



Ministry of Higher Education  
and Scientific Research  
University of Babylon



College of Materials Engineering

Department of Ceramic and Building Materials Engineering

# **Strengthening of Porcelain by Zirconia Addition**

A research

Submitted to the College of Materials Engineering of Babylon  
University as a Part of Requirements Achieve graduate  
Degree in Materials Engineering / Department of Ceramic  
and Building Materials Engineering

By

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وزارة التعليم العالي والبحث العلمي  
جامعة بابل – كلية هندسة المواد  
قسم هندسة السيراميك ومواد البناء



## تقوية البورسلين باضافة الزركونيا

بحث مقدم إلى جامعة بابل كلية هندسة المواد – قسم هندسة سيراميك ومواد البناء  
كجزء من متطلبات نيل شهادة البكالوريوس في هندسة السيراميك ومواد البناء

من قبل

**نور سعود عزيز**

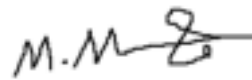
بإشراف

أ.م. مروة مرزة سلمان

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## CERTIFICATION

I certify that this project entitled "*Strengthening of Porcelain by Zirconia Addition*" was prepared by "*Noor Saud Aziz*" 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 .



Signature :

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(Supervisor)

Date: 25 / 5 / 2022

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

﴿إِقْرَأْ وَرَبُّكَ الْأَكْرَمُ﴾ الَّذِي

عَلَّمَ بِالْقَلَمِ ﴿عَلَّمَ الْإِنْسَانَ مَا لَمْ

يَعْلَمُ﴾

صدق الله العلي العظيم

سورة العلق . الآيات ٣ - ٤ - ٥

## الإهداء

أول من يُشكر ويُحمد أناء الليل وأطراف النهار هو العلي القهار  
الذي اغرقنا بنعمته التي لا تحصى واغدق علينا برزقه الذي لا يفنى  
وانار دروبنا فله جزيل الحمد والثناء العظيم

الى من علمني ان الدنيا كفاح وسلاحها العلم والمعرفة الى الذي لم  
ييخل علي بأي شيء الى من سعى لأجل نجاحي، الى اعظم واعز  
رجل في العالم ( أبي العزيز )

الى من ساندتني في صلاتها ودعائها الى من سهرت الليالي لتنير  
دربي الى من تشاركني افراحي واساتي الى نبع العطف والحنان الى  
اجمل ابتسامة في حياتي، الى ارواح امرأة في الوجود (امي الغالية)  
الى القلوب الطاهرة الرقيقة والنفوس البريئة، الى رياحين حياتي  
( اخوتي )

أقدم شكري وأمتناني الى من اعانتني وساعدتني في اخراج هذا  
المشروع بفضلها وجهدها وآرائها القيمة التي ابدتها الي، الى مشرفة  
البحث ( الست مروة مرزة )

الى كل من اضاء بعلمه عقل غيره أو هدي بالجواب الصحيح حيرة  
سائله فاطهر بسماحته تواضع العلماء وبرحابته سماحه العارفين  
( أساتذتي )

## ABSTRACT

In this study, the effect of zirconia additive on physical and mechanical properties of porcelain was evaluate to improve these properties. The porcelain samples were prepared by use (50% kaolin , 25% feldspar, and 25% silica) at first stage, then mixed with (0, 5, 10)% wt. of zirconia at second stage. After preparation of the porcelain samples with zirconia additive in the second stage, the samples were dried at temperature (110°C) for three hours, then sintered at temperature (1200°C) with heating rate (5°C/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 addition of zirconia to porcelain improves its physical and mechanical properties.

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# Chapter One

# ***INTRODUCTION***



# Chapter One

## Introduction

### 1-1 Introduction

porcelain is produced in many countries and its technology is well known and described in different textbooks and papers [1]. 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. 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 Zirconia**

