

Structural Properties of Mesoporous Borosilicate Bioactive Glass

N. S. Ramli^a, E. S. Sazali^{*}, F. M. Noor^b, N. A. N. N. Malek^c and S. K. Md. Zain^d

*Advanced Optical Materials Research Group, Department of Physics,
Faculty of Science, Universiti Teknologi Malaysia, 81310 UTM Skudai,
Johor, Malaysia.*

Email: nadiashafiraramli@gmail.com^a, ezzasyuhada@utm.my^{},
faizani@utm.my^b, niknizam@utm.my^c, ejakelly92@gmail.com^d*

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Abstract. A series of mesoporous borosilicate bioactive glasses (MBBG) with compositions of $(x)\text{B}_2\text{O}_3-(80-x)\text{SiO}_2-15\text{CaO}_2-5\text{P}_2\text{O}_5$ where $x = 0, 5, 10,$ and 15 mol% are functionalized using a combination of the sol-gel method and evaporation-induced self-assembly (EISA) process, along with a calcination temperature of 600°C at 7 hours. The amorphous nature of the glass is determined using an X-ray diffractometer (XRD) and, the structural properties are characterized using a Fourier Transform Infrared (FTIR) spectroscopy in the range of 400 to 4000 cm^{-1} . XRD spectra confirm that all the glass samples are amorphous. FTIR spectrum exhibited three major bands attributed to identifying chemical bonds in a molecule by producing an infrared absorption spectrum with varying percentages of boron oxide. The appearance of three distinct primary bands corresponding to the Si–O–Si bond vibration modes. The B–O symmetric stretching vibrations refer to the band at $1400 - 1500\text{ cm}^{-1}$.

Keywords: Mesoporous Borosilicate Bioactive Glass, EISA, XRD, FTIR

I. INTRODUCTION

Bioactive glass (BG) is a material made from amorphous silicate that is highly compatible with human limbs, binds bones, and is also able to stimulate the growth of unused bone when dissolved over time [1]. Hench *et al.* invented bioactive glasses in 1969 and they are a set of reactive materials that can bind to mineralized bone tissues in a physiological context [2]. Because of its high specific surface area, massive pore depth, and mesoporous composition, mesoporous bioactive glass (MBG) was recently synthesized and found to have improved bone-forming bioactivity, oxidation, and drug delivery properties as compared to traditional BG. As a result, MBG can be used as a bioactive material in the regeneration of bone tissue [3].

Previously, by using a mixture of surfactant templating, sol-gel techniques, and EISA processes, a series of alkali-free borosilicate glasses with mesopores resulted in short-ordered wormholes was successfully prepared. The mesoporous structure has greater stability in samples with lower boron contents [4].

However, one significant impediment to the development of new BG based on borosilicate occurs due to our lack of relevant understanding of the basic composition and structural drivers that regulate the kinetics of borosilicate dissolution in body fluids [5]. Ouis *et al.*, examined a borate glass, called 45S5B1, with the same composition as 45S5 but substitute for B_2O_3 for all SiO_2 , which found that the borate glass surface was hydroxyapatite (HA) layered on the dipotassium phosphate (K_2HPO_4) solution at $37^\circ C$ after immersion [6]. In addition, the development of the HA layer on borate glass is faster than on glass 45S5 [7]. An apparent change as boron content increases, the arrangement becomes more disordered, indicating that the mesoporous structure partly collapses [4]. These characteristics make such bioactive glasses produced from borosilicate are among the capable candidates for treating a variety of skeletal and nonskeletal biomedical issues.

II. EXPERIMENTAL

A series of mesoporous borosilicate bioactive glass with a composition of $(x)B_2O_3-(80-x)SiO_2-15CaO_2-5P_2O_5$ where $x=0, 5, 10,$ and 15 mol% with temperature calcination of $600^\circ C$ for 7h have been prepared by sol-gel with evaporation-induced self-assembly EISA method. 8 g of the P_{123} is dissolved in 120 g of ethanol, boric acid (H_3BO_3), calcium nitrate tetrahydrate ($Ca(NO_3)_2 \cdot 4H_2O$), and triethyl phosphate (TEP) in a fume hood at room temperature. The sample was intensely mixed then transferred to a petri dish and kept at $35^\circ C$ under a fume hood for 1 day. The gel sample was aged at $60^\circ C$ for another day after 1 day of EISA handling. The dry gel sample was then be calcined to induce a white solid form of mesoporous bioactive glass. The amorphous nature of mesoporous borosilicate bioactive glass (MBBG) is confirmed by the X-ray diffraction pattern from the Rigaku SmartLab High-Resolution X-Ray Diffractometer equipped with $CuK\alpha$ radiation with a wavelength in the 2θ range of $15-35^\circ C$ and 0.02 step size. The structural properties of MBBG have to be tested by Fourier Transform Infrared (FTIR). The FTIR spectrum was acquired using a pelletized sample produced with KBr at a ratio of 1:100 and a resolution of 4 cm^{-1} .

