Synthesis and Characterization of $35SiO_2$-$55CaO$-$10P_2O_5$ Bioactive Glass Composition

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Abstract— **Glass with the composition of $35SiO_2$-$55CaO$-$10P_2O_5$ has been prepared in the laboratory by using Stober method. Structural analysis of sample has been undertaken with the help of XRD and FESEM studies. Simulated body fluid has also been prepared in our laboratory. The growth of apatite layer has been confirmed by XRD and FESEM studies. Variation in pH values of sample during in vitro analysis has been analyzed. Rate of appetite layer growth and other mechanisms are also discussed in this paper and it is found that sample shows bioactive nature.**
Keywords-Bioactive glass, Biomaterials, Stimulated Body Fluid, Hydroxyl apatite

I. INTRODUCTION

Repair of the damaged and diseased human bones is a challenging problem. Some of the possible solutions can be bone grafting, metallic implants and bioactive glasses. Grafting of human bone is very common practice but due to several limitations like transfer of infection from donor to recipient, limited availability of graft material, damaging of healthy tissues etc., artificial bone regenerating materials are in demand. Metallic implants such as titanium, bronzes etc. can also be used for repair of bones but these metals also have several demerits such as temperature dependence, toxicity and surgical problems. L.L. Hench[1] demonstrated the possible solution for these problems with the invention of a special kind of material which have the ability to bond with the bones and soft tissues with hydroxyl apatite (HAp) layer in stimulated body fluid (SBF). This material is 45S5 (45% SiO_2, 24.5% Na_2O,24.4% CaO and 6% P_2O_5 in weight percentage) bioactive material and termed as Bioglass®. Bioactivity and biocompatibility of a biomaterial can be influenced by many factors including composition, texture, density and porosity. After the invention of Bioglass®, many research groups have tried to improve the mechanical and bioactive properties of bioactive glasses with different compositions and methods of preparation. Recently much attention has been paid to prepare bioactive glasses with nano particles to enhance bioactivity of biomaterials [2-4]. Appreciable bone bonding and load bearing abilities make nano particles based bioactive glasses as good biomaterials. In the present work, an attempt has been made to prepare bioactive glass with crystallite size of few nano meters with Stober method [5-7]. Biological response of sample is monitored by incubating in SBF.

II. MATERIALS AND METHODS

For the preparation of $35SiO_2$-$55CaO$-$10P_2O_5$ composition, bioactive particles have been synthesized by using Stober method under controlled temperature condition. For the synthesis purpose, TEOS, TEP and calcium nitrate tetra hydrate have been used as precursors of SiO_2, P_2O_5 and CaO (all Merck Company, AR grade). 2-Propanol and distilled water with citric acid have been employed as hydrolysis agents for TEOS and TEP. Molar ratio of TEOS: Water: 2-Propanol:: 1:4:8 is blended for 1 hour by adjusting pH (1~2) with the help of citric acid. TEP is added drop wise and continuously stirred for half an hour for preparation of solution 1. 0.1M solution of calcium nitrate tetra hydrate (pH: 10 ~11) with the help of NH_4OH has been prepared as solution 2. Subsequently, drop wise solution 1 is added into solution 2 for 1 hour under controlled temperature conditions. Prepared solution is heat treated at 200˚C and 700˚C for 40 minutes each for removal of solvents. SBF solution was prepared using the standard method given by Kokubo[8] having pH and ions concentration equivalent to human blood plasma. 150 mg of sample was incubated in 50ml of solution for 24 hours.

III. CHARACTERIZATION TECHNIQUES

A. *XRD Studies*

XRD study of prepared samples has been undertaken by Bruker D8 Focus. Cu Kα lines were the source of X-rays with high intensity. Wavelength (~1.54A^0) at scanning rate of 2°/min in the angle (2θ) range 10° to 70°for identification of amorphous and crystalline nature of samples has been employed.

B. *FESEM Studies*

Surface morphology of sample has been studied by using ZEISS Supera 55 scanning electron microscope. Change on the surface morphology of sample can be noticed clearly before and after in

vitro test. EDX patterns indicate presence of calcium and phosphorus. Intensity of EDX peaks increase gradually with increase in the time period of contact for samples in SBF.

C. pH Studies

Change in pH value was measured every two hours using pH meter (ESICO, model 1012).

