EFFECT OF LEAD CATIONS ON THE STRUCTURAL, PHYSICAL AND GAMMA-RAY SHIELDING PROPERTIES OF SOME IRON-LEAD-ZINC-BORATE GLASSES

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Abstract

A glass system with the molecular composition [60% B_2O_3; (25-x) % ZnO; x% Pb_3O_4; 15% Fe_2O_3; with x = 0, 5, ... 25] has been prepared by using the melt quenching technique. The melting and annealing temperatures were 1100 ^oC and 350 ^oC, respectively. The structure features of these glasses have been examined applying Mössbauer effect spectroscopy and infrared analysis, while their thermal stability was obtained using differential thermal analysis. Density, molar volume, and magnetic susceptibility, as well as their gamma-ray shielding properties have been also investigated. Mössbauer effect results indicated that iron appeared as ferric ions occupying the tetrahedral symmetry in the low spin state acting as glass network former. Infrared analysis showed that boron ions occupy different structural groups and most lead and zinc ions occupy the interstitial vacancies acting as a glass network modifier. It confirmed also that iron occupies glass network former positions. The differential thermal analysis results showed that as lead oxide was increased the characteristic temperatures showed slight decrease, and their thermal stability showed slight linear increase. Also, the relation between glass transition temperature and the quadrupole energy confirmed that iron entered the network as former ions. The molar magnetic susceptibility, density as well as the molar volume increased with increasing lead oxide. Finally, as Pb_3O_4 was increased at the expense of ZnO, the mass attenuation coefficient shows linear increase at all the studied gamma-ray energies, while the calculated values of both the half value layers (HVL) and mean free path (MFP) show a gradual decrease.

Keywords: *Iron-lead-zinc-borate glass; Mossbauer Effect spectroscopy; Infrared analysis; Structural, thermal and physical properties; Gamma-ray shielding properties*

Introduction

During the last few decades, much attention has been paid to investigate various glasses as useful solid materials, especially those containing transition metal ions (TMIs) and those containing heavy metal ions (HMIs) or those containing both TMIs and HMIs. These ions affect directly the glass structure and its physical properties [1-8]. Among all the transition metals, iron is of the highest interest, since it improves the glass durability as well as their electrical and magnetic properties [9]. Zinc is classified as an important transition metal, glasses containing ZnO being also of high importance especially for optical purposes as well as for manufacturing soft and soldering glasses [10]. Lead importance as heavy metal is due to its high gamma-ray attenuation coefficient [11, 12].

On the other hand, borate glasses, in particular, have been the subject of numerous infrared studies due to their structural peculiarities [13]. It is well known that the structure of borate glasses strongly depends on the rate of cooling around glass transition temperature as well as the annealing temperature and time [14, 15]. Doweidar et al [16] showed that the introducing of ZnO in lead borate glasses might change the distribution of Pb in lead-borate glass. More recently, Motke et al. [17] demonstrated that the structure of Zn-doped borate glass consists of randomly connected BO_3 and BO_4 groups and this structure is independent on the composition.

The paper main objective is to investigate thoroughly the effect of introducing lead at the expense of zinc ions on the structural, physical and gamma-ray shielding properties for some borate glasses containing iron. Mossbauer Effect (ME) spectroscopy, differential thermal analysis (DTA) and infrared spectral analysis were utilized to investigate the structure of the prepared glasses. The density, molar volume, magnetic susceptibility as well as gamma-ray shielding properties will be also studied.

Experimental Procedures and Methodology

Analytically pure H_3BO_3, Fe_2O_3, Pb_3O_4 and ZnO were used -as raw materials- to prepare some iron-borate glasses of the molecular composition, [$60B_2O_3$. (25-x) ZnO; x Pb_3O_4; 15 Fe_2O_3, where x = 0, 5, 10, 15, 20 and 25]. The glass batches were mixed well and were melted in porcelain crucibles using an electric muffle furnace at 1100 ^oC for 2 h. Melts were stirred several times during melting, and after the melting time, they were poured on stainless-steel molds. The obtained glass samples, after just sitting, were directly transferred to the annealing furnace at 350 ^oC for 2h in order to obtain stress and strain free glasses. The furnace was then turned off and left to cool to room temperature (RT) overnight, with a heating rate of about 0.5 ^oC/min.

A conventional constant acceleration Mossbauer Effect (ME) spectrometer was used to investigate the state of the iron ions in the studied glasses using 10 mCi ^{57}Co radioactive source diffused in chromium matrix. The analysis of the ME spectra were carried out using a least square fitting program based on Loranzia in line shape. The obtained ME parameters were calculated relative to a metallic iron calibration spectrum.

