



Effect of gamma irradiation on the FTIR of cement kiln dust–bismuth borate glasses



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ARTICLE INFO

Article history:

Received 23 October 2014

Received in revised form 5 February 2015

Accepted 18 March 2015

Available online 11 April 2015

Keywords:

Bismuth borate glasses;

Cement kiln dust;

FTIR;

γ -Radiation

ABSTRACT

Glasses from cement kiln dust and lead barium bismuth borate were prepared by the melt quenching method and studied by using FTIR and ultrasonic techniques before and after exposure to gamma irradiation at doses up to 120 kGy. The FTIR results indicated a deformation of the lead borate based glasses with the replacement of PbO by Bi₂O₃ which was attributed to the appearance of Bi–O stretching vibrations in [BiO₆] structural units and generated excess of non-bridging oxygens. This deformation affected the [BO₃] units which are indicated from the lower decreasing rate of N_4 as a function of Bi₂O₃. The high covalency of Pb–O and B–O bonds compared with that of Bi–O or Si–O attracts the excess of non-bridging oxygens and resulted in a relaxation of the glass network. The relaxation decreased the ultrasonic velocities and the bulk modulus and hence the rigidity of the glass despite the increase of the density which was attributed to the replacement of Bi₂O₃ instead of PbO. The structural and rigidity changes due to composition variations showed a decreasing manner to the effects of radiation. The density of the glass increased while the ultrasonic velocities decreased. This behavior modulated the compositional behavior of the elastic moduli on Bi₂O₃ content.

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1. Introduction

The structure of lead borate glasses had attracted scientific and industrial interests because the network of these remarkable glasses is capable of involving different mixed former/modifier oxides such as SiO₂, Bi₂O₃, CaO, ... [1]. Synthesis of these glasses revealed large glass forming regions and their vitreous network showed a significant variation with concentration depending on the composition of former/modifier oxides [2]. It was reported that heavy metal borate based glasses (HMG) such as PbO and Bi₂O₃ (HMO) are comparable and behaved as conditional glass network formers. Moreover, the glasses have a relatively large vitreous formation range and the forming property of Bi₂O₃ was greater than that of PbO. Moreover, incorporation of PbO or Bi₂O₃ with empty d orbital into borate based glasses colored these with colors that ranged from faint- to dark-yellow or brown, respectively [3,4]. The ions of these elements in vitreous network have a lone pair electron configuration and several stable coordinations with oxygens which enable more than one type of structural unit in the vitreous matrix to be created [5, 6]. It was reported that addition of Bi₂O₃ to lead borate glasses gave high stability against devitrification and chemical inertness due to the dual role of HMO [7,8]. These glasses have high refractive index, high infrared transparency, enhanced optical nonlinearity, and wide

transmission window, are functional for generation of photo-induced second harmonic and can exhibit zero stress optic properties [9]. Moreover, it could be used as laser medium for broadband fiber amplifier since they exhibit very broad luminescence, radiation sensitive dosimeters and calorimeter medium for the superconducting of super collider or as shielding candidates due to its high absorption cross-section for radiation [10].

A category of structural studies has been appointed to explore the influence of modifier/former oxides in lead borate glasses including spectroscopic studies, such as FTIR [1,11], Raman [12] and NMR [13], in addition to neutron diffraction, X-ray diffraction [14] and molecular dynamics simulation [15]. The convenience of spectroscopic studies of these glasses was due to the significant difference between the masses of Pb or Bi and B atoms. This difference allowed for a separation of vibrational modes of HMO polyhedra and the borate structural units [2].

On the other hand, modifier/former oxides can be acquired from cement kiln dust (CKD) which is a pollution problem. This polluter of the environment is a by-product during the normal production of cement industry. Recently, much research has been focused on utilization CKD as the raw material to produce glass and glass–ceramics because vitrification seems to be one of the most promising technological options for establishment of this industrial waste. CKD consists mainly from CaO and SiO₂. Thus, the use of this waste material for glass industry is of great economic technological and scientific importance with proper modulation of the chemical composition [16–19].

