Physical Characterization of Glasses based on Blast Furnace Slag (BFS)

A. G. Mostafa, M. A. Sayed, M.Y. Hassaan, K. A. Aly, Y.B. Saddeek, A. El- Taher

Abstract- Glasses based on Blast Furnace Slag (BFS) were prepared by conventional melt-quenching method. The ultrasonic velocities data of these glasses have been used to determine the elastic modulus. Densities of glass samples were measured by Archimedes's principle using Toluene as an immersion liquid. The composition dependence of the elastic properties of these glasses was discussed. Furthermore, based on the measured transmittance and absorption spectra in the wavelength range 350-2000 nm, the optical constants (optical band gap (E_g) and index of refraction (n)) have been determined. The addition of BFS produced significant changes such as an increase in the glass density, refractive index, ultrasonic velocities and elastic moduli. On the other side, the BFS additions shifts the absorption edge toward long wavelength side i.e., leading to a decrease in the E_g values. The obtained results were well discussed in terms of the electronic polarizability and the change in the glass structure with the addition of BFS content.

Keywords: BFS, prepared glasses, XRF for raw materials, density and molar volume, Ultrasonic measurements.

I. INTRODUCTION

Blast furnace slag is an industrial solid waste generated in the process of iron ore reduction in blast furnace and represents one of many types of wastes resulting from industrial metallurgical processes. The major amount of produced slag in Egypt is found to be 300,000 tons/year, where a little amount can be used as a raw material in cement industry and in road pavement. The largest amount is directly discharged into landfills, which consequently causes environmental problems. So, reducing the environmental impact of slag, scrap and dust resulting from iron and steel product will give its product further important benefit and other significant potential for cost savings profit if reintroduced into the industrial process through well planned programs [1, 2]. Recycling of BFS is the best way for eliminating their hazardous impact on the people and environment [3]. Many possible applications were suggested to incorporate proportions of the wastes in the processing of glass tiles and glass ceramics. These glasses can be used in many applications such as optical glasses, oven ware's, nuclear waste materials and in the electronics industry [4].

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The study of the elastic constants of glassy materials gives considerable information about the structure of these non-crystalline materials, since they are directly related to the inter-atomic forces and potentials [5]. The elastic properties, micro-hardness, Poisson's ratio, or other related parameters are of great interest to investigate the linear and anomalous variations as a function of composition of glass and have been interpreted in terms of the structure or transformation of cross-linkages in the glass network [6]. To study the structure of oxide glasses, the coordination number of the network former and the change of oxygen bonds in the frame work, induced by the different cations were needed to be investigated. Furthermore, many author's studies on borosilicate glass have been reported for structural properties of glass, by using ultrasonic techniques [7].On the other hand, the ultrasonic parameters such as velocity as a function of composition, besides the density and the molar volume are informative about the changes occurred in the structure of the glass network. The determination of elastic properties of glasses by ultrasonic pulse-echo method becomes a more interesting subject, due to the non-destructive nature and the high precision of the technique. This measurement yields valuable information regarding the forces operating between the atoms or ions in a solid. Since the elastic properties describe the mechanical behavior of the materials, so, the study of these properties is of fundamental importance in interpreting and understanding the nature of bonding in the solid state [8]. The optical properties of oxide glasses have been the subject of intense studies during the last decades specially the determination of the optical constants which frequently based on the optical transmission and reflectance spectra at normal incidence [9-13]. Therefore, the aim of the present work is to recycle wastes of blast furnace slag for glass production and studying the optical and elastic properties as a function of BFS content.

II. EXPERIMENTAL PROCEDURES

The used tailings of blast furnace slag were provided by Egyptian Iron and Steel Company, Helwan, Egypt. For preparation of a glass sample an appropriate amounts of reagent grade of Borax ($Na_2O_4B_7.10H_2O$) powder was thoroughly mixed with raw materials in an agate mortar and melted in a platinum crucible to obtain glass system. The chemical formula of the prepared glasses has the following form 100-x ($Na_2O_4B_7.10H_2O$) – x BFS (where x =10, 20, 30, 40, 50, and 60 wt.%). The electric furnace was kept at a temperature 1100 ^oC for 30 Minute under normal atmospheric conditions, after which the glass was poured in a preheated stainless steel mold and then slowly cooled to room temperature.

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To assure the homogeneity of the glass, the well-mixed components were added in small portions and the melt was swirled frequently. The glasses were annealed at 400ºC for 2 h to relieve the internal stresses and allowed to cool gradually to room temperature at a rate of about 30°C h^{-1}.

