Ministry of Higher Education and Scientific

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Department of Ceramic and Building Materials

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Effect of bioactive glass ceramic on mechanical behavior of hydroxyapatite

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وزارة التعليم العالي والبحث العلمي

جامعة بابل

كلية هندسة المواد

قسم هندسة السيراميك ومواد البناء

مشروع تخرج مقدم الى قسم هندسة السيراميك ومواد البناء في كلية هندسة المواد - جامعة بابل كجزء من متطلبات نيل درجة البكالوريوس في علوم هندسة المواد - السيراميك

تأثير السيراميك الزجاجي النشط بيولوجيًا على السلوك الميكانيكي لهيدروكسيباتيت

من قبل

هدى عبد الكريم عطية

إشراف

أ.د محسن عباس اسود

بسم الله الرحمن الرحيم

(وَالرَّاسِخُونَ فِي الْعِلْمِ يَقُولُونَ آمَنَّا بِهِ كُلٌّ مِنْ عِنْدِ رَبِّنَا)

صدق الله العلي العظيم

سورة آل عمران – الآية 7.

Dedication

To the one who created me and dressed me..... to whom

With his kindness and generosity, he revived me..... To whom?

Guide me and guide me..... to whom By remembrance of Him, souls will be refreshed, and hearts will be reassured_my Lord_

To you, my supporter in this life..... To you, O crown of time and all tenderness.... To you, who planted

I have ambition... that pushes me towards the future

Here's to you, the sweetest word that my tongue uttered....

_ My beloved father _

To my beautiful angel and my unfailing love....the symbol of tenderness and tenderness....to the spring of my life and the queen of my heart...

_ Dear mother_

To the most beautiful planets in my sky..... To the dearest thing in my life.

My brothers and friends

To everyone who taught me a letter and illuminated the path of the future before me..... To those with whom I went insane and to whom I belonged... To our dear teachers, I dedicate to you the fruit of what your hands have made.Our dear teachers

الاهداء

الى من خلقني فسواني .....الى من

بلطفه وكرمه احياني .....الى من

ارشدني وهداني .....الى من

بذكره تطيب النفوس ..... وتطمئن القلوب

ربي

اليك ياسندي في هذه الحياة.....اليك يا تاج الزمان وكل الحنان..... اليك يامن زرعت

في طموحاً ..... صار يدفعني نحو المستقبل

اليك يا اعذب كلمه نطق بها لساني.....

ابي الحبيب

الى ملاكي الجميل وحبي الذي لا يزول .... رمز الحنان والعطاء ..... الى ربيع حياتي وملكه قلبي .....كل الحب والوفاء الى شمسي المشرقة وبدري المنير ..... اجمل عطايا الرحمن

_ امي الحبيبة

الى اجمل كواكب في سمائي ..... الى اعز ما في حياتي .

_ اخوتي و اصدقائي _

الى كل من علمني حرفاً وانار امامي طريق المستقبل ..... الى من بهم جننت واليهم انتميت .... الى اساتذتنا الاعزاء اهدي لكم ثمرة ما صنعت ايديكم..

اساتذتنا الأعزاء

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First of all, I thank Allah who gave me the ability and desire to complete this work.

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for his endless supported, guidance and advice. His encouragement helped me to go on and complete this research. I was lucky to have such a supportive supervisor.

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Finally, I would like to thanks my family; my friends and everyone helped me to accomplish the present work.