The DTA measurements were carried out using Shimadzu, DTA-50 differential thermal analyzer under constant heating rate of 10 ^oC/min, in order to determine the glass transition (T_g), crystallization (T_c) and melting (T_m) temperatures, and to confirm the structural state of iron. The thermal stability of these glasses can be calculated as the differences between the crystallization and the glass transition temperatures (T_c-T_g) ^oC.

Infrared spectra of the prepared samples were recorded at RT utilizing KBr disk technique using Fourier transform infrared (FTIR) 580 Perkin-Elmer spectrometer.

The experimental density (ρ_{exp}) values were measured at room temperature using the liquid displacement method, applying Archimedes formula given below:

$$\rho_{\exp} = \frac{M_a}{M_a - M_l} \tag{1}$$

where M_a and M_l are the sample weights in air and liquid, respectively, and ρ_l is the density of the used emersion liquid (carbon tetrachloride (CCl_4) ρ_l = 1.593 g/cm^3).

The empirical density values (ρ_{emp}) were also calculated applying the following relation:

$$\rho_{emp} = \sum X_i \rho_i \tag{2}$$

where ρ_i are the densities of the oxides forming a glass sample and X_i is the mole fraction of corresponding oxide.

The molar volume $(V_m)_{exp}$ values were calculated using formula given as follows:

$$\left(V_m\right)_{\exp} = \sum W_m \frac{1}{\rho_{\exp}} \tag{3}$$

where W_m is the mean molecular weight of a glass sample and ρ_{exp} is its experimental density.

The empirical molar volume values $(V_m)_{emp}$ can be also calculated by replacing (ρ_{exp}) by (ρ_{emp}) in Eq. (3), [9].

Gouy method was applied to obtain the volume magnetic susceptibility of the studied glasses, using 4 tesla electro-magnets and an electric balance of five decimal digits.

A special tube of constant diameter (a) was filled with the powder (less than 160 mesh size) of a glass sample. The tube was then suspended between the two poles of the Gouy magnet, such that the powder of the sample will be located exactly between the two poles. The variation of (Δm) due to the magnetic pull, as a function of the magnetic field intensity change was recorded, and the molar magnetic susceptibility (X_{mol}) can be calculated applying the following equations:

$$X_V = \frac{M}{H},\ X_V = 2\left(\Delta m\right) g / aH^2 \tag{4}$$

where M is magnetization per unit volume, H is magnetic field intensity, g is acceleration due to gravity, ($\Delta\ m$) is variation in the weight of a sample due to the magnetic pull and a is diameter of the used tube [18].

The mass and molar magnetic susceptibilities (X_m & X_{mol}) were then calculated using the following equations:

$$X_m = \frac{X_V}{\rho} \tag{5}$$

where ρ is the experimental density of the glass sample.

$$X_{mol} = X_V \cdot W_m \tag{6}$$

where W_m is the mean molecular weight of glass sample.

The values of the total mass attenuation coefficients of the studied glasses were calculated using WIN X-COM program, based on mixture rule using the equation:

$$\left(\mu/\rho\right)_{m(total)} = \sum_i^n w_i \left(\mu/\rho\right)_{m(i)} \tag{7}$$

where $(\mu/\rho)_m$ is the total mass attenuation coefficient for a glass sample and w_i is the fractional weight of an oxide in such glass sample.

The half value layers (HVLs) of the studied glasses were also calculated according to equation:

$$HVL = 0.693/\mu_l \tag{8}$$

where μ_L is the linear attenuation coefficient.

The mean free path (MFP) values have been also calculated using the equation:

$$MFP = 1/\mu_l \tag{9}$$

Results and discussion

Mössbauer Effect Spectroscopy

It has been established early that ME spectroscopy is an effective tool to investigate the hyperfine structure of iron in crystalline and amorphous materials. Accordingly, it was utilized here to obtain exact information about the iron ions in the studied glasses, and Figure 1 shows some representative ME spectra (due to samples containing 0, 10, 20 and 25 mol % lead oxide).

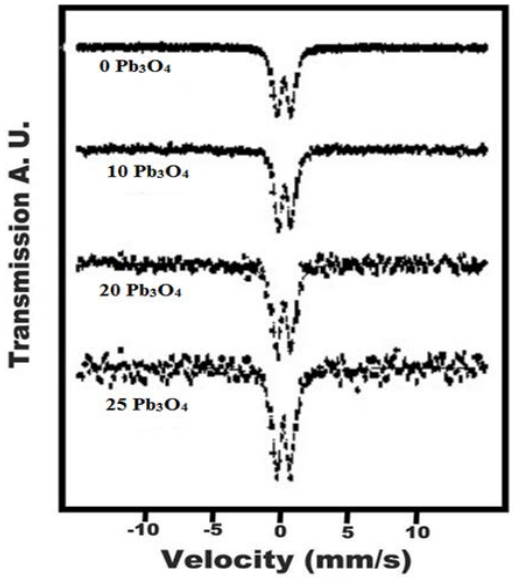


Fig. 1. ME spectra of the samples containing 0, 10, 20 and 25 mol % Pb_3O_4

The general features of these spectra are, as follows:

- All spectra appeared approximately similar with a central paramagnetic doublet.
- The doublet of all spectra seems to be approximately symmetric.
- The computer fitting of the obtained spectra reveals that all iron cations occupy only single symmetrical site.
- Calculated ME parameters [Isomer shift (IS), quadruple splitting (Δ) and line width (LW)] of these glasses are plotted in Figure 2 as a function of lead oxide content.

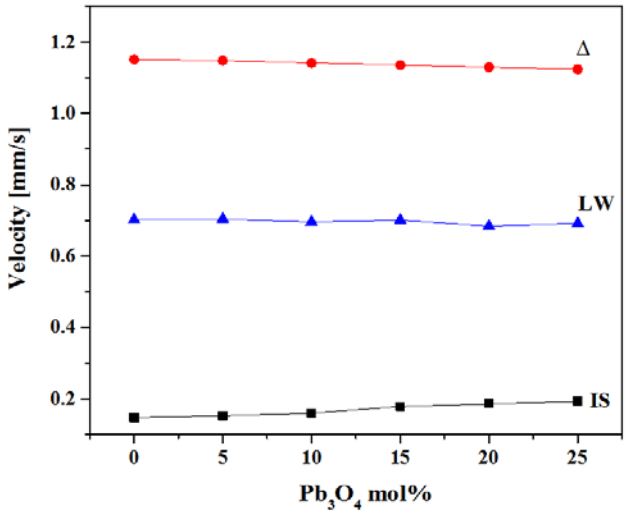


Fig. 2. The variation of obtained ME parameters with Pb_3O_4 content

It can be noted from Figure 2 that the isomer shift (IS) values show a slight increase from about 0.17 to about 0.20 mm/s with increasing Pb_3O_4. This trend reveals that the increase of lead oxide at the expense of zinc oxide cause a slight increase in the covalence character of the Fe-O bond, which may be due to the field strength and the polarizing power of lead [19].

The quadruple splitting (Δ) values of iron ions exhibit a slight decrease from about 1.16 up to about 1.12 mm/s, with increasing Pb_3O_4. This reflects a slight increase in the asymmetry around iron ions in the glass network as Pb_3O_4 increased, which in turn increase the electric field gradient due to the network around iron nuclei.

The line width (LW) values appear to fluctuate between about 0.70 and 0.72 mm/s; it appeared approximately constant as lead oxide was gradually increased. It can be stated that the LW values of these glasses reflect highly random structural features that exhibited usually by amorphous solids and glasses.

According to the calculated ME parameters, it can be stated that iron ions occupy the glass network former (GWF) positions [19]. Therefore, DTA technique will be applied here to confirm the state of iron that obtained by ME applying "T_g - Δ" rule (due to Nishida) [19, 20].

Differential Thermal Analysis

It was established early by Nishida [20-21] that a linier relationship between T_g and Δ, is usually obtained for glass, obeying the following formula:

$$T_g = a\Delta + b \tag{10}$$

In this relation the slope parameter (a) is considered to be effective in determining the structural role of iron ions in the glass network.

Nishida suggested that, if the obtained values of a are around 680 ^oC $(mm/s)^{-1}$, this means that iron ions occupy low spin ferric state in the tetrahedral coordination, that is iron appear in the glass network former (GNF) sites. While, if the obtained values of a are around 260 ^oC $(mm/s)^{-1}$, iron ions occupy low spin ferric state in the octahedral coordination, that is iron appear in the glass network modifier (GNM) sites.

Figure 3 shows the DTA thermo-grams of the samples containing 0, 5 and 25 mol % lead oxide.

Inspecting all the obtained thermo-grams, it can be stated that the general feature of all curves is the approximate similarity i.e. all these spectra showed an endothermic peak followed by an exothermic peak followed by another sharp endothermic one. These peaks can be attributed to the glass transition (T_g), crystallization (T_c) and melting (T_m) temperatures respectively.

The variation of T_g, T_c and T_m with the gradual increase of lead oxide content are depicted in Figure 4.