Table 1: XRF for raw materials by wt.%.

Constituent Oxide	BFS wt%	BORAX wt%
CaO	30.67	-
SiO_2	36.83	-
SO_3	2.37	-
K_2O	1.05	-
MgO	5.61	-
Al_2O_3	12.91	-
Na_2O	1.30	16.93
MnO	4.27	-
BaO	3.665	-
Fe_2O_3	0.44	-
TiO_2	0.51	-
SrO	0.232	-
ZrO_2	0.047	-
P_2O_5	0.04	-
Y_2O_3	0.016	-
Cl	0.03	-
$2B_2O_3$	-	35.97
L.O.I.	0.01	47.1
TOTAL	100.00	100.00

The amorphous state of the glasses was checked using X-ray diffraction using Philips X-ray diffractometer PW/1710 with Ni-filtered Cu-Kα radiation (λ = 1.542 Å) powered at 40 kV and 30 mA.

The prepared samples were polished with different grades of SiC emery powder on a soft leather piece fixed on a flat platform for the ultrasonic velocity measurements. Non-parallelism of the two opposite side faces was measured with a micrometer, which could measure down to 0.01 mm. The density of each sample was measured by Archimedes' method 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 ±25 kgm^{-3}.

The ultrasonic velocities, longitudinal (v_L) and shear (v_T), at room temperature (~300 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 ±10 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 = ρv_L^2 , and for pure transverse waves G = ρv_T^2 . The elastic bulk modulus (K) and Young's modulus (Y) can be determined using the standard relations adopted in previous work [14]. The uncertainty in the measurement of the elastic moduli is ± 0.15 GPa. The optical transmittance spectra have been measured at room temperature using a double beam (Jasco-V670) computer controlled spectrophotometer, at normal incidence of light and in the wavelength range 350 - 2000 nm.

III. RESULTS AND DISCUSSION

The chemical compositions of the BFS and Borax were analyzed by using X-ray fluorescence technique and their values were listed in Table 1. The amorphous state of the as-prepared glasses was confirmed by the absence of any sharp lines or peak as shown if Fig.1.

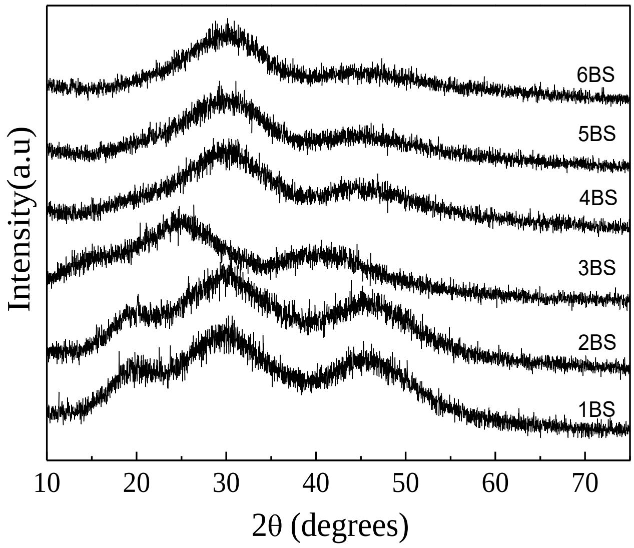

Fig.1. X-ray diffraction patterns for 1BS-6BS glasses.

A. Density and molar volume

The density is an intrinsic property capable of casting the light on the glass structure. It was reported that, modification of B_2O_3 glass causes a conversion of its basic structural unit [BO_3] into four-fold [BO_4]. The BO_4 structural groups are denser than BO_3 structural units and are responsible for the increase in the connectivity of the glass network and the degree of the structural compactness.

Analysis of the oxides in this study revealed that as the concentration of BFS was increased on the expense of the concentration of NaO_2 and B_2O_3, i.e., the concentration of CaO increased. In the studied glasses, it was observed that, the density increases linearly with a linear decrease in the molar volume as listed in Table 2 which was attributed to the higher density of CaO than that of NaO_2 and B_2O_3. The values of the density and the molar volume are near to other researches [15-17].

