Synthesis and Characterization of LI Doped Bioactive Glass

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Abstract:** Bioactive glass with Na_2O-Li_2O-SrO-CaO-B_2O_3-SiO_2-P_2O_5 was prepared by conventional melt-quench method. The prepared glass samples have been characterized by X-ray diffraction (XRD) shows amorphous nature of the glass composition. Simulated body fluid (SBF) was used to study the in vitro bioactivity of the prepared glass composition. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) were carried out to investigate bioactivity of the prepared glass. SEM and EDX results were used to reveal the formation of a hydroxycarbonate apatite (HCA) layer on the sample, which confirms the bioactivity nature of the sample.
***Keywords**: Bioactive Glass; Melt-quench; Simulated body fluid; In vitro; Hydroxycarbonate apatite;*

I. INTRODUCTION

In 1969, Hench et.al synthesized the first bioactive material of composition Na_2O-CaO-P_2O_5-SiO_2 known as 45S5 by conventional melt quench method [1]. Basically, a bioactive material is a synthetic material and is capable to bond with living tissue. Bioactive material is defined as "a nonviable material used in a medical device, intended to interact with biological systems"[1]. All bioactive materials when in contact with physiological fluids, a calcium phosphate rich layer is formed on their surface which, upon nucleation and growth of an apatite layer called hydroxycarbonate layer (HCA) is formed [2],[3]. Bioactive materials are known to bond to living bone through this hydroxycarbonated apatite layer [4]. This mechanism was successfully explained by many authors[5], [6].

Hydroxyapatite layer is a crystalline form of calcium phosphate similar to the mineral phase present in bone and is mainly responsible for interfacial bonding [7]. It is a compound with a definite crystallographic structure and of a definite composition $Ca_{10}(PO_4)_6(OH)_2$. The bonding mechanism of bioactive glass to living bone is very complex. However, in vitro studies explained the formation of hydroxycarbonate apatite layer on the surfaces of bioactive glass and glass ceramics when placed in an acellular, supersaturated simulated body fluid (SBF), known as kokubo's solution proposed by kokubo et al., in 1990 [8]. The ion concentration of simulated body fluid is nearly equal to that of human blood plasma. Hence in vitro studies in SBF have become very popular to test biocompatibility of bioactive glass materials [9].

In this paper bioactivity of glass-ceramic with composition Na_2O-Li_2O-SrO-CaO-B_2O_3-SiO_2-P_2O_5 has been evaluated. In vitro studies were carried out by examining the formation of hydroxycarbonate apatite layer on their surfaces when treated in SBF.

II. MATERIALS AND EXPERIMENTAL PROCEDURES

A. Preparation of Glass

Glasses with compositions $8Na_2O$-$8Li_2O$-8SrO-20CaO-$40B_2O_3$-$10SiO_2$-$6P_2O_5$ and $8Na_2O$-$8Li_2O$-8SrO-20CaO-$46B_2O_3$-$10SiO_2$ in Wt% were prepared by traditional melt-quench method. The analytical grade chemicals were weighed in appropriate amount and mixed in an agate pestle and mortar and were taken into porcelain crucibles. The samples were heated at 1100^oC in an electrical muffle furnace. The melts were poured on a preheated stainless steel plate and then pressed with steel rod for quenching.

B. Preparation of SBF

Simulated body fluid (SBF) was prepared by dissolving the appropriate portions of reagent grade components NaCl, $NaHCO_3$, KCl, $K_2HPO_4.3H_2O$, MgCl2.6H2O, $CaCl_2$, Na_2SO_4 in ion exchanged distilled water according to the method proposed by kokubo et al,[10]. It was buffered at pH of 7.4 with appropriate amount of tris-hydroxymethyl aminomethane and hydrochloric acid.

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C. In Vitro Bioactivity Test

The assessment of in vitro bioactivity of the prepared sample were tested with the help of simulated body fluid (SBF) . The samples were taken into plastic container. SBF was taken according to the ratio Sa/Vs approximately $10cm^{-1}$ [10]. The samples were kept in incubation chamber at temperature 36.5^oC. The samples soaked in SBF were kept in incubation chamber for 7 days. After 7 days the samples were removed from the incubator and cleaned gently with 100% ethanol and then with distilled water [11]. The samples were left to dry at ambient temperature in a dessicator. The dried samples were analysed by using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) unit attached to the SEM.

III. RESULTS AND DISCUSSION

The X-ray diffraction (XRD) pattern of prepared glass samples before soaking in SBF are shown in fig 1 and fig 2.The XRD results reveals the amorphous nature of glass ceramics.

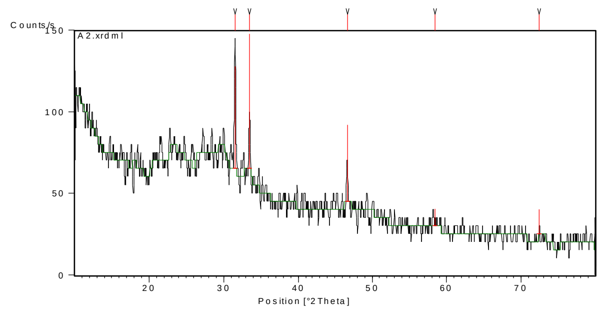

Fig. 1 XRD spectrum of glass sample $8Na_2O$-$8Li_2O$-8SrO-20CaO-$40B_2O_3$-$10SiO_2$-$6P_2O_5$

