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Optical spectroscopic analysis of Fe_2O_3 doped CuO containing phosphate glass

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Abstract The effect of Fe_2O_3 in sodium zinc phosphate glass system containing CuO with the chemical composition $40P_2O_5{:}38ZnO{:}1CuO{:}(21-x)Na_2O{:}xFe_2O_3$ (where $x = 1, 2, 3,$ 4, 5 and 6 mol%) has been studied. The glass formability of the prepared samples was examined by means of XRD which proved that there are no natural crystal contents. Archimedes method has been employed to measure the density of the prepared glass samples hence, the molar volume was calculated. The density and the molar volume were found to be increased by increasing Fe_2O_3 content. The optical spectroscopic analysis for the obtained glass samples has been carried out over the whole range (190–1000 nm) for studying the effect of bandpass absorption glass filter, its color peak center and UV cut-off. The center for bandpass filter is found to exhibit a red shift by increasing Fe_2O_3 content. Moreover, all glass samples showed a bandstop in UV-range which was increased by increasing Fe_2O_3 content. The results reveal the practicality of this glass composition in optical color glass bandpass filter for UV preventing applications such as UV-Laser protection.

Keywords Fe_2O_3 · Phosphate glass · Bandpass filter · UV-Laser protection

1 Introduction

Over the past few years, phosphate glass has opened a new science in optics called photonic glass science. This science covers all the new modern branches in optical materials like laser, optical amplifiers, nonlinear optics and optical glass filters (Yamane and Asahara 2005; Gan and Xu 2006; Koughia et al. 2007). In view of physical and

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chemical properties, the phosphate glasses possesses low glass transition temperatures, low melting and softening temperatures, high electrical conductivity and interesting dielectric properties (Jiménez 2016; Sreehari Sastry and Rupa Venkateswara Rao 2014; Mouss et al. 2016). Moreover, the structural role of zinc oxide in many oxide glasses is exclusive since zinc oxide can act either as a glass former and a glass modifier. When acts as a glass former, ZnO enters the network with ZnO_4 structural units. Meanwhile as a network modifier, zinc ion is octahedrally coordinated so that it behaves like conventional alkali oxide modifiers (Szumera et al. 2016). On the other hand, copper-doped phosphate glasses exhibit interesting electrical and optical properties that make them suitable for use as super-ionic conductors, solid state lasers, color filters, and can be doped with high level by metal ions and remain amorphous (Mugoni et al. 2016; ElBatal et al. 2013; Ouis et al. 2016). Copper phosphate glasses are usually used as bandpass filters (Elhaes et al. 2014a). The bandpass filter is the filter that can transmit a group of light and prohibit another. Light filtration by optical material can be classified into absorption glass filters or reflection glass filters, this filtration of light depends on the spectroscopic techniques and its applications (ElBatal et al. 2011). Copper phosphate glasses exhibit an optical absorption band in the visible-near infrared region and fundamental optical absorption edge in the ultraviolet region, i.e. it can be used as a bandpass filter. The center of the bandpass filter and its color play an important role for controlling the light pass (Aboulfotoh et al. 2014; Elbashar 2016; Elbashar et al. 2016a, 2017). Many studies have been performed on the effect of copper oxide with different glass former and with different transition elements (Elbashar et al. 2016b, c; Badr and Elbashar 2016; Elbashar 2015; Saeed et al. 2015; Elhaes et al. 2014b; Rayan et al. 2013, 2015). However, using copper oxide with some transition metals oxide like iron oxide is rare for bandpass filter applications. As the iron oxide has good optical and magnetic properties (Li et al. 2013, 2014; Lu et al. 2015; Joseph et al. 2015; Doweidar et al. 2005; Magdas et al. 2008; Moguš-Milanković et al. 2001, 2003; Moustafa et al. 2004), the recombination between copper oxide and iron oxide for changing the color of the filter can have a crucial interest for optical industry. The present work is devoted to characterize the chemical composition of sodium zinc phosphate glass containing copper oxide by doping it with iron oxide at different ratios; to obtain new properties of bandpass absorption glass filter used for preventing ultra-violet and other related applications in optics such UV-laser protection.

