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Effect of Aluminum Oxide on the Mechanical Properties of ceramic crucibles prepared by slip casting from local kaolin clay material.

Ammar Thamer Karim,
College of Environmental Sciences, Al- Qasim Green University, Babylon Province, Iraq.
ammarthamer1991@environ.uoqasim.edu.iq

Abstract: In this Article, the structural and mechanical properties were studied for the manufactured ceramics crucibles by the slip casting method from local clay materials after adding different percentages of aluminum oxide, where the adding percentages were ranged between (0.0 - 50 wt%). A number of parameters affecting the final properties of the local kaolinite sample were studied, such as the sintering temperature, which ranged between (1000-1500 ºC) and the adding percentages of the aluminum oxide. The apparent density and porosity analyze showed that increasing the adding percentages of the aluminum oxide led to an increase in the porosity at the high percentages, while low percentages lead to an increase in the density of the samples. On the other hand, the results showed that the increase of the aluminum oxide leads to the decrease of the samples hardness at the high sintering temperature, while the opposite occurs at the low sintering temperature.

Keyword: Ceramic crucibles, slip casting , sintering , alumina oxide, hardness

1. Introduction

Clays are involved in the manufacture of many ceramics products, such as Capacitors, furnaces lining, and crucibles, which are widely used in laboratories and scientific and technical workshops [1]. Clay is characterized by their chemical resistance, electrical properties, and resistance to heat caused by the nature of the internal structure, which is in the form of plates or sheets linked to each other, the most important of these clays is the kaolin which also called Chinese clay and its chemical formula is ($Al_2O_3.2SiO_2.2H_2O$) [2,3].

clay is a natural source of many industrial finished products. One of such products that have proved indispensable in the metallurgical industries is the refractory
material **[4].**

Many industrial finished items are naturally sourced from clay. Refractory material is one of these goods that has proven essential in the metallurgical industry. Clay is a complex mixture whose composition varies depending on geological location. It is a naturally occurring substance that is abundant. [5]. Clay can be traced back to either of two geological processes, namely sedimentation or weathering [5]. [6] According to Chester Kaolinite (Al2O3.2SiO2.2H2O), chromite (FeOCr2O3), magnesite (MgCO3), and numerous types of clays are the raw materials used to manufacture a variety of refractory products. The primary types of refractories utilized in the metallurgical industries are alumino-silicate and magnesite products [6].

kaolin is characterized by high smoothly and purity, it has a dark white color and it's melting point amount to (1755 ºC) **[7]**. Kaolin is found in the form of platelets, which have a pseudo-hexagonal crystal shape [1]. Kaolin is a "1:1″ clay mineral, with a layered structure formed by a single tetrahedral Si sheet covalently bonded to a single octahedral Al sheet. When kaolin is sintered above 1000 degrees Celsius, it transforms into mullite ($3Al_2O_3.2SiO_2$) and cristobalite (SiO_2) [3]. At temperatures above 1400 C, cristobalite transforms into amorphous phase [2].According to Chen [8], the degree to which the firing temperature affects the microstructure of sintered kaolin determines the degree to which the material's mechanical qualities are affected. Densification of kaolin bodies is primarily accomplished through a method known as liquid phase sintering. Therefore, the amorphous phase can be suppressed by adding a suitable amount of alumina (Al_2O_3), increasing the amount of mullite in the finished product [4].However, mullite made from organic clays frequently has a significant proportion of glass phase. In this situation,

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kaolin clay and alumina might be the best mix. It is true that more alumina can interact with the amorphous phase to produce more mullite [9]. In the Al2O3/SiO2 phase diagram, mullite is the only crystalline compound that is stable between ambient temperature and its melting point of 1810 °C [4]. It has interesting properties such as high thermal shock resistance, high refractory property, low thermal expansion, good creep resistance, and high mechanical resistance [10].

This article aims to study the effect of the adding of Aluminum Oxide on the mechanical properties for the samples of the manufactured ceramics crucibles by the slip casting method, which characterized by its ease in the manufacture of crucibles and to get a good shape without defects.

