Abstract. Glasses based on borosilicate system were prepared by melt-quenching method in the composition range of (mol%) 20Na₂O : 1.0Al₂O₃ : 13B₂O₃ : 6.3CaO : 0.2Sb₂O₃ : 4.5BaO : (55-X)SiO₂ : xFe₂O₃ (where x = 0, 5, 10, 15, 20 and 25) and the structural, optical and magnetic properties were investigated. The result showed that the density and molar volume of glasses increased with increasing of Fe₂O₃ concentration. This might be due to an increase of Fe₂O₃ concentration at the expense of SiO₂ caused the opened glass network structure. Fe₂O₃ also affected the change of B₂O₃ structure detected by FTIR technique. The magnetic measurement revealed that the glass sample exhibited a ferrimagnetic behavior when doping with 25 % mol Fe₂O₃.

Introduction

Borosilicate glass is a well known prevalent type of glass that can be used in many applications, for examples, laboratory equipment and glass cookware because of its excellent chemical and physical properties such as a low thermal expansion coefficient and a high chemical corrosion resistance. In the recent years, glasses doped with transition metal oxides have attracted a great deal of attention due to their important physical and chemical properties [1-3]. In particular, Fe₂O₃ has been considered as one of the most promising candidate for improving physical properties of glasses such as electrical, optical and magnetic properties [4-6]. In this work, glasses based on borosilicate system doped with Fe₂O₃ in a range of 0-25 % mol were prepared through a melt-quenching method. The effect of Fe₂O₃ concentrations on structural, optical and magnetic properties of the samples was examined by Fourier transformed infrared spectroscopy (FTIR) and vibrating sample magnetometer (VSM), respectively.

Materials and Method

The glass samples were prepared by using high purity Na₂CO₃, Al₂O₃, H₃BO₃, CaO, Sb₂O₃, BaCO₃, SiO₂ and Fe₂O₃ in a composition range of (mol%) 20Na₂O : 1.0Al₂O₃ : 13B₂O₃ : 6.3CaO : 0.2Sb₂O₃ : 4.5BaO : (55-X)SiO₂ : xFe₂O₃ (where x = 0, 5, 10, 15, 20 and 25). The starting powders were thoroughly mixed by grinding in a high purity alumina crucible for 30 minutes and then melted at 1,100 °C in an electrical muffle furnace for 3 h. After melting completely, the melted powders were then poured quickly into a preheated stainless steel mould and annealed at 500 °C for 3 h before
cooled to room temperature. Finally, the as-prepared glass samples were cut and finely polished to obtain a dimension of 1.0cm x 2.0cm x 0.3cm. By applying Archimedes principle, the weight of the prepared glass samples was measured in air and in xylene using a 4-digit sensitive microbalance (Denver, Pb214). Density, \( \rho \), was determined by the relation as follow.

\[
\rho = \frac{W_a}{W_a - W_b} \times \rho_b
\]

where \( W_a \) is the weight in air, \( W_b \) is the weight in xylene, and \( \rho_b \) is the density of xylene (\( \rho_b = 0.863 \) g/cm\(^3\)). The corresponding molar volume (\( V_M \)) was calculated using the relationship, \( V_M = \frac{M_T}{\rho} \), where \( M_T \) is the total molecular weight of the multi-component glass system. The FT-IR transmission spectra of Fe\(_2\)O\(_3\) doped glass were recorded with Elmer FTIR Spectrometer Spectrum 2000, using the KBr pellet technique. Magnetic property measurements for all samples were carried out at room temperature with a vibrating sample magnetometer (VSM, Lakeshore 7404).

**Results and Discussion**

The glass with no Fe\(_2\)O\(_3\) composition was shows colorless and transparency. When 5 % of Fe\(_2\)O\(_3\) add in glass, the high intensity yellow-brown color was appeared and decrease transmission in visible region. The glasses doped with Fe\(_2\)O\(_3\) was turned into an opaque when Fe\(_2\)O\(_3\) concentration at 25% mol. The variation of the density and molar volume with Fe\(_2\)O\(_3\) concentration is presented in figure 1 and figure 2 respectively. As can be seen in figure 1, the density of glasses show linear increase (with \( R^2 = 0.99 \)) with the content of Fe\(_2\)O\(_3\) incorporated in the network. This indicated that the substitution of SiO\(_2\) by Fe\(_2\)O\(_3\) resulted in an increase of the average molecular weight of oxide ions in the glass due to Fe\(_2\)O\(_3\) has a higher relative molecular mass than that of SiO\(_2\). The molar volume depended on both the changing rate of density and molecular weight. However, when the Fe\(_2\)O\(_3\) concentration was increased, the increasing rate of molecular weight is greater than the increasing rate of density. As shown in figure 2, the molar volume increased with increasing of Fe\(_2\)O\(_3\) content, this is attributed to an increase in the number of non-bridging oxygen (NBOs). The obtained results indicated that the Fe\(_2\)O\(_3\) could enter in the glass network and acted as a modifier by occupying the interstitial space in the network and generating the NBOs to the structure. It was observed that the addition of Fe\(_2\)O\(_3\) may accordingly result in an extension of glass network [7]. The following result shows a best fit was polynomial trend line to illustrate the relationship between molar volume and Fe\(_2\)O\(_3\) content. Notice that the \( R^2 \) value is 0.9858, which is a best fit of the line to the data.
**Figure 1.** The densities of Fe$_2$O$_3$-doped glass

