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Studying the effect of particle size on the physical and chemical properties of the ceramic material

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الاهـــــــــداء

بسم الله الرحمن الرحيم

(قل إعملوا فسيرى الله عملكم ورسوله والمؤمنون)

صدق الله العظيم

بدانا بأكثر من يد وقاسينا أكثر من هم وعانينا الكثير من الصعوبات وها نحن اليوم والحمد لله نطوي سهر الليالي وتعب الإيام وخلاصة مشوارنا بين دفتي هذا العمل المتواضع. إلى منارة العلم والامام المصطفى إلى الأمي الذي علم التعلمين إلى سيد الخلق إلى رسولنا الكريم سيدنا محمد صلى الله عليه وسلم. إلى الينبوع الذي لا يمل العطاء إلى من حاكت سعادتي بخيوط منسوجة من قلبها إلى والدتي العزيزة. إلى من سعى وشقى لأنعم بالراحة والهناء الذي لم يبخل بشيء من أجل دفعي في طريق النجاح الذي علمني أن أرتقي سلم الحياة بحكمة وصبر إلى والدي العزيز. إلى من حبهم يجري في عروقي ويلهج بذكراهم فؤادي إلى أخواتي وأخواني. إلى من سرنا سوياً ونحن نشق الطريق معاً نحو النجاح والإبداع إلى من تكاتفنا يداً بيد ونحن نقطف زهرة وتعلمنا إلى صديقاتي وزميلاتي. إلى من علمونا حروفا من ذهب وكلمات من درر وعبارات من أسمى وأجلى عبارات في العلم إلى من صاغوا لنا علمهم حروفا ومن فكرهم منارة تنير لنا سيرة العلم والنجاح إلى أساتذتنا الكرام.

الشكر والتقدير

لابد لنا ونحن نخطو خطواتنا الأخيرة في الحياة الجامعية من وقفة نعود إلى أعوام قضيناها في رحاب الجامعة مع أساتذتنا الكرام الذين قدموا لنا الكثير باذلين بذلك جهودا كبيرة في بناء جيل الغد لتبعث الأمة من جديد ...

وقبل أن نمضي تقدم أسمى آيات الشكر والامتنان والتقدير والمحبة إلى الذين حملوا أقدس رسالة في الحياة ...

إلى الذين مهدوا لنا طريق العلم والمعرفة ...

إلى جميع أساتذتنا الأفاضل.......

"كن عالما .. فإن لم تستطع فكن متعلما ، فإن لم تستطع فأحب العلماء ،فإن لم تستطع فلا تبغضهم"

وأخص بالتقدير والشكر الى :

الدكتورة شيماء جابر التي تفضلت بإشرافها على هذا المشروع لجهودها البناءة وتوجيهاته القيمة

Abstract :The continuous demand for use materials that have good properties and low costs that are available in nature, and their use in Many industrial and domestic applications, requires studying the factors affecting them such as granular size and increasing the area and their effect on the properties from silica and kaolin (clay), where kaolin was mixed with silica (16 micro), and the silica was ground in the Nano mill for 10 hours by a steel ball mill to reduce the granular size of the silica powder, where the size was reached. Granular (3 micro) and mixed with kaolin in the following proportions (16 micro + 3 micro) (16 micro) and (3 micro) in proportions of 10%, 30% for each proportion of the granular size of silica. The samples were pressed into a 20-drop mold, where the samples were prepared (12) and PVA was used to form the samples. The samples were pressed in the pressing device for a minute for each sample, and then the samples were dried at a temperature of 5000 degrees and the samples were entered into the incineration oven at a temperature of 1000 The mechanical and physical properties of the samples were examined, such as compressive strength, density, porosity, and absorbency), as results were obtained that the ratio of (16 +3) micro has the highest compressive strength.

4_RESULTS & DISCUSSION

5_ CONCLUSIONS & RECOMMENDATIONS

CHAPTER ONE INTRODUCTION

1.1 INTRODUCTION:

Clay consists of fine particles of less than 002.0 mm, most of which are crystalline and belong to a limited group of minerals known as Clay minerals. Clay minerals consist of hydrated aluminum silicate, and the clay material contains one or more minerals, some of which contain Magnesium ions, iron ions, alkali metal ions, alkaline earth. As well as non-clay minerals and minerals of which many are found in The clay part of the soil, and this part differs from the parts of sand and silt, not only in the size of the particles, but also in the mineral composition. The silt contains the primary minerals (mainly primary minerals).[1]

1.2 The importance of clay [2].

1- It is a natural ingredient and helps create a link between natural resources.

