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Assessment using of various sources of silica on the properties of porcelain

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CERTIFICATION

I certify that this project entitled " *Assessment using of various sources of silica on the properties of porcelain* " was prepared by " *Gufran Adel Waheed* " under my supervision at Babylon University/ College of Materials Engineering/ Department of Ceramic and Building Materials Engineering, in partial fulfillment of the requirements achieve graduate degree in materials engineering .

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Date: 29 / 5 / 2022

بِسْمِ اللهِ الرَحْمَنِ الرَحِيمِ

(يَرْفَعِ اللهُ الَّذِينَ آمَنُوا مِنْكُمْ وَالَّذِينَ أُوتُوا الْعِلْمَ دَرَجَاتٍ وَاللهُ بِمَا تَعْمَلُونَ خَبِيرٌ)

صدق الله العلي العظيم

سورة المجادلة/الآية ١

ABSTRACT

Various sources of silica were used in porcelain production to study their effect on the properties of porcelain. These sources of silica are silica fume, porcelanite and silica sand. The porcelain samples were prepared by use (50% kaolin , 20% feldspar, and 30% silica source). After preparation of the porcelain samples, the samples were dried at temperature (110ºC) for three hours, then sintered at temperature (1200ºC) with heating rate (5ºC/min) and soaking time for (2 hours). Linear shrinkage, apparent porosity, bulk density, the fracture strength and the hardness of the samples were measured. Results of these tests showed that an use of these sources of silica in the porcelain manufacture exhibits a good physical and mechanical properties and the environmental and economic interests.

Contents

Chapter One

INTRODUCTION

Chapter One
Introduction

1-1 Introduction

Although the term porcelain is sometimes applied to a variety of vitreous and near vitreous ware, it is more properly restricted to translucent vitreous ware. A wide range of triaxial ceramic compositions that are used in white ware industries basically contain kaolin, quartz and feldspar [1]. Porcelain insulators and porcelain shells are important equipment in the operation of power plants and transformer substations insulation and supporting wire [2]. Porcelain materials have very interesting properties for many industrial applications. Ceramics possess an extremely low thermal expansion; low thermal conductivity, and high mechanical strength, these properties give an excellent thermal shock [3].

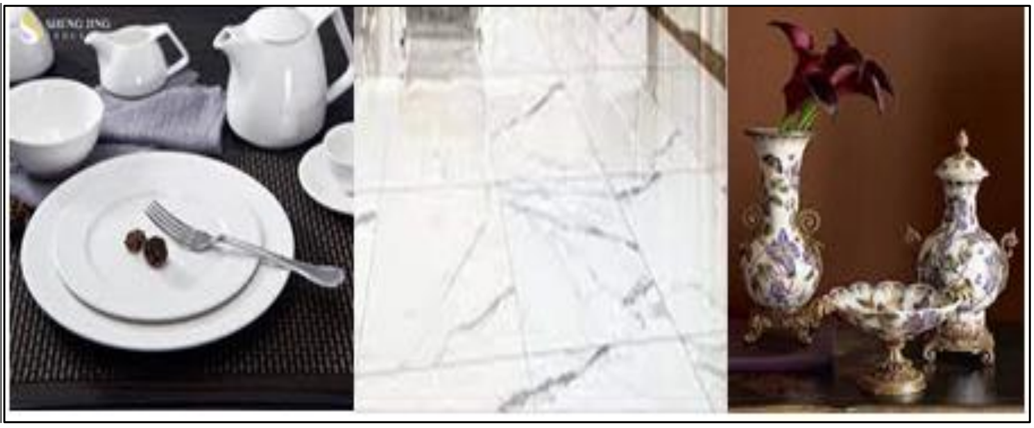

Figure (1-1) Some products of porcelain

It is well documented that nominally identical specimens of brittle materials such ceramics show a large variation of fracture stresses and in order to use brittle material as engineering ones, strength has to be characterized. Complicating factors affecting the strength of ceramics are manifested mainly in two main ways: First, strength is generally time dependent in that the applications of tension stress on a component causes a gradual dimension of its capability to withstand further stress without rupture. Second, there is a relatively large statistical variation in the strength of a batch of otherwise identical specimen [4].

