J. Nanoanalysis., 5(4): 227-231 Autumn 2018

ORIGINAL RESEARCH PAPER

Survey and Evaluation of Diopside Nanostructure (DSN) Bioactivity in Biomedical Application

Hassan Gheisari Dehsheikh[1,2,]*, Ebrahim Karamian[1] and Ali Soheily[1]

[1]Advanced Materials Research Center, Department of Materials Engineering, Najafabad Branch, Islamic Azad University, Najafabad, Iran
[2]Institute of Biomaterials and Biomedical Engineering, University of Toronto, Toronto, ON, Canada

Received: 2018-06-18 Accepted: 2018-07-23 Published: 2018-10-01

ABSTRACT

Diopside (DS) is a monoclinic pyroxene mineral with composition $MgCaSi_2O_6$. Lately,diopside (DS) has been introduced as a bioceramics due to its best bioactivity and biocompatibility. It has a good strength and toughness than those of hydroxyapatite (HT). In this project, bioactivity of diopside (DS) powder were evaluated and investigated. For synthesized of diopside (DS) powder, magnesium (Mg), calcite ($CaCO_3$) and nano silicium (SiO_2) powders was mechanically activate for different times. After that, the prepared powders were blended with ammonium chloride (NH_4Cl) and put on various temperatures. In this part, for survey of bioactivity evaluation, the obtained dioside (DS) powders were pressed and immersed in Kukobo solution (SBF)The results indicated that nano-struacture diopside powder with crystalline size is 40 nm. The apatite formation ability, bioactivity and better mechanical behavior make it a good candidate in bone implant materials and open new insights in biomedicalapplications.

Keywords: *Bioactivity; Diopside; Hydroxyapatite; Nano-Materials; SBF*

How to cite this article

Gheisari Dehsheikh H, Karamian E, Soheily A. Survey and Evaluation of Diopside Nanostructure (DSN) Bioactivity in Biomedical Application. J. Nanoanalysis., 2018; 5(4): 227-231. DOI: 10.22034/jna.2018.5442734

INTRODUCTION

The composition of hydroxyapatite (HT) is very similar to bone, it's very weak mechanical behavior limit the use of this material 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 system are bioactive used in dental prosthesis [3,6]. Dioside (DS) and enstatite are such good biomaterials that belong to olivine group. DS is machinable bioceramic with a chemical formula of $MgCaSi_2O_6$. That is different polymorphs. A metastable form, clinoenstatite, can be formed from protoenstatite and depending on temperature, pressure and internal stresses in size [7]. For changes of enstatite structure can cause volume changes and produce intrinsic stress, and this material used in medical application [8].

On the opposite, DS has not destroyed volume changes and, due to its good bioactivity, may be used as a good for enstatite bioceramic. While DS has good biocompatibility and better mechanical behavior than those of HT, its bioactivity is poor [9]. Moreover, nanostructure bioceramics is better bioactivity than micron-sized structure [10]. The goal of this project was to study the diopside (DS) nanostructure bioactivity from SiO_2, $CaCO_3$ and Mg in the presence of chlorine ion. Also, bioactivity of the single-phase nanostructure DS powder was investigated by standard method.

MATERIALS AND METHODS

The nanostructure DS powder was synthesized based on our previous study [11]. Concisely, SiO_2 powder was mixed with $MgCaCO_3$ to adjust the

* Corresponding Author Email: *Hassan.gh.d@gmail.com*

$MgCaO/SiO_2$ molar ratio to 2:1, which corresponds to the theoretical value of pure diopside (DS). After that the powder blend was milled in a planetary ball mill under ambient conditions for 10 hours. The ball to powder weight ratio was 20:1, and the rotational speed of the disc were set at 600 rpm. Then, NH_4Cl powder was added to the mechanical milling mixed and the mixture was milled for 10 minutes. The molar ratio of $MgCaCO_3$:SiO_2: ammonium chloride powders were set to 6:1:4. Annealing of this powder was carried out at 1250 °C for 2 hours.