2. Material and experimental part

2.1. Starting materials:

Kaolin powder (less than 200 meshes) was obtained from an Iraqi geological and mining survey. The kaolin powder was washed several times with distilled water to reduce the proportion of salts found in it. Alumina (Al_2O_3) powder was introduced into batch sample with different percentage. itis supplied from the local market, fine grain (>75µm).

The chemical composition of Kaolin was determined using the gravimetric wet method, which was accomplished by the Iraqi geological and mining survey (Table I).

Table I. Chemical analysis of the starting material

Oxides %	SiO_2	Al_2O_3	MgO	Fe_2O_3	CaO	K_2O	Na_2O	L.O.I
Kaolin	49.02	32.51	0.09	1.38	0.83	0.41	0.33	12.43

2.2. Sample preparation:

The production of ceramic crucibles was accomplished by the use of slip casting techniques. Table (2) shows the percentages of composition components of the studied batches. A standard sample was prepared with 100% of the Iraqi clay to compare the results with study samples. After the preparation of these batches and weighing it, a milling process has been conducted a grinding process by the ball mill for two hours with the presence of distilled water to get a good homogeneity for the batch, The batch was then dried by a drying oven at 100 °C, and then the batch was manually milled with a ceramics mortar to prepare the batch for the compressing process. The 10 g from each batch was then compressed into 3 cm diameter cylinder form to obtain six samples per batch for sintering at different temperatures.

Table 2**:** The percentages for batches.

No. of Batches	**Al_2O_3 (%)**	**Kaolin (%)**
1	10	90
2	20	80
3	30	70
4	40	60
5	50	50

A sample from each batch was taken to conduct the sintering process at 1000 °C for 2 hr., the sintering process was then repeated for sintering at 1100, 1200,1300,1400 and 1500 °C according to the thermal program as shown in Figure (1).

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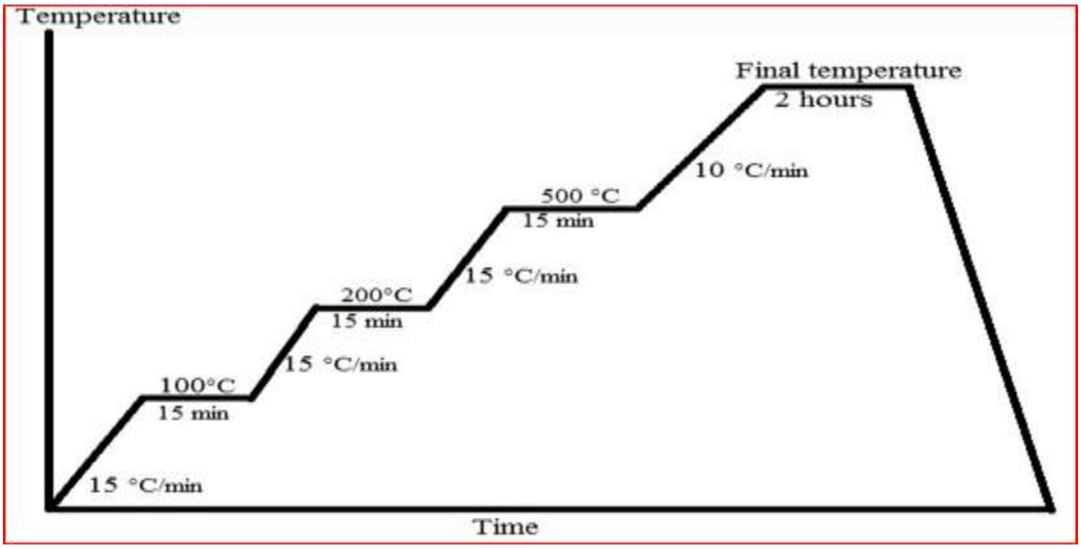

Figure 1: Thermal program for the sintering samples**.**

The casting mold was manufactured by mixing the gypsum material with the water, then forming it in the required form and drying it at 50 °C for three days to become ready for casting.

