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RESEARCH ARTICLE

Characterization and Investigation of Bredigite Nanostructures (BRN) Bioactivity as bio ceramic materials Using Medical Applications

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ABSTRACT

Bredigite Nanostructure (BRN) is a calcium silicate mineral with composition of $Ca_7MgSi_4O_{16}$. Lately, Bredigite Nanostructure (BRN) has been introduced as a bioceramic material due to its best bioactivity and biocompatibility. It has a good strength and toughness than those of hydroxyapatite (HA). In this project, bioactivity of Bredigite Nanostructure (BRN) powder was evaluated and investigated. For the synthesis of BRN powder, Magnesium nitrate hexahydrate (Mg $(NO_3)_2.6H_2O$), Calcium nitrate tetra hydrate (Ca $(NO_3)_2$ $.4H_2O$) powders and Tetraethyl ortho silicate liquid (TEOS) was used and using Sol-Gel process. In this part, for survey of bioactivity evaluation, the BRN synthesized powders were pressed and sintered then immersed in Kukobo solution (SBF) for 28 days at 37 °C in incubator. The results indicated that nano-struacture bredigite powder with crystalline size is 45 nm. The apatite formation ability, bioactivity and good mechanical behavior make it a good candidate in bone implant materials and open new insights in biomedical engineering.

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INTRODUCTION

The composition of hydroxyapatite (HA) is very similar to bone; its very weak mechanical behavior limits the use of these materials in load bearing applications [1, 2]. So, finding some substitutions is necessary for load bearing in medical applications. Recently, some researches indicated that some compounds from magnesia– silica and calcite (CaO-SiO_2-MgO) system are bio actively used in dental applications such as bredigite [3,4]. Bredigite (BR) is a machinable bio ceramic with chemical formula of $Ca_7MgSi_4O_{16}$ with bioactivity behavior and good biocompatibility, Fig. 1. In fact, it is as a bioceramic on both osteogenesis and angiogenesis in vitro [5-7]. The mechanical strength of bredigite is close to cortical bone. It causes the bredigite implant to be endure to mechanical pressure. This bioceramic containing silicon has shown rapid mineralization and surface apatite formation, excellent cell compatibility of osteoblaste cell proliferation and very strong osteogenetic potential in various studies [6, 8].

Therefore, the bredigite is considered a better bioceramic than HA and TCP (three calcium phosphate) for composites to make implant ceramics [9]. Moreover, nanostructured bioceramics have better bioactivity than micron-sized structure [3, 9]. The aim of this research was to study the bredigite nanostructure (BRN) bioactivity from. Calcium nitrate tetra hydrate and tetraethyl ortho silicate (TEOS) were used along with Sol-Gel process. Also, crystalline size and bioactivity behavior were investigated in using bone tissue applications.

EXPERIMENTAL

Materials and Methods

In this study, for the synthesis of bredigite nanostructure (BRN) powder according to the sol-gel method explained by Kouhi et al (2016) [10]. At first, TEOS in distilled water and nitric acid for 30 min with ratios of 1, 4 and 0.08 respectively. It was stirred on a magnetic stirrer and the hydrolysis process was carried out at this stage. In the next step, calcium nitrate tetra hydrate and magnesium nitrate hexahydrate were added to the previous solution. To prepare the gel, the created solution was kept at 60 °C for 24h, and then the gel was dried at 120 °C for 2 days. Also, in order to perform the calcination process, the dry gel was heated at 1150 °C for 2h. Zirconia ball mill was used to obtain homogeneous powder. The zirconia vial had a volume of 125 ml and 4 zirconia balls with a diameter of 20 mm were used for 5h. The ball to powder weight ratio was 20:1 and rotation speed was 150 rpm. Finally, the obtained uniform white powder subjected to XRD analysis with radiation, at current intensity of 30 mA, voltage difference of 40 kV with a scanning speed of 3 °/min.

