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Role of SrO on the bioactivity behavior of some ternary borate glasses and their glass ceramic derivatives

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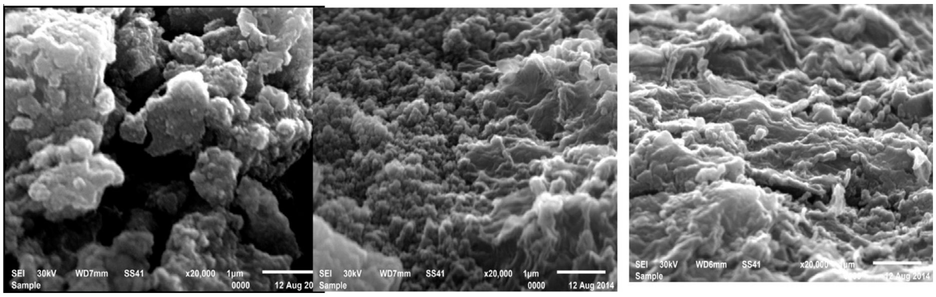
HIGHLIGHTS

- Borate glasses containing SrO substituting both CaO and NaO were prepared.
- Glass ceramic derivatives were prepared by thermal heat treatment process.
- Prepared glass and glass ceramics characterized for their bioactivity or bone bonding ability.
- Hydroxyapatite layer was formed after immersion in phosphate solution.

GRAPHICAL ABSTRACT

SEM of selected glass–ceramic samples after immersion in phosphate solution for two weeks.



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ABSTRACT

Borate glasses containing SrO substituting both CaO and NaO were prepared and characterized for their bioactivity or bone bonding ability. Glass ceramic derivatives were prepared by thermal heat treatment process. FTIR, XRD and SEM measurements for the prepared glass and glass–ceramics before and after immersion in sodium phosphate solution for one and two weeks were carried out. The appearance of two IR peaks within the range 550–680 cm^{-1} after immersion in phosphate solution indicates the formation of hydroxyapatite or equivalent Sr phosphate layer. X-ray diffraction data agree with the FTIR spectral analysis. The solubility test was carried out for both glasses and glass ceramics derivatives in the same phosphate solution. The introduction of SrO increases the solubility for both glasses and glass ceramics and this is assumed to be due to the formation of Sr phosphate which is more soluble than calcium phosphate (hydroxyapatite). SEM images reveal varying changes in the surfaces of glass ceramics after immersion according to the SrO content.

1. Introduction

Boron plays an important role in bone growth, so the addition of boron as borate glass is one of recent continuous interest research area in biomedical applications [1–4]. The poor chemical durability of borate glasses must be taken into consideration to avoid the toxicity of high release of boron. So, the chemical composition and bioactivity of borate glasses will be calculated well to prevent the rapid release of boron.

Pan et al. [1] have found that the incorporation of strontium into borate glass moderates the rapid release of boron and also induces the adhesion of osteoblast – like cells, $SaOS^{-2}$.

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The degradation mechanism within borate glass and 45S5 bioglass is completely different, where in Hench glass a porous SiO_2 – rich gel layer is formed due to the leaching of alkaline ions which is important for the apatite precipitation [2]. But in borate glass degradation there is no SiO_2-rich layer, thus facilitating the formation of the apatite [3] due to the rapid release of all the glass constituents.

Margh and Abdelghany [4] have prepared and characterized the bioactivity of ternary borate glasses and their glass–ceramics derivatives from the system $Na_2O{\cdot}CaO{\cdot}B_2O_3$ together with soda-lime borate samples containing 5 wt% of MgO, Al_2O_3, SiO_2 and P_2O_5. They have indicated and confirmed the formation of hydroxyapatite in the pure borate glass together with the glasses containing SiO_2, P_2O_5 and Al_2O_3 after immersion in phosphate solution.

Abdelghany et al. [5] have established the ability of the ternary borate glasses of the system ($B_2O_3{\cdot}CaO{\cdot}Na_2O$) together with substituted Li_2O or K_2O to form hydroxyapatite after the immersion in a sodium phosphate solution for a week.

Liu et al. [6] have assumed that the degradation of bioactive borate glass in an aqueous phosphate solution is accompanied by the release of ions, such as those of boron, and network modifier such as Na & K due to the degradation of the B—O bonds in the glass network resulting from the continuous and complete conversion of the glass to HA.