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In this work, waste cement kiln dust was used in the manufacture of glassy materials. This type of glass can be considered as a candidate for the production of novel glass resisted radiation when incorporated with oxides such as BaO, PbO and Bi₂O₃ that have high ability to absorb gamma radiation. It was also suggested that a dramatic improvement in reducing toxicity of lead borate based glasses was associated with the substitution of PbO by Bi₂O₃ into the borate network [3]. The use of Bi₂O₃ together with B₂O₃ increases the vitrification of the glasses. Therefore, the present work has been investigating the structural and rigidity properties of lead borate glasses containing CKD and different compositions of Bi₂O₃, by using ultrasonic and FTIR techniques before and after exposure to different doses of gamma irradiation.

2. Experimental procedures

The nominal glass compositions (the starting mixture [Table 1]) were prepared by the conventional melt-quenching method from powders of reagent grades of H₃BO₃, Bi₂O₃, PbO, BaO and cement kiln dust (CKD) according to the chemical formula $x \text{ Bi}_2\text{O}_3 - (40 \text{ B}_2\text{O}_3 - 32 \text{ CKD} - 3 \text{ BaO}) - (25 - x) \text{ PbO}$, ($0 \leq x \leq 25 \text{ mol\%}$) in an amount sufficient to produce 50 g glass. The details of the preparation of the studied glasses were mentioned in a previous work [20]. The amorphous state of the glasses was checked using X-ray diffraction. Philips X-ray diffractometer PW/1710 with Ni-filtered Cu-K α radiation ($\lambda = 1.542 \text{ \AA}$) powered at 40 kV and 30 mA was used. The XRD patterns as shown in Fig. 1 revealed the characteristic broad humps of the vitreous materials and did not reveal discrete or any sharp peaks.

Fourier transformation infrared (FT-IR) spectra for the glass powder were obtained using a spectrophotometer type JASCO, FT/IR-430 (Japan). For this purpose, each sample was mixed with KBr in the proportion of 1:100 (by weight) for 20 min and pressed into a pellet using a hand press. At least two spectra for each sample were recorded in the wavenumber range of 4000–400 cm^{-1} at a resolution of $\pm 1 \text{ cm}^{-1}$ by taking 256 scans. The spectra were corrected for dark-current noise, normalized and curve fitted to get quantitative values for the band areas of heavily overlapped bands. The details of curve-fitting procedure were mentioned elsewhere [21].

The density of each sample was measured by Archimedes' principle by using toluene as the immersion fluid. Four samples of each glass were used to determine the density (ρ). A random error in the density values was found as $\pm 25 \text{ kg m}^{-3}$. The ultrasonic velocities, longitudinal (v_L) and shear (v_T), at room temperature ($\sim 300 \text{ K}$) were obtained using the pulse-echo method. In this method, x-cut and y-cut transducers (KARL DEUTSCH) operated at a fundamental frequency of 4 MHz along with a digital ultrasonic flaw detector (KARL DEUTSCH Echograph model 1085) were used. The uncertainty in the measurement of the ultrasonic velocity is $\pm 10 \text{ m/s}$. The two velocities besides the density were utilized to determine two independent second-order elastic constants, L and G . For pure longitudinal waves $L = \rho v_L^2 Z$, and for pure transverse waves $G = \rho v_T^2 Z$. The elastic bulk modulus (K_e), and Young's modulus (Y) can be determined using the standard relations adopted in previous work [20]. The uncertainty in the measurement of the elastic moduli is $\pm 0.15 \text{ GPa}$.

Table 1

The glass compositions for BaO–B₂O₃–Bi₂O₃–PbO–CKD glasses (mol.%).