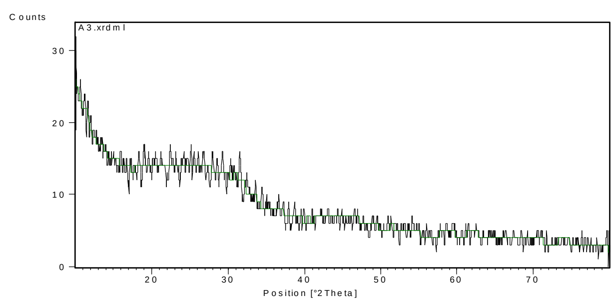

Fig. 2 XRD spectrum of glass sample $8Na_2O$-$8Li_2O$-8SrO-20CaO-$46B_2O_3$-$10SiO_2$

The surface morphology of the samples after incubation treated in SBF for 7 days was assessed by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) profiles at an accelerating voltage of 15kV. The micrograph provides visual evidence of the formation of an apatite layer on the surface of a glass samples. Fig 3 Shows the SEM micrograph of bioactive glass sample of composition $8Na_2O$-$8Li_2O$-8SrO-20CaO-$40B_2O_3$-$10SiO_2$-$6P_2O_5$. Fig 4 Shows the SEM micrograph of bioactive glass sample of composition $8Na_2O$-$8Li_2O$-8SrO-20CaO-$46B_2O_3$-$10SiO_2$.

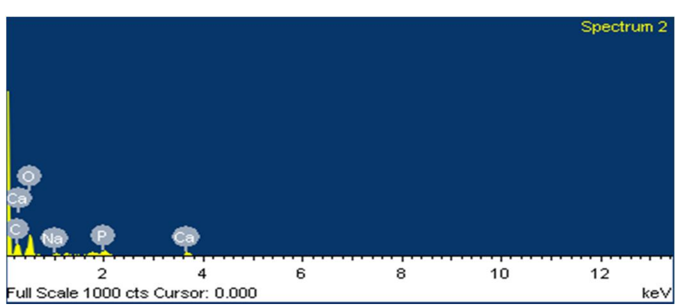

Fig. 3 SEM micrograph of glass sample $8Na_2O$-$8Li_2O$-8SrO-20CaO-$40B_2O_3$-$10SiO_2$-$6P_2O_5$

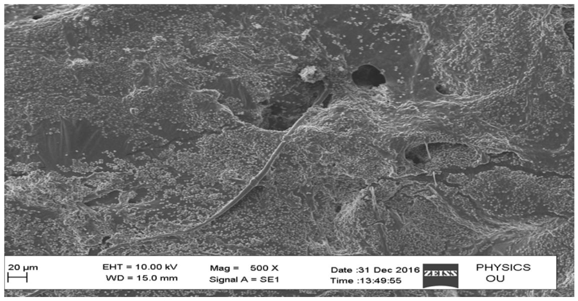

Fig. 4 SEM micrograph of glass sample $8Na_2O$-$8Li_2O$-8SrO-20CaO-$46B_2O_3$-$10SiO_2$

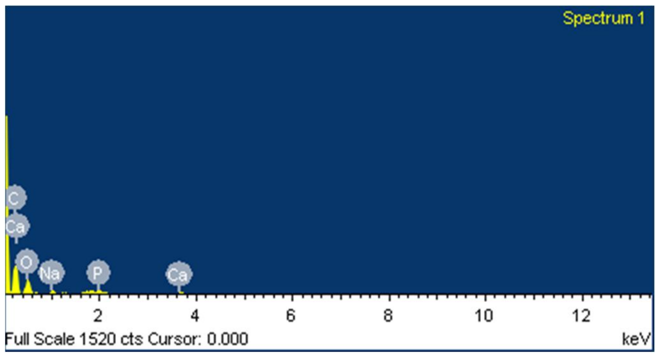

Fig. 5 EDS data of glass sample $8Na_2O$-$8Li_2O$-8SrO-20CaO-$40B_2O_3$-$10SiO_2$-$6P_2O_5$

The EDS analysis of synthesized glass samples reveals the formation of a Hydroxyapatite layer on the surface of glass samples after immersion in SBF for 7 days. The precipitates on the surface of the sample are made up of calcium and phosphorous. Micro analysis of the precipitates reveals the presence of small quantities of Na, P, Ca, C, O as shown in EDS . It may be concluded that the surface layer forms hydroxyapatite layer.

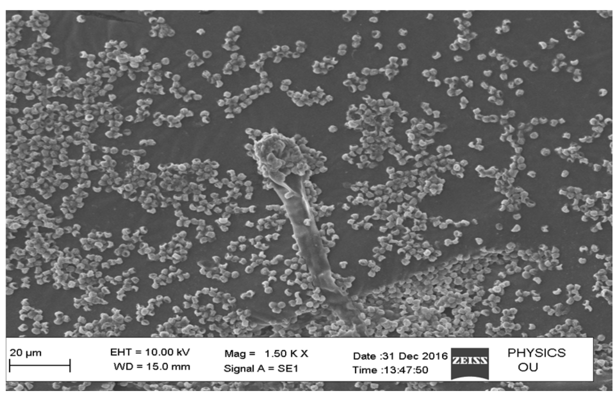

Fig. 6 EDS data of glass sample $8Na_2O$-$8Li_2O$-8SrO-20CaO-$46B_2O_3$-$10SiO_2$

IV. CONCLUSIONS

The in vitro bioactivity in the glass samples $8Na_2O$-$8Li_2O$-8SrO-20CaO-$40B_2O_3$-$10SiO_2$-$6P_2O_5$ and $8Na_2O$-$8Li_2O$-8SrO-20CaO-$46B_2O_3$-$10SiO_2$ were examined. The amorphous nature of the samples analysed using XRD before soaking in SBF. The hydroxyapatite forming ability of LiO2 has been investigated. The surface morphology was analysed using SEM micrograph and EDS data. SEM and EDS data revealed the biocompatibility of the synthesized samples. The Ca/P ratio values are in good agreement with biological apatite.

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