Bioactive glass microsphere의 합성 및 특성 분석

추고연, Pragya Shrivastava[1], S.Vijayalakshmi[1,*], Pratibha Sharma[1], D.Sridhar[1]

한국에너지기술연구원, [1]Indian Institute of Technology Bombay

(vlakshmi@iitb.ac.in*)

Synthesis and characterization of bioactive glass microspheres

Ko Yeon Choo, Pragya Shrivastava[1], S.Vijayalakshmi[1,*], Pratibha Sharma[1], D.Sridhar[1]

Korea Institute of Energy Research, [1]Indian Institute of Technology Bombay

(vlakshmi@iitb.ac.in*)

Introduction

Nanotechnology has immense potential for putting significant contributions in medical technology, in terms of disease detection, diagnosis, treatment, therapy and prevention. In this context various nanoparticles like nanorods, Q dots, functionalized nanoparticles, bioactive ceramics and glass etc are important and under extensive research for various biomedical applications like, controlled drug delivery vehicles, bone regeneration therapy and dental applications. Glass due to its inertness even in prolonged contact with body fluids, mechanical stability and non-toxicity, can be used in a wide range applications in biomedical field. All these advantages make the bioglass suitable for desired biomedical applications. Bioactive glass is known for the formation of a biologically active apatite layer on implantation with bone tissues.

Experimental

Tetra Ethyl Ortho Silicate dissolved in ethanol and Calcium nitrate tetrahydrate in water were mixed under continuous stirring and the pH was maintained at 1-2 by adding citric acid solution.

Further neutralized using ammonium dibasic phosphate and polyethylene glycol (PEG) was added. The pH was adjusted to 9-11 and the whole mixture after aging for 48 h, was centrifuged at 6500 rpm and freeze dried at -55^{o}C for 24 h [1,2,3]. The bioactive glass microspheres thus prepared were calcined at 300°C, 500°C, 700°C and 900°C and were labeled as BG unheated, BG 300, BG 500, BG 700 and BG 900. The characterizations of the samples were done using SEM, TEM, XRD and FTIR.

Results and discussion

The micro structural analysis of all the samples was done on FEGSEM (JEOL, JSM-7600F) and also on FEGTEM (JEOL, JEM-2100F). The SEM and TEM images confirmed the spherical nature of the particles in the bioglass prepared. The BG unheated had microspheres with effective diameters ranging from 0.2 to 5.0 microns, whereas when the samples were calcined, the particle size diminished and at 700^{o}C, the particle size was 0.5 to 1.5 microns. The TEM images also revealed the same phenomenon. The BG unheated showed an agglomerated mass of many spheres with non uniform particle size, but the BG 700 showed individual uniform sized microspheres. The SEM and TEM images of BG unheated and BG 700 are shown in Fig.1 a & b and Fig 2 a & b respectively. The effective diameter data obtained from the SEM images were used to calculate the particle size distribution in the samples. The BG 700 sample showed more than 80% spheres with diameter 1-1.5 and 1.5-2 microns. The BG 900 sample had more than 80% particles with diameter, 0.5-1 micron size. There was size shrinkage occurring to the particles in BG 900, due to sintering. The particle size distribution is shown in Fig.3 a & b. The FTIR spectrum of BG 700 showed transmittance at wave numbers, 1080 and 810 cm^{-1} corresponding to Si-O-Si and Si-O bond, symmetric and stretching vibrations [4] (Fig.4). No trace of nitrates or other impurities were observed in BG 700. The XRD spectrum (Fig.5) showed that all the samples were amorphous.