1-2 Silica

Silica, SiO_2, is a polymorphic raw material found in nature in an amorphous (opal, pebbles) or crystallized form (quartz, cristobalite and tridymite). Sand contains between 95 and 100% of quartz mass. It is the most frequently used temper in the ceramic industry. To contribute significantly to the mechanical strength of the raw parts, it must consist of much coarser particles than those of clay. In the modern manufacturing processes of stone wares and porcelains, it is customary to use relatively fine sand grains (20 to 60 μm).

When a ceramic is fired, the sand can react, particularly with the fluxes. This reaction is seldom complete. The transformation of residual quartz into cristobalite can then start from (1200°C) onwards. It is favored by the rise in temperature, the use of fine grained sand, the presence of certain impurities and a reducing atmosphere [5].

1-3 Aim of the study :

The aim of this study is to assess an effect of the various sources of silica on the properties of porcelain and determine the best among them.

Chapter Two

Theoretical Part

Chapter Two
Theoretical Part

2-1 Introduction

The most significant development in the history of ceramics many centuries ago was the production of a vitrified, translucent porcelain body in China. Today, porcelain is produced in many countries and its technology is well known and described in different textbooks and papers. Although the term porcelain is sometimes applied to a variety of vitreous and near vitreous ware, it is more properly restricted to translucent vitreous ware. A wide range of triaxial ceramic compositions that are used in white ware industries basically contain clay, quartz and feldspar. The triaxial porcelain is one of the most widely studied ceramic systems. It has got diverse applications like whiteware, stoneware and insulators. Extensive research on porcelain for a long time confirmed its complexities, so they remain significant challenges in understanding porcelain in relation to raw materials, processing science, phase and micro structural evolution [6].

2-2 Porcelains

Porcelain is a type of ceramics highly valued for its beauty and strength. The raw materials used for the body compositions of porcelain can be divided into three groups of minerals, each having its own function: the clay raw materials give plasticity to the body, while the complementary non-plastic ones include melting minerals and structural ones. The clay minerals of illitic-kaolinitic or montmorillonitic origin belong to the first group and show more or less remarkable plastic characteristics with regard to their mineralogical structure and to their particle-size distributions. The melting minerals are feldspars and feldspathoids, talc, pegmatites. The feldspar is the most abundant mineral group in the world, forming around 60% of earth‘s crust, and is found in igneous, metamorphic and sedimentary deposits in most countries. They are used in the production of glass, ceramics and in polymer, paper and paint industries as fillers and extenders. Silica is often associated with the feldspars, as quartz in pegmatic deposits and silica in feldspathic sand deposits. Additionally, quartz and generally quartzites are the most refractory ones of those having a structural function [6].

In porcelain composition, clay serves as a dual purpose of providing fine particle size and good plasticity for forming. Feldspar acts as a flux, forming a viscous liquid at firing temperature and aids in vitrification. The quartz is mainly an inexpensive filler material which remains unreactive at low temperatures of firing and forms a highly viscous liquid at higher temperatures [7].

2-3The main raw materials to manufacture porcelains

In order to select the suitable raw materials, the properties of the final product had to be taken into consideration. A sufficient amount of feldspar is necessary to obtain the desired glassy phase. As in all traditional ceramics, the presence of quartz is necessary in order to decrease shrinkage. It also reduces the body tendency to warp or distort during firing, whereas, kaolin is characterized by low plasticity. Hence, the raw materials used to prepare porcelain bodies play a vital role in the ultimate product quality. These raw materials will be described below [7].

2-3-1 Clays

Refractory clays are used in high temperature processes. Their composition is rich in alumina. Kaolins are the most refractory among these clays. Always purified, they contain little quartz, generally less than 2% alkaline oxides in combined form and a small quantity of mica. Their plasticity is ensured by kaolinite and, if necessary, a little smectite or halloysite [CAR 98]. Very low in coloring element, they are particularly suited for the preparation of products in white shard [5].

2-3-1-1 Kaolin

Kaolin (Al_2O_3,$2SiO_2$,$2H_2O$) is the most common among the argillaceous minerals used in ceramics. The degree of crystallinity of the kaolinite present in clays is highly variable. It depends largely on the genesis conditions and the content of impurities introduced into the crystalline lattice. During the heat treatment, kaolin undergoes a whole series of transformations [5].