2- Clay has an interesting and unique nature.

3- It has unique sculptural qualities.

4- It requires some strength and effort to shape it, so it is considered a sporting action.

5- Clay can be reused if stored properly.

6- Its prices are reasonable and it can be bought.

7- It helps to develop gross and small motor skills, and requires the use of the muscles of the arms even in small and even finger movements.

8- Clay work helps to develop a connection between the hand and the mind, as the hand acts on the commands of the mind.

9- It can help develop coordination and dexterity.

10- It can help develop mental focus.

11- It can help develop planning, decision-making, forward-thinking and problem-solving skills.

12- It provides an interesting tactile experience; it feels different when wet or dry, and different from clay or plasticize.

13- It can help develop self-expression and can be an area for creativity.

14- It also helps in building and developing mathematical and engineering problem-solving skills, for example when making 3D sculptures and geometric shapes, and can be used to construct buildings and other structures.

15- Clay allows for artistic experimentation, and has been used as an art medium for thousands of years.

1.3 THE MOST IMPORTANT USES OF CLAY [3].

1- Clay is the earth in everything. Clay pots are characterized by being rough to the touch and dull in color. The oven is heated at 900 ° C and does not rise above 1200 ° C. This is the lowest temperature among the three. Clay is heated enough to solidify but not so much that it becomes glass-like. This is observed when the ceramic bowl and glassware are broken, the glass will crumble into pieces. The ceramic pot has jagged edges and from here you can see the porosity of the clay.

2- Porcelain stoneware is heated at 1200 to 1350°C. This makes it harder than porcelain, but it is still not as vitreous as it is as it is non-porous and can have a vitreous-like coating, which makes it shiny.

3- The porcelain kiln operates from 1300 to 1450 degrees Celsius, which makes it the hottest. Examples include plates, teacups, and a beaker of warm water. It is usually painted white with motifs painted. The furnace at the highest temperatures allows the porcelain to reach a glass-like substance, making it harder and harder.

1.4 THE IMPORTANCE OF CONTROLLING PARTICLE SIZE

The importance of distributing the size of particles to the matter in understanding its physical and chemical properties, affecting the strength and characteristics of carrying the robbery and soil, and also affects the interaction of solids involved in chemical reactions, and must be controlled in many industrial products such as the inks of inks, cosmetics and pharmaceutical products ,Measurement of particle lighting using a light of the particle lighting (the size of the particle via the plantity). The plant of the particle size is aimed at determining the powder of the particle. The examples of the construction materials (sand and cement), pharmaceuticals, limestone, hypo, ceramics, columns, emulsions, emulsions and emulsions and many more The range of applications is increasingly increasing, the measurement of the methods of measurement on the range of size, measurement and the reaction of the particles of sizes are available in the range of measuring services, including the sample of the particles of solids of the season.**[4]**

1.2 AIM OF THE STUDY:

The goal of this research is to study the effect of grain size on physical and mechanical properties of the clays.

2.1 CLAY:

Clay is made up of very small, sheet-shaped particles of alumina and silica bound together by water. There are different substances in clay that can give it different colors. For example, iron oxide can make clay red. Carbon compounds give different shades of gray. Clay usually consists of minerals that are in the soil, and these components are alumina, which is estimated at 34%, silica, which amounts to 50%, as well as water, and other materials include iron oxide by 8%, lime and magnesium by 6%, 2% organic materials. One of the most famous types of clay in the industry is clay, that is, pure clay, and it usually consists of 14% of water, 46.5% of silica, and 39.5% of alumina, and the size of a drop of fine clay atom. 0.002 mm, where these particles are compacted to form a clay paste.**[5]**

2.1-1 PHYSICAL PROPERTIES OF CLAY

When clay is exposed to water, it is characterized by a doughy, plastic texture, and when exposed to a high temperature. It turns into a hard substance, this feature gives clay a great industrial importance, as the ductility property allows the clay to be formed in the desired shape, and it is grilled with fire until we get ceramic tools. Clay as cohesion that participates in maintaining the shape, body of the clay paste, the clay shrinks as a result of the high temperature. Its intensity varies according to its type, and the less shrinking clay is considered the finest species, and the clay melts as a result of the relatively low temperatures that range between (1000-1400) degrees Celsius [6].