الخلاصة

يعتبر السيراميك الزجاجي النشط بيولوجيًا ذا فائدة لاستخدامه كمواد بديلة لأنسجة العظام نظرًا لتوافقه والنشاط الحيوي ؛ بالإضافة إلى القدرة على تكوين طبقة هيدروكسيباتيت متبلورة. تشبه هذه الطبقة في التركيب والهيكل المكون غير العضوي لمرحلة العظام المعدنية. في هذه الدراسة ، تم إنتاج السيراميك الزجاجي الحيوي باستخدام تقنية الصهر التي اشتملت على خلط 45 SiO2 و 24.5 CaO و 24.5 Na2O و 6 P2O5)بالوزن٪) في مطاحن الكرات الكوكبية لمدة 4 ساعات تقريبًا. بعد ذلك ، صُهرت المساحيق بعد ذلك في بوتقة الألومينا عند 1200 درجة مئوية لمدة ساعتين بمعدل تسخين 10 درجات مئوية / دقيقة. ثم تركوا في الفرن للتبريد البطيء لدرجة حرارة الغرفة.

Abstract

Bioactive glass-ceramic is of interest for using as a replacement material for bone tissue due to its compatibility, bioactivity; in addition to the ability of formation a crystallized hydroxyapatite layer. This layer is similar in composition and structure to the inorganic component of the bone mineral phase. In this study, the bioglass-ceramic was produced using a melting technique that included mixing of 45 SiO2, 24.5 CaO, 24.5 Na2O, and 6 P2O5 (wt%) in a planetary balls mills for about 4 hours. Next, the powders were then melted in an alumina crucible at 1200°C for 2 h at a heating rate of 10°C/min. Then, they left in the furnace for slow cooling to room temperature.

Chapter One

Chapter one

Introduction

Many substances that were recognized as suitable for use in human bodies are named biomaterial. The biomaterial can be defined as materials that are utilized for replacing or restoring functions to tissues of bodies and are constantly in contact with the fluid in the body. Certain substantial features materials should possess to be utilized in the bodies. It should have a biocompatible chemical composition that does not induce adverse tissue reactions. In other words, the material must be non-carcinogenic, nontoxic, not inflammatory, and non-allergic. The material should be bio-functional, with enough strength and wear resistance to withstand the environment in which it will be implanted. Relying on the applications for which the materials are utilized, might also require stabilization over a long period, has a certain degradations rate, or encourage bone ingrowths [1].

Bioactive ceramic is an expression involving bioactive glass, glass - ceramic, and calcium phosphate (hydroxyapatite). The oldest bioactive glass is 45S5 which was invented by Larry Hench in the late 1960s, to fill bone defects that the body would not be rejected. It is chemically bond with bones and has been used mainly as reconstructive materials for damaged hard tissue of bones [2]. Because the mechanical properties of bioactive glass are low compared to bioactive glass-ceramic, glass-ceramic was used as a replacement material for bone repair. It is partially crystalline materials that contain at least one or more

crystal phases embedded into a residual glass. Their crystallinity differs somewhere in the range of 0.5% and 99.5%, most much of the time somewhere in the range of 30% and 70%, and shows good biocompatibility and bioactivity[3].

The central point of limiting the use of bioactive glass-ceramics is their low mechanical strength and fracture toughness. One approach to using

ceramics as implants in load-bearing applications is reinforcing the ceramic with a second phase[4]. Bioglass-ceramic mechanical feature is managed by adding oxides such as magnesia, alumina, zirconia, or titani

.Failure of engineering materials is one of the most important research studies of all-time due to the cost of undesirable damages and accidents that are resulted from it. Engineers are often required to understand the causes of failures and try to minimize the probability of failures in the designed components. Fracture mechanics is one of the engineering science subjects that deal with the failure of solids resulting from initiation and propagation of crack [6], cracks are everywhere which often exist from industrial flaws or a variety of environmental conditions during loading. Crack opening displacement and crack propagation are the most important parameters in fracture mechanics[7].

The sample dimensions of brittle material to be tested are excessively minor to enable extensometers or electromechanical to be used for determining the displacement of the opening crack [8]. Measuring the crack opening

displacement is difficult and as values obtain smaller, distinct equipment is needed and for a ceramic, SEM, and AFM have been utilized. These methods require precise sample preparation and distinctive care to determine accurate values of displacement [9][10]. To overcome these obstacles, a process for measuring the displacement of mini sample and accurate strain measurements can be achieved by non-contact optical method, it's called Digital Image Correlation (DIC) which can be used to determine the displacement and strain of sample to detect crack propagation and measurement crack opening displacement[11].