2-3-2 Feldspars

Feldspar is a group of anhydrous alumina silicate minerals called orthosilicates. These silicates are in general igneous rocks. Igneous rock was at one time molten and cooled to its present form. Some examples of igneous rocks are granites, feldspars, rhyolites, basalts, etc. The composition of feldspars can vary significantly, having different end-members such as K, Na and Ca. These feldspars are orthoclase ($KAlSi_3O_8$) albite ($NaAlSi_3O_8$) and anorthite ($CaAl_2Si_2O_8$). Feldspar is often left unaltered in certain amounts during the formation of clay deposits [8].

Feldspars play an important role in ceramic materials, acting as fluxing agents to reduce the sintering temperatures of the clays. Potassium is a very powerful flux during firing and determines the fusibility of the feldspars and their ability to form eutectics with other components. The formation of eutectics makes it possible to reach a high densification of the ceramic materials even at low temperatures.

Feldspars are often used in formulations of porcelain pastes for production of stoneware tiles, tableware and sanitary ware bodies. The raw materials for the manufacture of such products are mixtures composed mainly of white kaolin, talc, feldspar and quartz. In fact, the large densification and high mechanical resistance showed by these ceramic materials after firing are due to the action of feldspars [8].

2-3-3 Silica

Silica (SiO2) is the most abundant components in the earth's crust and is made by way of synthesis for use in the application of technology [9].

Silica is a polymorphic raw material found in nature in an amorphous (opal, pebbles) or crystallized form (quartz, cristobalite and tridymite). Sand contains between 95 and 100% of quartz mass. It is the most frequently used temper in the ceramic industry [5].

Generally, there are a many sources rich with silica, from them silica fume, porcelanite and silica sand. These sources, which were used in this study to manufacture porcelain samples, will be described below.

2-3-3-1 Silica fume

Silica fume (SF), also known as microsilica, is a waste product from the metallurgical industry that occurs in the production of metallic silicon, ferrosilicon and other silicon alloys. It consists of microscopic spherical particles which have a diameter of approximately 0.1 μm and a specific surface area of approximately 20 m^2/g.

Silica fume is characterised by flabby nature (thus behaving like a fume or smoke when dispersed in air) and glassy nature [10].

Table (1) Physical and chemical properties of silica fume.

Chemical properties	Value	Physical properties	Value
SiO_2 (%)	90-95	Color	Grey
C (%)	0.8-3	Diameter (μm)	<1μm
Loss on ignition (%)	1.5-2.5	Specific gravity	2.2
		Surface area (m^2/g)	15-30

Figure (2-1) Silica fume (SF).

2-3-3-2 Iraqi Porcelanite Rocks

Porcelanite is a term used by Iraqi geologist to identify siliceous rock resemble to diatomite. These rocks are found in Iraq in different places. Porcelanites these rocks, composed of Opal-CT (cristobalite-tridymite crystal stratification) derived from biogenic amorphous opal silica (mainly from diatoms). They are part of the phosphorite-bearing sequences of the mastrichtianin the Western Desert (Digma and Akaashat formations respectively several Porcelanite horizons were identified as 0.5-1.5 m thick layers associated with shale phosphorite and chert [11]. Porcelanite rocks are largely composed of sponge spicules (pore) and some other siliceous microfossils (diatoms and radiolarian), as well as silicified foraminifera and non-plankton. Diatomite deposits have been found in many different countries and varies in their quality, purity, and uses from one area to another [12]. Table (2) shows the chemical composition of the Iraqi porcelanite rocks [13, 14].

Table (2) Chemical composition of the porcelanite.

Oxide %	Average for three beds of porcelanite
SiO_2	70.36
Fe_2O_3	1.0
Al_2O_3	2.6
CaO	6.1
MgO	5.4
SO_4	0.37
L.O.I	10.8
Na_2O	0.73
K_2O	0.14
Cl	1.08
P_2O_5	1.42
Total	100.0

Figure (2-2) The porcelanite rocks.

2-3-3-3 Silica sand

Silica sand is natural sand which contains high content of silica, generally higher than 98% silicon dioxide (SiO_2). It is defined as one of the commonest minerals in the earth's crust. In nature, silica sand occurs as a crystalline mineral in many and varied forms. The most common is quartz, commonly clear or white, with a specific gravity of 2.65. In many applications, silica sand is important as an exploitable industrial mineral. It is used mainly as construction materials, foundry materials, in the glass manufacture and the chemical industries [15].

