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In-vitro bioactivity studies of chitin/diopside composites

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Abstract: Silicates are well known for their bioactivity, here we are reporting the bioactivity of diopside ($CaMgSi_2O_6$) synthesized by sol-gel combustion process by using citric acid and succinic acid as a mixed fuel. The phase formation of the prepared sample was confirmed by powder X-Ray Diffraction. In-vitro bioactivity of the diopside in conjugation with biopolymer chitin in various proportions were analyzed by immersing the scaffolds in simulated body fluid (SBF) solution. The biopolymer to bioceramic ratio which mimics human bone shows more bioactivity.
Keywords: Diopside, Sol-gel combustion, Bioactivity, Hydroxyapatite.

1. Introduction

Since the discovery of Bioglass in 1971 by Hunch[1],researchers were focused for the preparation of silica containing materials for Biomaterial applications, because these silicates are highly encouraging for the growth and differentiation of osteoblasts cells. Diopside is a type of bioactive silicate material which can release silicate ions. The in-vitrobone like apatite layer formation in SBFsolution and in-vivo bone formationof diopside has already been reported[2]. Compared to other silicate ceramics like akermanite and bredigite, diopside has shown much improved bioactivity3. It can be said that silicate materials in conjugation with biopolymers like chitin and chitosanare widely used in medical field because of their good biological functions[4].

Synthesis of diopside can be achieved through many conventional preparation techniques. Whereas, sol-gel combustion method is found to be more suitable as it forms the product at low temperature[5]. The nature of the product mainly depends upon the starting materials and the fuel used in the reaction. In this content, for the first time we are preparing the diopside using citric acid/succinic acid as mixed fuel by sol-gel combustion method. The prepared diopside bioactivity was analyzed in conjugation with chitin in SBF solution.

2. Materials and methods

2.1Materials:$Ca(NO_3)_2.4H_2O$ (98 %, SDFCL, Pure), $Mg(NO_3)_2.6H_2O$ (99 %, Qualigens), TEOS (98 %, Acros organics), HNO_3(SDFCL), Citric acid anhydrous (99.5-101 %, SDFCL, AR) , Succinic acid (99 %, Qualigens), NaCl (99.9 %, SDFCL, AR), $NaHCO_3$ (99 %, Nice chemicals, LR), KCl (99.5, SDFCL, AR), $K_2HPO_4.3H_2O$ (SDFCL), $MgCl_2.6H_2O$ (98-100.5 %, Qualigens), HCl (SDFCL), $CaCl_2$ (90 %, SDFCL, LR), Na_2SO_4(99.5 %, SDFCL, AR) Tris buffer (99.8 %, SDFCL, AR), Chitin (Himedia).All these chemicals wereused as such without any further purification.

2.2 Synthesis of diopside: The stoichiometric ratios of calcium nitrate solution (1M), magnesium nitrate solution (1M), citric acid solution (1M) and succinic acid solution (1M) solution were taken and mixed thoroughly. Tetraethyl orthosilicate (TEOS, 2M) was added to the above mixture and then 10mL concentrated nitric acid was added. The resultant solution was stirred constantly at room temperature until the formation of the gel. Thus formed gel was dried at 55 ^{0}C for 20 h in hot air oven. Dried gel was combusted 400 ^{0}C for 30 min in the preheated furnace and combusted product was grinded and calcined.

2.3 Characterization: Bruker D8 advance X-Ray Diffractometer with Cu Kα, Ni filtered radiation was used for XRD analysis.

2.4 Bioactivity: The SBF solution was prepared according to chao et al. procedure. The chemicals, NaCl (7.996 g), $NaHCO_3$ (0.350 g), KCl (0.224 g), $K_2HPO_4.3H_2O$ (0.228 g), $MgCl_2.6H_2O$ (0.305 g), HCl (1M, 40 mL), $CaCl_2$ (0.278 g), Na_2SO_4 (0.071 g) and Tris buffer (6.057 g) were dissolved to the 1 L double distilled water one by one in the sequence. Then the pH of the solution was adjusted to 7.4 by adding 1M HCl solution.

For checking the bioactivity of the diopside, pellets were made with different proportions of the biopolymer chitin. The sample depicted as M20, M40 and M60 comprises 20 mg, 40 mg and 60 mg of chitin and 180 mg, 160 mg and 140 mg of the diopside sample respectively. These pellets were immersed in 30mL of SBF solution maintaining a temperature of 37^{0}C in an incubator.