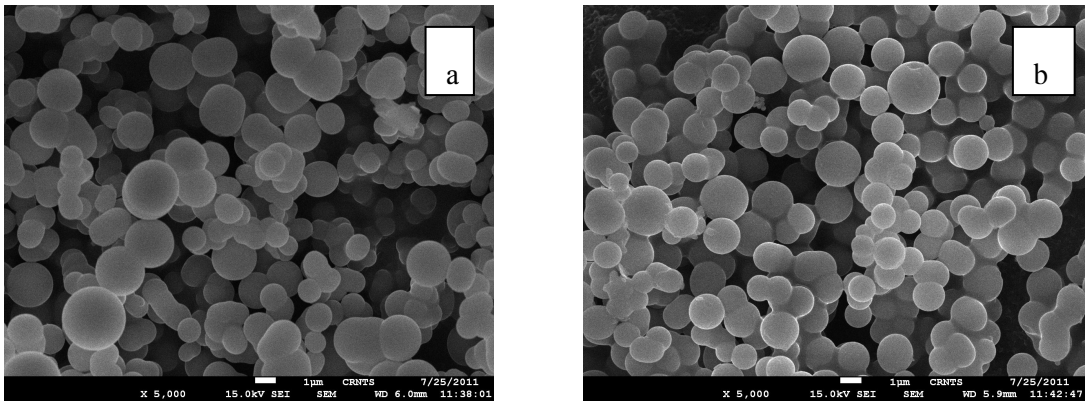

Fig.1 a & b SEM images of BG unheated and BG 700

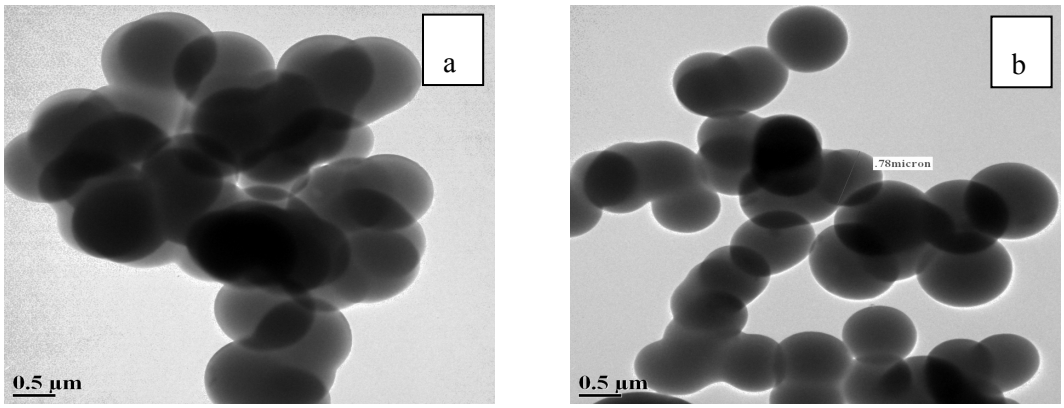

Fig.2 a & b TEM images of BG unheated and BG 700

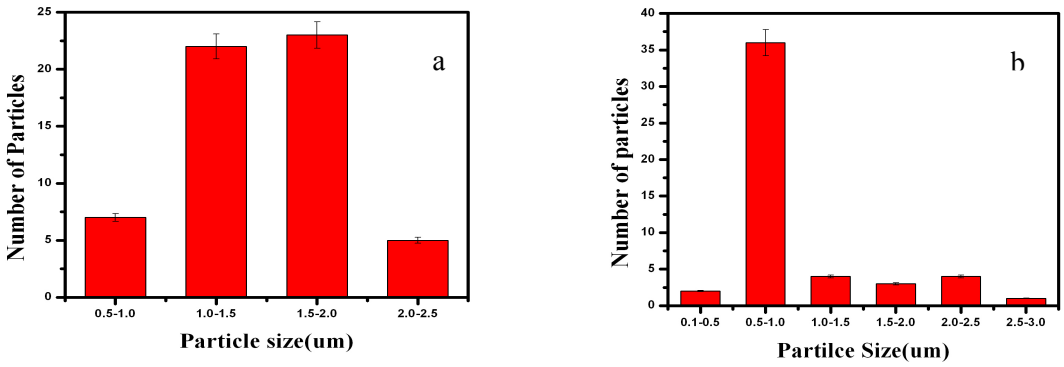

Fig.3 a & b Particle size distribution of BG 700 and BG unheated

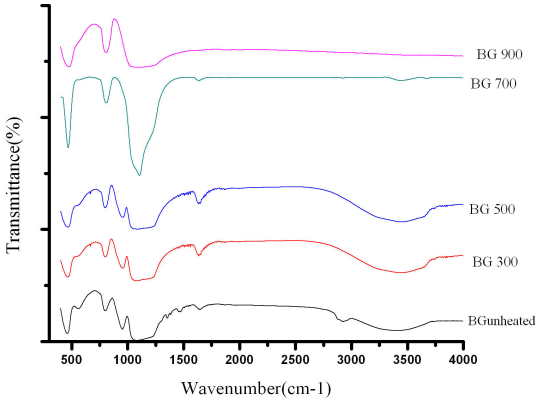

Fig. 4 FTIR of bioglass

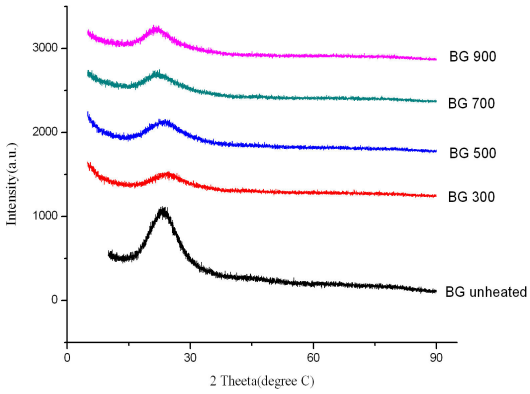

Fig. 5 XRD of bioglass

Conclusions

Bioglass microspheres were successfully prepared using sol-gel process. The size and shape of the bioglass particles are very much depended on the hydrolysis and aging time of the sol-gel. Highly uniform and clean bioglass spheres are formed when the samples were calcined at 700^{o}C for 3h. These samples can be made bioactive for desired biomedical applications by further modifications on the sample. Coating of hydroxy apatite layer is very much feasible on the BG700 sample prepared in this work.

Reference

1.M. Peter, Pandian Thodi Sudheesh Kumar, Nelson Sathy Binulal, Shanti V. Nair, Hiroshi T amura, Rangasamy Jayakumar. Carbohydrate Polymers 78, 926 (2009).

2. S.Padilla, J.Roman, A.Carenas, M.Vallet-Regi, Biomaterials 26, 475 (2005).

3. K.K.Flora and J.D.Brennan, Chem.Mater., 13, 4170 (2001).

4. D.Baciu, J.Simitzis, J.optoelectronics and advanced materials 9, 11, 3320 (2010)