2 Experimental method

In the present work, the conventional melt quenching technique had been employed to prepare the glass samples with chemical composition $40P_2O_5$:38ZnO:1CuO:$(21 - x)$ Na_2O:xFe_2O_3 (where $x = 1, 2, 3, 4, 5$ and 6 mol%). The analytically pure grade chemicals were mixed and grinded using mortar for 30 min, and then calcinated in a porcelain crucible using muffle furnace for 1 h at 250 °C to release the gases from the chemicals like CO_2 and NH_3. Then the samples in the porcelain crucible was placed into a melting furnace for 1 h at 1000 °C, and shaken clockwise to ensure that the material is in high homogeneity (Rayan et al. 2013; Li et al. 2013; Lu et al. 2015). Finally, the casting was quenched and annealed at 250 °C using stainless steel mould with pressing plate to obtain thin disks in order to study the optical properties. The annealing technique is required in order to remove the internal stress that remained in the glass during the quenching. The crystallite phases of the obtained transparent samples were identified by means of X-ray diffraction

(XRD) technique on a Brucker axis D8 diffractometer with crystallographic data software Topas 2, using Cu-Kα (λ = 1.5406 Å) radiation operating at 40 kV and 30 mA at a rate of 2°/min. The diffraction data were recorded for 2θ values between 4° and 70°.

The glass samples were obtained almost as disks of diameters ≈2.75 cm. The glass density was determined at room temperature using the conventional Archimedes method, with toluene as an immersion liquid of stable density (0.868 g/cm^3) as (Rayan et al. 2013):

$$\rho = \frac{W_{air}}{W_{air} - W_{liq}} \rho_0 \tag{1}$$

where, W_{air} and W_{liq} are the weights of the sample in the air the liquid respectively. The molar volume was calculated from the molecular weight, M_W and the density, ρ as (Shelby 2005):

$$V_M = \frac{M_w}{\rho} \tag{2}$$

The relative error in the density and molar volume measurements were ±0.0002 g/cm^3 and ±0.0023 cm^3/mole respectively.

The optical absorption and transmission spectra were recorded at room temperature using UV/VIS absorption (JASCO V570) spectrophotometer over the wavelength range (190–1000 nm).

3 Results and discussion

The glass samples have been investigated by XRD technique to examine their amorphous nature as shown in Fig. 1. The pattern showed fluctuations and no sharp peaks were found that the prepared samples exhibit an amorphous trend.

The density of the glass samples and the corresponding molar volume for different ratios of iron oxide are listed in Table 1 and the dependence of density and molar volume on Fe_2O_3 content is shown in Fig. 2. The density is found to be increased as Fe_2O_3 content is increased. This can be attributed to the replacement of Na_2O by Fe_2O_3 and the change in density is most likely related to the difference in atomic weights of Na and Fe. Moreover, the molar volume is found to be increased proportionally to Fe_2O_3 content. This is unusual behavior between density and molar volume; because the molar volume and density are

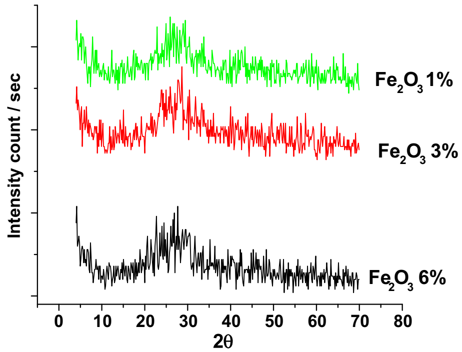

Fig. 1 XRD pattern for the glass samples

Table 1 Density, molar volume, oxygen packing density and the optical band gap of glass samples

Fe_2O_3 content (mole %)	Density (g/cm^3)	Molar volume (cm^3/mol)	Oxygen packing density (OPD)	Optical band gap (eV)
Fe_2O_3 1%	3.029	33.839	77.427	3.28
Fe_2O_3 2%	3.031	34.137	77.336	3.17
Fe_2O_3 3%	3.034	34.422	77.276	3.12
Fe_2O_3 4%	3.037	34.708	77.215	3.04
Fe_2O_3 5%	3.041	34.992	77.162	2.99
Fe_2O_3 6%	3.046	35.257	77.148	2.85