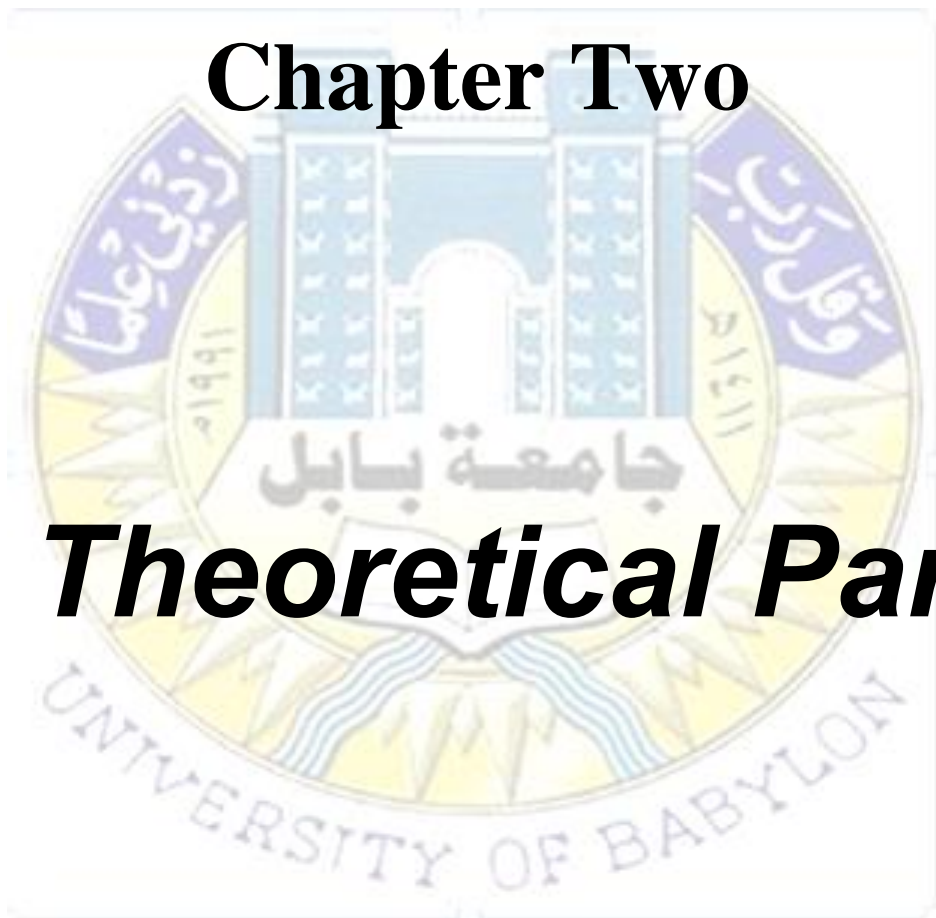
Zircon is a shiny gray-white metal, which may look blue-black when in powder form. Zirconia is an oxide which has a high tensile strength, high hardness and corrosion resistance. It is not found as a pure oxide in nature. The main sources of zirconium are zirconate ( $ZrO_2-SiO_2$ ,  $ZrSiO_4$ ) and baddelyite ( $ZrO_2$ ), and most of the material used is chemically extracted from these two minerals. The zirconate is more abundant, but less pure, requiring significant processing to get zirconia. Baddelyite already contains levels of zirconia ranging from 96.5% to 98.5%. As this mineral shows significant levels, it is known as a source of extreme purity in obtaining zirconium metal and its compounds. Zirconium dioxide ( $ZrO_2$ ) resulting from baddelyite, which is also known as zirconia, is a coarse oxide

that presents a monoclinic crystal structure at room temperature. However, the powder can be purified and processed synthetically at high temperatures, forming a cubic structure called cubic zirconia. The resulting material is hard, optically flawless and translucent, usually used for making precious stones or gas sensors, P. ex. Zirconia is a polymorphic material occurring in three temperature-dependent forms: monoclinic or baddeleyte (room temperature to 1170°C), tetragonal (1170–2370°C) and cubic (2370–2700°C, which corresponds to the melting point) [5].

**1-3 Aim of the study :**

The aim of this study is to evaluate an effect of the zirconia additive on some properties of porcelain, especially physical and mechanical properties, to improve these properties.

## Chapter Two



# *Theoretical Part*

## Chapter Two Theoretical Part

### **2-1 Introduction**

Ceramics have some attractive properties compared to metals and polymers, which make them useful for specific applications. Their physical properties have been utilized for many applications. In other applications their mechanical properties are important. The main drawbacks of ceramics are their brittleness and the large scatter in the mechanical properties.