In this part, to evaluate the apatite formation ability of prepared DS powder, and also to unify the experimental situations, the prepared DS powders were pressed into pellets in hardened steel mould at a pressure of 1000 MPa.

To evaluate of bioactivity of obtaining nanostructure DS was characterized by soaking the prepared bulk samples in the simulated body fluid solution for 1, 3, 7, 14, 21, 28 and 30days. 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 the Kokubo formula [12]. After soaking, the samples were gently rinsed with deionized water to remove simulated body fluid solutions followed by drying at 200 °C for 14 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), and X-Ray diffraction , energy dispersive spectroscopy (EDS), Fourier transitioned-infrared spectroscopy (FTIR; TAMKEN, GS 223).Also, the concentrations of calcium and magnesium ions of the simulated body fluid after soaking were determined by atomic absorption spectrometer (AAS; Hitachi, 1205), and the changes in pH of soaking solutions was also measure an electrolyte type pH meter.

RESULTS AND DISCUSSION

X-RAY diffraction pattern and crystallite size of nanostructure DS powder are shown in Fig.1a and b. All the X-RAY peaks showed of DS structure. As it is clear in Fig. 1b, the crystallite sizes of the DS powder obtained were in 40 nm.

For determining the magnesium and calcium ion concentration in the simulated body fluid, atomic absorption spectrometer test was performed. The results of the atomic absorption spectrometer test and the pH of the simulated body fluid are shown in the Fig. 2. As you have seen, the pH and magnesium ions concentration and decreasing the calcium ions concentration in the simulated body fluid were the overall consequence of soaking the samples. The findings revealed that in the first day of soaking, ions concentration and pH of the simulated body fluid solution had the most changes. With increasing the soaking time, calcium ions concentration decreased with a smooth slope due to the consumption of these ions and the formation of apatite on the surface of DS samples.

Fig. 2d compares the FTIR of the surfaces of the prepared DS bulk samples before and after soaking in the simulated body fluid solution. The bands related to the investigation bands of DS appeared at 1010, 971, 882, and 842 cm^{-1} (SiO4), at 621, 531, and 512 cm^{-1} (SiO4 bending), and at 481 cm^{-1} for modes of octahedral MgO_6, which is in a better agreement with previous studies [12]. By

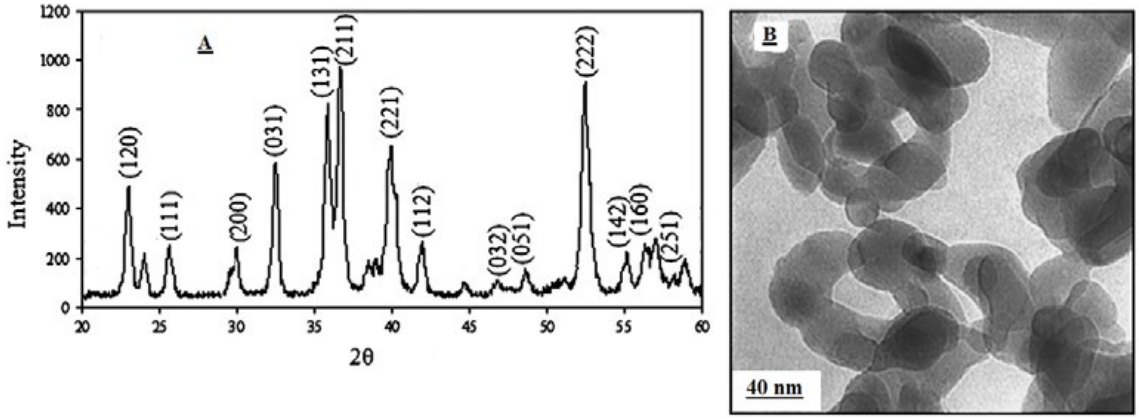

Fig. 1. (A).X-RAY diffraction pattern and (B) Transmission electron microscopy (TEM) image of obtained diopside powder after 10 h mechanical activation

