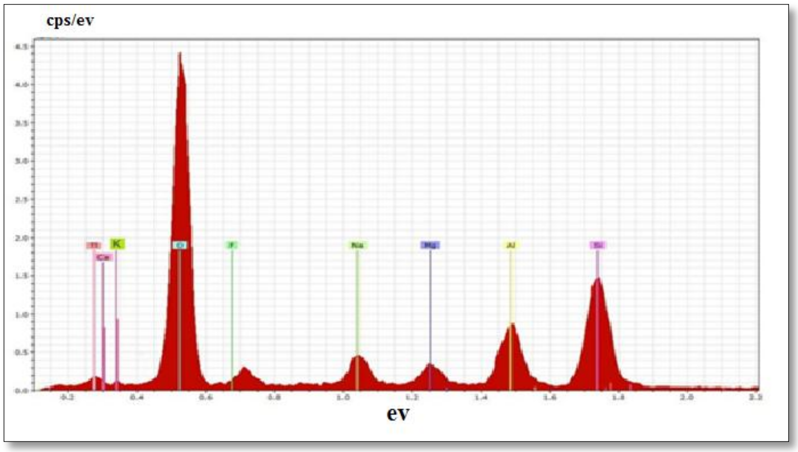

Figure 8: explain Kaolin before exposure thermal treatment.

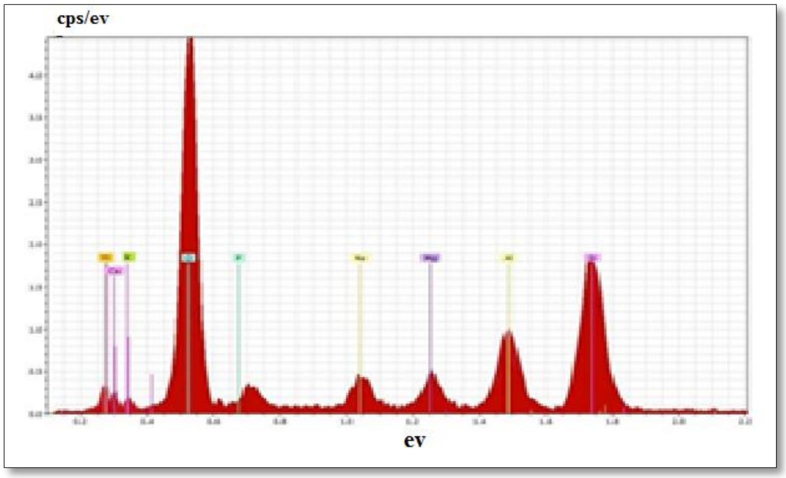

Figure 9: explain Bentonite before exposure thermal treatment.

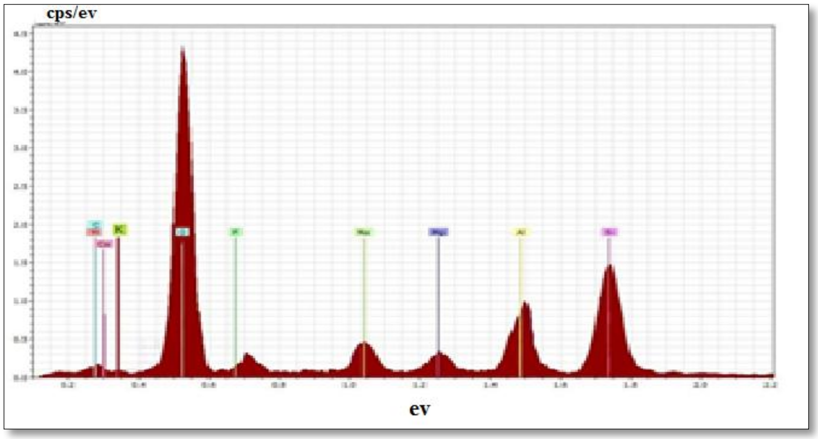

Figure 10: explain Kaolin-mortar after exposure thermal treatment.