An appropriate quantity of alumina is weighed with a quantity of Iraqi clay according to batches. These materials are mixed with a percentage of distilled water and then they are milled well in a ball mill for 24 hr. to obtain high smoothness and then add a percentage of sodium silicate as dispersant agent [11]. The mixture was left for 24 hr. to be fermented. This mixture is called the slurry. After that, the slurry is poured into the mold and left for 10 min to occurring the permeability process, where the liquid is partially absorbed by the mold and form a layer of semi-solid materials on the internal surface for the hole of the mold, the excess of the slurry was then poured out of the hole for the mold and then left to dry at 50 °C for 24 hr. We notice the occurrence of shrinkage in the material and separating it from the mold easily after that, the crucible is then removed from the mold and the sintering process is conducted at 1500 °C at a slow sintering rate to avoid the thermal shock.

Physical mechanical tests were then conducted (Apparent density, apparent porosity, and the Vickers hardness method for measuring hardness).

3. Results And Discussion

3.1. Physical properties

Figure (2) shows the effect of the adding percentage of aluminum oxide in the apparent density of the samples, which sintered at different temperatures. The figure also shows that the temperature of the sintering has a clear effect in the apparent density, where the sintered samples at a temperature of 1500 C have high density, which amounted to (2.5 $g.cm^{-3}$), While the samples containing (20 wt.%) of alumina have the highest apparent densities. This procedure was applied to all prepared samples at different sintering temperatures. The reason may be due to the fact that alumina particles contain internal porosity from one side and on the other hand the addition of alumina leads to reduce the Packing factor for the particles due to possessing its particles a semi-spherical structure compared to the structure of the particles for the kaolin.[9]

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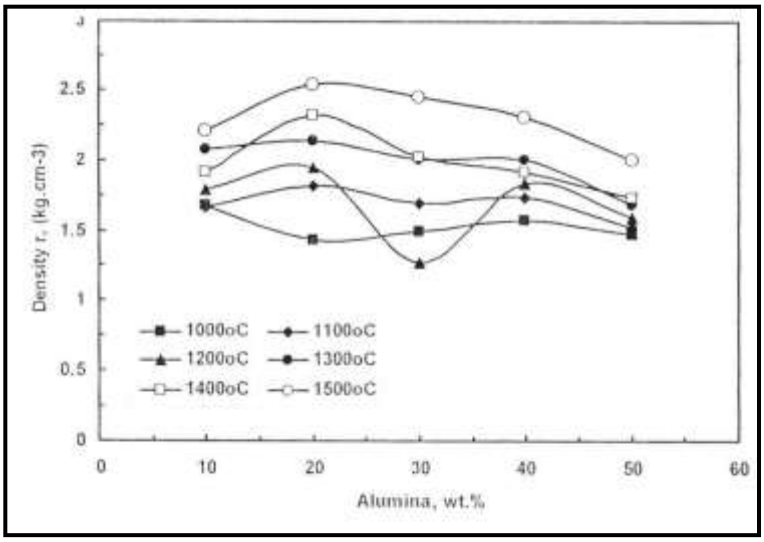

Figure (2): The effect of adding percentages of aluminum oxide in the apparent density of the samples at different sintering temperatures

Figure (3) confirms this conclusion by a clear increase in porosity of the samples through an increase in the percentage of alumina, while the behavior of density and porosity as a function of the sintering temperature as shown in Figures (4,5), which show clearly that increasing the temperature of the sintering leads to increasing density and low porosity. This behavior appears to be a result of the sintering phenomenon for the samples with increasing temperature. However, this behavior is the result of a range of transformations and reactions chemical that occurs when temperatures rise. Kaolinites lose part of the structural water at a temperature of (300-600 °C) and the amorphous silicon oxide begins to separate from the total structure at a temperature of (900-1050 °C), the behavior of the clays with the temperature is not only considered a function for the sintering process but a group of reactions and transformations that lead together to obtain structures with great complexity.