Chapter Two

Chapter Two

Theoretical part and literature review

This chapter explains the definition of bioceramics materials, their properties, and applications, an overview of fracture mechanics, the technique used in fracture mechanics for identified crack propagation and opening, and the last section consist of a literature review related to the subject of the thesis.

2.2 Bioceramic Materials

Bioceramic is a class of advanced ceramic that is defined as a ceramics product or component utilized in a dental and medical application [13]. Bioceramic is manufactured in various phases and shapes and provides functions in protecting objects. For several purposes, used ceramics in the form of a bulk material with special shapes named implants or prostheses or prosthetic devices. In addition, bioceramic is employed for filling spaces although the natural repair method restores functions. In other situations, Ceramics are used also as a substrate coating or as second stages in compounds and bind the features of each in novel materials using advanced biochemical and mechanical features [4].

2.3 Bioceramic Classification

Bioceramics are grouped as shown in figure (2.1) according to the living host response:

2.3.1 Bioinert Ceramics

Bioinert ceramics are those experience very little chemical changes when attacked by physiological environments. They preserve their properties (both mechanical and physical) while being in the host. The host forms a very thin fibrous tissue (in several micrometers or less) that encapsulates the implanted material. Bioinert ceramics are fixed in the body via mechanically powerful

interlock, by growing the tissue within the surface. The most common examples are Alumina, Zirconia, Alumina-Zirconia, and Carbon [14]. 2.3.2 Biodegradable or Resorbable Ceramics

Refers to materials that degrade (by hydrolytic breakdown) in the body while they are being replaced by regenerating natural tissue; the chemical by- products of the degrading materials are absorbed and released via metabolic processes of the body [15]. These materials are perfect implants because they stay in the body during their function and disappear after regeneration, and the result is a very thin interface. The drawback of these implants is that their mechanical strength is reduced through the whole time of the re-absorption process [14].

2.3.3 Bioactive or Surface Reactive Ceramics

It refers to substances that form bonds with living tissues [16]. These materials- other than hydroxyapatite that bonds to the bone directly- bond to the bones by a biologically active layer called (carbon-hydroxyapatite layer). This layer is structurally, and chemically similar to the bone minerals phases and it is responsible for interfacial association [17].

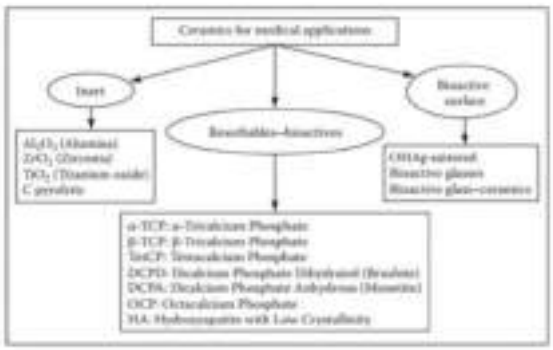


Figure (2.1): Classification of Bioceramic [18]

2.4 Bioactive Glasses and Glass-Ceramics

Glass and glasses-ceramics are bioactive materials, which directly attach to bone tissue by the carbohydroxyapatite (CHA) layer which is a biologically active layer and provides an interfacial bonding. This layer is similar to the minerals phase of bones chemically and structurally. Bioactive glasses and glass-ceramics have been developed to respond to the need to suppress interfacial mobility in embedded bioinert ceramics. In 1967, a new material was developed that binds with bone called Bioglass, which is used to repair strong, weakened bone tissue and conduct research to modify the chemical composition of glasses to enable them to communicate with the physiological system and to provide chemical bonding between living tissue and the surface of the implant [19].