In second step, to evaluate the apatite formation ability of prepared BRN powder, and also to unify the experimental situations, the prepared BRN powder was pressed into pellets in hardened steel mould at a pressure of 1000 MPa.

To evaluate the bioactivity of obtained bredigite nanostructure, they were characterized by soaking the prepared bulk samples in the simulated body fluid solution for 1, 3, 7, 14, 21 and 28 days. For this aim, the prepared bulk samples were soaked in 100 ml simulated body fluid solution. The simulated body fluid (SBF) solution was prepared based on the standard procedure described by Kokubo formula [11]. After soaking, the samples were gently rinsed with deionized water to remove simulated body fluid solutions followed by drying at 110 °C for 2 h.

The apatite formation on the surface of the samples as a result of the precipitation process of calcium phosphate was investigated by scanning electron microscopy (SEM; Philipps, TJP3347), X-Ray

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diffraction , and energy dispersive spectroscopy (EDS).

To calculate crystalline size and examine the nanostructure of the synthesized powder Scherer's relation (1-1 eq.) was used.

L = K.ƛ/Ɓ. CosΘ (1-1)

If we take a logarithm from both sides of the equation, we will have (1-2 eq.):

Ln Ɓ = Ln (K.ƛ/L) + Ln (1/Cos Θ) (1-2)

After drawing the graph according to the results of phase peaks (XRD data) of BRN powder, the width was obtained from the origin. It is Ln (K.ƛ/L), where K is shape factor (0.89), ƛ is CuKα radiation (0.154 nm) and L is crystalline size (nm) [12].

Also, the changes in pH of soaking solutions were measured by an electrolyte type pH meter.

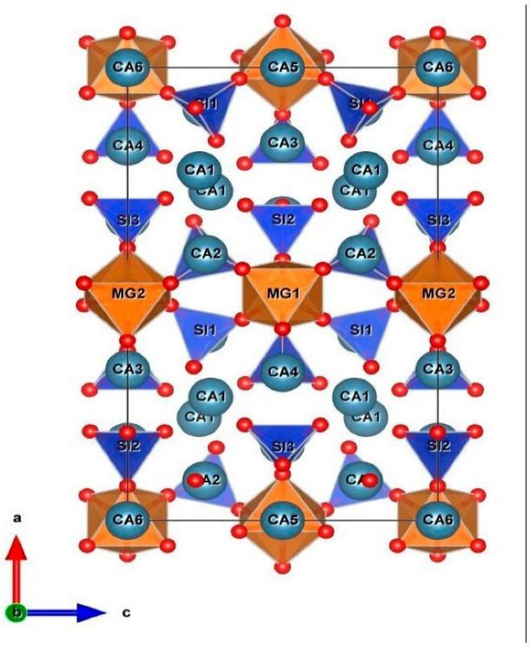

Figure 1. Crystal structure of bredigite [7].

RESULTS AND DISCUSSION

X-RAY diffraction pattern and crystallite size of nanostructure BRN powder are shown in Fig.2. All the X-RAY peaks showed are related to bregidite structure. As it is clear in Fig. 3 the crystallite sizes of the BRN powder obtained were in 25 nm. In fact, after drawing the graph according to the results of phase analysis (XRD data) of bregidite synthesized powder, the width from the origin was obtained as -5.255. So, considering that the width from the origin is equal to -5.225 and the values of ƛ$_{\mathrm{CuK}\alpha}$ (1.54059 A= 0.154 nm) and K (0.89), the crystalline size of the bregidite phase of synthesized powder was 25 nm on average.

The results of the simulated body fluid (SBF) pH are shown in the Fig. 4.

As it can be seen in Fig.4, pH increased during first week and started to decrease after 7 days. In fact, after just 7 days pH decreased and actually Ca^{+2} ions (basic ion) and phosphate ions (PO_4^{-3}) left the SBF solution as an acidic ion and accumulated and precipitated on the sample to form calcium phosphate compound like apatite , which leaded to a drop in pH even lower than the pH of pure SBF solution (PH=7.22). In the other words, the beginning of nucleation and apatitization on the surface of the bredigite sample starts from the second week (7th day), which most of bioceramics indicate the power of nucleation and apatitization almost after the 14_{th} day.