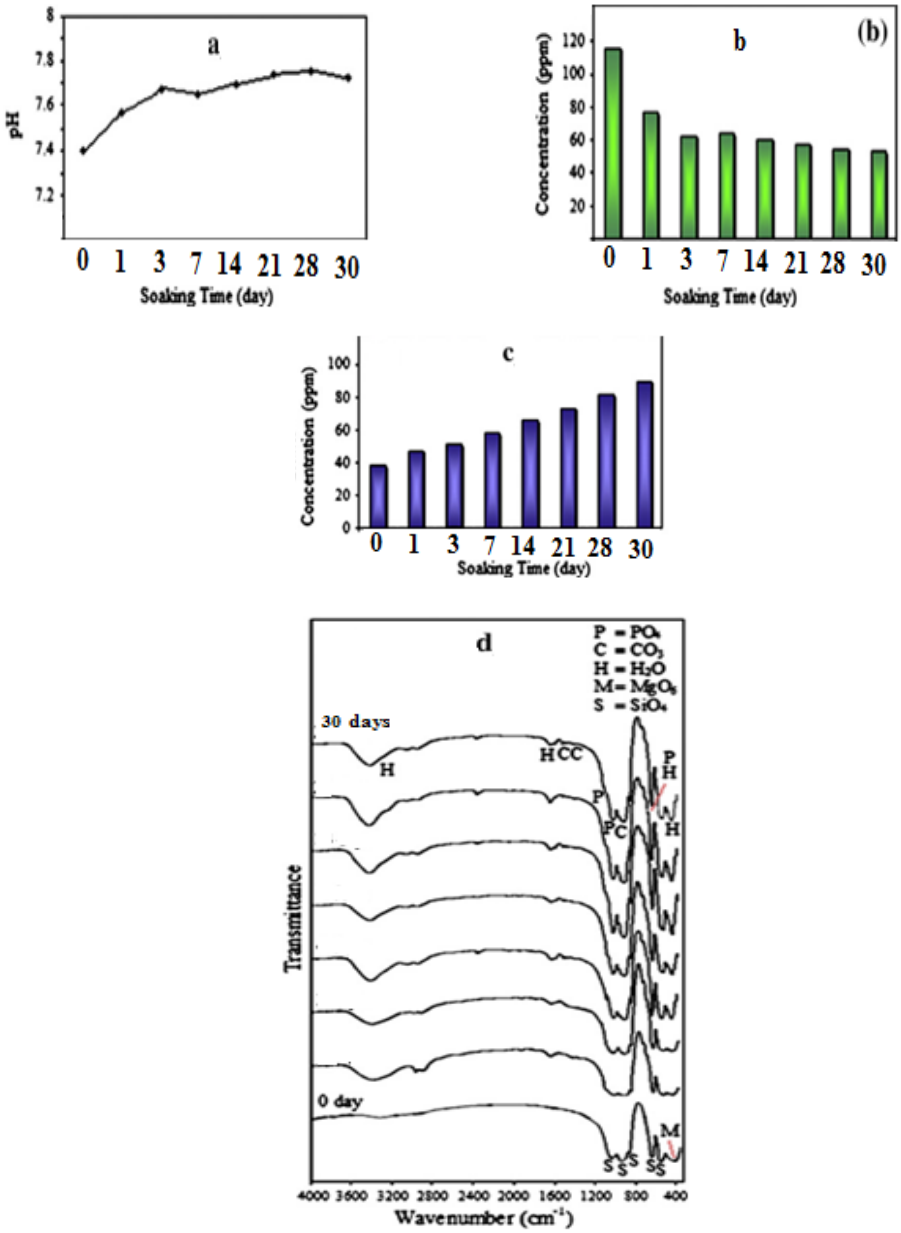

Fig. 2. The variations of (a) pH, (b) Ca, and (c) Mg ions concentration of the simulated body fluid, after soaking the nanostructure DS ceramics and (d) FTIR spectra of nanostructure DS soaked in the SBF for different periods of time.

immersing the samples in the simulated body fluid solution for different times, new absorption bands relating to O–H, C–O, and P–O were observed. The bands at 3650 and 1632 cm^{-1} belonged to hydroxyl ion groups in the HT.