2.4.1 45S5 Bioactive Glass

It is a soda-lime–phosphate–silicate glass that was prepared by Hench et al and embedded in rats femurs, the implants bonded to the living bone and it was difficult to remove it from their site. This glass composition contains 45% SiO_2 by weight, with the presence of 24.5% Na_2O and 24.5% CaO as network modifiers. The glass composition was denoted as 45S5 to signify the wt% of silica as the network former and a fivefold molar ratio of Ca/P [20].

There were three key compositional features to these glasses that distinguished them from traditional Na_2O-CaO-SiO_2 glasses: (1) less than 60 mol% SiO_2, (2) high-Na_2O and high-CaO content, and (3) high CaO/P_2O_5 ratio [21].

Figure (2.4) illustrates the Na_2O-CaO-P_2O_5-SiO_2 glasses dependence on bone tissue bonding. Region A can form a bond with the bone and term as a bioactive boundary. Region B is the silicate glasses (such as window, bottle, and glasses of the microscope) that have the behavior of nearly inert materials.

Region C is resorbable glasses that disappear within one day of implantation as maximum. Region D not realistic in a technical way

and never been tested as implanted materials. Region E adhere to the collagen part of soft tissues [22].

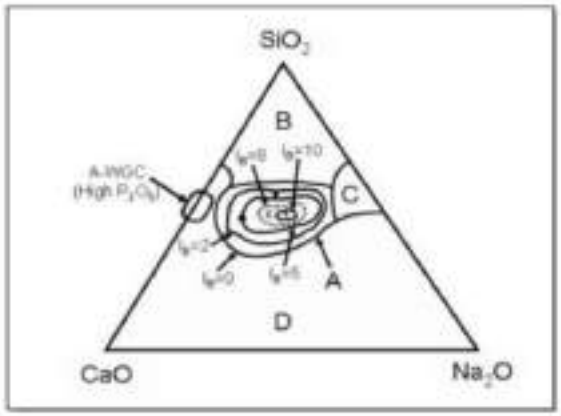


Figure (2.2): Compositional dependence of bioactive glass and glass ceramics for bone bonding and soft tissue bonding [22]

2.4.2 Structure of 45S5 Bioactive Glass

Silicate glass is an amorphous solid, it's characterized by the covalent networks [SiO4]4- tetrahedral building block, consolidated via connecting oxygen (BO) atom, and each BO shares with two Si4+ ions as shown in figure (2.3) [23].

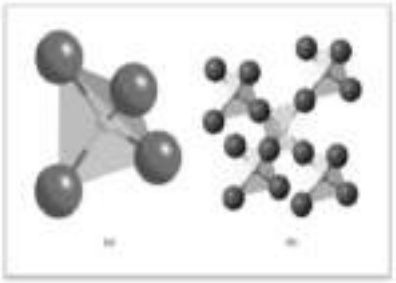


Figure (2.3): Building block of silicate glass network. (a) Single SiO_4 tetrahedron, (b) tetrahedral connected with a central tetrahedron within bridging oxygen ions [23]

Amorphous silica has a continual network, completely inter-connected in 3D, each tetrahedron joint by bridging oxygen (BOs) to four neighboring tetrahedra. Adding alkaline earth and alkali (as modifier cations) break the net system by supplying bonds of Si-NBO instead of Si-BO-Si. The work of the formers network is building bonds of (Si-O-Si), while modifiers break those bonds, and this allows the solidification of the fuse at a high level of disorder. Figure (2.4) shows the influence of Na2O in breaking the bridging oxygen. Modifiers reduce melting temperature and viscosity values. Ionic bonds between non-bridging oxygen and modifiers assurance the charge balance as well as the total charge neutrality. This interaction (ionic interaction) is critical toward the glasses stabilizing having a low level of silica (e.g. bioactive glasses) [13].