2.1-2 MECHANICAL PROPERTIES OF CLAY

Clay is characterized by low permeability as a result of small pores, and this characteristic directly affects its behavior, in the case of sand or coarse soils with high permeability. They can be compressed for many years, but at the same time, they are eventually compressed at high rates, and this causes high subsidence measured from the Earth's surface, such as those that occurred in Mexico City. Clay is distinguished like other materials, because its texture is related to its moisture content, and its volumetric weight. For example, when naturally, pressed clay is exposed to artificially high pressure, and then this pressure is removed, the lower value is equal to the previous normal pressure [7]. Clay is characterized by having a texture that is highly connected to its structural structure. In the case of measuring the resistance of clay soil on surfaces parallel to the surfaces of deposition for a specific geological position, we notice that the resistance of the soil is less than the resistance associated with the surface dependent on the deposition surfaces, and the structural structure is formed in an environment that has a specific chemical nature. If this environment changes, the clay soils are directly affected, such as the sensitive clay soils in the Scandinavian countries [8].

2.1-3 USES THE CLAY

Clay is used in many industries, such as building materials, electrical insulating materials, electric heating and heating equipment, as well as high-temperature furnaces and smelting. It is used in the manufacture of paper, rubber, oil purification and oil stain removers. Bentonites clay with water is also used in drilling wells to stabilize the soil from collapsing [9].

2.2 SILICA

Silica (SiO_2) is the name given to a group of minerals made up of silicon and oxygen (eg quartz), which are the two most abundant elements in the Earth's crust. Silica is commonly found in the crystalline state and rarely in the amorphous state. The substance is composed of a silicon atom and two oxygen atoms, and is represented by the chemical symbol SiO_2. The majority of silicates are composed of igneous magma, and this magma turns into silicates when it rises to the surface and cools. There are two main factors that determine the type of mineral that magma will turn into next, which are its chemical composition and the method of its crystallization [10]. Silicates are chemical compounds that contain ions of the elements oxygen and silicon. It is the largest, most complex and rich type of minerals in the earth's crust at all. Geologists estimate that 30% of all types of minerals are silicate, and that 90% of the earth's crust is composed of silicate. Thus, oxygen and silicon are the most abundant elements in the earth's crust [11].

Characteristics Silica gel has a high specific area (about 800 m^2/g) and is usually transparent in colour. One of the most important features of silica gel is that it is a desiccant and absorbs gases. For this reason, it is found in most products. Density is 2.20 grams per cubic centimeter and Molar mass is 60.08 g/mol [12].

Silica powder is a very fine pozzolanic material, consisting of amorphous silica, produced as a by-product from furnaces for the production of elemental silicon or ferrosilicon alloys. It can be used in several fields of cement products such as concrete, special mortar, as well as in the manufacture of some flexible plastics, polymers, ceramics and some rubber applications. Silica gel is a granular, lustrous, porous substance that is a form of silicon dioxide artificially created from sodium silicate. Silica gel is hard and tough. It is more solid than the common household silica gel and forms gelatin or agar[13]. A natural mineral is purified, processed, and converted into any form other than granules or beads. Because of its dryness, it has an average pore size of 2.4 nanometers, and has a strong affinity for water molecules. Moreover, silica gel is more common in daily life towards small beads. Silica gel consists of silica, a substance that is absorbent of moisture, and has a high acidity, which shows us the purpose of placing it in shoes, handbags, as well as inside electronic device boxes. This type of goods for a longer period of time because they are exposed to damage from moisture, especially electronic leather goods and food as well, as it is added in deliberate proportions to some foodstuffs, and some medicine pills contain a quantity of silica gel to protect them from moisture that affects them harmfully. This substance remains somewhat toxic, so it must be kept out of the reach of children [14].

2.3 LITERATURE REVIEW

Ceramics hold all traditional, scientific and engineering raw materials or fabricated products used in different applications especially that performed at high temperatures through high-tempers. The remarkable progress made recently in the advancement of technology has related a prerequisite for a seriously huge number of parts with controlled porosity and grain size.