The most important advantageous features of ceramic materials are:

- low electrical conductivity,
- low thermal conductivity,
- low density,
- high strength at high temperatures,
- wear resistance,
- corrosion resistance,
- specific physical properties (optical, electrical, magnetic).

These properties lead to applications in many technical areas.

The main disadvantages of ceramics are [6]:

- low tensile strength at room temperature for some materials,
- brittleness,
- large scatter of strengths,
- subcritical crack extension.

From ceramic materials, porcelain which explained below:

## **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].

Silicate ceramics can be formed in various ways: by casting in a mould aqueous suspension called slip, by extrusion or jiggering of a plastic paste, or by unidirectional or isostatic

pressing of slightly wet aggregates. The quantity of water contained in the sample therefore depends on the method of forming. Generally, water is eliminated during a specific drying treatment. The raw part is transformed into ceramic by sintering, also called firing, carried out under suitable conditions of temperature, heating rate and atmosphere. Depending on the application considered, this ceramic, also called shard, can be dense or porous, white or colored [7].

### **2-3 The main raw materials to fabricate porcelains**

Each mineral raw material has a specific influence on the rheology of the paste, the development of the microstructure, the phases formation during the heat treatment and the properties of the finished product. The manufacture of all silicate ceramics requires such a large number of raw materials. Those most commonly used, i.e. clays, feldspars and silica, will be described below [7].

#### **2-3-1 Clays**

Clays are hydrated silico-aluminous minerals whose structure is made up of a stacking of two types of layers containing, respectively, aluminum in an octahedral environment and silicon in tetrahedral coordination.

All clays do not exhibit the same attitude towards manipulation and behavior during firing. Ceramists distinguish vitrifying plastic clays, refractory plastic clays, refractory clays and red 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 [7].

### **2-3-1-1 Kaolin**

Kaolin ( $\text{Al}_2\text{O}_3, 2\text{SiO}_2, 2\text{H}_2\text{O}$ ) 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 [7].

### **2-3-2 Feldspars**

Feldspar is by far the most abundant group of minerals in the earth's crust, forming about 60% of terrestrial rocks. Most deposits offer sodium feldspar as well as potassium feldspar and mixed feldspars. Feldspars are primarily used in industrial applications for their alumina and alkali content.

The term feldspar encompasses a whole range of materials. Most of the products we use on a daily basis are made with feldspar: glass for drinking, glass for protection, fiberglass for



insulation, the floor tiles and shower basins in our bathrooms, and the tableware from which we eat. Feldspar is part of our daily life [8].

Chemically, the feldspars are silicates of aluminum, containing sodium, potassium, iron, calcium, or barium or combinations of these elements, which are [8]:

- Orthoclase, ( $K_2O, Al_2O_3, 6SiO_2$ );
- Albite, ( $Na_2O, Al_2O_3, 6SiO_2$ );
- Anorthite, ( $CaO, Al_2O_3, 2SiO_2$ );
- Petalite, ( $Li_2O, Al_2O_3, 8SiO_2$ ).

### **2-3-3 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 stonewares and porcelains, it is customary to use relatively fine sand grains (20 to 60  $\mu m$ ).

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 [7].

## **2-4 Zirconia**

Zirconia is a very important industrial ceramic for structural applications because of its high toughness, which has proven to be superior to other ceramics. In addition, it has applications making use of its high ionic conductivity. The thermodynamically stable, room temperature form of zirconia is baddeleyite. However, this mineral is not used for the great majority of industrial applications of zirconia. The intermediate-temperature phase of zirconia, which has a tetragonal structure, can be stabilized at room temperature by the addition of modest amounts (below ~8 mol%) of dopants such as  $Y^{3+}$  and  $Ca^{2+}$ . This doped zirconia has mechanical toughness values as high as 17 MPa. On the other hand, the high-temperature phase of zirconia, which has a cubic structure, can be stabilized at room temperature by the addition of significant amounts (above ~8 mol%) of dopants. This form of zirconia has one of the highest ionic conductivity values associated with ceramics, allowing the use of the material in oxygen sensors and solid-oxide fuel cells. Research on this material actively continues and many improvements can be expected in the years to come.