These disarranged structures lead to high reactivity in aquatic environments. Reactivity considers as an advantage for the application of bone growth and periodontal repairing, the reaction between these glasses products and physiologic fluids produce in the forming of an apatite-like structure that looks similar to the inorganic parts in the bone [24].

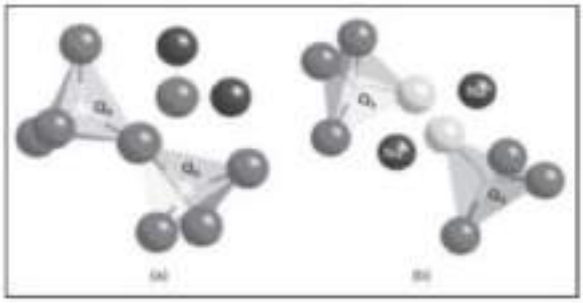


Figure (2.4): Connections in silicate glass network. (a) The bridging oxygen between two tetrahedral, (b) Non-bridging oxygen formed by addition Na_2O [24]

2.4.3 Bioactive Glass-Ceramics

Bioactive glass-ceramics are partially crystalline materials that contain one or more than one crystal phase in addition to a residuals glass [25]. They are a group of predestined osteoconductive biomaterials for medical applications, especially orthopedic and dental implants, because of their excellent bioactivity and biocompatibility [26].

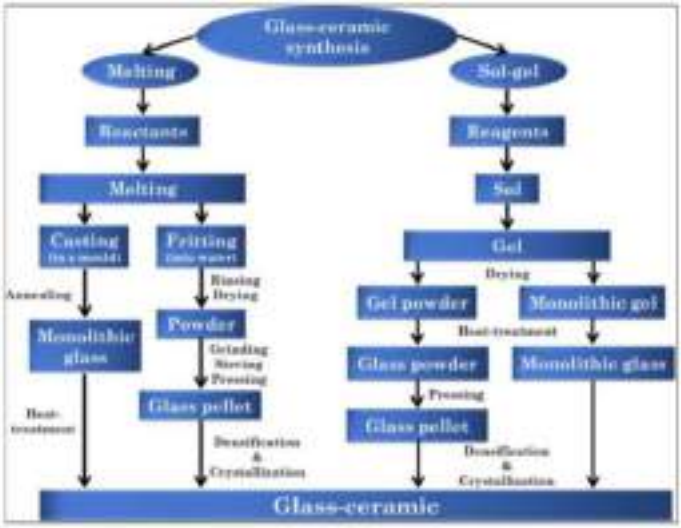
Bioactive glass is one that, at the interface of the material, induces a particular biological reaction that promotes cell proliferation, gene reaction, and the formation of bonds between the livings tissues, and the materials. A commons feature of bioactive ceramic glass is that a coating of biologicals actives hydroxycarbonates apatites (HCA) binding to the bones is formed on its surface. The apatite stage of hydroxycarbonate that forms on bioactive glass- ceramic is chemicals and structurally, equivalents to the bones minerals process [25].

2.4.4 Bioactive Glass-Ceramic Fabrication Methods

They are generally produced by two methods: melting and sol–gel followed by some heat treatment as displayed in Figure (2.5). Bioglass -ceramics fabricated by the sol-gel method, which provide several advantages such as a wider range of compositions, high purity and homogeneity, and low processing temperature[27]. The typical precursors are tetraethylortho silicate (TEOS), triethylphosphate, and calcium nitrate [28]. The precursor undergoes a hydrolysis process to form a sol. After polycondensation process, a silicate network is created. During the gel formation; the viscosity raises and network connectivity increases. The gel is then dried at 600 oC-800 oC [69]. Finally, the gel is densified at a temperature in the range of 900 oC -1300 oC, which leads to the development of glass-ceramic [29].