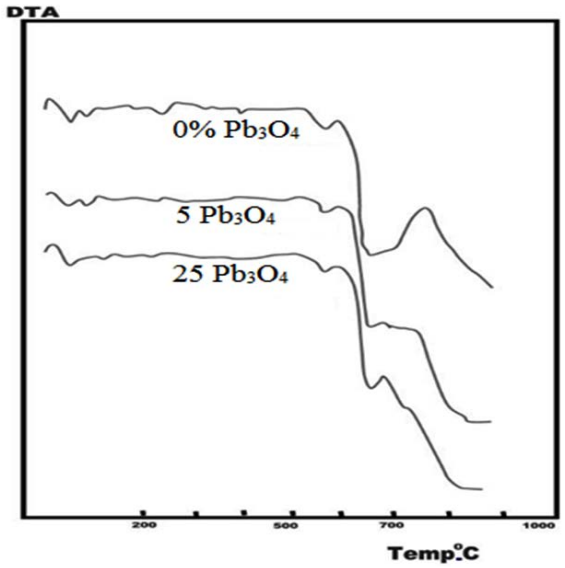


Fig. 3. DTA curves for the samples containing 0, 5 and 25 mole % Pb_3O_4

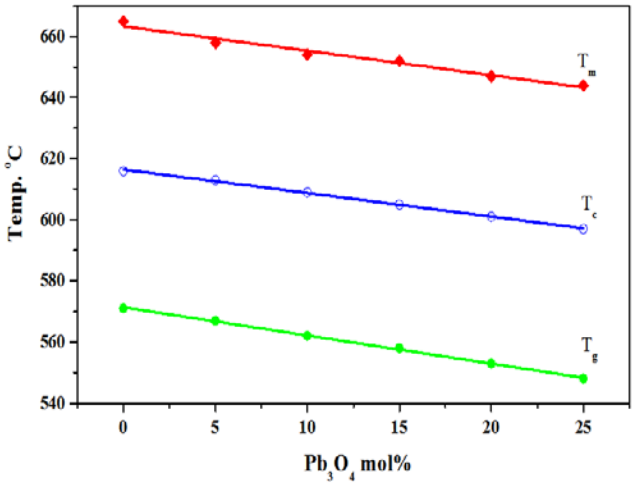


Fig. 4. The variation of T_g, T_c and T_m temperatures as a function of Pb_3O_4 content

It can be noted that as lead oxide content was increased, all the characteristic temperatures (glass transition; T_g, crystallization; T_c, and melting; T_m) show approximately a slight linear decrease. On the other hand, it is well known that the glass stability can be calculated as the differences between the crystallization and the glass transition temperatures (T_c-T_g) °C [21, 22].

However, the effect of the gradual substitution of ZnO by Pb_3O_4 on the thermal stability of these glasses is presented in Figure 5. It is seen that the thermal stability shows a linear increase with the increases of lead oxide content.

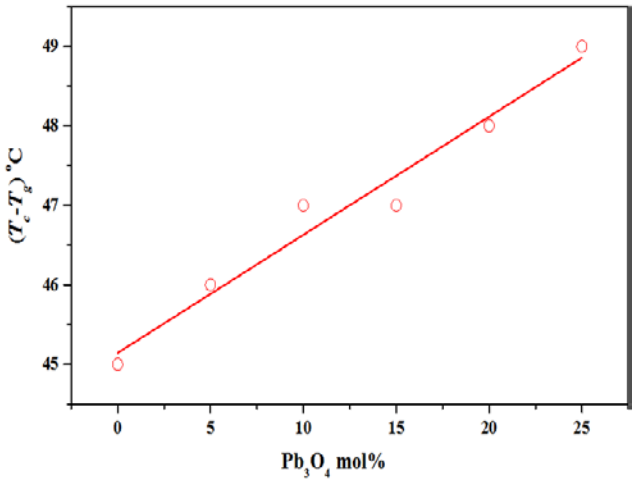


Fig. 5. The variation of the thermal stability with lead oxide content

Figure 6 represents the variation of the obtained values of T_g and Δ for the studied glasses (solid circles). The solid line represents the best fit where the calculated slope was about 789 °C $(mm/s)^{-1}$. Accordingly, it can be stated that, iron ions occupy mostly the tetrahedral coordination state acting as GNF ions and make covalent bonds to the nearest oxygen ions. The difference in slope parameter value between the present work and that obtained by Nishida may be due to the differences in composition and preparation method.