Figure (2-3) Silica sand.

2-4 Literature review

In 2003, C.S. Prasad et al. studied an effect of substitution of quartz by rice husk ash (RHA) and silica fume (SF) on the properties of whiteware compositions. The influence of the addition on the thermo-mechanical properties, vitrification behavior and microstructure has been investigated. It has been found that the improvement of properties of the whiteware compositions is attributed to sharp changes in the microstructural features as a result of significant reduction in the content of the quartz phase and the simultaneous increase in glassy phase. [16].

The effect of silica fume (SF) addition as a substitution of quartz on the thermo-mechanical properties and microstructure of traditional whiteware body compositions was studied by C.S. Prasad et al. in 2002. Noticeable improvement in the mechanical strength was observed on replacing quartz by 10% silica fume in whiteware body with the reduction of maturing temperature by

50˚C whereas addition of 25% silica fume reduced by 100˚C. The lower maturing temperature of the body would be helpful to reduce the fuel consumption and thus reduce production costs [17].

Other studies were made for the same purpose, such as the study titled with "Waste silica from aluminum fluoride industries used for ceramic whitewares " which worked by R. Sarkar et al. in 2007 [18], and the study titled with " Agricultural wastes as a source of silica material" which worked by N. Permatasari et al. in 2016 [9].

Chapter Three

Experimental Work

Chapter Three
Experimental Work

3-1 Introduction

This chapter explains the experimental work which was made in this study in laboratory, as it was described below.

3-2 The materials and equipment used in this study:

1) Kaolin clay
2) Feldspar (Albite)
3) Silica fume
4) Porcelanite
5) Silica sand
6) Sieve
7) Sensitive Balance
8) Mixing device
9) Stainless-Steel die
10) Compression device
11) Drying Oven
12) Burning furnace
13) Verna
14) Microhardness device
15) Archimedes device
16) General test machine

3-3 Experimental work

The porcelain samples were prepared from a mixture of kaolin clay, feldspar and one of the silica sources which used in this study (which are silica fume, porcelanite and silica sand) . The experimental work includes the following stages:-

3-3-1 Preparation of primary materials:

1. Sieving process: Kaolin powder was sieved by a (98μm) sieve to obtain a fine powder. Figure (3-1 a) shows the sieving device which used in this process.
2. Weighting process: The weights of kaolin, feldspar, silica fume, porcelanite and silica sand were taken by sensitive balance, which shown in figure (3-1 b). Table (1) shows the mixing percentages of materials which were used in this study. Where the total weight of one sample was (5) g.
3. Mixing process: The powders of materials were mixed according to the mixing percentages in table (1) by use the electrical mixer which shown in figure (3-1 c) for (3) hour to obtain a homogeneous mixture.

(a) (b) (c)

Figure (3-1): a) The sieving device, b)The sensitive balance, and c) The electrical mixer

Table (1) shows the mixing percentages of materials

Samples	Raw materials for porcelain		
	Kaolin	Feldspar	Silica source
A (with Silica fume)	50	20	30
B (with Porcelanite)	50	20	30
C (with Silica sand)	50	20	30

3-3-2 Forming of samples

Single direction Semi-dry pressing method was used in samples formation by using hydraulic uniaxial pressing machine which shown in figure (3-2 a) at a pressure of (70 MPa) with using stainless-steel die with (d= 20mm). Liquid paraffin wax was used as the lubrication to reduce the friction between the two parts of the die and to prevent adhesion between the particles with die wall during getting out the sample from the die after the pressing. Polyvinal alcohol (PVA) binder was used to prepare the samples as the pressing is semi-dry.

3-3-3 Drying process

The samples were dried at temperature (110ºC) for three hours by use the drying furnace which shown in figure (3-2 b) to remove the moisture from the samples.

3-3-4 Burning process

An electrical furnace, which shown in figure (3-2 c), was used for burning the samples in this study at temperature (1200ºC) with heating rate (5ºC/min) and soaking time for (2 hours). Figure (3-3) shows the samples after firing.

(a) (b) (c)

Figure (3-2): a) Pressing machine, b) Electrical drying Oven, and c) Electrical firing furnace.