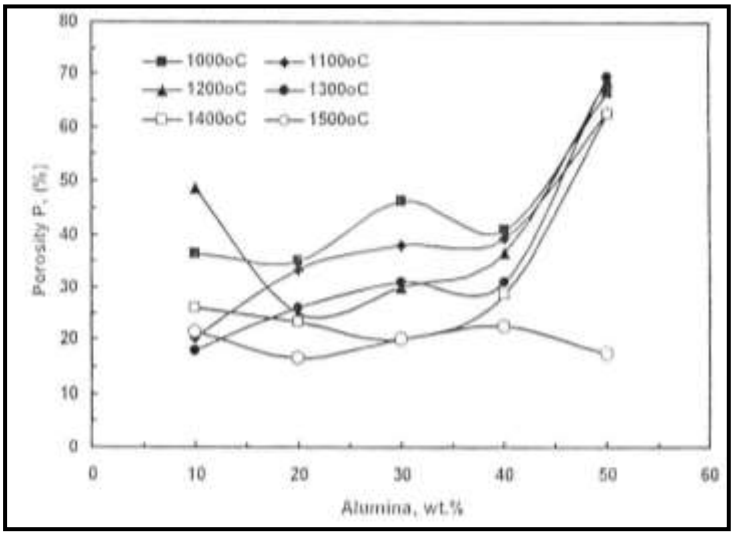

Figure (3): The effect of adding percentages of aluminum oxide in the porosity of the samples at different sintering temperatures.

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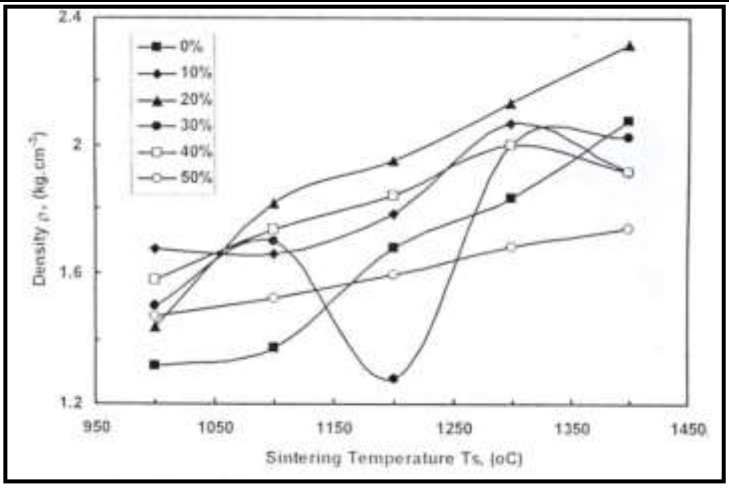

Figure (4): Effect of Sintering Temperature in the Apparent Density of the samples at Different Adding percentages.

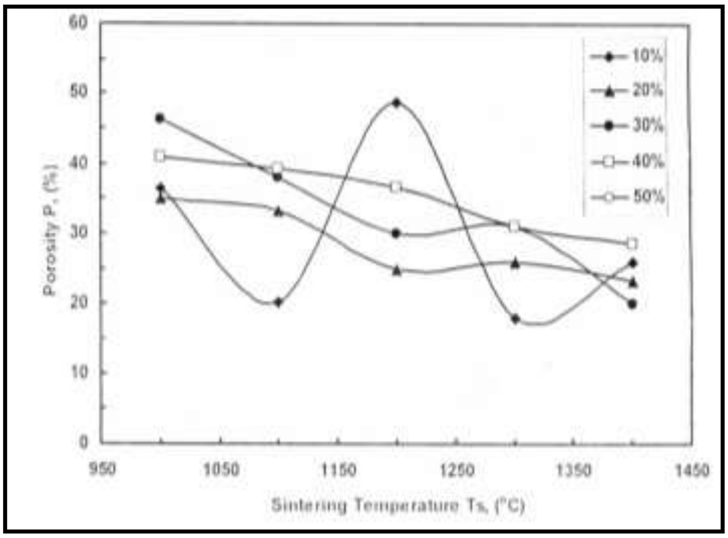

Figure (5): Effect of Sintering Temperature in the porosity of the samples at Different Adding percentages