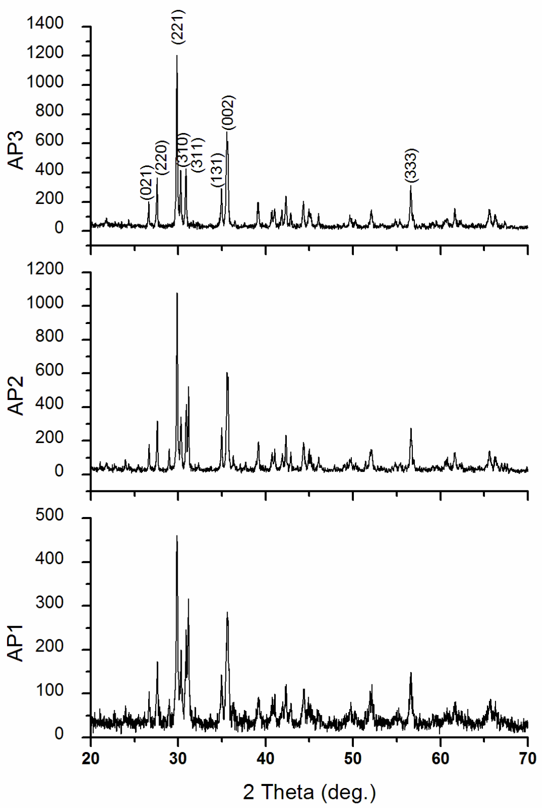

Figure 1 XRD pattern AP1: diopside calcined at 900 ^{0}C for 6h, AP2: diopside calcined at 1000 ^{0}C for 6h, AP3: AP2 sample socked in the SBF solution for 5 days.

3. Results and discussion

A complex product i.e. gel is obtained after the reaction between the metal salts and the fuels as a result of poly condensation reaction. This gel was decomposed in the preheated furnace and resultant precursor was calcined at different temperatures to study the phase evolution.

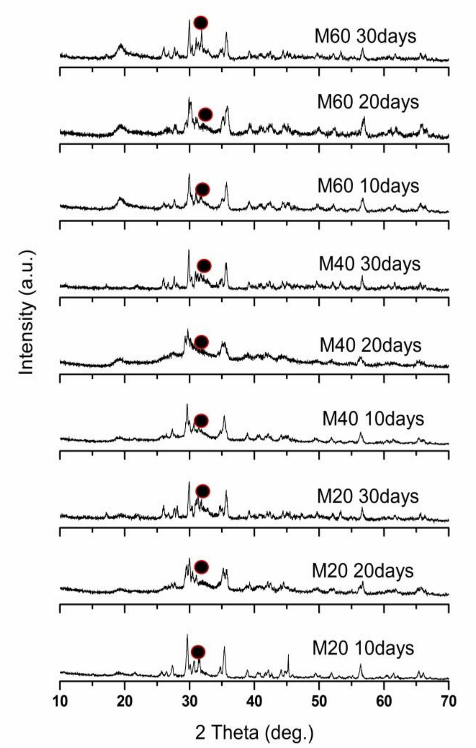

Figure 2 XRD pattern of the scaffolds immersed in the SBF solution

The calcined product wasanalyzedby powder X-ray diffractometer.The XRD pattern of the sample calcined at 900^0Cfor 6 hours (AP1) shows the diopside as major phase and akermanite as a secondary phase.

The XRD pattern of the sample calcined at 1000 ^{0}C shows (AP2) reduced content of akermanite.

When the powder sample was soaked in SBF for five days and dried at hot air oven for 6 h and analysed by powder X-ray diffractometer. The XRD pattern (AP3) of the powder soaked in SBF shows the absence of the akermanite phase.The XRD pattern is in agreement with the JCPDS data card 01-075-0945. Even for all ratios when soaked in SBF for longer duration shows enhancement in its bioactivity.

Scaffolds of the diopside sample was made with different proportions of biopolymer chitin. The bioactivity was studied through the formation of the hydroxyapatite layer on the surface of scaffolds. The hydroxyapatite layer formation was analysed bypowder XRD. The XRD pattern (Figure 2) of scaffolds immersedin the SBF for various durations shows very good bioactivity. Among 3 different ratios the one mimics the ratio of biopolymer to bioceramic as the bone, shows higher bioactivity (M60 30days).

4. Conclusion

Diopside was successfully synthesized by sol-gel combustion method by using succinic acid and citric acid as mixed fuel for the first time. In-vitro bioactivity studies of the scaffolds shows better bioactivity for the ratio which mimics natural bone.

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