Figure (3-3) The samples after firing

3-4 Tests

Several tests were carried out for the samples produced to study their properties, and these tests can be divided into:

1- Physical tests.

2- Mechanical tests.

3-4-1 Physical tests:

a) Linear shrinkage

Linear shrinkage on firing (L.S.%) was evaluated for samples by measuring the outer diameters of the samples before and after sintering process (which are $D_1$and D_2 respectively). Linear shrinkage (L.S.%) was calculated by the equation below according to (ASTM C326):

$$L.S.\% = \frac{D_1 - D_2}{D_1} \times 100\% \quad \ldots\ldots\ldots\ldots\ldots \quad (1)$$

b) Porosity

Calculation of porosity for the samples was based on ASTM standard (C373-88) [19]. The percentage of porosity was calculated by the equation (2). Archimedes device was used in this test, which shown in figure (3-4 a)

$$Porosity\% = ((M-D)/(M-S))*100 \quad \ldots\ldots\ldots\ldots \quad (2)$$

Where:

M: saturated weight (g),

D: dry weight (g),

S: Suspended weight (g).

c) Density

The density of samples (ρ) was calculated according to ASTM standard (C373-88) by use the following equation:

$$\rho = D/ M-S \quad \ldots\ldots\ldots\ldots\ldots \quad (3)$$

3-4-2 Mechanical Test:-

a) Fracture Strength

The fracture strength of the samples was calculated according to the ASTM (C 773-88) standard by use the following equation [20]:

$$б_c = F / A_r$$

where: $б_c$= Fracture strength in (MPa).

F = Applied load until fracture (N).

A_r= Cross section area (mm^2).

b) Hardness

The hardness of samples was tested by Vickers hardness. Vickers hardness values were measured on surfaces by Vickers indentation technique at (10) kg load applied for (12) seconds using the device shown in figure (3-4 b).

(a) (b)

Figure (3-4): a) Archimedes device, and b) Microhardness devise

Chapter Four

Results and Discussion

Chapter Four
Results and Discussion

4-1 The physical tests

Figure (4-1) shows the effect of silica source used in manufacture of porcelain samples on the linear shrinkage of these samples. Where, the linear shrinkage of porcelain samples with silica fume was higher than that of porcelain samples with silica sand or porcelanite, and the linear shrinkage of porcelain samples with silica sand was the least. While, figure (4-2) shows the effect of silica source used in porcelain samples on the apparent porosity of these samples. Where, the apparent porosity of porcelain samples with silica sand was higher than that of porcelain samples with silica fume or porcelanite, and the apparent porosity of porcelain samples with porcelanite was the least. Also figure (4-3) shows the effect of silica source used in porcelain samples on the bulk density of these samples. Where, the bulk density of porcelain samples with porcelanite was higher than that of porcelain samples with silica fume or silica sand, and the bulk density of porcelain samples with silica sand was the least. This difference in the densification behavior of porcelain samples with various sources of silica due to the difference in the chemical and mineralogical composition of these sources, which was mentioned previously in the chapter two. Where, it can be shown that the chemical composition of porcelanite contains on the large amount of alkalies, such as a CaO, MgO, K_2O and Na_2O. These oxides may play a significant role towards vitrification, phase transformation and mullite grain growth in the porcelain body as reported by other studies [21, 22], where the presence of such oxides in a porcelain body and kaolin-alumina silica mixture enhances their properties. Also the chemical composition of porcelanite indicated a loss on ignition 10.8% which may be due to the presence of carbon. That leads to the increase in the content of glassy phase which fills the pores, thus improvement of the densification behavior of porcelain samples. Also with respect to silica fume, where the densification behavior of porcelain samples which contains on silica fume depends on the chemical composition of silica fume, and the fineness and the flabby nature of silica fume particles in comparison with silica sand and porcelanite.