(2013) Ravi Mathur, et al, studied produced the white wares from porcelain bodies fabricated from triaxial mixtures of clay, quartz and feldspar with different size and amounts of nano particles were investigated. Although the purity of raw materials has a strong effect on the colour of the fired bodies, the particle size of raw materials also effect the whiteness The raw material mining minerals china Clay, Feldspar, quarts were prepared of various sized nano particles contains 10.60 -20.22%, 56.84- 70.80 % and

34.87-50.76 % of 100nm respectively. The fired bodies of raw mining minerals and triaxial bodies were subjected to colour measurement. The differences in whiteness were compared and discussed. The studies so far carried out is upto 400 mesh size while the present study has included up to 100nm particle size. A statistical correlation between whiteness of feldspar and triaxial body was also carried out. The correlation between china clay and triaxial body are 0.53, 0.57 and 0.66 for china clay similarly, correlation for feldspar is 0.49, 0.73 and 0.83 for triaxial body it are 0.97, 0.84 and 0.75 for A1, A2 and A3 samples. Correlation between china clay and feldspar with triaxial body are 0.79 and 0.92 respectively.[15]

(2019) Perdinan Sinuhaji, et al, studied the conducted on the characterization of raw materials and traditional ceramics from clay, feldspar, quartz, and kaolin. In this research raw materials made with a grain size of 100 mesh; 150; 200 and 300 mesh, with the composition of the raw material clay: feldspar: quartz: kaolin = 20: 30: 30: 20, plus 10% water, then printed with the Die Pressing method at a pressure of 5 Pa for 10 minutes . The sample was dried, then the sample was burned in a stove to a temperature of 1000^{o}C, hold for 5 hours. Characterization of ceramics at 300 mesh grain size showed that the density of ceramics tends to increase linearly with decreasing grain size, porosity and water absorption of ceramics tends to decrease linearly with decreasing grain size. The ceramic has iron element Fe cubic structure with lattice parameter a = 2.87Å, $AlKO_2$ compound, orthorhombic structure with lattice parameter a = 5.48Å, b = 11.03Å and c = 15.55Å; SiO2 compounds, trigonal structures, with lattice parameters a = 18.45Å and c = 7.48Å and aluminum elements, have cubic structures with lattice constants a = 4.04Å, with an average ceramic pore diameter of 3.199 μm [16].

(2020) MUNA NOORI ISMAEL, et al, studied the powder-mixing system was utilized to fabricate kaolin-silica fired composites. The main purpose of the paper was to prepare ceramic bodies rich in pores with the goal that it will have a lightweight structure. Different wt.% of silica having distinctive two-particle sizes are blended with kaolin, and afterwards, they shaped by semi-dry pressing in a hardened steel mold under 3-ton loads and sintered at four various temperatures, 900, 1000, 1100 and 1200 °C. Evaluating of composite's physical properties showed that a good level of porosity was obtained, and it was extended; especially in mixtures contain a coarse particle size. These results are enhanced by acceptable evident comes from good apparent solid density and good strength [17].

3.1 STARTING MATERIALS:-

They are silica (SiO_2) and kaolin (clay) for the manufacture of ceramic material, were summarized in Table (3-1):

Table (3-1) Chemical formula starting materials.

Raw Material	formulation
silica	SiO_2
clay	Al2Si2O5(OH)4
PVA	(C2H4O)x

3.2 EQUIPMENTS

1- Sensitive scale

2- Press mold (20 diameter)