B. Optical properties

Fig.2 depicts the measured transmittance and reflectance spectra for the studied glasses. It is found that, the optical absorption edge is not sharply defined in the present glasses, which clearly indicates their glassy nature. As illustrated in this figure, the addition of CaO shifts the optical spectra to the low wavelength side (i.e. to the blue shift of the optical band gap) [18, 19]. Based on the measured A values, the absorption coefficient was calculated by [20]:

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$$\propto(\lambda) = \frac{1}{x}\ln\left(\frac{1-R}{T}\right), \qquad (1)$$

where x is the sample thickness. The calculated values of the absorption coefficient α(λ) were plotted versus wavelength (λ) for different BFS content as shown in Fig.3. In the region with high absorption region, the photon energy dependence of the absorption coefficient obeys Tauc's formula [18, 21]:

$$\alpha . h\nu = B(h\nu - E_g)^p, \qquad (2)$$

where E_g is the optical band gap, B is an energy independent constant, and the exponent P takes different values depending on the mechanism of inter-band transitions (P =1/2 for direct and P=2 for indirect transitions) [20]. The linear relation of the absorption coefficient parameter $\sqrt{\alpha h\nu}$ versus hν for 1BS-6BS glasses were represented in Figure 4. The relation confirmed the transitions in the forbidden gap are indirect, i.e., P = 1/2. The intercept of $\sqrt{\alpha h\nu}$ versus hν at $\sqrt{\alpha h\nu} = 0$ denoted the value of E_g. The values of E_g as listed in Table 2 decreases with the increase of CaO content. According to the theory of reflectivity of light, the refractive index (n) values as a function of the reflectance (R) and the extinction coefficient (k) as [22]:

$$n = \frac{(1-n)^2+k^2}{(1+n)^2+k^2}. \qquad (5)$$

The k values have been determined using the relation ($k = \alpha\lambda/4\pi$). Fig. 5 shows the refractive index as a function of the wavelength for the studied glasses. The refractive index increases in the wavelength range of 200–2000 nm with the increase of CaO content [12]. The obtained results of the optical band gap and refractive index can be discussed according to Fajans's rules [23-25]. The polarizing power of the cation increases with decreasing its size and number of filled orbitals and with increasing it is positive charge. The polarizability of the anion increases with increasing its size and with increasing its negative charge. The polarizability of a cation is increased in the coulomb field of an anion, while the polarizability of an anion should be decreased in the field of a cation. Here, we replace the B_2O_3 and Na_2O (the atomic radius of B and Na are 0.8 and 1.9 Å [26]) with CaO (Ca with atomic radii is 1.97 Å [26]). Therefore, the electronic polarizability and consequently the refractive index of the as-prepared 1BS-6BS glasses were increased with increasing CaO content. The electronic polarizability and the optical basicity for the studied glasses were according to another work [14, 24]. Figs. 6 and 7 show that, both the electronic polarizability and optical basicity are increased with increasing CaO content. This behavior of the electronic polarizability would be expected due to the larger polarizability and basicity of CaO ((α_e = 2.505 and Λ= 1.0) than that of Na_2O and B_2O_3 (α_e = 0.18 and 1.345, Λ = 1.15 and 0.425, respectively) [24].

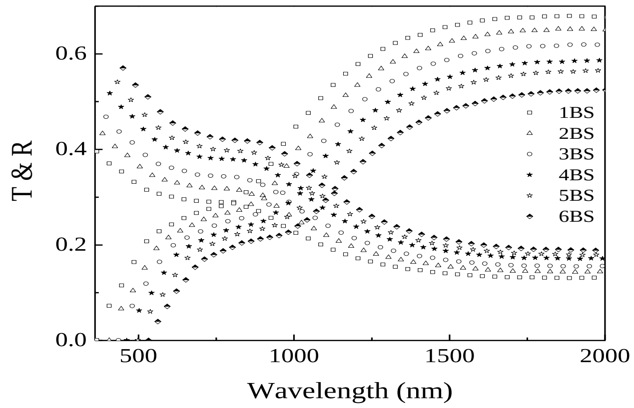

Fig.2. The measured T and R spectra versus λ for the 100-x Borax – x BFS (10 ≤ x ≤ 60) glasses.

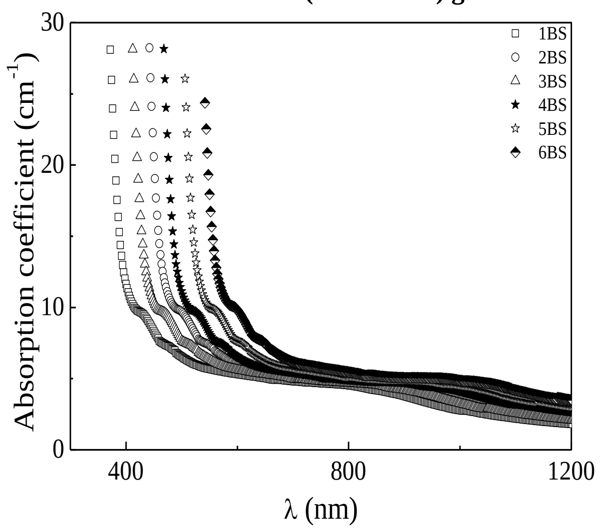

Fig.3. The absorption coefficient (α) versus λ for the 100-x Borax – x BFS (10 ≤ x ≤ 60) glasses.

