Fabrication and Characterization of Bioglass

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Abstract - **Glasses are common in use now days. These are used in different applications like domestic, automobile, telecommunication etc. The glasses are very useful materials because of their impressive properties. Few years back a new generation of glasses were developed i.e. bioactive glasses and bioactive glass ceramics. The glasses are used for bone grafting now-a-days because of their impressive bioactive properties. These glasses have tendency to form bonds with the living tissue organs. The future of these glasses will be bright in dental, orthopedics and prosthetic applications. In the present work borosilicate glasses of different compositions have been studied. The different elements were added with appropriate mol% to compose a new bioglass composition. The samples were prepared by melt quench route. The samples were immersed for 21 days in SBF. The samples were characterized before and after immersion in SBF by different techniques. The XRD technique was done to confirm the amorphous nature of glass before immersion and after immersion. The SEM and EDX were done to check the changes on the surface after immersion. The sample S1 has better biocompatibility results than S2 andS3. The formation of apatite on the glass samples were confirmed by all techniques mentioned above.**
Keywords: **Bioactive glass, Characterization, Orthopaedic applications.**

I. INTRODUCTION

The bioglasses are highly promising materials which are used now days because of these have tendency to bonds with living tissue organs [1].These materials have tendency to generate apatite on their surface after exposed to the SBF or human body plasma. The bioglass of composition elements silica (SiO_2), sodium (Na), calcium (Ca) and phosphorous (P) oxides was prepared by Hench for the first time and named as 45S5 bioglass. The 45S5 bioglass was prepared with 45% of silica(Si), 24.5% of sodium oxide (Na_2O),   24.5% of calcium oxide(CaO) and 6% phosphorous pent-oxide (P) (in weight%) composition elements [2]. In later the additions were done to the compositions to enhance the various properties of the bioglass.The bioactivity of bioglasses is concerned with the apatite generation on their surfaces when exposed to the biological fluids[3]. The generation of apatite may be of form HA (hydroxyapatite) or HCA (hydroxyl carbonate apatite). The apatite generation on their surface after exposure to the biological fluids make them useful in dental and orthopedic (such as bone replacements and bone regenerations) applications [5]. The apatite layer is generated due to several reactions taken between the biological fluids and the bioactive glass [6].The apatite generation on the surface of bioglass depends upon various

factors like composition of glass, structure of glass, temperature at which it is prepared and solution used for immersion etc. [6, 7, 8]. The changes in compositional elements can affect the properties of bioglass and its melting conditions [9, 10]. The melting temperature for bioglasses is taken as higher i.e. above 1400°C but the 45S5 is fabricated at lower temperature (1300°C) and longer time period [11].Thus, the work is done to develop a new compositions based upon silica, borate, sodium, calcium and phosphorous oxide system. The melting temperature is taken as lower. The apatite generation was tested after immersion in tris-buffer solution. Table I Composition of samples (at%.)

II. EXPERIMENTAL PROCEDURE

A. Sample Preparation

The powders of reagent grade were taken for the preparation of samples. Silicon dioxide (Si), borate (B), calcium carbonate (C), sodium carbonate ($Na\ CO_3$), and phosphorous pentoxide (P) were used to produce three different compositions of bioglass, which are described in Table I.

TABLE I THREE DIFFERENT COMPOSITIONS OF BIOGLASS

Samples			CaO		
S1	40	10	20	25	5
S2	35	15	20	25	5
S3	30	20	20	25	5

The total weight of each powder mixture was 25 gm. The powder mixtures were mixed and homogenized well with an agate mortar. Each powder was melted in an alumina crucible at 1350°C. The borate and silica content were varied to check the effects of both on apatite generation. Each sample was initially placed inside the furnace at the temperature of 1000°C for half an hour to evaporate the moisture content from the melt. Then the temperature was increased to 1200°C and held for half an hour to escape all the gasses from it. Finally the temperature of melt was held at the temperature of 1350°C and kept for half an hour to make it more uniform and defect free. The melt of samples were poured in a graphite mold and then annealed at 500°C for 5 hours in another electric furnace to remove thermal stresses. The each sample was taken out from the furnace, when the furnace was off and having same temperature as

atmosphere. The mold was pre heated at the annealing temperature, because it can cause thermal shock to samples when there is a huge temperature difference between melt and mold. Samples were taken out from the furnace when it has been cooled down to the atmospheric temperature, because slow cooling of glasses will allow it to settle well.