Zirconia ( $ZrO_2$ ) is an extremely versatile ceramic that has found use in oxygen pumps and sensors, fuel cells, thermal barrier coatings, and other high-temperature applications, all of

which make use of the electrical, thermal, and mechanical properties of this material. Proof of the interest and usefulness of zirconia can be seen from the voluminous literature found on this material. This chapter is intended to provide a concise summary of the physical and chemical properties of all phases of zirconia that underlie the appropriate engineering applications. The three low-pressure phases of zirconia are the monoclinic, tetragonal, and cubic, which are stable at increasingly higher temperatures. Calculated energy vs. volume data at zero absolute temperature confirms the higher stability of the monoclinic phase. However, most engineering applications make use of the tetragonal and cubic phases, even though their stability at low temperatures is quite low. In fact, the engineering use of all three phases of zirconia in pure form is rare. Generally, zirconia is doped with oxides such as Y<sub>2</sub>O<sub>3</sub> that stabilize the high-temperature phases at room temperature. This has enormous consequences for both the mechanical and electrical properties of zirconia, even though the local atomic and electronic structure of Zr<sup>4+</sup> in all three polymorphs is for the most part dopant independent [9].

## **2-5 Literature review**

In 2013, F. A. Chyad investigated scatter characteristics of fracture strength (splitting strength) of porcelain toughened by zirconia and the effect of sintering temperature. Many specimens were tested by Brazillian method to obtain the scatter

data of fracture strength. The probability distribution of fracture strength is evaluated by using Weibull distribution function. Fracture strength and Weibull modulus increased with zirconia content, and also with the increasing of sintering temperature [10].

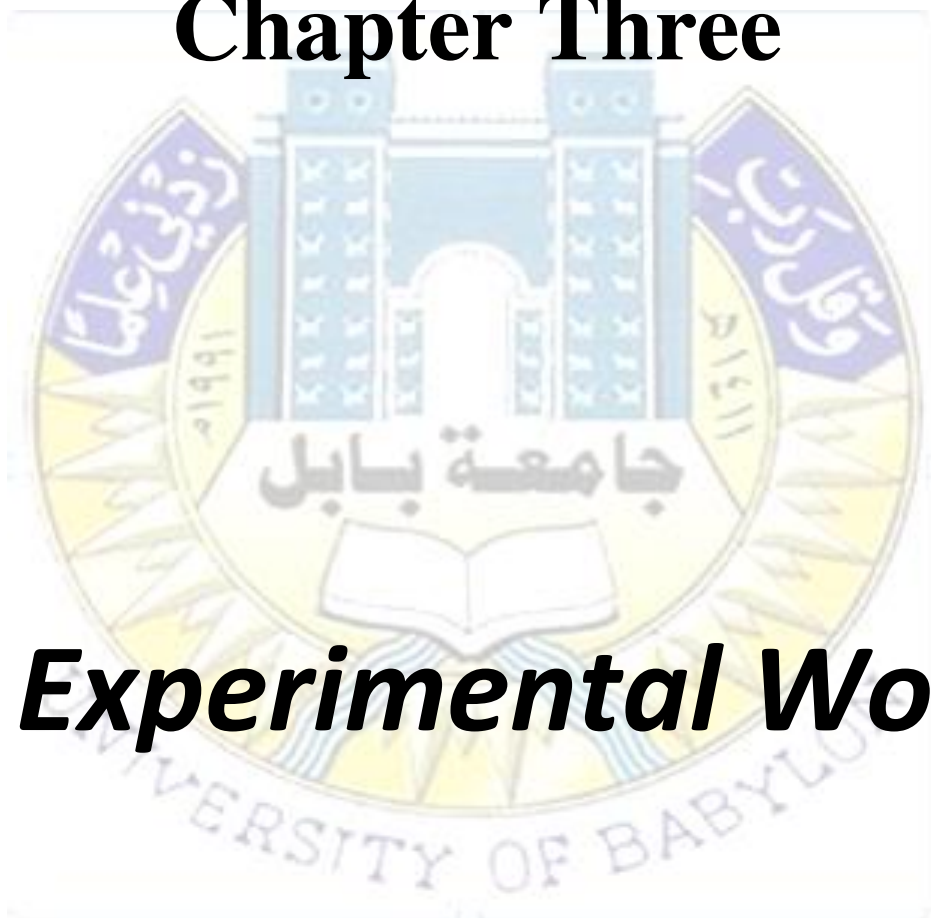
Also in 2013, I. M. Hamouda and M. M. Beyari studied an effect of addition of Yttria partially stabilized Zirconia powder to dental porcelain. The zirconium powder was incorporated in 3, 5 and 7-percent by weight to dental porcelain powder. The measured properties were flexural strength, modulus of elasticity, fracture toughness and hardness. The results indicated that the addition of Yttria partially stabilized Zirconia to the conventional dental porcelain powder resulted in increased flexural strength and fracture toughness of the porcelain. On the contrary, the addition of zirconia decreased the modulus of elasticity. It was concluded that zirconia-modified dental porcelain was stronger and tougher than the conventional unmodified ceramic [11].