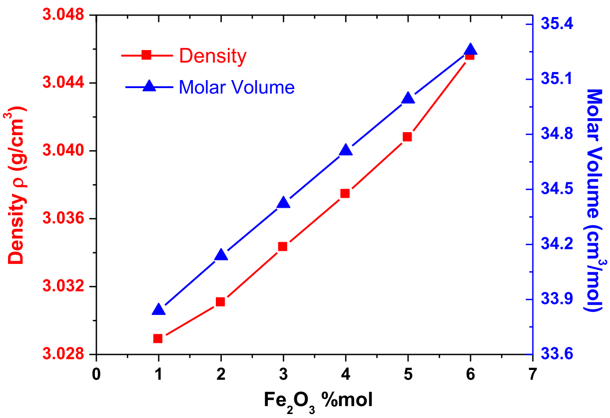

Fig. 2 Effect of Fe_2O_3 content on the density and molar volume

changed with inverse direction of each other's direction, but in the present study the trend of density and molar volume has the same increments (Mekki et al. 2003; Bray and O'Keefe 1963; Rani et al. (2009)). Although Fe is denser than Na, another competing factor must be considered; namely, the number of non-bridging oxygen (NBO) atoms which leads to open the glass structure, i.e. increasing of the molar volume. Calculation of oxygen packing density (OPD) is mainly considered for measuring the tightness of packing of the oxide network. The oxygen packing density was calculated using the following relation (Doremus 1994; Rao 2002; Garrett 1998; Mauro 2000):

$$OPD = \left(\frac{\rho}{M_w}\right) \times n_O \qquad (3)$$

where ρ is the measured density, M_W molecular weight and n_O the number of oxygen atom per formula unit. The calculated values oxygen packing density for different Fe_2O_3 content is listed in Table 1. It is evident that the oxygen packing density decreases by increasing the Fe_2O_3 content. In this respect, the structure of glass network became less tightly packed and the degree of disorder is increased by increasing Fe_2O_3 contents, i.e. formation of open structure, which explains the observed results of molar volume.

In order to understand the chemical environment around iron ions in the glass matrix, optical absorption and transmission studies have been carried out. Figure 3 shows the optical absorption spectra of the glass samples over the wavelength range (190–1000 nm)

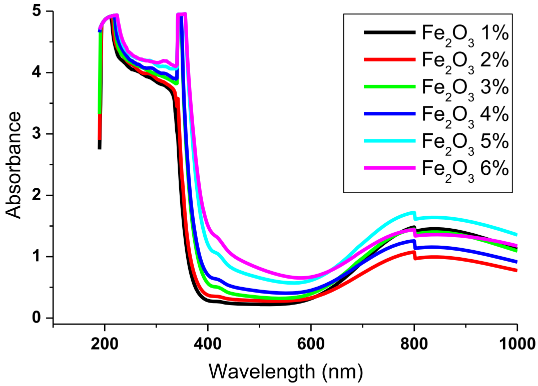

Fig. 3 Absorption spectra for the glass samples with different Fe_2O_3 content

for different ratios of Fe_2O_3. It can be noticed that the absorbance increases with increasing the contents of Fe_2O_3. It is evident that all samples exhibit an optical absorption band in both the visible-near infrared region and fundamental optical absorption edge in the ultraviolet region. The increase in absorbance with increasing the contents of Fe_2O_3 in the ultraviolet and infrared bands can be attributed to the coupling between the CuO and Fe_2O_3; since CuO has double absorption band in ultraviolet and infrared. This absorption bands in phosphate glass containing CuO can be assigned to the energy transitions: $^2B_{2g} \rightarrow {}^2B_{1g}$, $^2A_{1g} \rightarrow {}^2B_{1g}$ and $^2E_g \rightarrow {}^2B1\ _g$ (Rayan et al. 2013; Bae and Weinberg 1994; Takebe et al. 2007).

