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Preparation of Bioactive Glass Ceramic by using Sol – gel method

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ABSTRACT

Bioactive glass ceramic with quaternary system of $\text{SiO}_2\text{-Ca}_2\text{O-Na}_2\text{O-P}_2\text{O}_5$ composition" was prepared by "sol-gel method using" aquatic "sodium metasilicate" ($\text{Na}_2\text{SiO}_3\cdot 5\text{H}_2\text{O}$) as a silica "source". "Monolith was gotten after sintering temperature at 900°C , for 1 hour, as on the resulting gel was aged at room temperature for 5 days, dried at 60°C for 72 hours to give the monolith phase. The monolith was further heat treated as follows ; 200°C (40 hours) to evaporating water and ethanol from the monolith , 600°C (5 hours) to preamble monolith to access the begging formation of glass ceramic, 800°C (3 hours) to complete the phase of glass ceramic and 900°C (1 hour)the sintering degree to obtain the final phase of glass ceramic ($\text{Na}_2\text{Ca}_2\text{Si}_3\text{O}_9$) .The X-ray diffraction analysis presented the occurrence of the combeite ($\text{Na}_2\text{Ca}_2\text{Si}_3\text{O}_9$) as a crystalline phase". Fourier "Transformation" InfraRed (FTIR) spectroscopy facilitated a confirmation of the hydroxyapatite (HA) "foundation.

The topography of the surface and grain size measurement was obtained using Atomic Force Microscope (AFM). In the present study, the "large number of a benefits such as low dispensation temperature, the higher cleanliness and similarity were obtained, therefore, the better controller of a bioactivity process". Thus," the obtained monolith solid could be the good candidate in tissue engineering scaffolds applications".

1. Introduction

The "bioactive ceramic glasses are the groups of biomaterials that are used in the field of orthopedics and dentistry", since in the last decades ago. These glasses modified a jobs and abilities of a biomaterials from bioinert to the bioactive by motivating the strong response after planting in a human body [1]."

The" materials "can be categorized a bioactive if the above-mentioned biological response results in formation of the strong chemical bonds between an entrenched material and the soft or hard tissue" [2]. "Silica-based the glasses with their interesting characteristics have been discovered for using such substitute a second generation for both bone defects and soft tissue action biomaterials due to as their "biocompatibility" [3,4].

Just as "dissolution products from the bioactive glass up regulate expression of genes that control osteogenesis [5,6]. which explains their higher rate of bone formation in comparison with the other

inorganic ceramics such as hydroxyapatite [7]."

When the "glass contains more than 60% SiO_2 , a bonding to the tissues is no longer observed [8]. Additional and on the other hand, it is expected that bioactivity increases depending on increasing with amount" of CaO "in the composition, because that the chemical bond formation depend generally on the dissolution calcium ion from the glass [9].

The melt method is the conventional method to produce the bioactive glasses from blends of compounds such as phosphates and carbonates at high thermal treatment before the quenching to synthesis glass". Presence Na_2O or K_2O suggest reducing melting temperature [10]. Host tissue-material interaction generally depending on solubility materials in aqueous media [6,11-14].

Furthermore ,it is envisioned that inclusion of sodium oxide (Na_2O) will contribute in crystallizing of certain sodium calcium silicate phases, such as Ca_2

Na₂Si₃O₉ [15], which ultimately will improve their mechanical properties. This approach however, often leads to heterogeneous materials with incipient crystallization and some degree of possible contamination from chemicals used. Style though, often mains to chemically varied compounds with emerging crystallization and approximately degree" of the likely pollution from "chemicals that using, through the cooling or grinding procedures" [16]. Hench in the Florida University introduced the first bioactive glass" in 1969.

Those days, the available implant materials (metals and polymers) designed to be bioinert had a problem; They initiated fiberboard encapsulation after establishment, rather than creating a constant bond with the tissues. Hench work began to overcome this problem by obtaining the materials that could tie the bone and live exacting environment of human body [17].

Hench tried making the degradable of glass in composite (Na₂O-CaO-SiO₂-P₂O₅) system with high content of calcium [17]. A viable another is a sol gel process with numerous advantages rather than the dissolve and hardening path presentation higher homogeneity and the purity, and an extensive range of structures [18,19].

"Synthesis of sodium" containing the quaternary system (SiO₂-CaO-Na₂O-P₂O₅) bioactive glasses are healthy documented [16,20] founded on alkoxysilanes, likewise tetramethyl" orthosilicate (TMOS) and tetraethyl orthosilicate (TEOS) as the source. However, alkoxides are hazardous and expensive [21-24] and are therefore not suitable for large-scale production."