While in 2016, Chen et al., investigated improvement of bond strength between zirconia framework and veneering porcelain. Shear strength test, Scanning Electron Microscope (SEM), and Energy Dispersive Spectrometer (EDS) were adopted in the investigation. A remarkably enhance of average bond strength from 24.1 MPa to 37.2 MPa was obtained by performing humps on zirconia substrate [12].

Also in 2017, Almeida Junior et al. studied effect of the cooling rate on the properties of veneer porcelain for zirconia dental prosthesis. All cooling group showed residual tensile stress. It was concluded that cooling rate produce minor changes in the porcelain properties [13].



# Chapter Three



# *Experimental Work*

## Chapter Three Experimental Work

### **3-1 Introduction**

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

### **3-2 The materials and equipment used in this work:**

- Kaolin clay
- Quartz
- Feldspar (Albite [ $\text{Na}_2\text{O}, \text{Al}_2\text{O}_3, 6\text{SiO}_2$ ])
- Zirconia
- Sieve
- Sensitive Balance
- Mixing device
- Stainless-Steel die
- Compression device
- Drying Oven
- Burning furnace
- Milling machine
- Verna
- Microhardness device
- Archimedes device
- General test machine

### **3-3 Experimental work**

The porcelain samples used in this study were prepared from a mixture of kaolin clay, quartz and feldspar as a first stage, then mixed with zirconia as second stage. The experimental work includes the following stages:-

#### **3-3-1 Preparation of primary materials:**

1. Sieving process: Kaolin powder was sieved by a (98 $\mu$ m) 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, quartz, feldspar and zirconia 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.

In the first stage, the porcelain samples were prepared only. While, the samples of porcelain with zirconia were prepared in the second stage with the same method of preparation in the first stage.





(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 in the first stage						Raw materials in the second stage			
	Kaolin clay		Quartz		Feldspars		Porcelain		Zirconia	
	%	Weight (g)	%	Weight (g)	%	Weight (g)	%	Weight (g)	%	Weight (g)
A	50	2.5	25	1.25	25	1.25	100	5	0	0
B	50	2.5	25	1.25	25	1.25	95	4.75	5	0.25
C	50	2.5	25	1.25	25	1.25	90	4.5	10	0.5

### **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= 13mm). 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. Polyvinyl 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 (110°C) 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 (1200°C) with heating rate (5°C/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.