Figure 4 shows the optical transmission spectra of the glass samples in the wavelength range 190–1000 nm for different ratios of Fe_2O_3. The transmission spectra are consistent with the absorption data. The bandpass in optics is a technique that allows us to pass a band of spectral lines through a filter. The obtained results reveal a bandpass filter in the visible range with band stop in the ultra violet range. It should be mentioned that all glass samples show high absorption in UV region started from 190 nm. As shown in Fig. 5 and listed in Table 2, the UV cutoff is found to be increased from 344 to 381 nm by increasing Fe_2O_3 content. The shift of absorption edge or cutoff wavelength to longer wavelengths with increasing Fe_2O_3 content can be attributed to conversion of bridging oxygen atoms to non-bridging oxygen atoms. Therefore the negative charges present on the non-bridging oxygen atoms cause electron excitations with higher wavelengths (Chanshetti et al. 2011). It is well

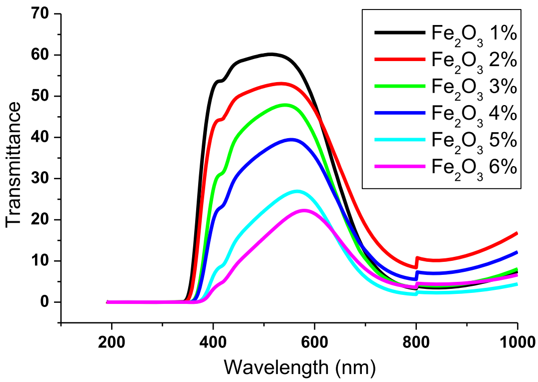

Fig. 4 Transmission spectra for the glass samples with different Fe_2O_3 content

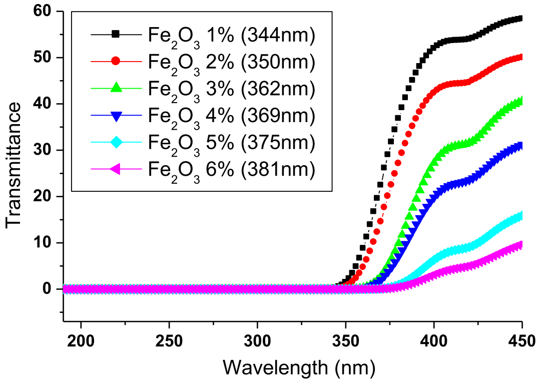

Fig. 5 UV band stop for glass samples with different Fe_2O_3 ratios

Table 2 The peak analysis of the bandpass filter

Fe_2O_3 content (mole %)	Area	Center (nm)	Width (nm)	Height	UV cutoff (nm)
Fe_2O_3 1%	14,787	508.71	188.59	62.559	190–344
Fe_2O_3 2%	15,706	520.03	219.52	57.087	190–350
Fe_2O_3 3%	9917.5	526.20	169.75	46.616	190–362
Fe_2O_3 4%	9793.7	534.75	195.33	40.004	190–369
Fe_2O_3 5%	5804.7	549.51	171.95	26.935	190–375
Fe_2O_3 6	4077.9	565.82	162.25	20.053	190–381

known that the UVC ranges up to 280 nm while, the UVB ranges from 280 to 315 nm and the UVA ranges from 315 to 380 nm. It can be concluded that the present study offers an excellent optical filters for preventing the whole UV range i.e. UVC, UVB and UVA. Considering the transmission height ranged from 62 to 23% as shown in Fig. 4, the obtained optical filters can be perfect for UV preventing applications such as UV-Laser protection.