Those bands at 1471 and 1431 cm^{-1} fit with bands in carbonate groups of apatite. Also, the band at 881 cm^{-1} was assigned to carbonate groups that could be distinguished at higher soaking times in the simulated body fluid solution. Also, the bands related to phosphate groups were situated at 1110–1040, 611, and 581 cm^{-1}. By increasing the soaking time, the absorption bands of all O–H, C–O, and P–O bands got stronger because of formation of higher amount of HT on the surface of DS samples.

Fig. 3 indicates the surfaces morphology and energy dispersive spectroscopy spectra of the DS samples after immersion in the simulated body fluid

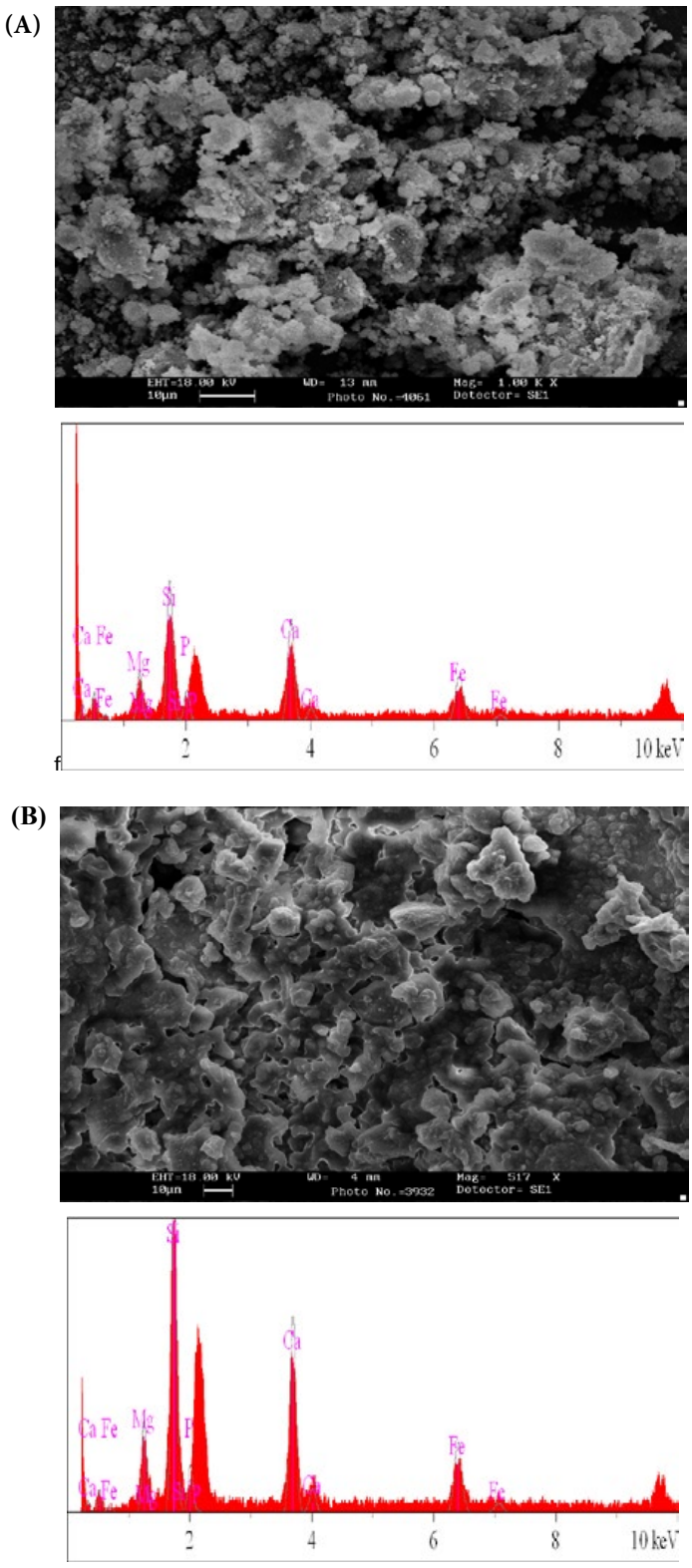

Fig. 3. SEM micrographs and EDS spectra of the surfaces of nanostructure DS after immersion in SBF for (A) 14 days and (B) 30 days.