"In this work, our objective is to prepare sodium containing quaternary bioactive glass-ceramic material using aquatic sodium metasilicate as cheap silica source".

2. Materials and Experimental Procedures

2.1. "Materials"

"The chemicals used for synthesizing of bioactive glass are: aquatic sodium metasilicate", "Na₂SiO₃·5H₂O with composition (24.9) SiO₂, (20.9) Na₂O and (54.2) wt.% H₂O" (obtained locally), "nitric acid (Fluke, Germany), phosphorus pentoxide (BDH Laboratory, 98%) and calcium nitrate tetrahydrate (LOBA, 98%)".

2.2. "Experimental Procedures"

"Bioactive glass having the composition (mol%); 50SiO₂:25Na₂O: 21CaO:4P₂O₅ was synthesized

through the sol-gel process from aquatic sodium metasilicate, "Na₂SiO₃·5H₂O according to the following procedure". The Na₂SiO₃·5H₂O (7.5g) "was stirred in a beaker in deionized water (22.5 ml) followed by addition of ethanol (3.75 ml)" to "give a clear solution.

In concentration(2M) of HNO₃ as drop wise was added to the mixture, stirring process was "continued further for 1 hour to allow complete hydrolysis. P₂O₅ (0.75 g) and Ca (NO₃)₂·4H₂O (6.15 g) were added in sequence under constant" stirring.

Each reagent was allowed 45 minutes to react before adding the next reagent, finally" the "mixture was stirred for 1 hour after the last addition. The" resulting "gel was aged at room temperature for 5 days, dried at 60°C for 72 hours to give the monolith phase. The monolith "was further heat treated as follows; 200°C (40 hours) to evaporating water and ethanol from the monolith, 600°C (5 hours) to preamble monolith to access the begging formation of glass ceramic, 800°C (3 hours) to complete the phase diagram and 900°C" (1 hour) the sintering degree to obtain the final phase of glass ceramic (Na₂Ca₂Si₃O₉).

2.3. Characterization: "The sintered specimen was analyzed by X-ray diffraction (XRD, Mini Diffractometer MD-10) using Cuka (1.54184Å) radiation source operated at 40 kV and 40 mA. The diffraction patterns were obtained in the 2θ" range from (20° - 60°). Surface topography of specimen was assessed by Atomic Force Microscope AFM (SPM -AA3000). "Fourier transform infrared (FTIR, SHIMADSU model-8400S) spectroscopy employing KBr. potassium bromide. pellets operating in the reflectance mode with a 4 cm⁻¹ resolution was used to monitor the types of bonds present in the glass" network.

3. Result and discussion

3.1. "XRD Analysis": The "XRD pattern of the final phase of glass ceramic (Na₂Ca₂Si₃O₉) is "shown in Fig.1.

Both angular location and intensity of the peaks" have a good matching "to the standard PDF 96-900-7718 which indicating to "formation of the crystalline phase of Na₂Ca₂Si₃O₉. Similar phase has been previously identified" [26,27]. The "extensive densification and fine crystallinity of Na₂Ca₂Si₃O₉ confers the good mechanical property" of scaffold (burnished) so that the resulting monolith became ready to bioactivity testing [28].

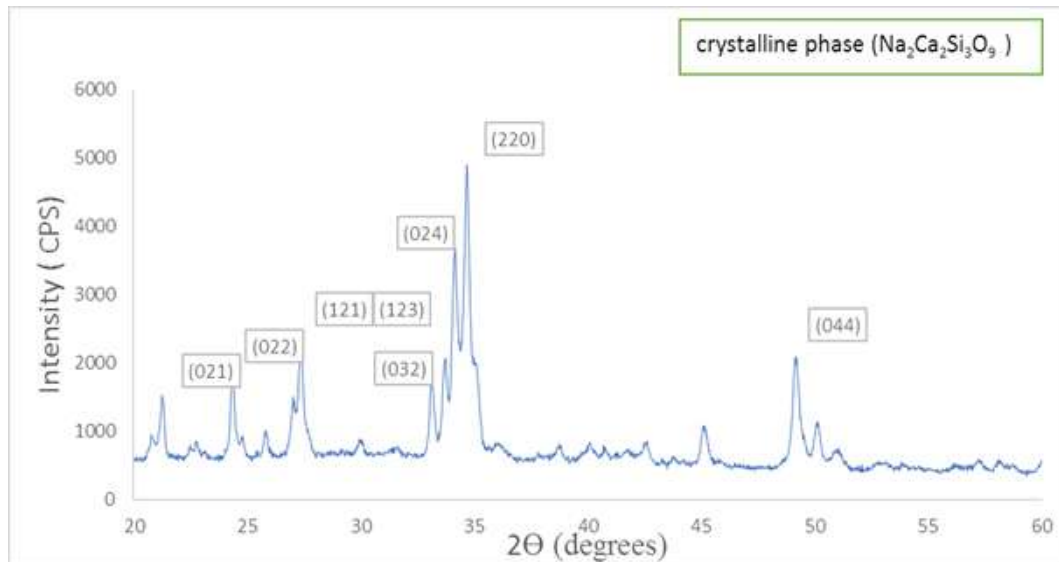


Figure (1) XRD Analysis for bioactive glass sintered at 900°C.