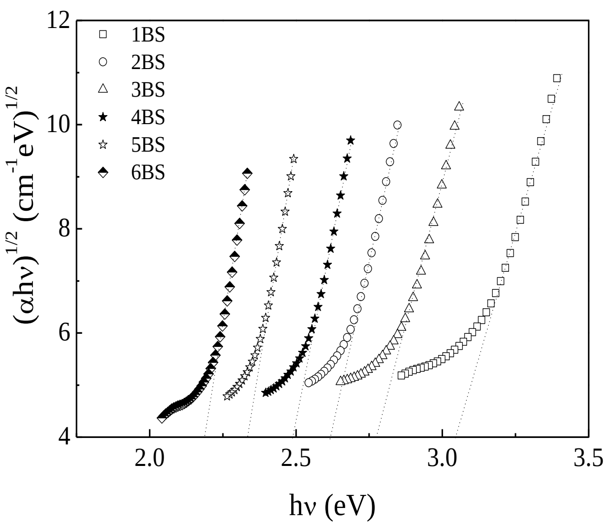

Fig.4. The absorption coefficient parameter $(\alpha h\nu)^{1/2}$ versus photon energy (hν) for the 100-x Borax – x BFS (10 ≤ x ≤ 60) glasses.

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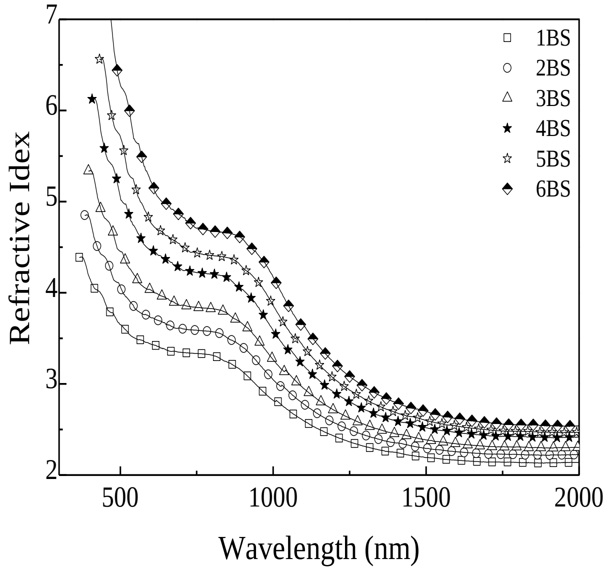

Fig.5. The refractive index (n) vs. the wavelength (λ) for 100-x Borax – x BFS (10 ≤ x ≤ 60) glasses.

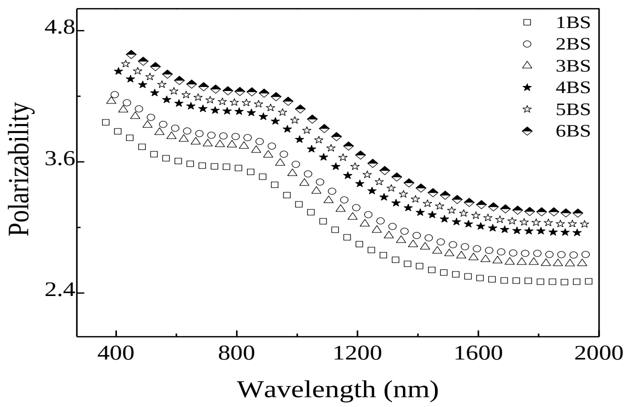

Fig.6. The optical polarizability as a function of the wavelength (λ) for the 100-x Borax – x BFS (10 ≤ x ≤ 60) glasses.