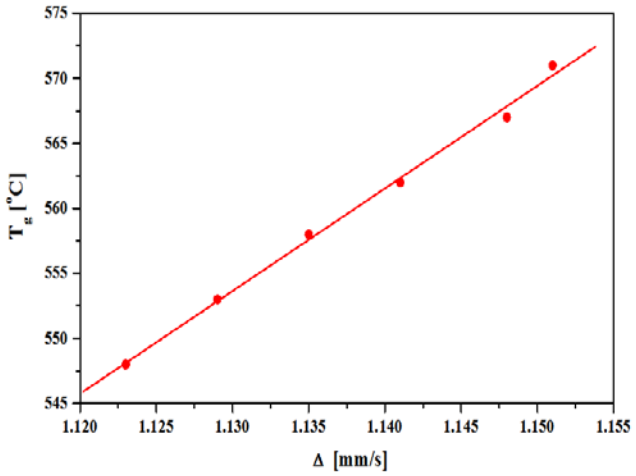


Fig. 6. T_g versus Δ for the studied glasses

Infrared Spectroscopy

Figure 7 represents the obtained IR spectra of the samples containing 0, 10 and 25 mol% Pb_3O_4, where all spectra appeared to be approximately similar. This means that, the replacement of zinc oxide by lead oxide does not create major changes in the glass networks.

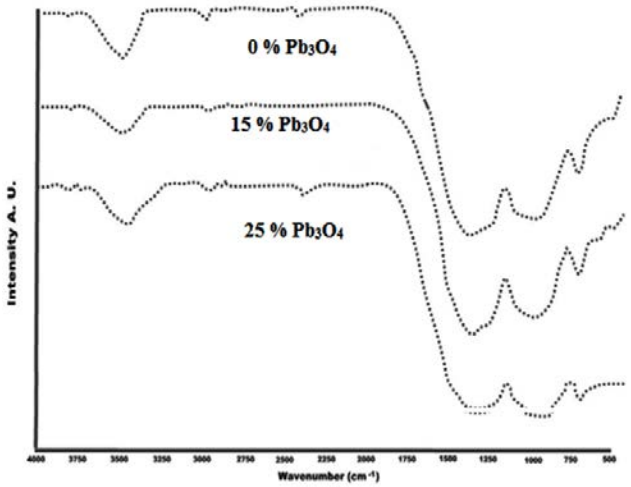


Fig. 7. The obtained IR spectra for the samples containing 0, 15 and 25 mol% Pb_3O_4

Inspecting all the obtained spectra, it is noticed that, a band appeared at 460 cm^{-1} in the spectrum of the sample containing 25 mol% lead oxide, shifts to higher energy region on replacing ZnO by Pb_3O_4, to appear at 469 cm^{-1}. This band can be attributed to the presence of some four-fold coordinated lead ions [17, 22].

A shoulder band appeared at 570 cm^{-1} in all glasses and can be attributed to some deformed modes of borate network including plain bending of boron oxygen triangles [18, 19], as well as presence of iron ions in the tetrahedral coordination state (FeO_4) acting as GNF ions [23].

The band that appeared at 687 cm^{-1} in the spectrum of the sample containing 25 mol% lead oxide, show a shift towards higher energy, and it can be attributed to bending vibrations of the bridging oxygen atoms (B---O) [24].

Another broad band appeared at 928 cm^{-1}, in the sample containing 25 mol% lead oxide also. This band was found to shift towards higher energy as zinc oxide was gradually replaced by lead oxide to appear at 987 cm^{-1} in the lead free sample. The band can be attributed to the stretching vibrations of B-O bond in BO_4 tetrahedral units in penta-borate groups [25].

Moreover, there is a small shoulder band appeared approximately at 1240 cm^{-1} which can be attributed to either the stretching vibrations involving mainly the oxygen linkage connecting different groups [18] and / or the present B-O bonds which act as bridges between B_3O_6 rings and BO_3 triangles [19].

The band appeared at 1325 cm^{-1} in the sample containing 25 mol% lead oxide showed a slight shift towards higher energy with the gradual increase of zinc oxide. This band can be attributed to the stretching vibrations of some meta-borate groups [17].

Furthermore, the bands which appeared between 2200 and 3750 cm^{-1} may originated as a result to the presence of some hydrogen bonds as well as some water content [16].

The apparent decrease of the bands intensity on replacing zinc oxide by lead oxide can be attributed to the gradual increases of lead oxide that act to decrease gradually the water content in the glass networks [14, 24, 25].

Based on the previous analysis of the obtained IR spectra, it can be stated that, in all the studied glasses, boron forms different structural groups in a highly distorted glass network [17-19]. Most of the lead ions occupy the interstitial positions while little lead forms some PbO_4 groups [17, 23]. Iron ions appeared in the tetrahedral coordination state as GNF former ions [22]. On replacing zinc oxide by lead oxide (increasing lead oxide), this causes gradual increase of the band due to PbO_4 groups. Therefore, it can be concluded that Pb^{2+} ions occupy the interstitial positions that previously occupied by Zn^{2+} ions [14, 15, 24, 25]. The observed shift of the bands towards higher energy indicates that the deformation of the glass network increased, which may be due to the polarizing effect of the introduced lead ions. This is in fact due to the high field strength and the large ionic size of lead, and this indicated also the strengthening of the B-O bonds in the glass network [15, 19].