IV. RESULTS AND DISCUSSION

A. XRD Studies

An x-ray diffraction pattern of prepared sample is shown in Fig. 1. Absence of any sharp peaks confirms the amorphous nature of the prepared sample. Crystallite size of prepared particles has been evaluated as 28 nm by using Scherrer formulae[9, 10]. After incubation in SBF for 24 hours, sharp peaks of apatite layer appear at 26.2˚,31.9˚,45.7˚,56.7˚ and 66.3˚ which matches with calcium hydroxide phosphate (Hydroxyl apatite), JCPDF no. 074-0566. XRD results indicate the bioactive nature of prepared glass composition.

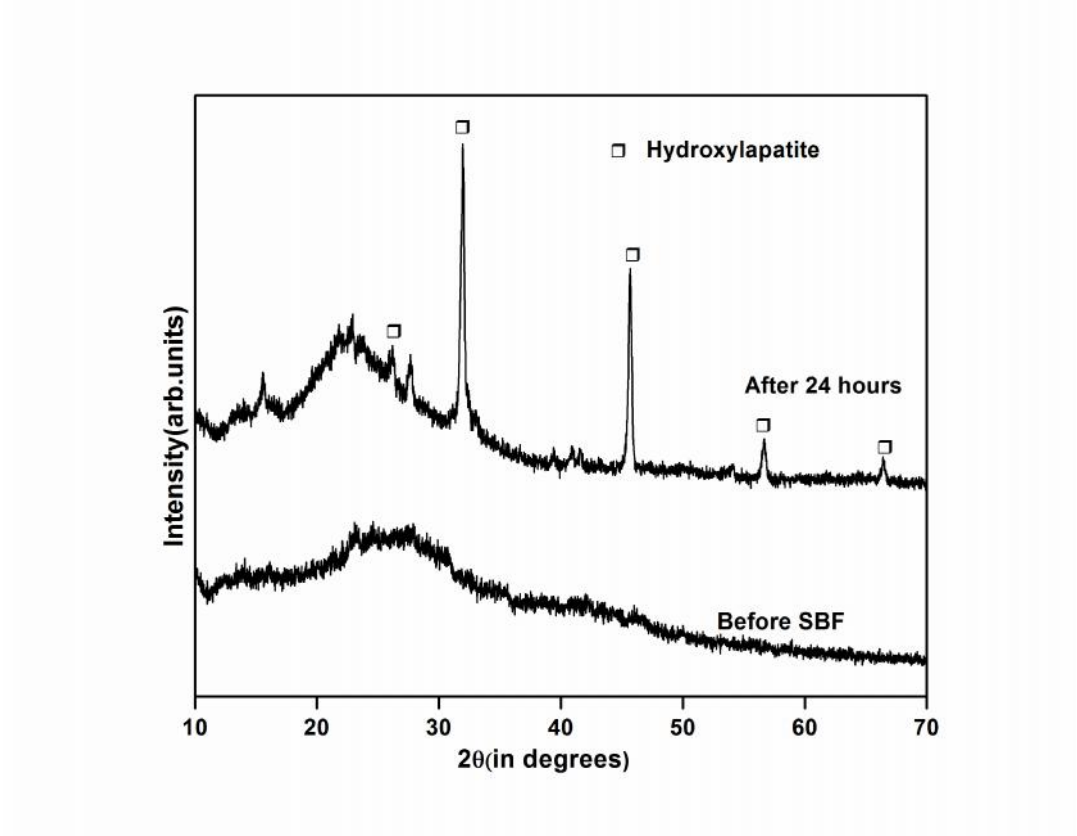

Figure 1: XRD pattern of prepared particles before and after in vitro analysis

B. FESEM Studies

Surface morphology of prepared sample has been investigated before in vitro analysis and after incubation in SBF for 24 hours. Change in the surface has been noted clearly. EDX analysis shows the variation in intensities of calcium, phosphorus and silicon before in vitro and after incubation in SBF as shown in Fig. 2.