Sample	Composition (mol.%)				
	BaO	B ₂ O ₃	Bi ₂ O ₃	PbO	CKD
G0	3	40	0	25	32
G1	3	40	5	20	32
G2	3	40	10	15	32
G3	3	40	15	10	32
G4	3	40	20	5	32
G5	3	40	25	0	32

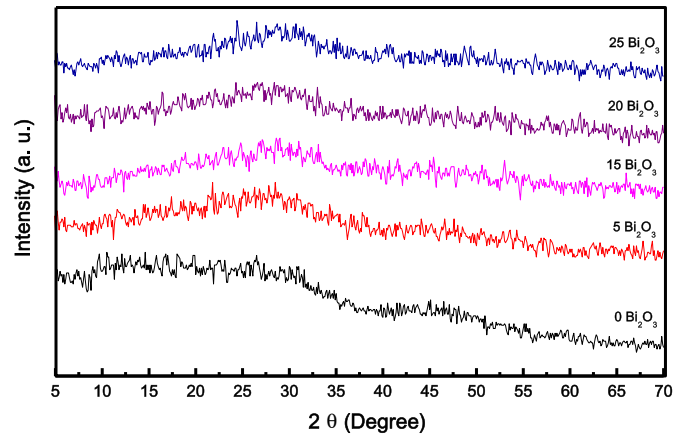


Fig. 1. X-ray diffraction pattern of the investigated $x \text{ Bi}_2\text{O}_3 - (40 \text{ B}_2\text{O}_3 - 32 \text{ CKD} - 3 \text{ BaO}) - (25 - x) \text{ PbO}$, ($0 \leq x \leq 25 \text{ mol\%}$).

At room temperature, a ^{60}Co gamma cell (5Ci) as a gamma ray source with a dose rate of 1.15 kGy/h was used. Samples were subjected to six radiation doses in the range of 10–120 kGy. The investigated glass samples were exposed to the same gamma dose every time. Using a Fricke dosimeter, the absorbed dose in glass is expressed in terms of absorbed dose in water, rather than in terms of dose in glass. No cavity theory correction was made. Each glass sample was subjected to a total dose of 10, 20, 30, 60, 90 and 120 kGy according to previous work [22, 23]. FT-IR spectra of all the powdered samples after irradiation were recorded using the same conditions as before irradiation.

3. Results

The FTIR spectra of the studied glass system in the range 4000–400 cm^{-1} as shown in Fig. 2 revealed the characteristic broad absorption bands of borates and some small absorption band at $\sim 3443 \text{ cm}^{-1}$ brought by air moisture during the preparation of KBr pellets of the samples [21] and attributed to the fundamental stretching of hydroxyl groups.

The FTIR spectral features of the base glass as shown in Fig. 2 have exhibited two intense absorption envelopes in the region 1515–1163 cm^{-1} and 1163–758 cm^{-1} and two peaks with lower intensity at 700 cm^{-1} and 466 cm^{-1} . Absorption of the FTIR spectra in these regions was related to vibrations of specific structural groups in the borate network. According

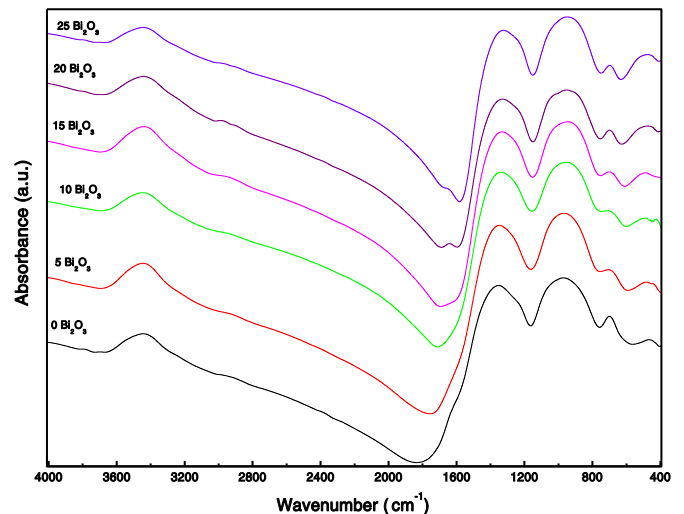


Fig. 2. FTIR spectra of the investigated $x \text{ Bi}_2\text{O}_3 - (40 \text{ B}_2\text{O}_3 - 32 \text{ CKD} - 3 \text{ BaO}) - (25 - x) \text{ PbO}$, ($0 \leq x \leq 25 \text{ mol\%}$). Numbers at the plots represent Al_2O_3 content in mol%.

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