solution for 21 and 30 days. After 14-day soaking, small particles were observed on the surface of DS samples with cauliflower shape. The energy dispersive spectroscopy spectra showed that these particles are composed of Ca and phosphorus. By increasing the soaking time, the DS grew and their size increased. Moreover, the energy dispersive spectroscopy (EDS) spectra proved the presence of Ca and phosphorus in this specimen. With the results of FTIR patterns and changes on the ions concentration in the simulated body fluid solution, it is expected that the formed deposits were hydroxyapatite (HT).

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 DS powder had apatite formation ability and was bioactive. With dissolving of DS in simulated body fluid solution, some preferable locations were formed on the ceramic surface, that improved the apatite formation ability of nanostructure diopside (DS). Finally, the Release of Mg ions from nanostructure DS ceramics into simulated body fluid solution medium were quantitatively estimated to support its in vitro bioresorbability.

CONCLUSION

In this research paper, the behavior of nanostructure DS ceramic of the simulated body fluid solution was studied to evaluate its bioactivity. These results indicated that prepared nanostructure DS with crystallite size of about 40 nm, was bioactive and had the ability of apatite layer formation. In addition to, atomic absorption spectrometer; AAS test of simulated body fluid showed that nanostructure DS ceramic released Mg ions into simulated body fluid and had biodegradation behavior. In sum up, our findings indicated that nanostructure diopside (DS) bio-ceramic is bioactive and might make an good candidate for biomedical purposes.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

REFERENCES

1. H.Gheisari, E.Karamian and M.Abd, A novel hydroxyapatite –Hardystonite nano composite ceramic, Ceramics International.,4, 5967 (2015).
2. H. Gheisari and E.Karamian, Preparation and characterization of hydroxyapatite reinforced with hardystonite as a novel bio-nanocomposite for tissue engineering, Nanomed.,1, 298 (2014).
3. K.Thakkar, Biological synthesis of metallic nanoparticles. Nanomed., 6,62 (2010).
4. A.Hosseini and A.Emtiazi. Development of an eco-friendly for biogenesis of silver nanoparticles using spores of Bacillus athrophaeus, J. Microbiol Bio., 3, 64 (2013).
5. A.Hosseini and A.Emtiazi, Biosynthesis of silver noparticles by Bacillus stratosphericus spores and the role of dipicolinic acid in this process, Biotechnol.,4, 282 (2014).
6. E.Karamian and H.Gheisari. Fluorine-substituted HA reinforced with zircon as a novel nano-biocomposite ceramic: Preparation and characterization.Nanomed., 3, 8 (2015).
7. H.Gheisari Dehsheikh and S.Ghasemi-Kahrizsang, Performance improvement of MgO-C refractory bricks by the addition of Nano-ZrSiO4.Materials Chemistry and Physics., 4, 376 (2017).
8. S. Ghasemi-Kahrizsangi, H.Gheisari Dehsheikh and E.Karamian, Impact of Titania nanoparticles addition on the microstructure and properties of MgO-C refractories, Ceramics International., 5, 477(2017).
9. S.Ghasemi-Kahrizsangi, H.Gheisari Dehsheikh, Effect of micro and nano-Al2O3 addition on the microstructure and properties of MgO-C refractory ceramic composite, Materials Chemistry and Physics. 2, 236 (2017).
10. M. Bagi, S.Adak. Study on low carbon containing MgO-C Refractory Use of nano carbon, Ceramics International., 3(3) 2339-2346 (2012).
11. M. Bagi, S.Adak. Nano carbon containing MgO-C refractory and effect of graphite content, Ceramics International., 4(1) 4909-4914 (2012).
12. S.Ghasemi-Kahrizsangi and H.Gheisari Dehsheikh, A Review on Recent Advances on Magnesia-Doloma Refractories by Nano-Technology, JWEN., 3 , 222 (2017).