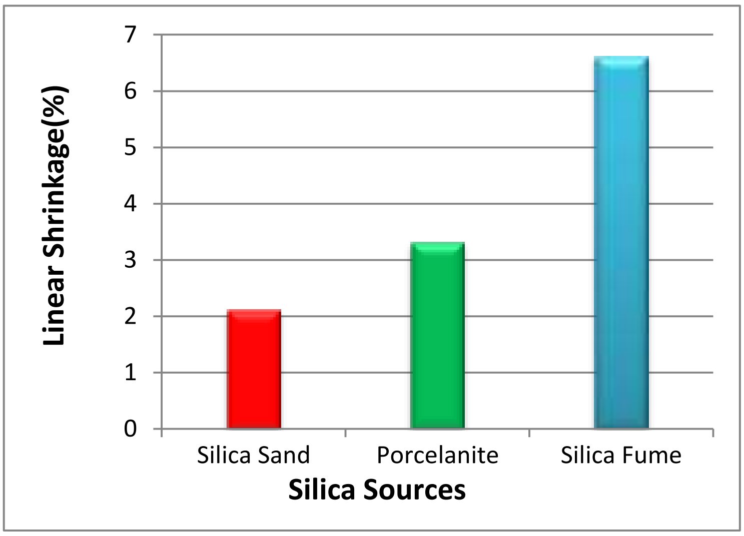

Figure (4-1) Linear shrinkage of porcelain samples with various sources of silica.

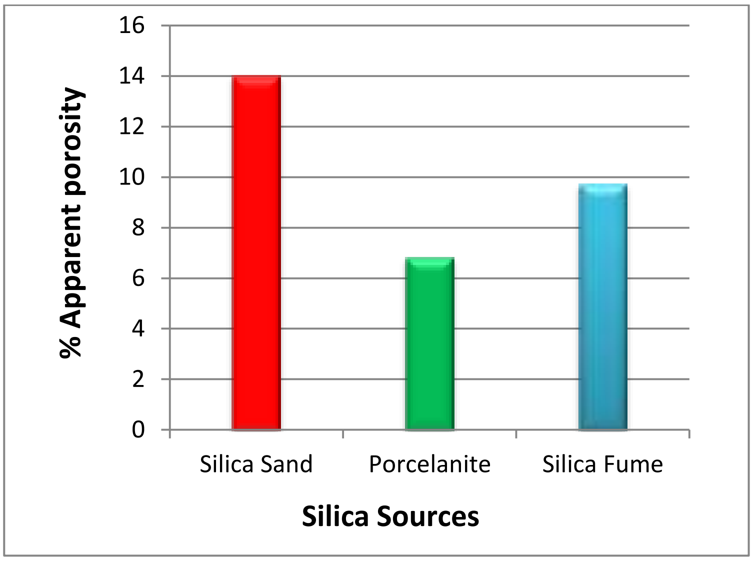

Figure (4-2) The apparent porosity of porcelain samples with various sources of silica.

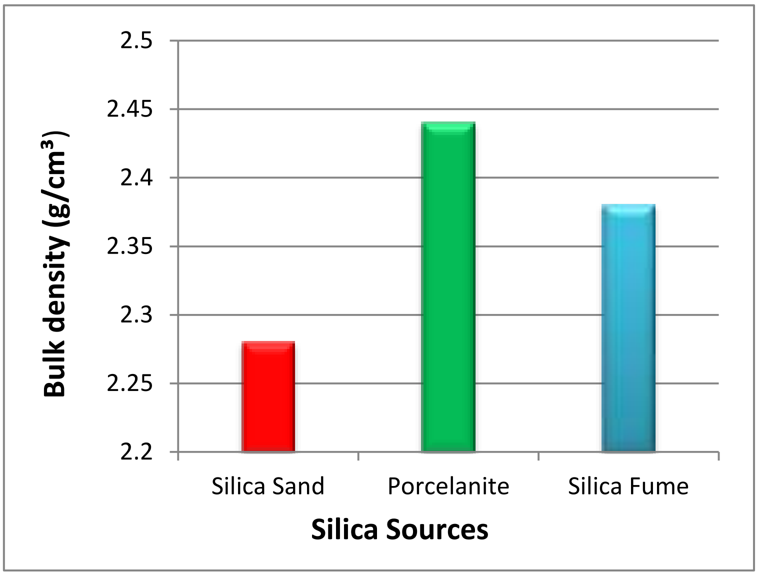

Figure (4-3) The bulk density of porcelain samples with various sources of silica.