3.2. Microstructure Characterization

Figure (6) Shows x-ray diffraction patterns for batch number (2) of adding 20% aluminum oxide Al_2O_3 for sintering samples prepared by slip casting method and fired at 1300 °C in which that samples showed the optimum properties. It is observed that percentage of alumina component in batches reflects an occurrence of a crystalline phase of corundum. also, the result is showed quartz mineral is considered a stable phase in this range of temperature. Also, appears some peaks belong to phase transformation of kaolinite clay at sintering temperature of 1300 ^{0}C in which mullite phase, cristobalite and illlite phases appears at this sintering temperature in which phase transformation occurs of kaolin gradually with increasing sintering temperature. Mullite begins to form at a temperature greater than 1100 °C [12]. Moreover, amorphous silica is transformed into a crystalline structure (cristobalite) at a temperature greater than 1200 °C, Primary mullite is formed by the transformation of kaolinite, whereas secondary mullite is formed by the reaction of alumina and the excess silica in kaolin. [4]. although illite phase appears refers to unreacted alumino – silicate layers

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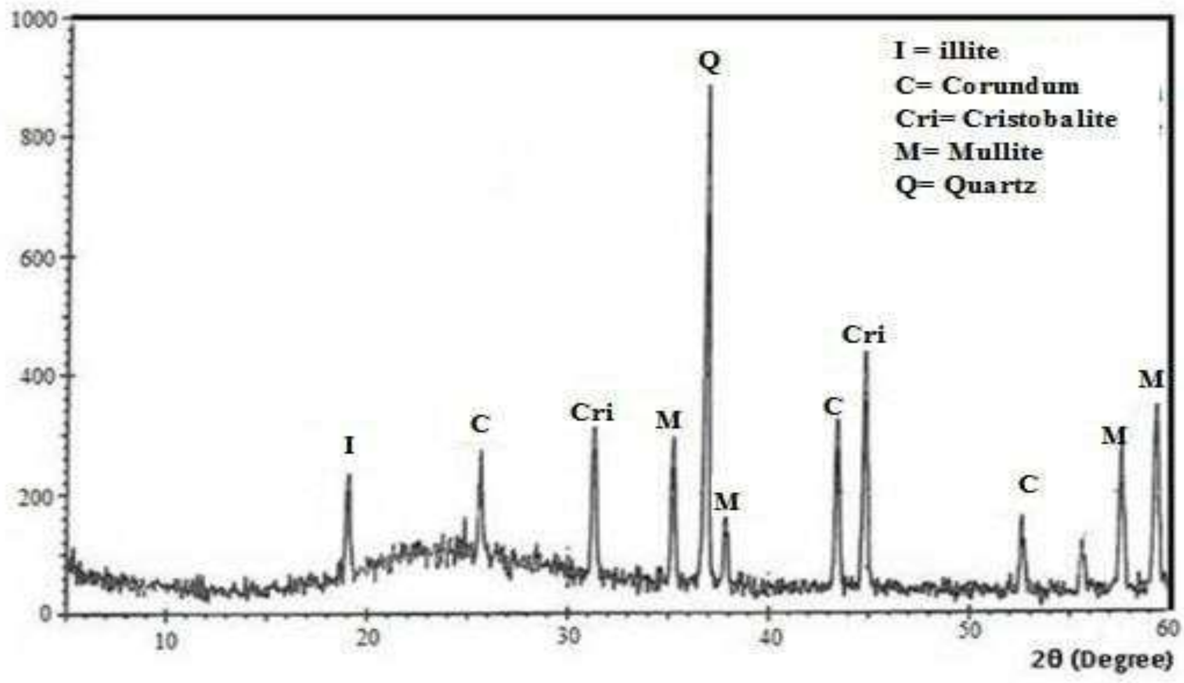

Figure (6): XRD analysis for batch number 2 fired at 1300 ^{o}C .