III. RESULTS AND DISCUSSION

The mesoporous borosilicate bioactive glass (MBBG) samples with compositions with different percentages of boron are naming as MBBG 0, MBBG 5, MBBG 10, and MBBG 15, respectively. Figure 1 depicted the X-ray diffraction pattern for MBBG 0, MBBG 5, MBBG 10, and MBBG 15 samples, showing the wide-angle XRD pattern of calcined MBBG. A strong halo apparent between 15° and 35° indicates that the sample is amorphous [8]. A wide-angle XRD analysis confirms that the calcined glasses are amorphous, with no diffraction peaks visible except a large band [9].

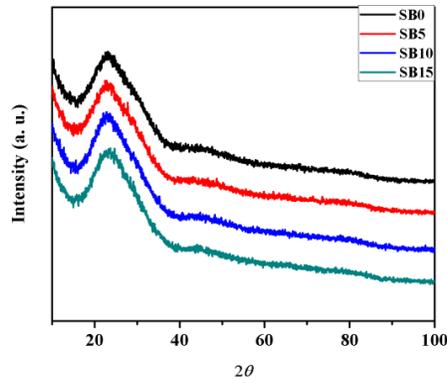


FIGURE 1. The X-ray diffraction pattern of MBBG 0, MBBG 5, MBBG 10, and MBBG 15 samples with different percentages of boron.

The FTIR spectra of the MBBG with various percentages of mol of boron oxide are shown in Figure 2. The 400 to 4000 cm^{-1} range was observed for MBBG that were calcined at 600°C with varying percentages of boron oxide. The O–H stretching vibration, which causes absorption between 3710 and 3300 cm^{-1} , has been discovered and is thought to be linked to adsorbed water [10]. The appearance of three different primary bands corresponding to separate Si–O–Si bond vibration modes [11] At the first peak level, at 470 cm^{-1} , it corresponds to the rocking vibration from the Si-O-Si bending mode. Si-O symmetrical voltage vibrations are allocated directly in the second path at 800 cm^{-1} . Si - O - Si asymmetric stretching mode represents the third peak, which is about 1650 cm^{-1} [12]. The B–O symmetric stretching vibrations become sharper according to the increase of boron percentage that refer to the band at $1400\text{--}1500\text{ cm}^{-1}$ [13]. The existence of stretching modes of C-O-C on P_{123} and O-H on P_{123} [14].

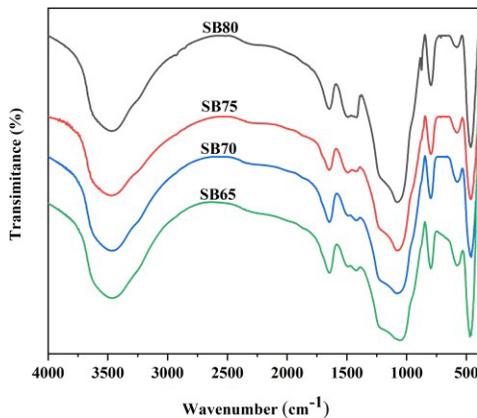


FIGURE 2. The MBBG's FTIR spectra pattern of MBBG 0, MBBG 5, MBBG 10, and MBBG 15 samples with different percentages of boron.