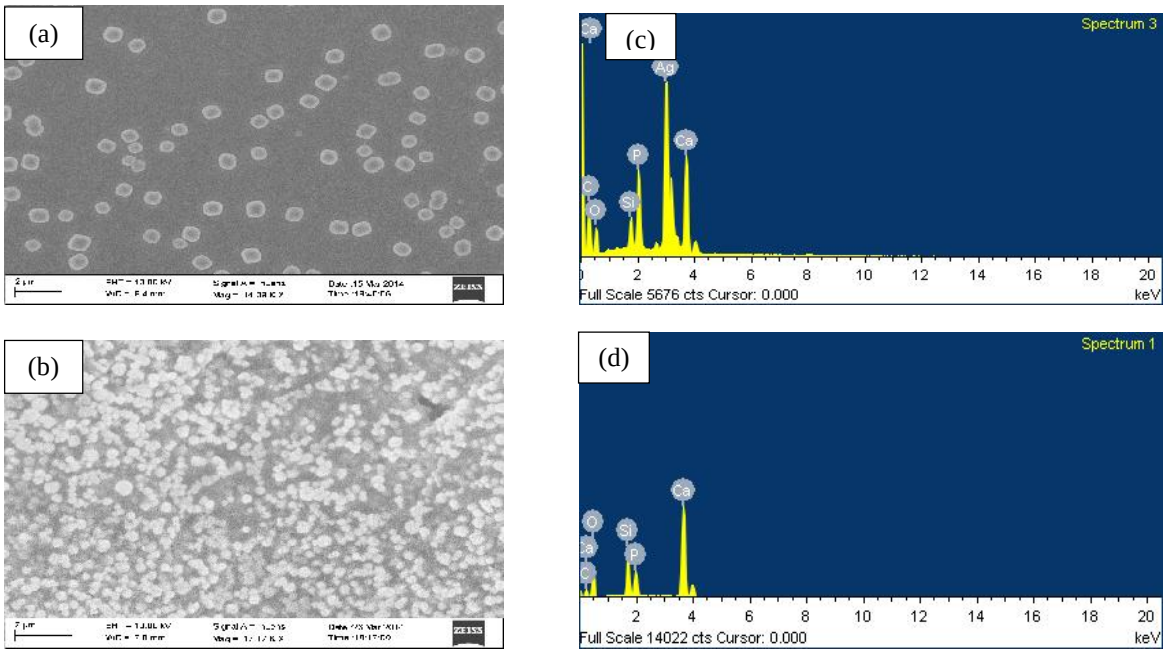

Figure 2: FESEM images of prepared nanoparticles (a) before and (b) after in vitro analysis, EDX patterns of sample (c) before and (d) after in vitro analysis.

C. *pH Studies*

pH value of sample has been studied after regular intervals of time. Trends of pH value are provided in Fig. 3. pH of sample had started from 7.4 and reached up to 8.9. pH of sample has varied with in small narrow range of 1.5. pH values indicate the non-acidic nature of sample which is very useful for the growth of hard and soft tissues.

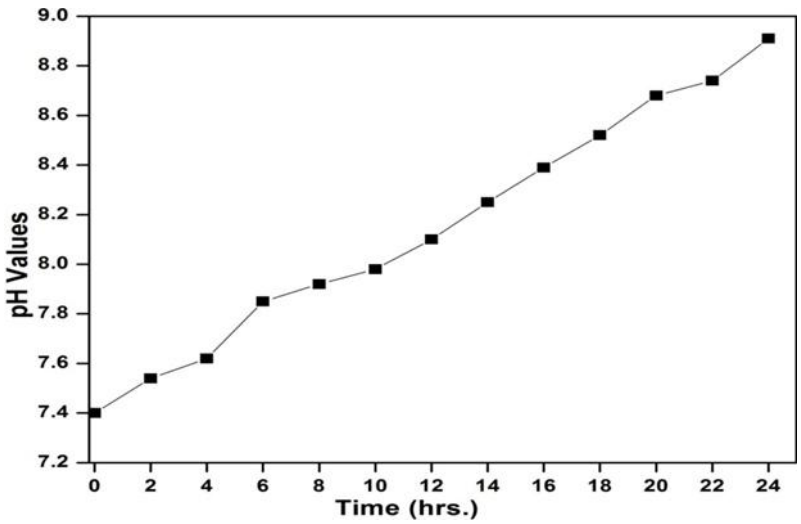

Figure 3: Variation of pH value with time in hours

CONCLUSIONS

From above results and discussion, it can be concluded that

1) Crystallite sizes of particles are in nanometre range and the sample is bioactive in nature.

2) pH values of the sample indicate non-acidic nature of the prepared particles indicating that particles are useful for growth of soft and hard tissues.

3) It is observed that sample shows good value of apatite growth rate. Traces of apatite layer have been observed within 24 hours.

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