Glasses containing conventional network modifiers are usually completely colorless in the visible region of the spectrum. It changes if the glasses contain at the same time a transition element such as: Cu, Ti, V, Cr, Mn, Fe, Co, and Ni are the most important. Among the theories that explain the coloration phenomenon is that of the Ligand-field theory of Hartmann. This theory predicts that the glasses coloration by the transition metals “3d” which is due to electronic transitions between energy levels of the electron-degenerate d. The color is also affected by the concentration of the transition metal. The effect of the concentration of is clear: increasing of concentration causes more absorption and consequently less transmission. The decrease in transmission height with increasing Fe_2O_3 content can be attributed to the replacement of Na_2O by Fe_2O_3 which results in attenuation of light in the transmission band. However, increasing Fe_2O_3 content exhibits a red shift in the center of the transmitted band as shown in Fig. 4 and listed in Table 2. The center of the bandpass filter is changed from 508 to 565 nm which is most likely related to the coupling between CuO and Fe_2O_3. This is very important for the color glass filters industry; because the variation of color is always needed in color glass optical filters.

The optical band gap, E_g of the glass samples was determined using the relation (Elhaes et al. 2014a; Aboulfotoh et al. 2014):

$$\alpha h\nu = B\left(h\nu - E_g\right)^2 \quad (4)$$

where, B is constant and α is the absorption coefficient that was calculated using the relation (Rayan et al. 2013; Punia et al. 2011; Varshneya 1994):

$$\alpha(\nu) = (1/d)\ln(I_0/I) = 2.303(A/d) \quad (5)$$

where, I_0 and I are the intensities of the incident and transmitted beams respectively, A the absorbance spectrum and d is the thickness of the glass samples. However, for non-crystalline systems it is customary to plot $(\alpha h\nu)^{1/2}$ as a function of photon energy ($h\nu$) in order to find the optical band gap as shown in Fig. 6. As listed in Table 1, the optical energy gap is found to be decreased from 3.28 to 2.85 eV by increasing the Fe_2O_3 content. This can be related to the progressive increase in number of non-bridging oxygen (NBO) atoms. It can be noticed that the optical energy gap and the UV cutoff have a reversible trend.

The study of refractive index is very important for any optical material. In glasses, the electronic polarization frequencies are in the UV, where they induce a strong absorption, and the molecular polarization frequencies are in the infrared, where they cause multi-phonon absorption. In between, the refractive index has weak frequency dependence, decreasing with increasing wavelength (normal dispersion) (Simmons and Potter 1999).

However, the measurement of refractive index as a function of wavelength was determined from the relation (Simmons and Potter 1999):

$$R = \frac{(n-1)^2}{(n+1)^2} \quad (6)$$

with, R is the reflectance which can be calculated by the following simple equation (Simmons and Potter 1999):

$$A + T + R = 1 \quad (7)$$

where, A is the absorbance and T is the transmittance.

The chemical composition of glass is considered as the main factor that can affect the refractive index. However, the refractive index is a function of ion refraction as produced by Clausius–Mossotti method (Simmons and Potter 1999; Elbashar and Saeed 2015). As

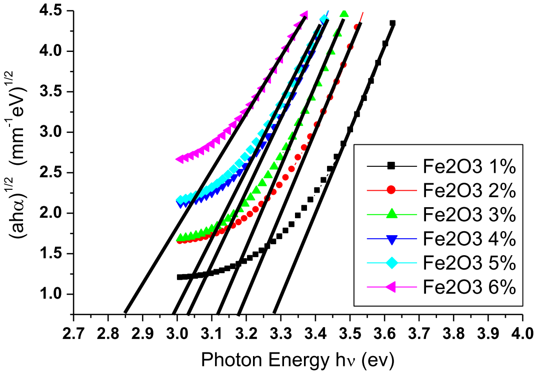

Fig. 6 Determination of the optical band gap

shown in Fig. 7, the refractive index is found to be increased by increasing the Fe_2O_3 content. This can be attributed to the replacement of Na_2O by Fe_2O_3 and the change in refractive index is most likely related to the difference in ion refraction of Na and Fe. It can be concluded that the ion refraction of Fe is higher than that of Na which explains the increasing of refractive index by increasing Fe_2O_3 content.