B. In-Vitro Testing

The in-vitro testing of bioglass samples was done with tris buffer solution for 21 days immersion. The temperature of the solution was kept at 37°C. the pH of tris-buffer solution was 7.1 before immersion of samples.

C. Sample Characterization

The characterization of samples was done before and after immersion to check the behavior of samples in the solution.

D. X-Ray Diffraction (XRD)

XRD – (X'Pert Pro Panalytical PN – 3040/80 Almelo, Netherlands) was used for the XRD analysis of the samples. The Cu Kα radiation was (λ=1.540560) and the 2θ values were taken in the range of 20° to 80°. The speed of scanning was 2° per minute for each sample. The inter planner distances can be calculated by using the Bragg's formula.

E. Scanning Electron Microscopy (SEM)/ Energy Dispersive Spectroscopy (EDS)

In the proposed work testing was performed on SEM (JEOL JSM – 6610LV, A KISHIMA, JPN) machine. The samples were coated with conductive layer for this purpose.Both SEM and EDX were done before and after immersion in SBF at same magnification

III. RESULTS AND DISCUSSION

A. pH Measurements

The pH of samples was recorded at the interval of 72 hrs repeadeatly. The total incubation time of imersion was 21 days (504 hrs). Initially the pH values were recorded decreased from 7.1 to 6.3 upto 220 hrs of immesion. After that the values incresed gradually between 6.8 to 7.2 upto 432 hrs of immersion and then decreased as shown in figure 1.

Initially the attack of $H_3^-O^-$ and OH^- ions on the bioglass takes place and then bioglass releases Ca^{2+} and Na^{2+} ions to the solution. The alkali ion addition to the SBF solution will cause increase in pH values. The release of sodium ions to the SBF exhibits in initially stages, that's why the pH of the solution decreased rapidly in initial stages.
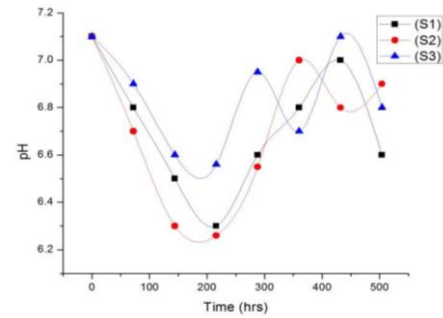

Fig. 1 pH values of SBF at different intervals

After that the solution pH values increases and it also increases the kinetics of formation of Ca layer and apatite formation on the surface of the bioglass [12].

B. XRD Analysis

1. Before Immerson: The XRD data patterns shown that the all samples have clear and amorphous structure when melted at lower temperature. XRD data of all samples before and after immersion are showen in figures 2, 3 and 4. The sharp peaks were absent in the diffraction data patterns in all three samples before immersion. The amorphous hump was recorded in all samples before immersion. The amorphous hump of S1, S2 and S3 samples were recorded between 23° to 35°.

2. After Immersion: The sharp peaks were appeared after immersion in SBF at 23.7°, 31.9°, 34° and 72° in S1, S2 and S3 XRD diffractin data patterns as shown in figures 3, 4 and 5. The peaks in S1, S2 and S3 XRD diffractin data confirmed the apatite generation on the surfaces of the samples because these are caused by reactions in SBF [13].
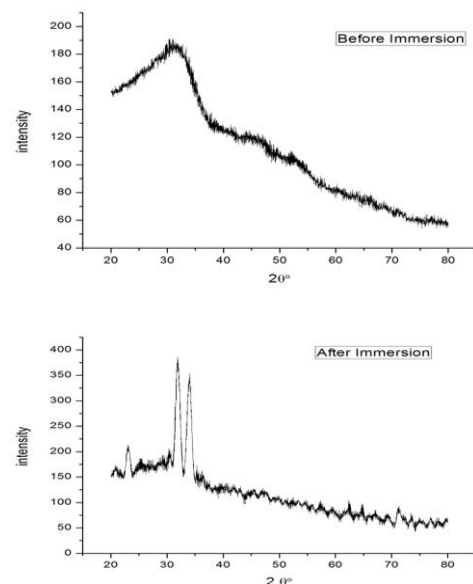