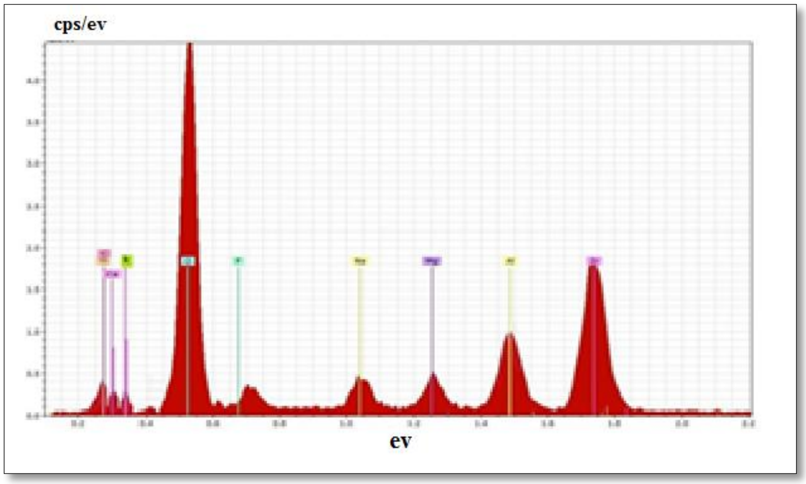

Figure 11: explain Bentonite- mortar after exposure thermal treatment.

Using Scanning Electron Microscope (SEM), the microstructure was studied for cross-sectional of prepared mortar specimens as shown in Figure 12, which illustrated the fracture surface for pure specimens.

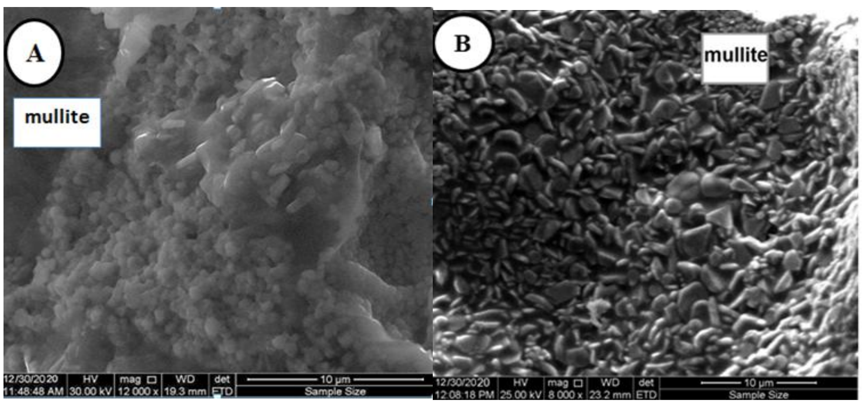

Figure 12: SEM images for prepared mortar specimen's surfaces (A) Bentonite Mortar (B) Kaolin Mortar.

From image A: The crystals of the mullite phase are small due to the high percentage of the glass phase. The appearance of the glass phase in high percentage is clear from the appearance that occurs in clay ores due to the presence of oxides that help in smelting and that silica partially fills the pores at the last stages of the firing process. That works on the cohesion of the final ceramic body. The crystalline phases are linked together by the appearance of the mullite phase crystals with a small percentage. The mortar produced by adding 40% suitable to acceptable bentonite during testing, the results of chemical analysis of bentonite with the results of microstructure are achieved by scanning electron microscope, Small irregularly shaped mullite phase crystals

From image B: The microstructure shows K- mortar. The crystals of the mullite phase are larger and have a longitudinal or tetrahedral shape. Because new crystals are formed during the burning process, and the growth of the originally existing crystals has occurred from the grog of the medium. As a result, the glass phase is formed in a high percentage with a low porosity ratio.

5. Conclusions

Best physical and mechanical properties were obtained when kaolin was added to (grog) due to crystalline ceramic phases such as the mullite and crystal phases due to the regularity of the crystal structure and the presence of strong ionic bonds between atoms. Hence, the mechanical resistance to the appearance of the glass phase decreases with the crystalline ceramic phases that bind the atoms with high efficiency and contribute to the distribution of pressures applied to the ceramic body and transfer them to phases with high mechanical resistance. The mechanical properties decrease despite the presence of pores inside the ceramic body.

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Conflict of Interest
There are no conflicts of interest regarding the publication of this manuscript.

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