4-2 The mechanical tests

Figures (4-4) and (4-5) show the effect of silica source used in manufacture of porcelain samples on the fracture strength and hardness of these samples respectively. Where, the fracture strength and hardness of porcelain samples with silica sand were higher than that of porcelain samples with silica fume or porcelanite, and the fracture strength and hardness of porcelain samples with porcelanite were the least. This is due to the formation of a more glassy phase in the porcelain samples with silica fume or porcelanite at the expense of the mullite formation, and the fineness of silica fume particles which effects on the size of quartz grains. Where, it is known that quartz grains in different sizes have significant effects on mechanical strength of porcelain bodies. Especially, it is proposed that flexural strength of the porcelains increases with an increase in inter planar spacing of quartz crystals.

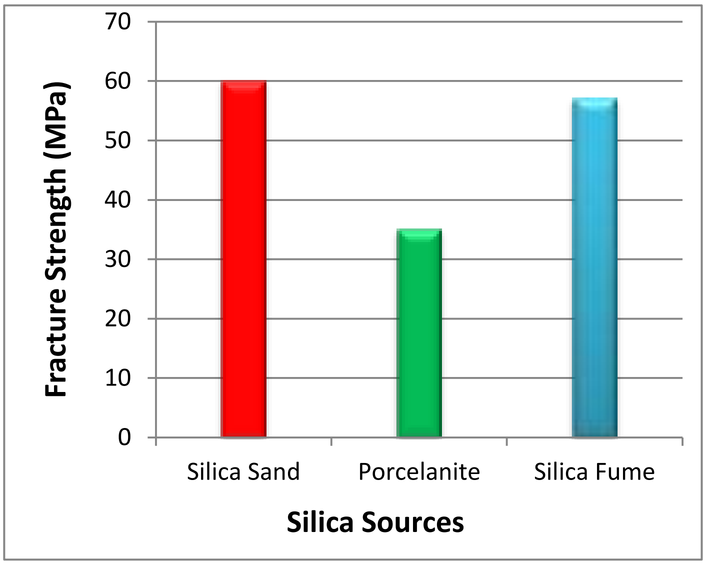

Figure (4-4) Fracture strength of the porcelain samples with various sources of silica.

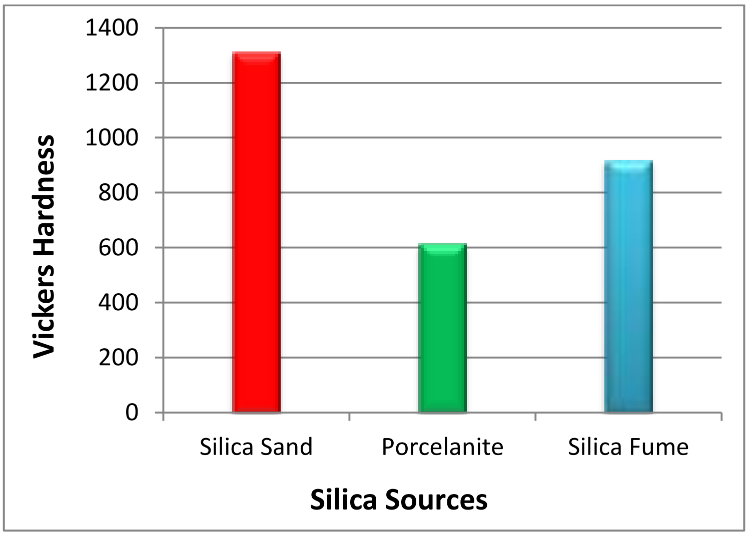

Figure (4-5) Hardness of the porcelain samples with various sources of silica.

Chapter Five

Conclusions and Recommendations

Chapter five
Conclusions and Recommendations

5-1 Conclusions:-

1- In the densification behavior of porcelain samples, the porcelain samples with silica sand were the least and with porcelanite were the highest.

2- The fracture strength and hardness of the porcelain samples with silica sand were the highest.

3- These sources of silica can be used in the porcelain manufacture to reduce the production cost because of these materials are cheaper than the pure silica (quartz) and they don't need to refine, thus reduction of the energy consumption.

4- The porcelain samples with silica fume showed the intermediate values of physical and mechanical properties, in addition to the silica fume is the byproduct for many industries (i.e. waste), thus its use in the porcelain manufacture exhibits the environmental and economic interests.

5-2 Recommendations:

1- Study the effect of other silica sources to improve the mechanical properties of porcelain.

2- Study the effect of the silica sources used in this study on the thermal properties of porcelain.

3- Study the effect of the silica sources used in this study on the properties of other ceramic products.

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