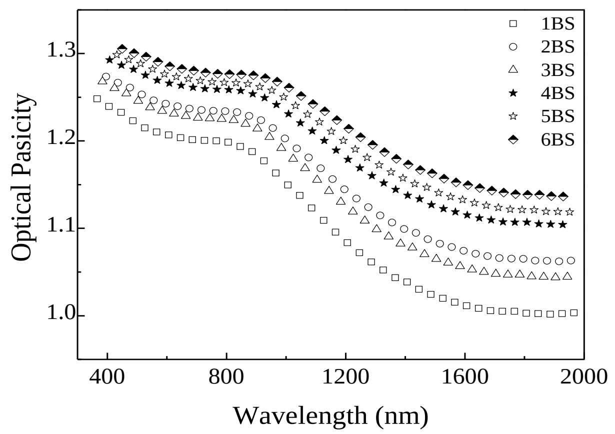

Fig.7. The optical basicity as a function of the wavelength (λ) for the 100-x Borax – x BFS (10 ≤ x ≤ 60) glasses.

B. Ultrasonic measurements

The longitudinal (V_L) and shear ultrasonic (V_T) velocities of the glass system with different wt. % of BFS content are listed in Table 2. The changes of glass structure were depending on the propagation of both longitudinal and shear wave velocities in the bulk samples [27]. Hence, filling of Ca^{2+} ions into the sodium borate glass network would result in the conversion of BO_3 structural unit into BO_4 structural unit which strengthen the glass structure and decrease the bond length between the different structural units as a result of creating bridging oxygens. As a result, both velocities (V_L) and (V_T) were increased, respectively with the increase of BFS concentration. It can be seen that the values of (V_L) are higher than (V_T). The increase in the ultrasonic velocity of the studied glass revealed the fact that the addition of BFS would cause a swift movement of the ultrasonic wave inside the network of the glass structure. Due to this factor, the ultrasonic velocity of the glasses would increase as the BFS content was increased. This behavior was confirmed with the decrease in the optical band gap of the glasses as the content of BFS is increased. The independent elastic constants for isotropic solids and glasses are longitudinal modulus (C_{11}) and shear modulus (C_{44}), where calculation for other elastic constants and Poisson's ratio depend on the density and both the two ultrasonic velocities. Young's modulus (Y) was defined as a ratio of the linear stress over the linear strain [27], whereby this Young's modulus was related to the bond strength of the materials. The bulk modulus (K_e) was defined as the changing in volume when a force is acted upon it at all directions [27]. As shown in Table 2, the values of elastic moduli were showing an increasing trend with the increase of BFS content. The attainment of a higher value of *Y* than *K* indicated that the glasses were able to with stand a higher longitudinal stress than transverse stress. The increase in K was due to the changing in the coordination number with an increasing in the BFS content and to the increase of the rigidity of the glasses. Finally, the obtained Poisson's ratio based on the elastic moduli as listed in Table 2 was affected by the crosslink density of the glass structure. A decrease in Poisson's ratio as a function of BFS content suggested an increase in the crosslink density as a direct result of the conversion of BO_3 into BO_4 structural units and to the increase of the concentration of CaO_6 structural units [28].

Table 2: The values of density (*d*), molar volume (V_m), sound velocities (v_L and v_T), elastic moduli and Poisson's ratio (σ) of the studied glass system.

Sample name	D	E_g	v_L	v_T
	g.cm^{-3}	eV	m.s^{-1}	
1BS	2.51	3.05	6300	2247
2BS	2.55	2.78	6370	2300
3BS	2.62	2.63	6417	2337
4BS	2.67	2.49	6460	2452
5BS	2.75	2.34	6477	2800
6BS	2.83	2.19	6490	2840

Sample name	C_{11}	C_{44}	K_e	Y	σ
	GPa				
1BS	100.76	14.27	93.17	40.9	0.432
2BS	111.47	14.53	92.09	41.42	0.425
3BS	109.74	14.56	90.33	41.44	0.424
4BS	109.34	15.752	88.33	44.61	0.416
5BS	105.81	19.18	80.24	53.29	0.389
6BS	105.51	20.2	78.57	55.83	0.382

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IV. CONCLUSIONS

Huge amounts of tailings were produced in every year in the process of BFSin Egypt. Glass industry is one of the solutions of this problem which is the aim of this study. As the content of BFS was increased in the glass batch, the concentrations of CaO increase at the expense of sodium diborate concentration. Therefore, the glass transmittance was decreased leading to an observed decrease in the optical band gap. On the other side the refractive index (n), the electronic polarizability (α_e) and the optical basicity (Λ) were increased with increasing the BFS content. As the content of BFS was increased, the density was increased and the ultrasonic velocities (V_L) and (V_T) were increased, respectively. The values of elastic moduli were showing an increasing trend with the increase of BFS content.

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