3- Sample pressing device

4- Drying oven

5- Burning oven 1000 degrees Celsius

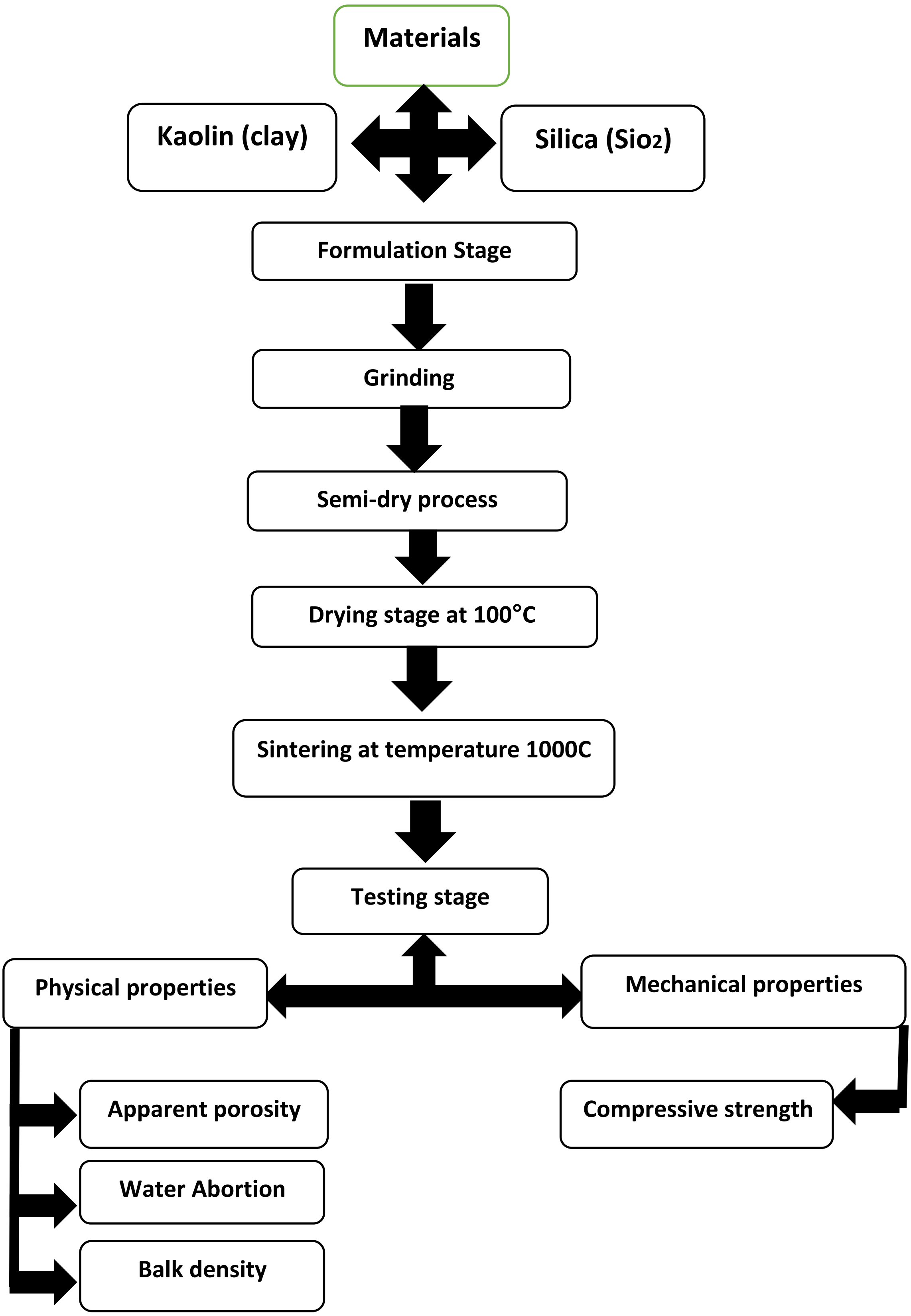

Fig. (3.1): flow chart for preparing

3.3 Formulation stage

3.3-1 Grinding

It is used to produce nano materials in the form of powder, where the basic material is exposed to very high energy, and then grinded using balls made of steel that move in a vibratory, planetary or vertical manner, and the size of the nano materials that are manufactured range from 3 to 25 nanometer The granular size of silica was reduced by 16 micros by grinding it in the nano mill for 10 hours until the required powder was obtained, which is 3 microns.

3.3-2 Particle Size Analysis

Ceramic filter is basically affected by the particle size of their constituents and its size distribution of the mineral which are present. Many characteristics of ceramic firing shrinkage, porosity, density and water absorption properties are dependent upon the particle size and size distribution. Particle size analyzer (Type Better 2000) which is available in the Ceramic and Building Materials Department, Engineering Materials College, University of Babylon, was used to measure the particle size and size distribution of both red clay and additives.

3.3-3 Mixture stage

The batches were prepared to represent the mixtures, which consist of finely clay, Silica and additive PVA each batches mixing to get a homogeneous mixture.

Table (3.2) Batches Proportion of Clay + SiO_2, which represented the constituents of mixtures.