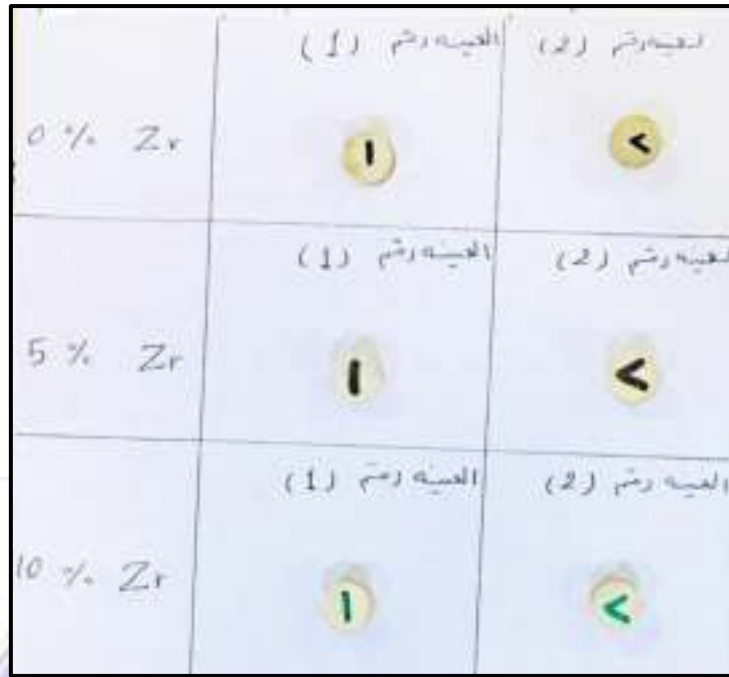


Figure (3-3) The samples after firing

### **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):

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

**b) Porosity**

Calculation of porosity for the samples was based on ASTM standard (C373-88) [14]. The percentage of porosity was calculated by the equation (2). Archimedes device was used in this test, which shown in figure (3-4 a)

$$\text{Porosity \%} = ((M-D)/(M-S)) * 100 \dots\dots\dots (2)$$

Where:

M: saturated weight (g),

D: dry weight (g),

S: Suspended weight (g).

**c) Density**

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

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

**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 [15]:

$$\sigma_c = F / A_r \dots\dots\dots (4)$$

where:  $\sigma_c$  = Fracture strength in (MPa).

F = Applied load until fracture (N).

$A_r$  = Cross section area ( $\text{mm}^2$ ).

**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 device

# Chapter Four



# *Results and Discussion*

## Chapter four Results and Discussion

### 4-1 Results and Discussion

#### 4-1-1 The physical tests

Figures (4-1), (4-2) and (4-3) show the effect of zirconia additive on the linear shrinkage, apparent porosity and bulk density of porcelain samples. Where, the porosity of samples decreases with increasing of zirconia content. While the linear shrinkage and density of the samples increases with increasing of zirconia content. Generally, the presence of the liquid phase facilitates the sintering process. This may be due to the formation of a liquid phase and due to the filling of most of the open pores with zirconia particles, in addition to the transformation of residual open porosity into closed porosity. Thus, good dense body was produced.

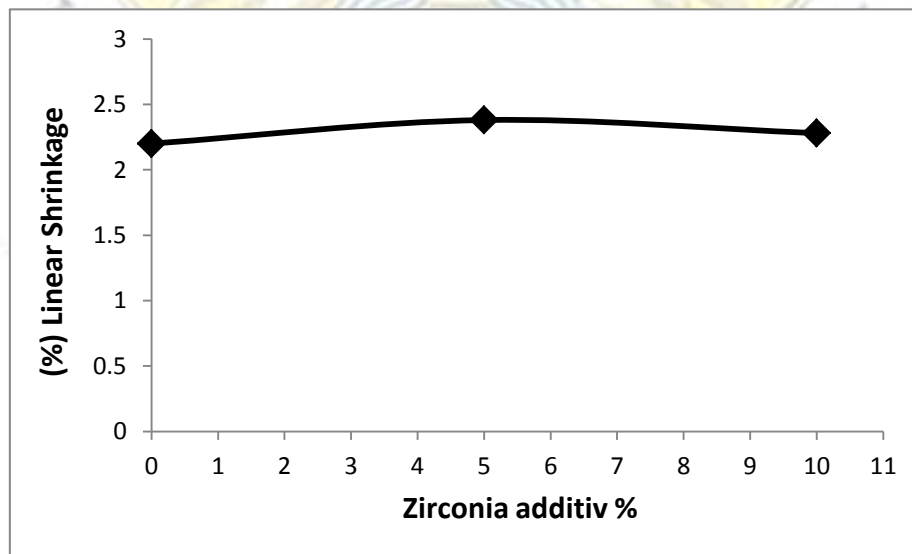


Figure (4-1) the effect of zirconia additive on the linear shrinkage of porcelain samples.