The permittivity (ε), the electric susceptibility (χ) and the polarizability (γ) were respectively calculated using the following equations (Simmons and Potter 1999):

$$\varepsilon = n^2 \tag{8}$$

$$\chi = \frac{(\varepsilon - 1)}{4\pi} \tag{9}$$

$$\gamma = \frac{3}{4\pi N}\frac{\varepsilon - 1}{\varepsilon + 2} \tag{10}$$

The extinction coefficient (k) and the dielectric constants (ε' and ε'') were respectively calculated using following equations (Simmons and Potter 1999):

$$k = \frac{\alpha\lambda}{4\pi} \tag{11}$$

$$\varepsilon' = n^2 - k^2 \tag{12}$$

$$\varepsilon'' = 2nk \tag{13}$$

where, α is the absorption coefficient, λ is the wavelength, n is the refractive, index ε' the real part and ε'' is the imaginary part.

The wavelength dependence of permittivity, electric susceptibility, polarizability, extinction coefficient and the dielectric constants exhibit higher values for higher Fe_2O_3 contents as indicated by Figs. 8, 9, 10, 11, 12, and 13. According to Clausius–Mossotti, this can be attributed to the increase ion refraction due to the change in chemical composition of the glass material (Fowler and Pyper 1985; Duffy 1989, 2002).

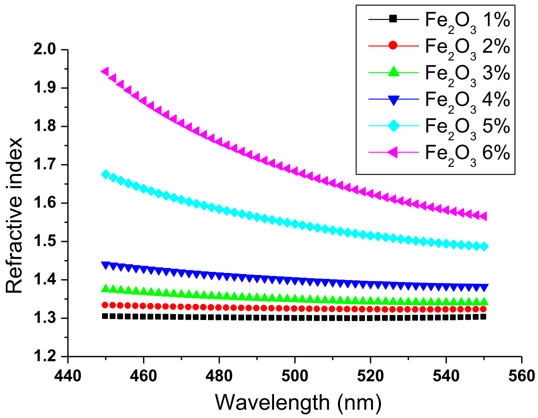

Fig. 7 Wavelength dependence of the refractive index with different Fe_2O_3 content

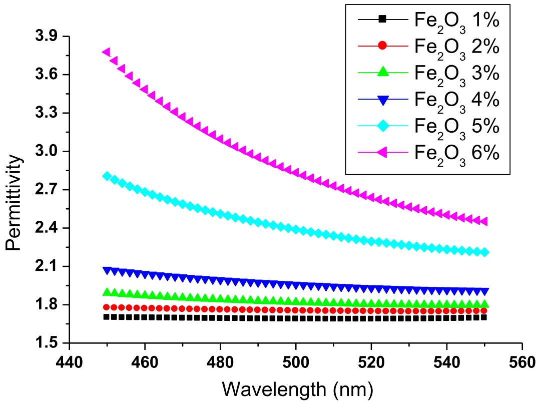

Fig. 8 Wavelength dependence of the permittivity with different Fe_2O_3 content

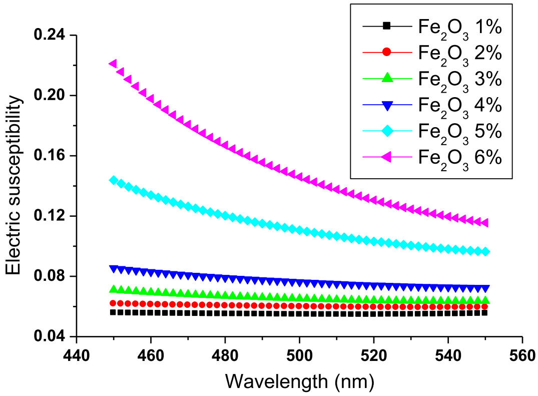

Fig. 9 Wavelength dependence of the electric susceptibility with different Fe_2O_3 content

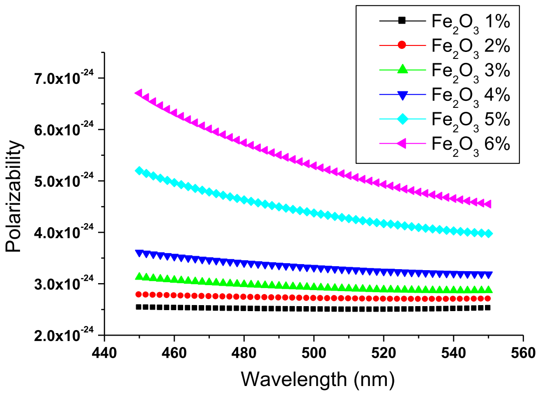

Fig. 10 Wavelength dependence of the polarizability with different Fe_2O_3 content