Density and Molar volume

Density of glass is a property of importance, since it reflects usually any structural transformation, phase separation or crystallization that can be appeared during preparation.

However, Figure 8 exhibits variation of both experimental and empirical density values as a function of lead oxide content for comparison. It is observed that the empirical density values are usually larger than those obtained experimentally. This can be taken as evidence for the pure glassy state of the present samples.

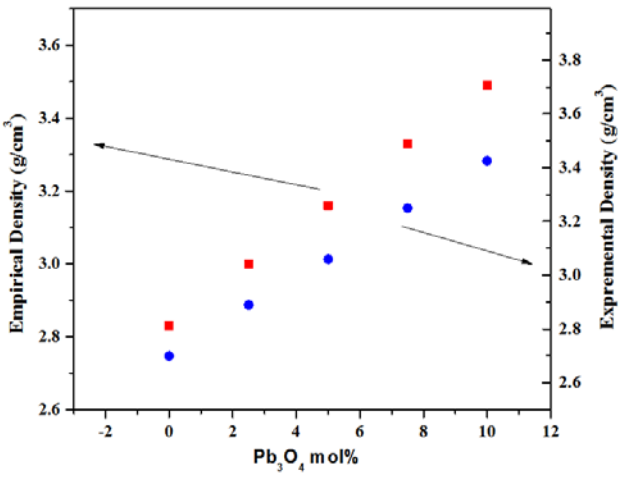


Fig. 8. The variation of the experimental and empirical densities as a function of Pb_3O_4 content

On the other hand, Figure 9 shows the change of both the experimental and empirical molar volume values as a function of Pb_3O_4 content, also for comparison, where both values show a gradual linear decrease and the empirical molar volume values appeared usually lower than those obtained experimentally.

Such decrease in the molar volume may be due to [26, 27]:
- the formation of Pb–O bonds, at the expense of Zn–O bonds, which act to reticule the glass network structure;
- the gradual increase of the oxygen ion density in the glass network, since one oxygen atom (in ZnO_molecule) is replaced by four O atoms (in Pb_3O_4 molecule).

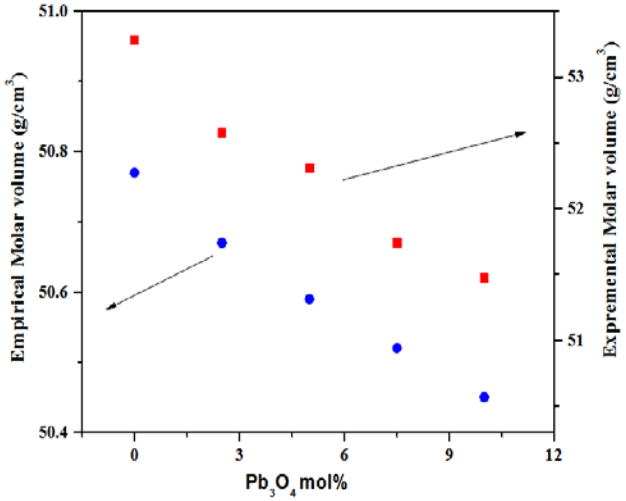


Fig. 9. The variation of the experimental and empirical molar volume values as a function of Pb_3O_4 content

The higher values of the experimental molar volume in comparison to those obtained empirically confirm also the amorphous nature and the glassy state character of the studied samples.

Magnetic susceptibility

It is interesting to investigate the magnetic properties of these glasses since they contain two transition metal ions, iron and zinc.

With respect to iron, it participates in all glasses with a constant concentration (15 mol %), and the obtained structural information revealed that, it enters the glass networks in the tetrahedral coordination state. It can be seen that all the magnetic susceptibility values (volume-, mass- & molar susceptibilities) show gradual increase. Figure 10 shows the variation of the molar magnetic susceptibility as lead oxide was gradually increased.

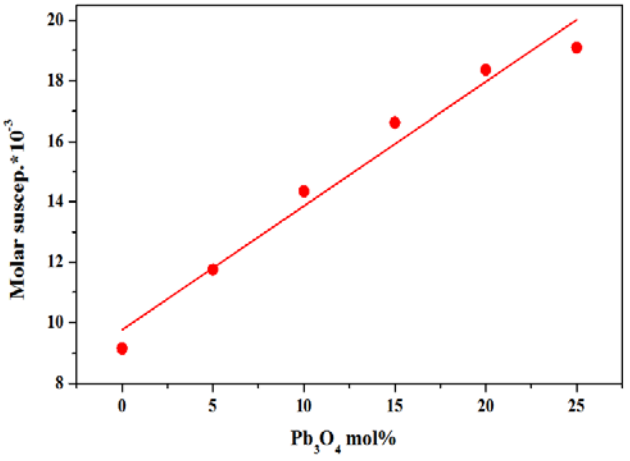


Fig. 10. The variation of the molar magnetic susceptibility with Pb_3O_4 content

It can be seen that the molar magnetic susceptibility showed gradual linear increase. It is supposed that the existence and the gradual increase of lead zinc cations in the interstitial positions makes its magnetic properties somehow high, remembering that Zn is a diamagnetic ion. This in turn can be attributed to the distribution of their d-electrons since most of them are spin paired [19].