3.3. Mechanical Characterization

Structural changes caused by the addition of aluminum oxide led to changes in the mechanical properties for the samples. This can be confirmed by one of the important mechanical properties of ceramic materials, namely hardness, which give good indications for the resistance of the material surface to the Scratching and penetration, thus give other indications for the material strength and its resistance to cracks. Figure (7) shows the results of measurements of Vickers hardness for the sintering samples at different temperatures and the effect of the percentages of the added aluminum oxide. It is clear from the figure that increasing the aluminum oxide leads to increasing the hardness values until reaching an adding percentage amounted to 20% and then decreasing with increasing the adding percentage. Although aluminum oxide has a very high hardness compared to the hardness of kaolinite, the porosity of the samples described above Makes the samples structure more brittle, thus reduce hardness. On the other hand, it is noticed that increasing the sintering temperature as shown in Figure (8) leads to a decrease in the hardness values clearly until reaching a temperature of 1200 °C, Thereafter, stability or a slight increase in hardness values occurs at higher temperatures. As previously indicated, raising the temperature of the samples leads to the decomposition of the kaolinite into Metakaolin and then transforming it into Mullite and amorphous silica, which in turn weaken the mechanical properties of the samples [10]. At temperatures above 1200 °C, the thermal decomposition and transformations stop to begin the sintering process, which in turn increase the cohesion of samples, thus increase their hardness

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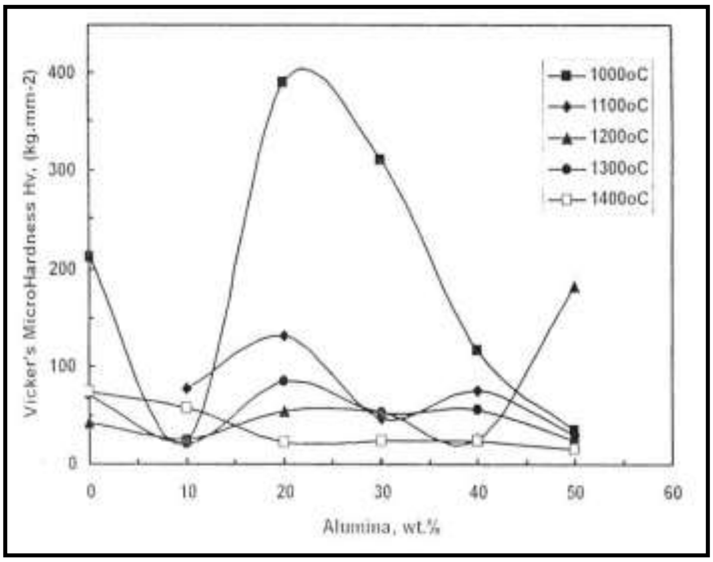

Figure (7): The effect of adding of aluminum oxide in the Vickers microhardness of the samples at different sintering temperatures.

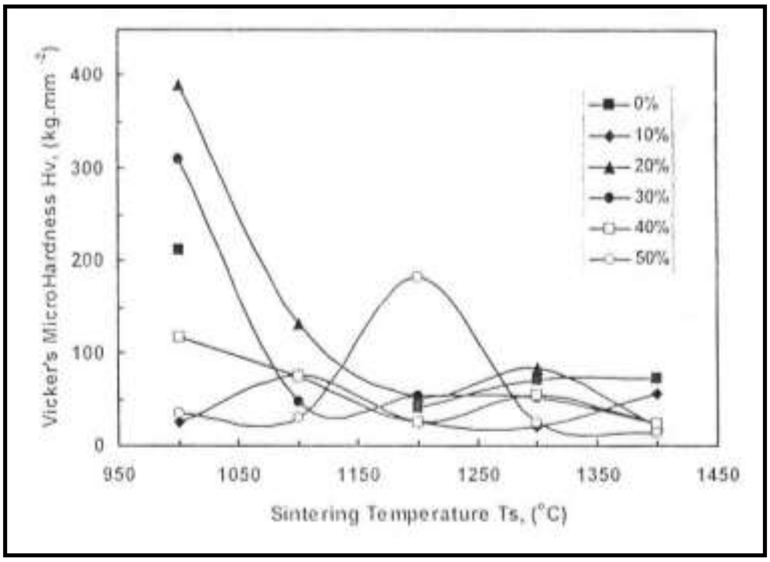

Figure (8): Effect of Sintering Temperature in the Vickers microhardness of the samples at Different Adding percentages.

4. Conclusions

1- The addition of alumina led to increasing the apparent density of the prepared samples, which recorded the highest averages at 20% and the Continuation of the adding led to reduce the apparent density.
2- Increasing the sintering temperatures of the prepared samples led to an increase in the apparent density
3- Adding alumina led to increasing the hardness values of the samples while the hardness decreased at the adding percentages which exceed (20% wt.).

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