Mix no.	Clay wt.%	SiO_2 (3µm) wt.%	SiO_2 (16 µm) wt.%
Batch 1	100	0	0
Batch 2	90	10	0

Batch 3	**70**	**30**	**0**
Batch 4	**100**	**0**	**0**
Batch 5	**90**	**0**	**10**
Batch 6	**70**	**0**	**30**
Batch 7	**90**	**5**	**5**
Batch 8	**70**	**15**	**15**

3.4 FORMING

The forming stage was applied to prepare the clay specimens by Semi-dry pressing method

3.4-1 Semi-dry Pressing Method

Different proportions of silica SiO_2 were pressed with Clay, at first, the powder was weighed and dry mixed thoroughly in a mortar pestle as shown in table (3-2). Then, followed by addition of desired volume of PVA solution so that final PVA content of the batch was 2%. Using a mold with a diameter of 20 mm and was pressed by a pressing device to prepare the samples with a carrying force of 5 KN for one minute as shown in figure (3.2).

Fig. (3.2) the samples after forming

3.4 -2 DRYING

All the formed cylindrical shape are direct dried in the oven at 100 °C for 24 hr.

3.4 -3 Sintering

An electrical digital furnace was used for sintering the prepared of all samples sintered at a temperature of 1000 ° C for 5 hours with heating rate 10 °C/ min and soaking time (2) hour, after that the furnace was switched off for cooling.

3.5 TESTS

3.5-1 Physical Test Measurement

3.5-1.1 Apperat porosity, Water absorption and bulk density

The density and apparent porosity were measured according to Archimedes method according to (ASTM C373-88) as follow [18]:

1-The test specimen was dried in an drying oven at (110) for (24) hours and allow to cooled to room temperature and their dry mass (D) were record

2-All the specimen was immersed in a beaker of distilled water and boil for (5) hour, and then allow to soak for an additional 24 hr., to record the suspended weight (S).

3-Every specimen was blotted lightly with a moistened cotton cloth to remove the excess water from the surface, and the saturated mass was determined (M)

4-The apparent porosity, bulk density, can be measured using following equation:

Apparent porosity =[(M-D)/(M-S)]*100% …………………….(3-1)

Bulk density =[D/(M-S)]………………………………………...(3.2)

Water Absorption =[(M-D)/D100% ………………………….. (3.3)

3.5 -2 Mechanical Test

3.5-2.1 Compressive test)

The strength of a material is its capacity to withstand destruction under the action of external loads. The compressive test was used for determining the strength and deformation properties of the specimen under uniaxial load.

It is commonly known that most ceramic materials have high compressive strength. The compressive of sample was determined by using (universal testing machine) in the polymer laboratories / College of Material Engineering/ University of Babylon).

Cylindrical samples of the length is twice. This test is done according to the ASTM (C-733-88) standard. Compressive strength of each sample is then calculated using the formula [19]:

$$\sigma_C = \frac{F}{A} \qquad \text{……………………….(3-6)}$$

where:

σ_C : compressive strength in (MPa),

F : Applied load until fracture (N),

A : Cross sectional area (mm^2).

4.2 INTRODUCTION

This chapter includes the main results obtained from the experimental work. The results and their discussion are illustrated the effect of different of additives particle size of SiO_2 of clay samples on them physical and mechanical properties.

4.2 PARTICLE SIZE ANALYSIS

It is necessary to identify the particle size of the starting materials (SiO_2). Figure (4-1) shows the particle size analysis of red clay. It may be noted from this figure, there are three populations of particles size distribution. Their diameters, relative occurrence and an average particle size are summarized in the table below.