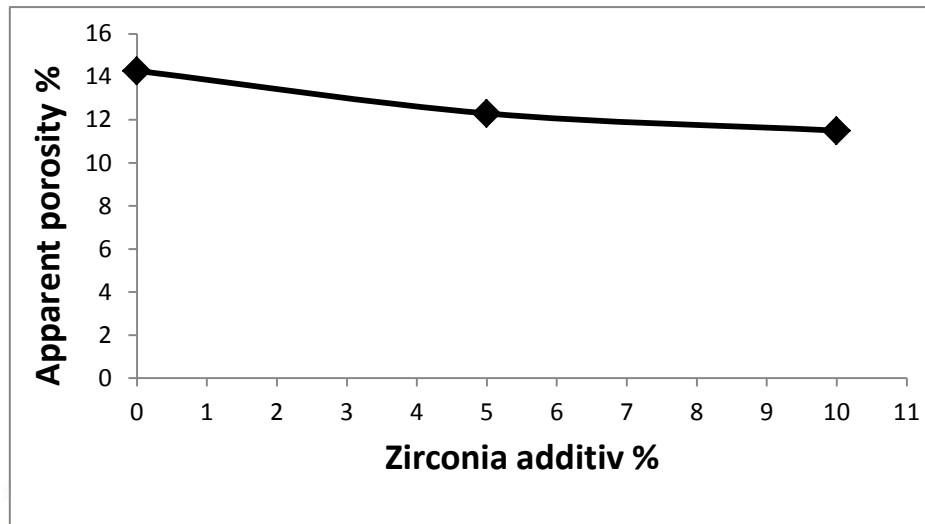


Figure (4-2) the effect of zirconia additive on the apparent porosity of porcelain samples.

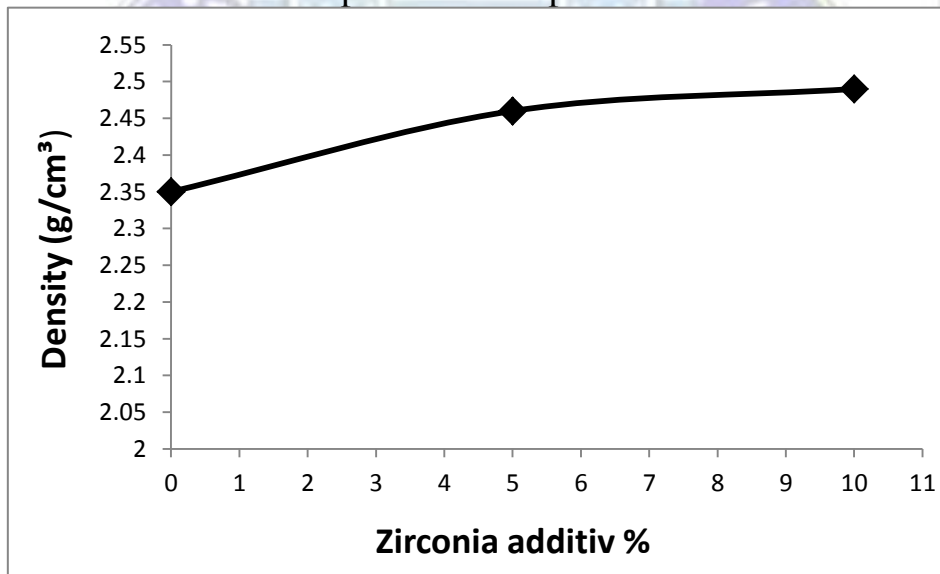


Figure (4-3) the effect of zirconia additive on the bulk density of porcelain samples.

#### **4-1-2 The mechanical tests**

Figures (4-4) and (4-5) show the effect of zirconia additive on the fracture strength and hardness of porcelain samples. Where, the fracture strength and hardness of the samples increase with increasing of zirconia content. This is because of the formation of glassy phase in the porcelain matrix for the presence of an amount of different fluxes which means the



increase in the bond between the material particles and the decrease the pores between them for this reason and due to the filling of most of the open pores with zirconia particles, that makes the network more rigid, which lead to increase in mechanical strength of the samples.