**Figure 2.** Molar volumes of Fe$_2$O$_3$-doped glass
Figure 3 shows FTIR spectra of 20Na2O : 1.0Al2O3 : 13B2O3 : 6.3CaO : 0.2Sb2O3 : 4.5BaO : (55-x)SiO2 : xFe2O3 (where x = 0, 5, 10, 15, 20 and 25) glass samples in a range of 400-2000 cm⁻¹. It is clearly seen that the main absorption band in a region of 900–1100 cm⁻¹ is attributed to the combined stretching vibrations of Si–O–Si bonds and the stretching vibrations of B–O bonds in BO₄ structural units [8-9]. The absorption band in a range of 1200-1500 cm⁻¹ can be assigned to the B–O stretching vibrations of BO₃ structural units [9, 10]. The intensity of this band increased as a function of Fe2O₃ concentration. This is due to the structural changes involving the conversions of the BO₄ into BO₃ structural units with an increase of the Fe2O₃ glass modifier in good agreement with molar volume result. The absorption peaks at about 700 cm⁻¹ and 460 cm⁻¹ are assigned to the bending vibration of B-O-B and Si–O–Si bonds, respectively [8,9]. The IR spectrum of 25% mol Fe2O₃-doped glasses show the appearance of absorption band in a range of 550-580 cm⁻¹ which is ascribed to stretching vibrations of Fe–O bonds [8]. This peak might come from the formation of iron oxide or ferrite phase in the glass network.

Figure 4. Magnetization for 0-20 % mol Fe2O₃-doped glass

Figure 5. Magnetization for the 25% mol Fe2O₃-doped glass
The magnetization (M) as a function of magnetic field (H) of 20Na₂O : 1.0Al₂O₃ : 13B₂O₃ : 6.3CaO : 0.2Sb₂O₃ : 4.5BaO : (55-x)SiO₂ : xFe₂O₃ (where x = 0, 5, 10, 15, 20 and 25) glass measured by a vibrating sample magnetometer (VSM) at room temperature are presented in figure 4 and 5. It was observed that undoped glass exhibited a diamagnetic behaviour while the glass doped with Fe₂O₃ at 5-20 % mol, showed a paramagnetic behaviour and the magnetization increased with increasing Fe₂O₃ concentration. The paramagnetism originates from the presence of permanent magnetic dipole moments of iron in the glass (see in figure 4). The magnetization curve of the 25% mol Fe₂O₃-doped glass was shown in figure 5. It is obviously seen that the hystерetic loop indicated a ferromagnetic behavior. The coercive field of hystерetic loop is about 240 Oe. The magnetization and FTIR results of 25% mol Fe₂O₃-doped glass could confirm that the formation of iron oxide or ferrite phase formed in the glass sample. For high concentration (25 % Fe₂O₃), each Fe³⁺ ion in glass network become closer and Fe³⁺ ion is not fully compensated by NBOs which tend to aggregate with each other and finally, cluster or particle of Fe₂O₃ was formed.

Conclusions

In this present work, glasses based on borosilicate system doped with Fe₂O₃ in the range of 0-25 % mol were prepared by a melt-quenching method. The structural, optical and magnetic properties of glass samples were studied. The result showed that the density and molar volume of glasses increased with increasing of Fe₂O₃ concentration. This might be due to the increase of Fe₂O₃ concentration at the expense of SiO₂ caused the opened glass network structure. The FTIR results showed that the BO₃ structural units increased with increasing of Fe₂O₃ concentration. Herein, The FTIR and magnetic results could confirm the presence of iron oxide or ferrite phase in the glass sample when doping with 25 % mol Fe₂O₃.

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