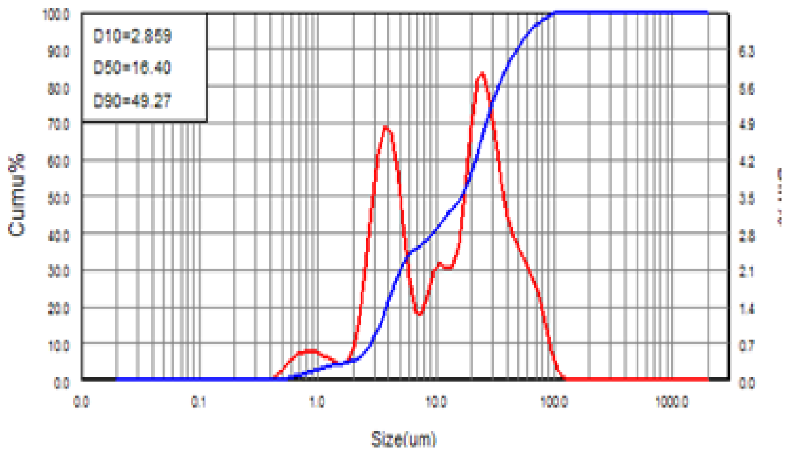

Figure (4-1) Particle size analysis of SiO_2

4.3 PHYSICAL PROPERTIES:

4.3.1 Apparent porosity:

Figures (4.2), (3.2) show the results of apparent porosity and water absorption of the clay samples with different SiO_2 content. They show that the porosity and water absorption of the samples increased with increasing SiO_2 content and when increase the particle size of SiO_2**.** Furthermore, that properties decrease when sample contains mixed particle size of SiO_2 (3+16) µm) may be that because the small particles will be filled the voids between large particles.

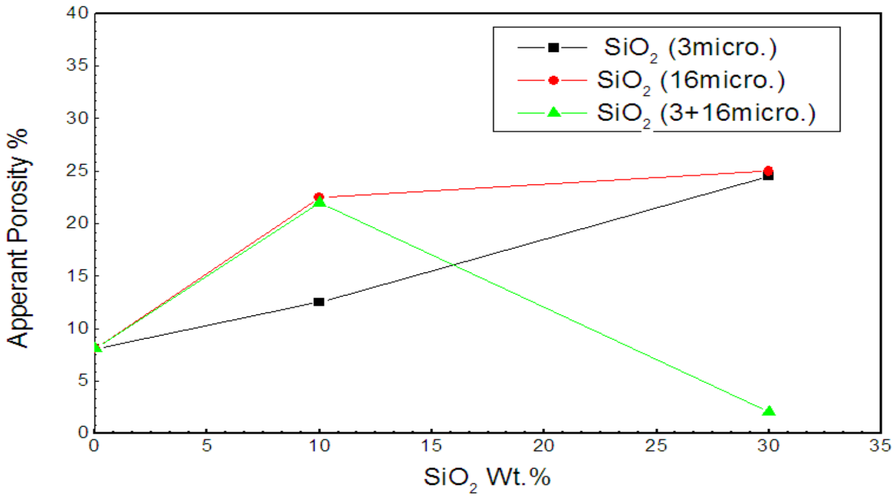

Figure (4.2): Apparent Porosity with different ratio of SiO_2

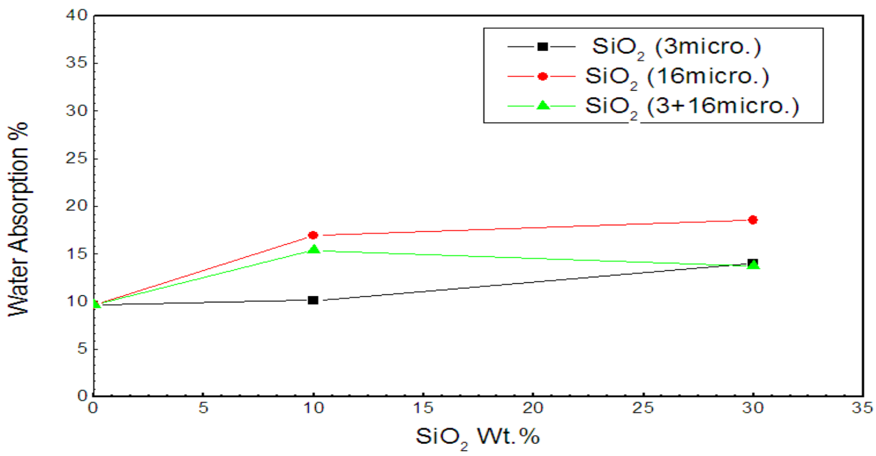

Figure (4.3): Water Absorption with different ratio of SiO_2

4.3.2 Bulk Density:

Figure (4.4), shows the bulk density of the clay samples with different SiO_2 content. The results show that bulk density decreased with increased SiO_2 content and when increase the particle size of SiO_2 that because of increasing of porosity. Furthermore, the density increase when sample contains mixed particle size of SiO_2 (3+16) μm) may be that because the small particles will be filled the voids between large particles.