Figure 3 shows the deconvolution of FTIR spectra of MBBG samples. Based on the data that have been reported in Table 1, a Gaussian shape consistent with the FTIR band has been observed. The wide vibration band located at 1647 to 1651 cm^{-1} corresponds to the tensile vibration of the H - O of H_2O bond absorbed in the material or silanol (SiO - H) group. Water removal causes the loss of H - O bonds when the temperature is increased to a higher level [12]. The carbonate path is more abundant at MBBG 0 and MBBG 5 at 1008 and 968 cm^{-1} . The strip at 1498 to 1521 cm^{-1} corresponds to the symmetrical stretching vibration of the B-O unit $[\text{BO}_3^{3-}]$. The appearance of the $[\text{BO}_3^{3-}]$ band shows that $[\text{BO}_3^{3-}]$ has partially replaced $[\text{SiO}_4^{4-}]$ sites in the glass structure, especially when considering that increasing the boron content in MBBG, at the expense of CHA, improves the look of this material. [10]. This identifies the existence of boric acid in the MBBG with increased boron concentration, as seen by the wide-angle X-ray powder diffraction results presented above [4]. The presence of more C-O bands compared to the other powders in MBBG 15 is consistent with the XRD findings in the presence of hydroxyapatite and calcium carbonate phases [13]. A small band located at a distance of 460 cm^{-1} corresponding to the Si-O-S bending vibration mode in the SiO_4^{4-} group shows the presence of amorphous silicates [14]. The Si-O band located at 799 cm^{-1} is visible, given Si-O along with two non-bridging oxygen per tetrahedron $\text{SiO}_4(\text{Si-O-2NBO})$ called group Q^2 [15] and at 1070 - 1081 cm^{-1} determined the symmetrical stretch of the Si-O group in a silicate tetrahedron where it is characterized by the formation of a silicate-rich phase [16].

TABLE 1. The assignment of the different vibrational bands from FTIR spectra of samples.

Wavenumber (cm^{-1})				Assignments	Reference
SB0	SB5	SB10	SB15		
461	461	465	469	The rocking vibration from the Si-O-Si unit	[15]
799	795	795	799	Si-O symmetrical voltage vibrations	[16]
1008	968	952	928	Stretching modes of C-O-C on P123	[17]
1081	1070	1070	1074	Si - O - Si asymmetric stretching mode	[18]
1498	1509	1517	1521	B-O symmetric stretching vibrations	[13]
1654	1647	1651	1651	Stretching vibration of the H - O bond of H_2O	[19]

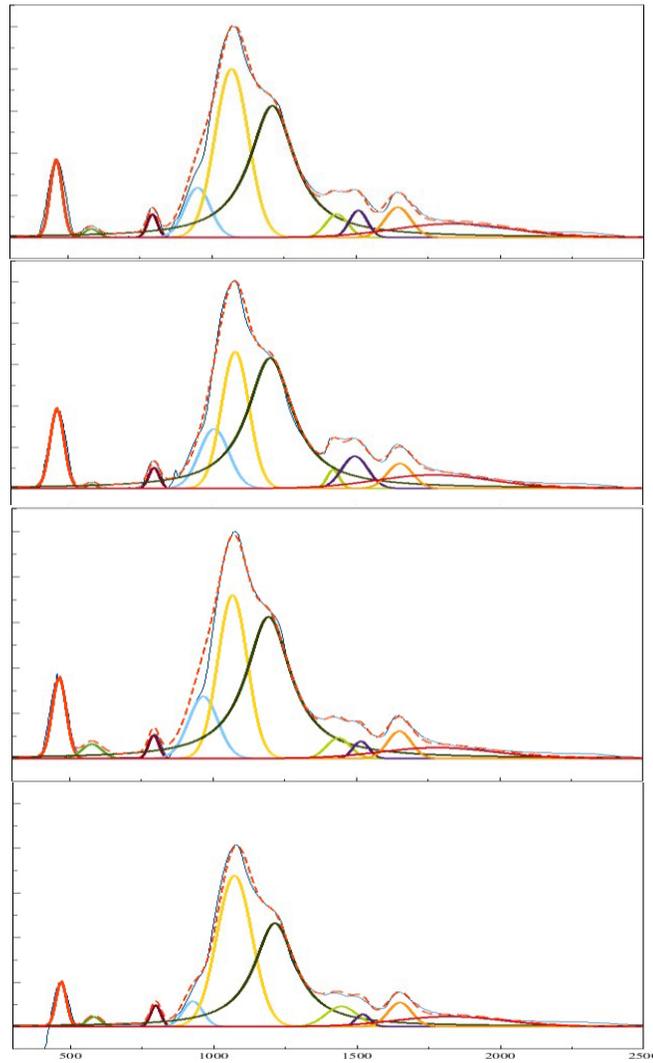


FIGURE 3. Deconvoluted FTIR spectra of the bioactiveglasses for MBBG samples.

IV. CONCLUSION

Mesoporous borosilicate bioactive glass with different various percentages of mol of boron oxide has been prepared by the EISA method. A noticeable difference in the morphology and structure of MBBG may be noticed when boron oxide is added at a different percentage. The results from the X-ray diffraction pattern reveal that all glasses are amorphous, with no diffraction peaks visible except a large band. Besides that, FTIR spectra of the MBBG also resulted in the increased absorbance intensity of symmetrical stretching vibration of the B - O unit from 1498 to 1521 cm^{-1} when the increasing of percentages of mol of boron.

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