3.2. FTIR Analysis"

The FTIR spectroscopy analysis of bioactive sample prepared by Sol-gel method is shown in Fig.2., the peak at 748.38 cm^{-1} is as-signed to the symmetric Si-O-Si stretching vibration in 44SiO_2 , typical of crystalline silicates. The 478.35 cm^{-1} peak is assigned to Si-O-Si bending vibration which is attributed to normal vibration modes of Si-O in "the 44SiO_2 group "indicating the presence of unstructured silicate" [29]. The peak at 613.36 cm^{-1} "can be attributed to a crystalline phase which corroborates the XRD result thereby suggesting the presence of the crystalline

phosphates" [30]. Furthermore, "a new absorption band due to bending mode of 34PO_4 group and attributed to the formation of hydroxyapatite (HA) is seen" at 555.50 cm^{-1} "[31]". A "very broad OH absorption band from $(2500 - 3700) \text{ cm}^{-1}$ and a weak water absorbed band around" 1639.49 cm^{-1} "appeared in the spectrum. The bands at" 1438.90 and 1558.48 cm^{-1} "were considered for the absorption of carbonate group" (ν_3). "The peak around 900 to 1300 cm^{-1} were mainly attributed to the phosphate absorptions [16,32]".

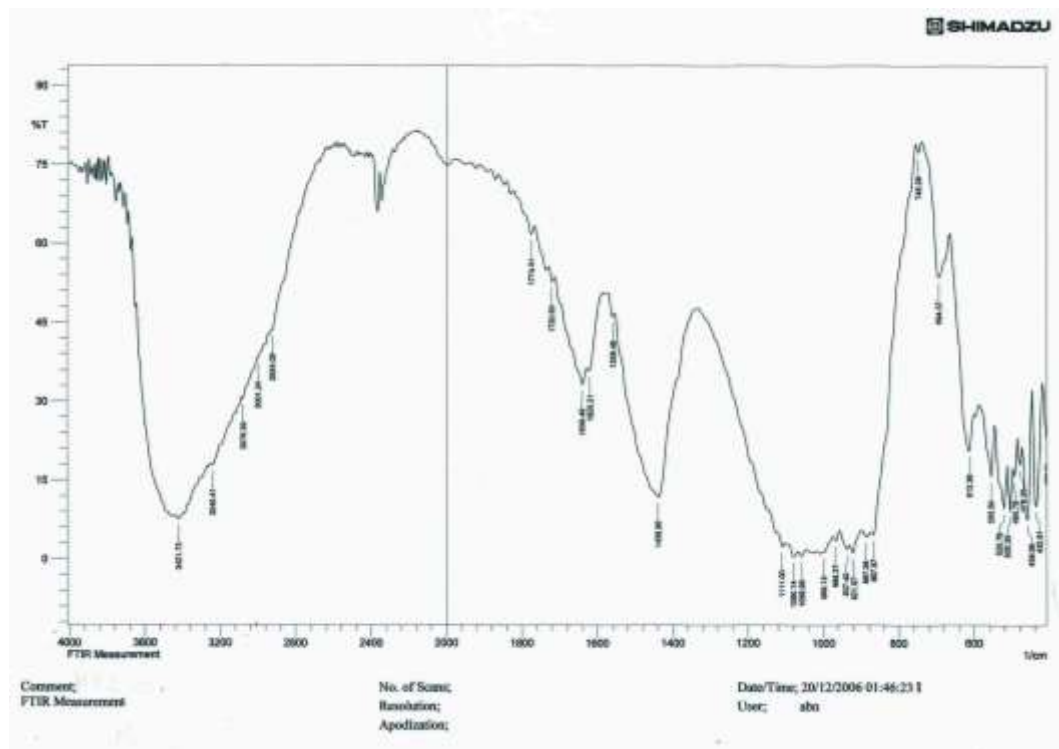


Figure (2) FTIR Analysis for glass ceramic"

3.3 "The AFM Analysis"