The objective of this study is to investigate the mechanism of converting some sodium–calcium–borate glasses containing different amounts of strontium oxide replacing both sodium oxide and calcium oxide to HA or similar strontium phosphate phases in aqueous phosphate solution at room temperature for 1 and 2 weeks. Structural and compositional changes were characterized by FTIR, X-ray and SEM measurements for the original glasses. The conversion of the studied glasses to their corresponding glass–ceramics by controlled heat treatment was carried out. The same process of the immersion in phosphate solution was repeated out for the prepared glass–ceramic derivatives. A final aim of the work is to compare the bioactivity behavior of the prepared glasses and their corresponding glass–ceramic derivatives together with clarifying the role of SrO.

2. Materials, preparation of glasses

All the glasses were prepared using chemically pure materials. The materials include H_3BO_3 for B_2O_3, Na_2O and CaO were introduced in the form of their respective anhydrous carbonates. Strontium oxide was also introduced as heavy strontium carbonate. The composition of prepared glasses is shown in Table 1.

The accurately weighed batches were introduced into platinum crucibles and were calcined at 500 °C for 1/2 h to remove water then the melting was carried out in an electric furnace at 1150 °C for 2 h. Homogenization was achieved by rotating frequently the melts. The melts were cast into preheated stainless steel moulds of the required dimensions. The prepared glass samples were immediately transferred to a muffle furnace regulated at 400 °C for annealing. The annealing muffle after 1 h was left to cool to room temperature at a rate of 30 °C/h.

Table 1
Chemical composition of prepared glass.

Glass No.	Na_2O (wt%)	CaO (wt%)	B_2O_3 (wt%)	SrO (wt%)
G-1	20	20	60	–
G-2	17.5	17.5	60	5
G-3	15	15	60	10
G-4	12.5	12.5	60	15
G-5	10	10	60	20

2.1. Infrared absorption measurements

The FT Infrared absorption spectra before and after immersion were measured at room temperature in the range 4000–400 cm^{-1} by an infrared spectrophotometer type (Nicolet-*is*10 spectrophotometer, USA), using the KBr disc technique. Two milligrams of powdered glass was mixed with 200 mg of KBr and the mixture was subjected to a load of 5 $tons/cm^2$ to produce clear homogeneous discs. The infrared absorption spectra were measured immediately after preparing the desired discs. The same measurements were repeated for the samples after immersion for 1 or 2 h. The IR measurements were also carried out for the prepared glass–ceramics before and after immersion in the phosphate solution.

2.2. Differential thermal analysis

Differential thermal analysis (DTA) measurements were carried out on powdered glass samples which were examined up to 1000 °C using a powdered alumina as a reference material. A DTA Perkin Elmer DTA-7 apparatus was used in this study. The DTA data was used to find out the proper heat treatment temperatures to obtain the appropriate glass–ceramic derivatives with high micro-crystallinity. Briefly, the values obtained for glass nucleation temperatures and the onset of crystallization temperatures are given in Table 2.

2.3. Heat treatment regime (conversion to glass ceramics)

The glass samples were thermally treated in two step regime at the mentioned temperatures (Table 2). Each glass was heated slowly to the first nucleation temperature (5 °C/min) for the formation of sufficient nuclei sites and after holding for 6 h, was then further heated or raised to reach the second chosen crystal growth temperature, and after holding also for another 6 h, the specimen was left to cool inside the muffle to room temperature at a rate of 20 °C/h.

2.4. X-ray diffraction measurements

The heat treated glass–ceramics specimens were analyzed by X-ray diffraction in order to identify the crystalline phases that precipitated within these samples during the heat treatment and conversion to glass–ceramic derivatives. The glass–ceramic samples were ground and the fine powder was examined using a diffractometer adopting Ni-filter and Cu-target. The X-ray diffraction patterns were obtained using a Philips PW 1390 X-ray diffractometer. The diffraction patterns were identified with comparison with standard ASTM cards and published related data.

2.5. Morphological analysis using scanning electron microscopy

Scanning electron microscopic (SEM) investigations were carried out on glass samples at room temperature using an SEM model JEOL-840A attached with EDAX unit, accelerating voltage 30 kV. All

Table 2
Heat treatment scheme of conversion to glass–ceramics.

Glass No.	Nucleation temperature (°C)	Holding time (h)	Crystal growth temperature (°C)	Holding time (h)
G-1	525	6	650	6
G-2	530	6	650	6
G-3	535	6	660	6
G-4	540	6	660	6
G-5	550	6	670	6