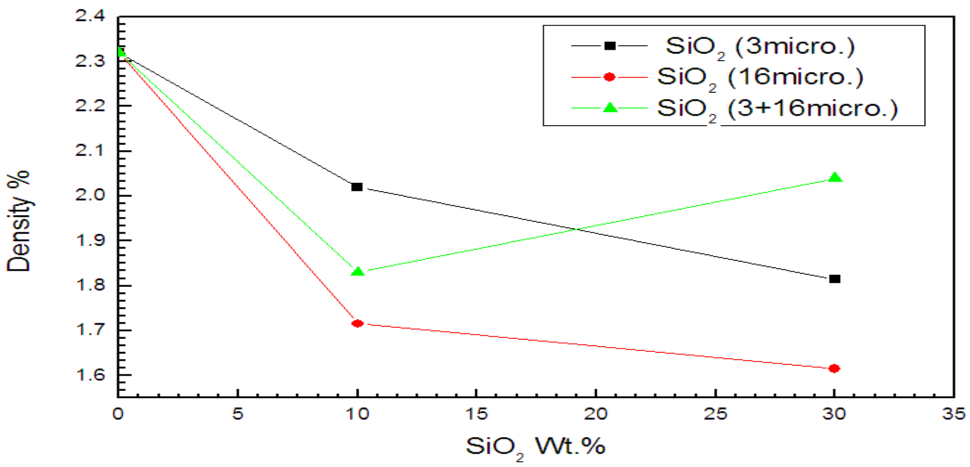

Figure (4.5): **Bulk density with different ratio of SiO_2**

4.4 Mechanicl Properties:

4.4.1 Compressive Strenght:

Figures (4.5) show the compressive strength of the clay samples with different SiO_2 content. The results show that comperssive strength decreased with increased additives content SiO_2, but the comperssive strength increase on sample contains mixed particle size ofSiO_2 (3+16) μm) because of increasing of porosity and decreasing density.

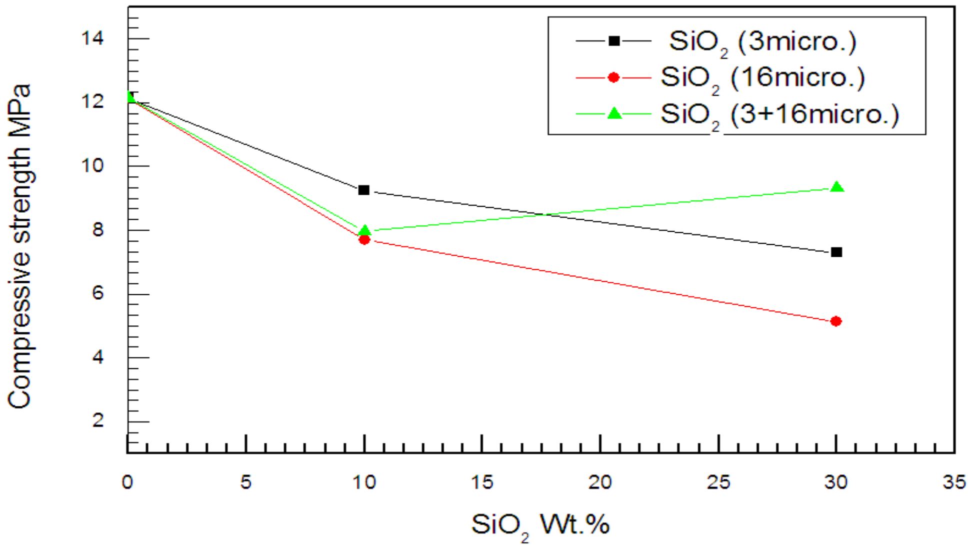

Figure (4.5): **Compressive strength with different ratio of SiO_2.**

CHAPTER FIVE CONCLUSIONS & RECOMMENDATIONS

5.1 Conclusions

1- Synthesis of macro-porous clay with different ratio of SiO_2 by semi-dry pressing.
2- The porosity and water absorption increase as the proportions of additive of SiO_2 will increase and with increase particles size of SiO_2. While decrease on sample contains mixed particle size of SiO_2 (3+16) µm).
3- The density decrease as the proportions of additive of SiO_2 will decrease and the density with increase particles size of SiO_2. While increase on sample contains mixed particle size of SiO_2 (3+16) µm)
4- The compressive strength of samples decreasing with increasing additives content of SiO_2. While increase on sample contains mixed particle size of SiO_2 (3+16) µm).

5.2 Recommendations

1- Studying the effect of nano additives on porous clay of the same raw materials.
2- Studying the effect of different calcinations temperature on porous clay body properties.

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