The bone-bonding ability of a material is evaluated by examining the ability of apatite to form on its surface in a simulated body fluid with ion concentrations nearly equal to those of human blood plasma. It was proposed that the examination of apatite formation on a material in simulated body fluid solution is useful for predicting the in vivo bone bioactivity of a material. Our findings suggested that nanostructure BRN powder had apatite formation ability and was bioactive. With dissolving of BRN in simulated body fluid solution, some preferable locations were formed on the ceramic surface that improved the apatite formation ability of nanostructure berdigite. Finally, the Release of Mg ions from nanostructure berdigite bioceramics into simulated body fluid solution medium were quantitatively estimated to support its in vitro bioresorbability.

Fig. 5 indicates SEM micrographs and EDS spectra of the surfaces of bredigite nanostructures (BRN) after immersion in SBF for 21 days and 28 days. As can be seen in the SEM (Fig.5), the density of white apatite deposited in the 28-day sample is higher than of the 21-day sample. On the other hand, in both EDS spectra images, the simultaneous presence of calcium and phosphate together indicates the formation of calcium phosphate compounds (apatite participates).

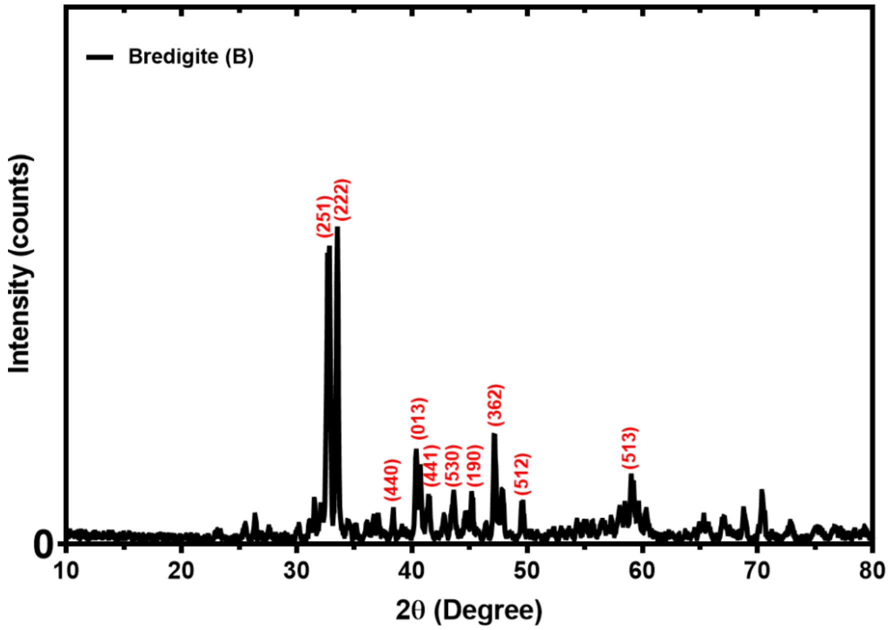

Fig. 2. X-RAY diffraction pattern of bredigite synthesized

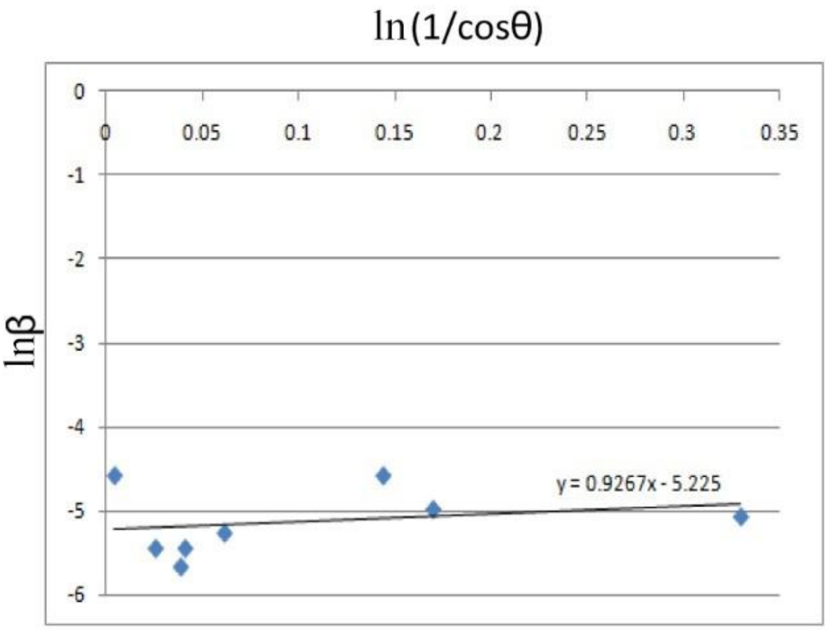

Fig. 3. Ln Ɓ curve vs Ln (1/ CosΘ) based on the XRD data of bredigite synthesized phase peaks

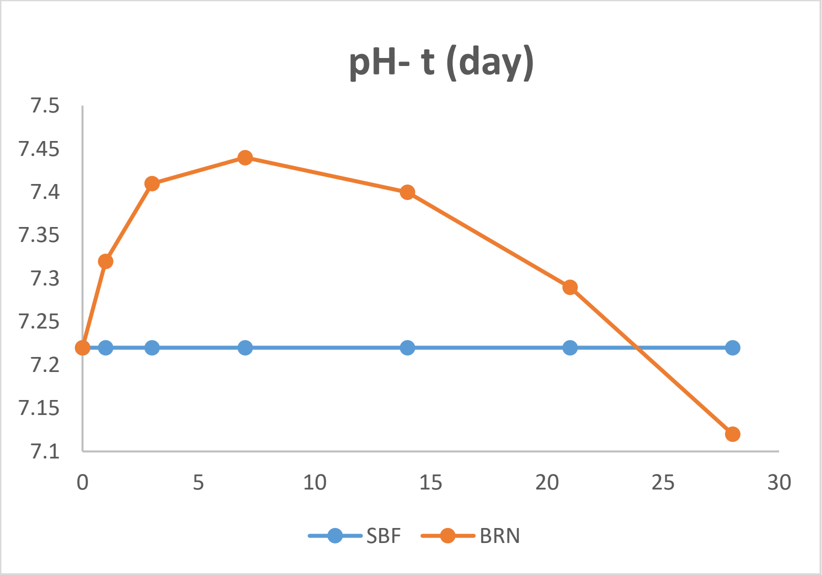

Fig. 4. The graph pf PH vs time of bredigite synthesized sample immersed in SBF solution for 28 days