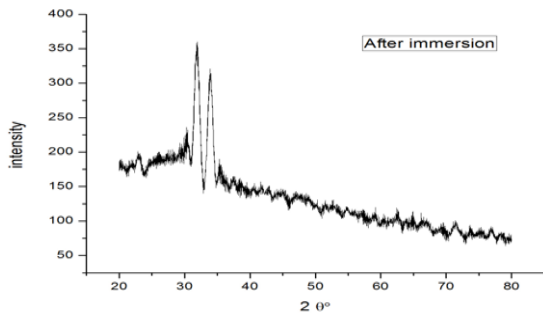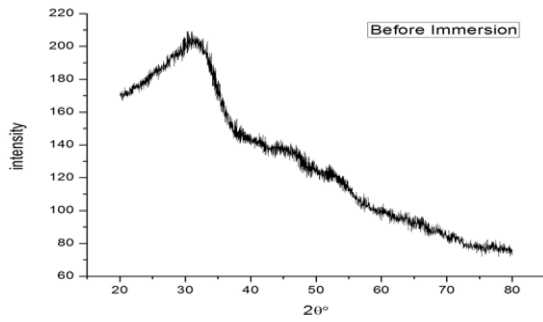Fig. 2 XRD patterns of S1 before and after immersion

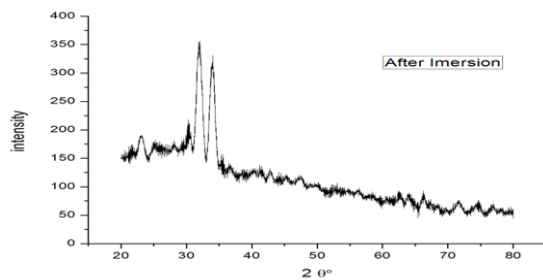Fig. 3XRD patterns of S2 before and after immersion
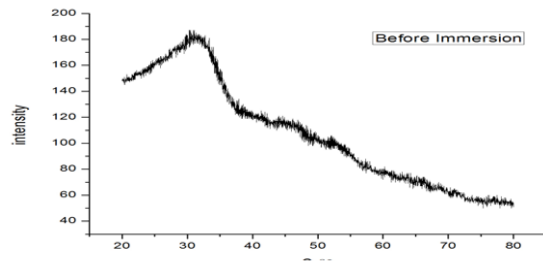

Fig. 4 XRD patterns of S3 before and after immersion

C. SEM Analysis

The changes were noticed by both SEM and EDX surface identification techniques. The results of samples S1, S2, S3 were recorded as positive index of apatite generation.

1. Before Immersion: The surfaces of the samples were clearly amorphous before immersion as shown in the figures 5, 6 and 7. All glass samples have clean surface and there is no aptite layer present on the surfaces of the samples. The samples are amorphous and the EDX data shown that the Ca/P ratio of S1, S2 and S3 samples is 2.37, 2.32, 2.41and respectively.
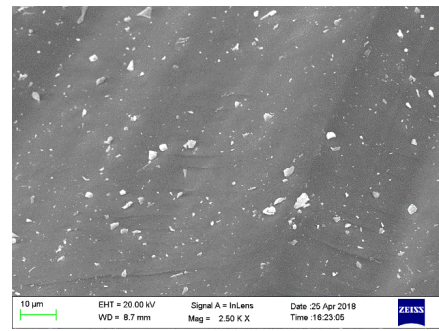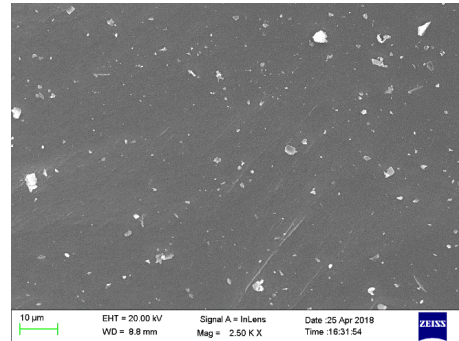