An alternative technique to fabricate bioglass-ceramics is the melting method. Melted and formulated using methods similar to traditional soda-lime glasses are bioactive glasses obtained from the melt. However, when the necessary

amounts of oxides, carbonates, or phosphates are homogenized and then melts at 1350-1450°C in a platinum crucible, the processing requirements must comply with the standards, for products used in medicals applications [28]. To produce solid glasses, the molten glasses are either poured into graphite molds or quenched in waters or oils, resulting in fried glasses. Subsequent mechanical grindings (in planetary mills) can be used to obtain powdered glasses that can be used directly as bone defects fillers materials. Meltings is a flexibles technique that enables various glass formulations to be created simply by changing the raw material number and proportion ratio [30]. Moreover, During the sintering process, the regular 45S5 bioglass obtained by the melting process tends to crystallize, forming a predominantly crystalline phase (Na2Ca2Si3O9)[31].



Figure(2.5): Schematic diagrams of the main stage in the syntheses of bioglass-ceramic powder [29]

Chapter Three

Chapter three

practical part :-

Tools and materials used:

1- Hydroxy apatite powder

2- A cylindrical mold with dimensions D=13 mm in diameter

3-piston

4-drying oven

5-burning oven

6-sensitive scale

7- A glass flask

8-distilled water

9- Specific density scale

The method of work ::-

(Prepare three samples of pure hydroxyapatite without any additives through the following steps)

1- We take (600) grams of hydroxyapatite powder

2- We weigh (300) grams for each sample, then put drops of (PVA) on it and mix it

Mix well

3- We put the mixed powder in the pressing mold, then apply a pressure of (20mpa).

4- After extracting the sample from the pressing mold, its dimensions are measured

5- We dry the three samples at (100c) for (24h) in a drying oven

6- We measure the dry weight of the three samples

7- We put the dried samples in the burning oven at a high temperature... in order for them to form and acquire prayer after the shaping process

8- Conducting laboratory tests such as points

* Microstructure

Density check

9- We get acquainted with the microscopic structure of the material, by grinding it from the smallest paper to the largest, and then we perform a polishing process for the samples

10- Immerse the samples in a pot of distilled water and boil for a period of (5h). We pay attention to the samples until they are covered with water all the time. After (5h) of boiling, we allow the samples to drink or soak for an additional (24h) after turning off the heat. The pot is closed with a lid.

11- We measure the wet weight of the samples

12- The glass beaker is filled with distilled water, then we put the samples on the suspended clamp after ensuring that the samples are completely immersed in water, and the weight is taken and it is called the suspended weight, and it is approximately equal to half of the saturated weight

13- We extract the samples from the water and weigh them in the specific gravity balance after wiping them with a damp cloth. The weight is taken after placing it in the upper part of the scale, and it is called the saturated weight.

Prepare three samples

of hydroxyapatite powder at a rate of 2.4 + bioactive glass ceramics at a rate of 0.6) according to the following steps-

1- Weighs 2.4 of hydroxyapatite with 0.6 of bioactive glass ceramics

2- We mix the hydroxyapatite with the glass ceramics by adding a few drops of (PVA) solution.

3- We put the mixture in a cylindrical mold with a dimension of D = 13 mm, then press it using a cylindrical piston with a stress of (17 MPa).

4- The compressed sample is extracted and sintered at 1200C for a

period of (24h).

5- We dry the three samples at (100c) for (24h) in a drying oven

6- We measure the dry weight of the three samples

7- We put the dried samples in the burning oven at a high temperature... in order for them to form and acquire prayer after the shaping process

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11- We measure the wet weight of the samples

12- The glass beaker is filled with distilled water, then we put the samples on the suspended clamp after ensuring that the samples are completely immersed in water, and the weight is taken and it is called the suspended weight, and it is approximately equal to half of the saturated weight

13- We extract the samples from the water and weigh them in the specific gravity balance after wiping them with a damp cloth. The weight is taken after placing it in the upper part of the scale, and it is called the saturated weight.