The magnetic susceptibility results showed that the replacement of zinc oxide by lead oxide act to increases the magnetic susceptibility while lead cations act to close the glass structure and hence to increase the magnetic susceptibility of the whole glass in addition to the factor due to the differences between the molecular weights of both lead and zinc molecules.

Relation between the molar susceptibility and the molar volume

Recently, it was supposed that there may be a relation between the magnetic properties (a microscopic property) of a solid glass and its molar volume (a macroscopic property) [26].

However, in this paper, Figure 11 exhibits the relation between the molar magnetic susceptibility and the molar volume of the studied glasses, where it shows a linear increase with slope equal 0.8935 esu / cm^3 mol^{-1}. This means that any change in the molar volume of a glass affect directly its magnetic properties (volume, mass, & molar susceptibilities).

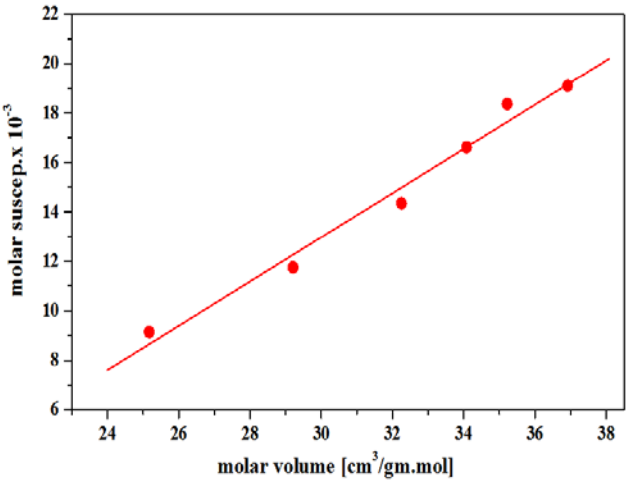


Fig. 11. Relation between the molar magnetic susceptibility and molar volume of the studied glasses

Gamma-ray shielding properties

In order to check the ability of studied glasses to attenuate the ionizing radiation, especially gamma-ray and other dangerous radiations, WIN-X COM program (based on the mixture rule) was used. The mass attenuation coefficients values have been calculated using Eq. (7) [7, 8].

Figure 12 exhibits the variation of the mass attenuation coefficient $(\mu/\rho)_m$ calculated values as a function of Pb_3O_4 content, at gamma-ray energies equal to 356 keV, 662 keV, and 1173 keV and 1333 keV, emitted from ^{133}Ba, ^{137}Cs, and ^{60}Co, respectively.

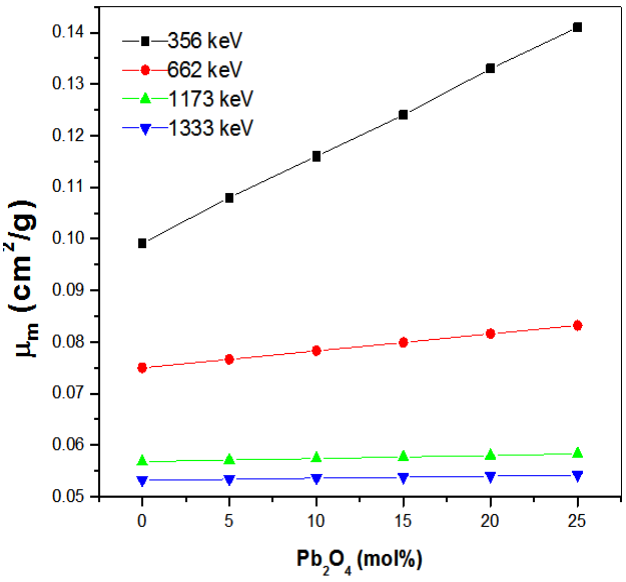


Fig. 12. The variation of the mass attenuation coefficient calculated values as a function of Pb_3O_4 content

It is seen that $(\mu/\rho)_m$ shows a gradual linear increase as Pb_3O_4 was gradually increased for all the applied gamma-ray energies. On the other hand, the half value layer (HVL) was also calculated for all the studied glass samples and at all the selected gamma-ray energies [20].

Figure 13 shows the variation of the HVL as a function of Pb_3O_4 content, at all the supposed gamma-ray energies.