Fig. 5 SEM image of sample S1 before immersion
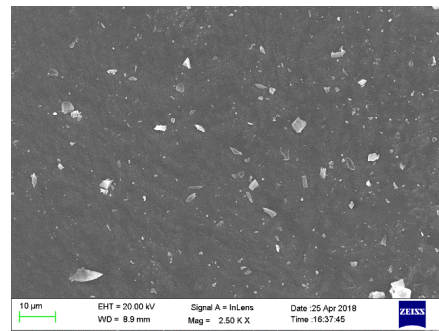

Fig. 6 SEM image of sample S1 before immersion

Fig. 7 SEM image of sample S1 before immersion

2. After Immersion : The apatite generation after immersion is appeared on the surfaces of S1, S2 and S3 after 21 days of immersion. The EDX reports also stated the change in Ca/P ratio after immersion. The Ca/P ratio of the bioglass should be higher than 1 to use in biomedical applications [13]. All the samples has Ca/P ratio more than 1 before immersion. All the samples shown that the apatite layer begins to be appeared after immersion but did not covered the entire surface as shown in figures 8, 9 and 10.
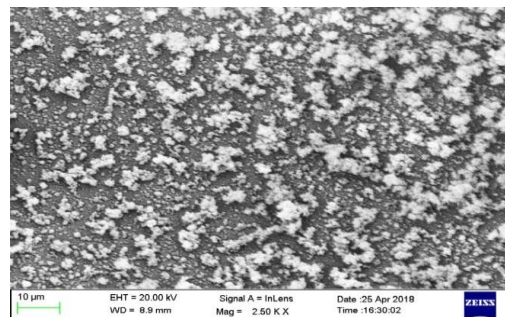

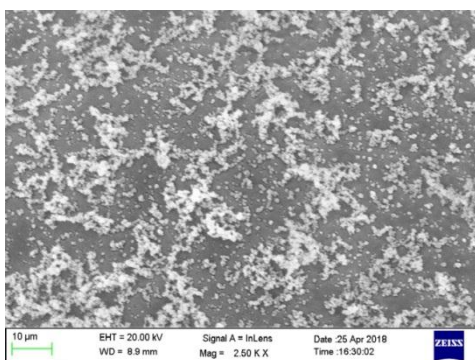Fig. 8 SEM image of sample S1 after immersion
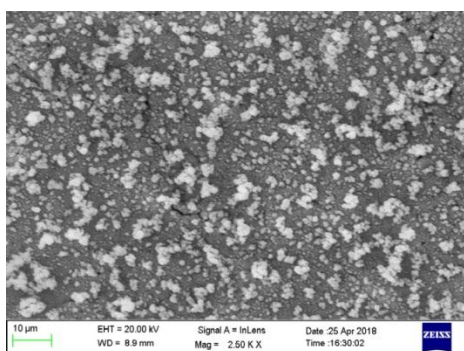
Fig. 9 SEM image of sample S2 after immersion

Fig. 10 SEM image of sample S1 after immersion

The Ca/P ratio values of All samples before and after immersion were also different as shown below in the table 2 below;

TABLE II CA/P RATIO BEFORE AND AFTER IMMERSION

Sample	Ca/P Ratio before immersion	Ca/P Ratio after immersion
S1	2.37	2.82
S2	2.32	2.59
S3	2.41	2.67

The glass has higher Ca/P ratio has higher tendancy to form Ca layer which is useful in biomedical applications than lower Ca/P ratio after immersion [14].

VI. CONCLUSION

In this present work three bioglass samples were prepared with mol% variability of SiO_2 and B_2O_3 to test their activeness in SBF. The different testing techniques SEM/EDX, XRD, pH measurements, were performed for characterization. The samples were prepared using melt quench route. The samples were soaked in tris buffer solution for 21 days to check pH measurements due to reactions between samples and SBF. The SEM, XRD and EDX were reported before and after immersion of samples.

The apatite generation was founded on the surfaces of S1, S2 and S3 samples after 21 days of immersion. The changes on XRD data patteren clued the change of amorphous to crystalinity of samples. The SEM and EDX reports further confirmed the apatite generation on surfaces. The bioglass having 10% silica and 40% in mol% (S1) borate content has more ability to form apatite layer on surface than S1 and S2.

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