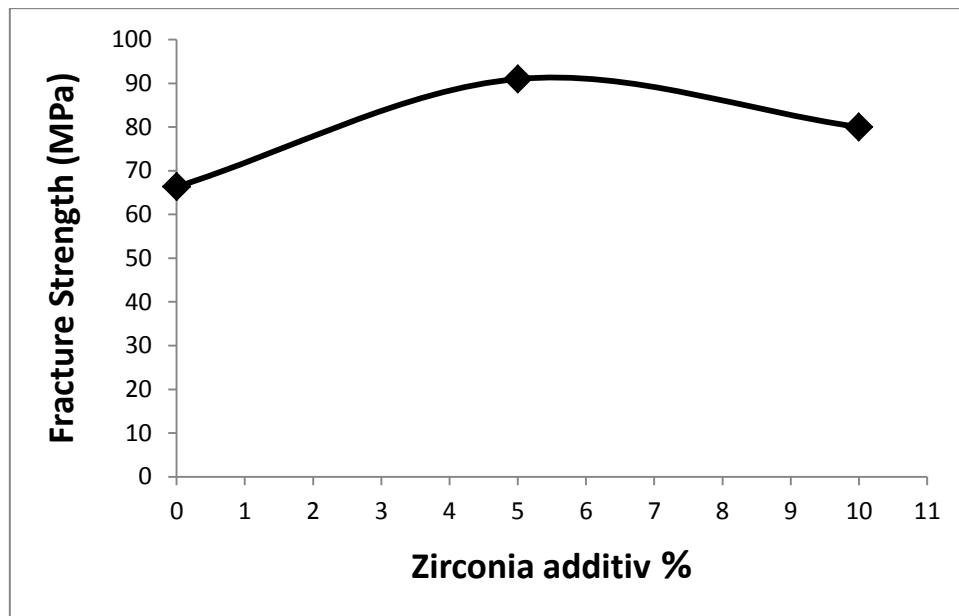


Figure (4-4) the effect of zirconia additive on the fracture strength of porcelain samples.

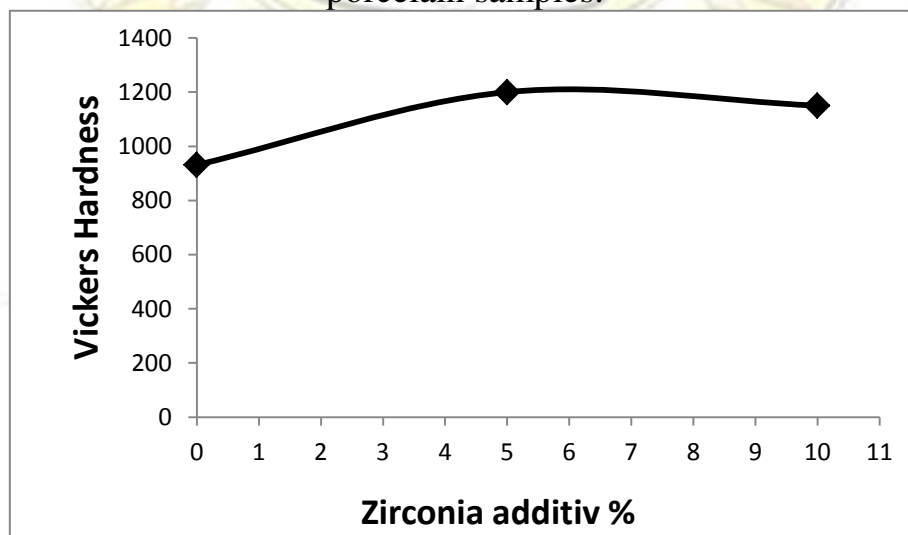


Figure (4-5) the effect of zirconia additive on the hardness of porcelain samples.

## Chapter Five



The logo of the University of Babylon is a circular emblem. It features a central blue archway with a white opening, flanked by two blue towers. The archway is set against a yellow background. The text 'جامعة بابل' (University of Babylon) is written in Arabic script across the archway. The years '1991' and '1465' are inscribed on the left and right sides of the emblem, respectively. The emblem is surrounded by a decorative border of stars.

# ***Conclusions and Recommendations***

Chapter five  
Conclusions and Recommendations

**5-1 Conclusions:-**

- 1- The linear shrinkage on firing and density of the porcelain samples increase with increasing of zirconia content. While the porosity of samples decreases with increasing of zirconia content.
- 2- The fracture strength and hardness of the porcelain samples increase with increasing of zirconia content.
- 3- The improving of some properties of porcelain by addition of zirconia to it.

**5-2 Recommendations:**

- 1- Study the effect of another material to improve the mechanical properties of porcelain products.
- 2- Study the effect of increasing of zirconia percent in the porcelain samples on its properties
- 3- Study the effect of zirconia additive on the thermal properties of porcelain.
- 4- Study the effect of zirconia additive on the properties of other ceramic products.

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