Prepare three samples of hydroxyapatite powder at a rate of 1.8 + bioactive glass ceramics at a rate of 0.2) according to the following steps-

1- Weighs 1.8 of hydroxyapatite with 0.2 of bioactive glass ceramics

2- We mix the hydroxyapatite with the glass ceramics by adding a few drops of (PVA) solution.

3- We put the mixture in a cylindrical mold with a dimension of D = 13 mm, then press it using a cylindrical piston with a stress of (17 MPa).

4- The compressed sample is extracted and sintered at 1200C for a period of (24h).

5- We dry the three samples at (100c) for (24h) in a drying oven

6- We measure the dry weight of the three samples

7- We put the dried samples in the burning oven at a high temperature... in order for them to form and acquire prayer after the shaping process

8- Conducting laboratory tests such as points

* MicrostructureDensity check

9- We get acquainted with the microscopic structure of the material, by grinding it from the smallest paper to the largest, and then we perform a polishing process for the samples

10- Immerse the samples in a pot of distilled water and boil for a period of (5h). We pay attention to the samples until they are covered with water all the time. After (5h) of boiling, we allow the samples to drink or soak for an additional (24h) after turning off the heat. The pot is closed with a lid.

11- We measure the wet weight of the samples

12- The glass beaker is filled with distilled water, then we put the samples on the suspended clamp after ensuring that the samples are completely immersed in water, and the weight is taken and it is called the suspended weight, and it is approximately equal to half of the saturated weight

13- We extract the samples from the water and weigh them in the specific gravity balance after wiping them with a damp cloth. The weight is taken after placing it in the upper part of the scale, and it is called the saturated weight.


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Chapter Four

Chapter Four Results and Discussion

The results of the experimental tests are their explanation presented in this chapter.

4.1 Powder Characterization

4.1.1 Particle Size Analysis

Figure (4.1) illustrates the particle size distribution as a histogram and as an accumulative curve of bioactive glass-ceramic powder. The bioactive glass- ceramic had a particle size distribution in the average of 0.1 to 75μm. The d10, d50, and d90 of the powders are 0.794, 3.288 and 4.760, respectively. It shows that the accumulative curve is not smooth. This can be attributed to the existence of agglomerates with different sizes although the milling