The AFM analysis was carried out using (SPM-AA3000) with magnification of 106X, by angstrom advance incorporation. The AFM image of glass ceramic prepared was showed in figure (3a). the granularity normal distribution report estimating the average diameter 64.94 nm. Meanwhile, the roughness

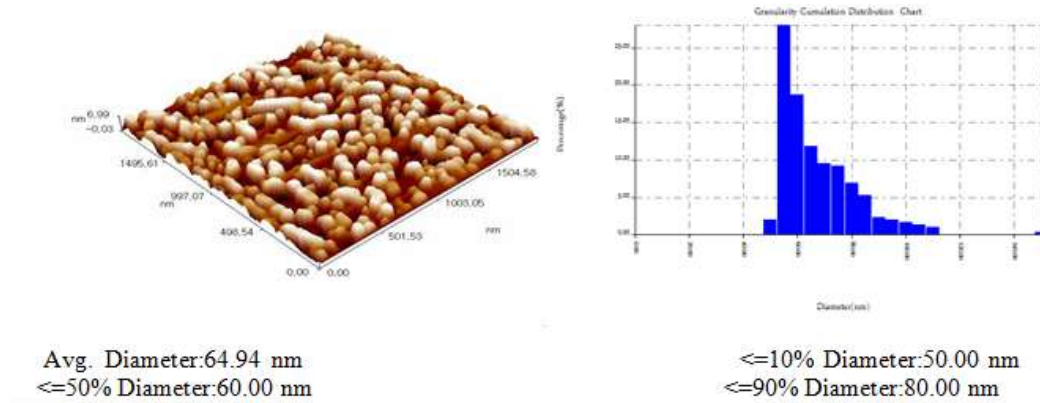


Figure (3): AFM Analysis for bioactive glass sintered at 900°C.

4. Conclusion

A bioactive material with compositions of "SiO₂-CaO-Na₂O-P₂O₅ based glass-ceramics was synthesized through sol-gel process using aquatic sodium metasilicate as a silica source". "The material was sintered at" 900°C for 1 hour " the resulting in

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average 1.51 nm, and the root mean square 1.79 nm.

These results appear that the quaternary system in glass ceramic leads to nucleation a crystal growth in different places through the sample. So that quaternary system was responsible for this statement [33].

formation of the sodium calcium silicate crystal Na₂Ca₂Si₃O₉". Therefore, low cost precursor of aquatic sodium metasilicate was used for synthesis of sodium containing quaternary bioactive glass-ceramic materials" by sol-gel technique.

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تحضير السيراميك الزجاجي الفعال حيويًا بطريقة السول - جل

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الملخص

تم تحضير مركب السيراميك الزجاجي (SiO₂-Ca₂O,Na₂O,P₂O₅) ذي الفعالية الحيوية بطريقة (سول - جل) باستخدام ميثاسليكات الصوديوم المائية كمصدر لتوليد السليكا. وقد تم الحصول على المونولث بعد المعاملة الحرارية (تليبيد) بدرجة حرارة 900 درجة مئوية ولمدة ساعة واحدة، إذ تدرج العمل وعلى شكل مراحل ابتداءً من 60 م لمدة 72 ساعة للحصول على المونولث (المادة المتصلبة الناتجة عن تجفيف الجل) بعدها مرت بدرجة 200 م لمدة 40 ساعة للتخلص من الماء والايثانول في المادة، ثم بدرجة 600 م لمدة 5 ساعات لغرض وصول المونولث الى بداية تكون السيراميك الزجاجي، ايضا مرورا بدرجة حرارة 800 م لمدة 3 ساعات لاكتمال طور السيراميك الزجاجي، واخيرا بدرجة حرارة 900 م التي تمثل درجة حرارة التليبيد للحصول على الطور النهائي للسيراميك الزجاجي Na₂Ca₂Si₃O₉، واطهرتحليل حيود الاشعة السينية بأن المركب Na₂Ca₂Si₃O₉ بالطور البلوري. وبمساعدة مطياف تحويلات فورير للاشعة تحت الحمراء تم تأكيد تشكل الهيدروكسي- بتايت (HA). بطوغرافية السطح والحجم الحبيبي لقد قيست باستخدام مجهر القوة الذرية. من فوائد الدراسة الحالية، الحصول على معالجة حرارية بدرجات واطنة، و نقاوة وتجانس عاليين. وعليه فان هذه الدراسة تفضل للسيطرة على العمليات الحيوية. وهكذا فان مادة المونولث التي تم الحصول عليها يمكن ان ترشح للاستخدام في تطبيقات هندسة الانسجة الحيوية.