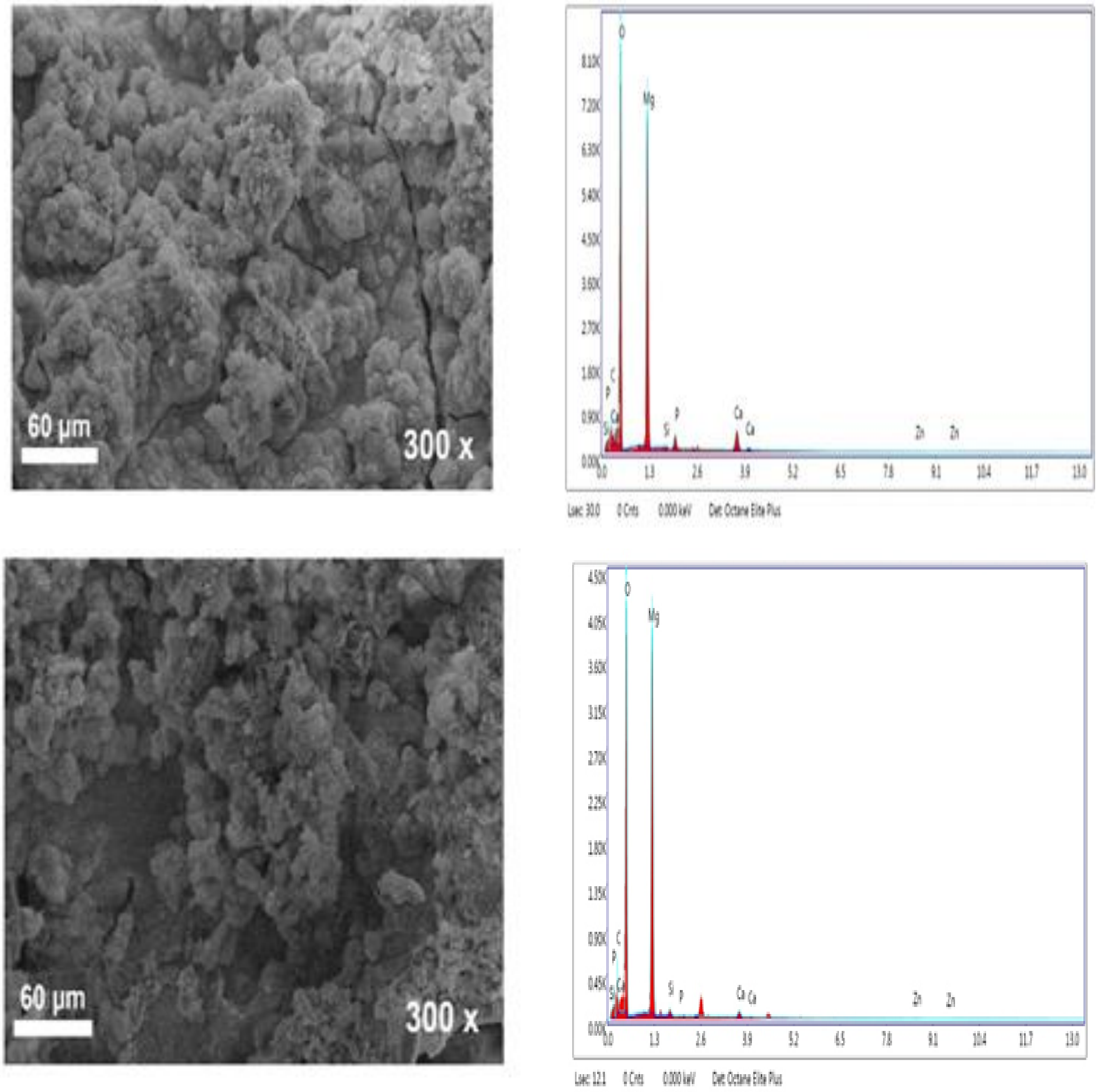

Fig. 5. SEM micrographs and EDS spectra of the surfaces of bredigite nanostructure (BRN) after immersion in SBF for (up) 21 days and (down) 28 days

Conclusion

In this research paper, the behavior of bredigite nanostructures (BRN) ceramic in the simulated body fluid solution was studied to evaluate its bioactivity. In general, the following results can be obtained:

- According to XRD results powder synthesized with crystallite size of about 25 nm, has nano structure.
- In addition, the pH results increased during first week and started to decrease after 7 days. In the other words, the beginning of nucleation and apatitization on the surface of the bredigite sample starts from the second week (7_{th} day).
- As can be seen in the SEM, the density of white apatite deposited in the 28-day sample is higher than that of the 21-day sample. On the other hand, in both EDS spectra images, the simultaneous presence of calcium and phosphate together indicates the

formation of calcium phosphate compounds (apatite participates).

- In general, it can be concluded that the powder synthesized has nanostructure (25 nm in crystal size) and has a strong bioactive behavior (the beginning of apatite nucleation and apatitization from 7_{th} day).

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