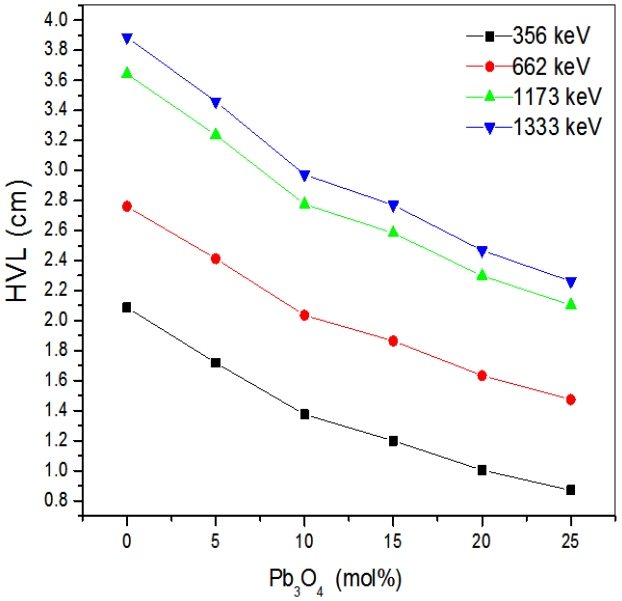


Fig. 13. The variation of calculated HVL values as a function of Pb_3O_4

As expected, it shows a gradual decrease with increasing Pb_3O_4 content. In addition, the mean free path values were calculated, and Figure 14 illustrates its variation with Pb_3O_4 content, showing also a gradual linear decrease.

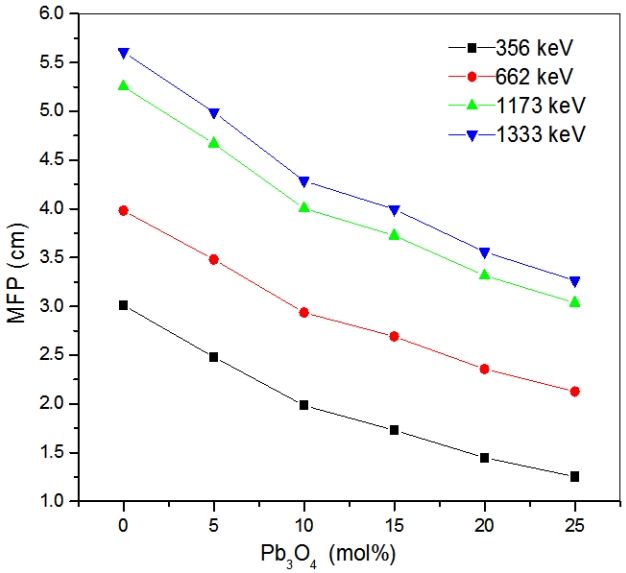


Fig. 14. The variation of the calculated MFP values as a function of Pb_3O_4 content

The observed increase of $(\mu/\rho)_m$, and the gradual decrease of both HVL and MFP values can be attributed to the gradual replacement of ZnO by Pb_3O_4, since the total mass attenuation coefficient of PbO is very high compared with that of ZnO, as well as the gradual increase of the density of the studied glass samples.

However, it is worth to recognize that the increase of Pb ions in the glass network act to increase the shielding parameters of the studied glasses.

Conclusions

In order to study shielding properties for this type of glass composition, as Pb_3O_4 content was increased at the expense of ZnO, as well its structural, thermal and physical properties modified.

ME and IR analyses, as well as the verification of quadrupole energy (T_g - Δ) rule confirmed that, most iron ions occupy the tetrahedral coordination state (FeO_4), acting as NWF. Also, from IR analysis it can be concluded that boron ions occupy different structural groups in the network of the studied glasses.

The characteristic temperatures (glass transition - T_g, crystallization - T_c, and melting - T_m) of the studied glasses show a slight gradual decreasing, while their thermal stability (T_c - T_g) shows gradual increases.

Both the increase of density and the decrease of molar volume may be due to the difference between the molecular weights of Pb_3O_4 and ZnO, as well as the gradual increase of both the number of oxygen and lead ion in the glass networks.

All the studied glass samples exhibit paramagnetic character and the molar magnetic susceptibility was found to increase due to the diamagnetic character of Zn. Also, a, linear correlation was established between the molar magnetic susceptibility and the molar volume of the studied glasses.

The dependence of the radiation shielding properties on Pb_3O_4 content has been theoretically undertaken. Both the linear and mass attenuation coefficients increased with increasing Pb_3O_4 concentration in the glass network for various low gamma-ray photon energies, but both the HVL and MFP values decreased. It can be stated that, all the lead containing glasses can act as good shielding materials.

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