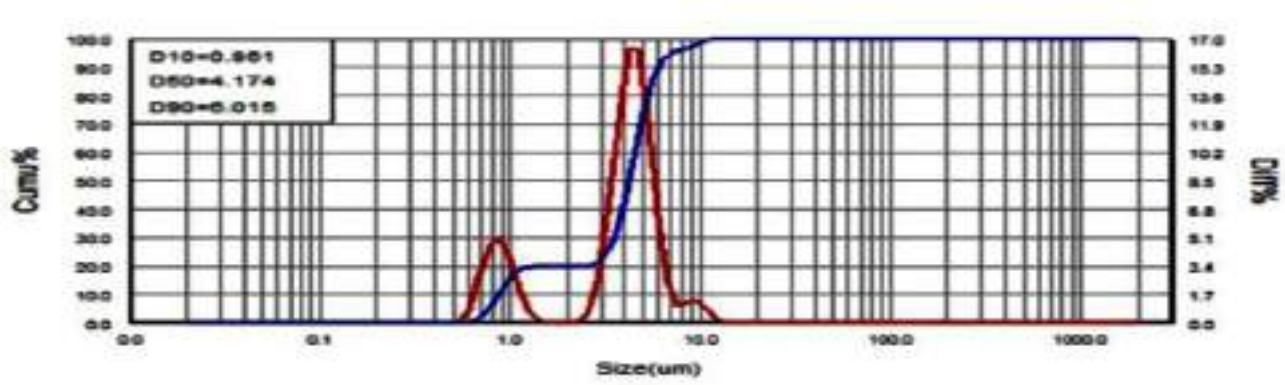
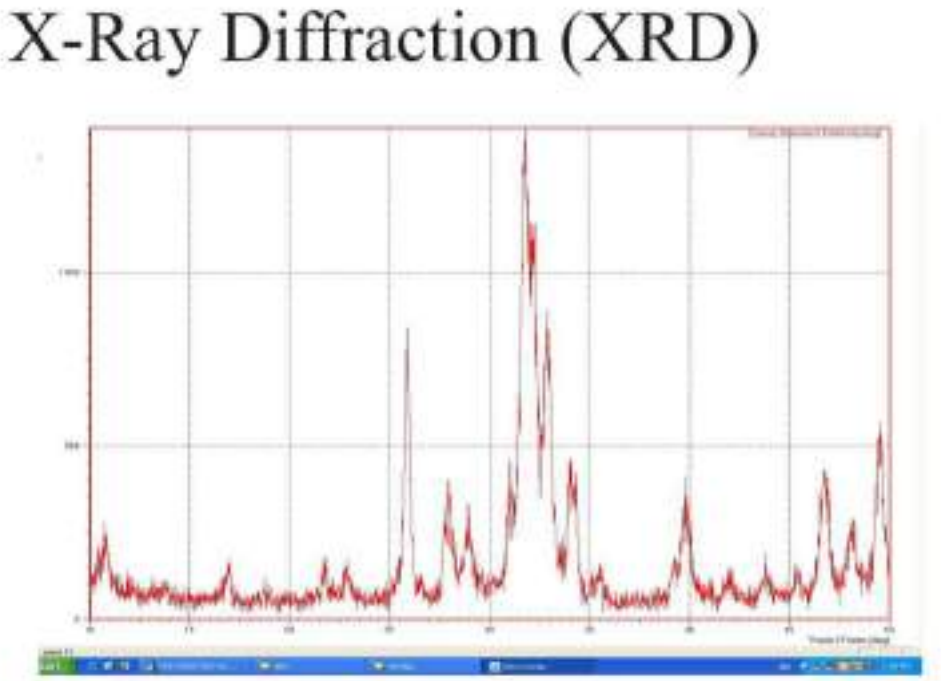


Figure (4.1): Particle size distribution of bioactive glass-ceramic powder



4.1.2 X-Ray Diffraction

The phases present in the materials were identified using the XRD technique, Figure (4.2) represent the XRD patterns of bioglass-ceramic powder at 1200oC with a heating rate of 10 oC /min, and (2h) as a soaking time of, the obtained three types of sodium-calcium silicate phase (Na2Ca2Si3O9), 50

(Na2CaSi3O8) and (Na6Ca3Si6O18) that scanned in a diffraction angle ranging from 10o to 60o and agree with (JCPDS, card NO. 22-1455), (JCPDS, card NO. 12-0671) and (JCPDS, card NO. 77-2189) respectively.

Figure (4.2): XRD pattern of bioglass-ceramic powder melted at 1200oC

Chapter Five

physical characterization of the samples :

absorbance	apparent porosity %	density (g/cm3)	sample
8.9	0.2	2.22	A1
4.5	0.6	3.14	A2
5.6	0.73	2.28	A3

absorbance	apparent porosity %	density (g/cm3)	sample
8.3535	0.2023	2.4226	A1
9.1280	0.23572	2.5842	A2
29.8912	0.46025	1.53975	A3

absorbance	apparent porosity %	density (g/cm3)	sample
2.8662	0.076625	2.673375	B1
12.1663	0.18077	1.4858	B2
25.9912	0.18077	1.5874	B3

Mechanical characterization of the Sample:

sample	hardness
A1	33.33
A2	53.82
A3	53.52

sample	hardness
B1	58.12
B2	73.70
B3	72.84

Conclusion

Praise be to God Almighty who helped us present this research, and here are the last drops in the course of this research. The research was talking about (the effect of biologically active glass ceramics on the mechanical behavior of hydroxyapatite), and we have made every effort and effort to produce this research in this form.

الخاتمة

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