4 Conclusion

The present work is devoted to study the effect of Fe_2O_3 in a glass system of chemical composition $40P_2O_5$:38ZnO:1CuO:$(21 - x)Na_2O$:xFe_2O_3 (where $x = 1, 2, 3, 4, 5$ and 6 mol%). XRD pattern confirm the glass formability of the prepared samples. Both the

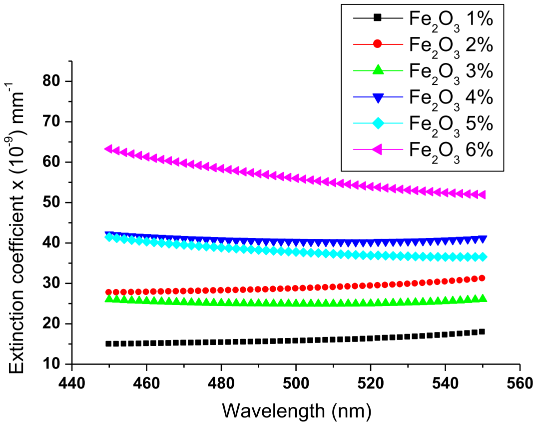

Fig. 11 Wavelength dependence of the extinction coefficient with different Fe_2O_3 content

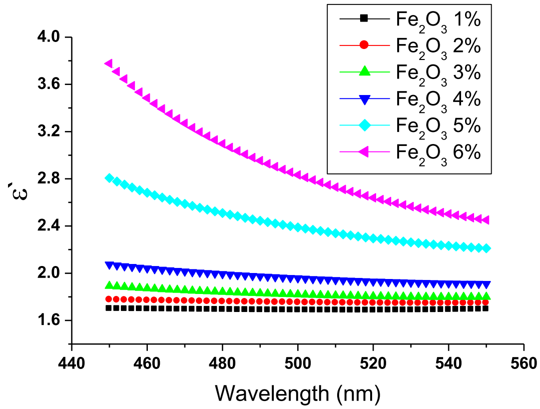

Fig. 12 Wavelength dependence of ε' with different Fe_2O_3 content

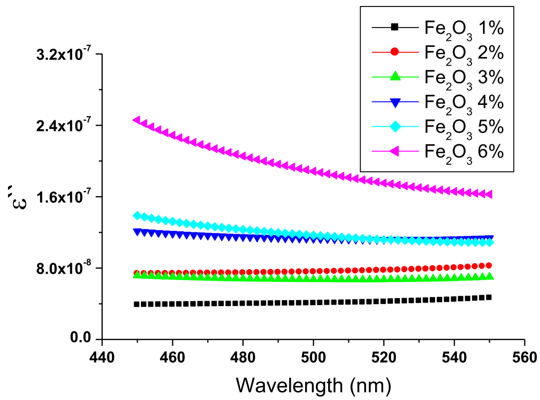

Fig. 13 Wavelength dependence of ε'' with different Fe_2O_3 content

density and molar volume of are found to be increased by increasing Fe_2O_3 content. The optical studies had concluded that the Fe_2O_3 acts as a network modifier with sodium zinc copper phosphate glasses. By increasing Fe_2O_3 content, the UV cutoff is increased from 344 to 381 nm meanwhile, the transmission peak centre is shifted from 508 to 565 nm. The optical energy gap is found to be decreased from 3.28 to 2.85 eV by increasing Fe_2O_3 content. The results assert a progressive increase in number of non-bridging oxygen (NBO) atoms. The wavelength dependence of the refractive index and extinction coefficient and some other related optical properties had been studied. The results reveal the practicality of using the prepared glass system as bandpass filters in the visible region with excellent band stop in UV range which can be perfect for UV preventing applications such as UV-Laser protection.

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