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Optical Spectroscopic Study of Cobalt Oxide Doped Boron Glass and Its Ion Effect on Optical Properties

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IN this paper, the optical spectroscopic properties of Co_3O_4 doped borate glass with chemical composition: 60 B_2O_3, 30 ZnO, (10-x) Na_2O, x Co_3O_4, (where x= 0 (S0), 0.1 (S1), 0.2 (S2), 0.3 (S3), 0.4 (S4), 0.5 (S5) mol %) were investigated. The glass samples (S0-S5) were prepared by conventional melt quenching technique. The formability of all prepared glass samples was examined by XRD and UV–visible spectroscopy techniques. The amorphous nature of glass structure was confirmed by XRD- technique. Archimedes method was used to measure density and calculate the molar volume of the prepared glass composition. It was observed that the density and molar volume were found to be increased by increasing concentration of Co_3O_4. The optical spectroscopic characteristics of the obtained borate glass have been carried out over the whole range (190-2500nm) for studying the bandpass glass filter and UV cut-off. Through experimental data it was observed that the optical energy gap decreased from 3.25 to 3.03 eV by increasing doped cobalt oxide contents on borate glass. Furthermore, UV cut-off regions increased by increasing concentration of cobalt oxide. The effect of doping concentration on the optical properties of the prepared glass samples (refractive index, extinction coefficient, permittivity, dielectric constants, electric susceptibility and polarizabilities) was determined and studied.

Keywords: Borate glass. Co_3O_4. UV-VIS spectroscopic measurements. Bandpass filter.

Introduction

Glasses are super cooled liquids, transparent [1], high chemical inertness [2] and amorphous in nature so they do not exhibit optical anisotropy that is characteristic of some crystals [3]. Borate glass is characterized by low melting point, high bond strength, high, transparency and complex dependences of the properties on the composition [4-7]. Boric oxide (B_2O_3) acts as the best glass former, because of its higher bond strength, lower cation size and smaller heat of fusion [8]. Sodium borate glass with different modifiers are both of scientific and technological interest due to their structural peculiarities [5]. B_2O_3 glass is formed from BO_3 triangles that composed of three – membered (boroxol) rings [9]. Addition of some oxides to the glasses lead to a lot of new applications and improve the glass optical, electrical and physical properties [10, 14]. The introduction of oxide as a modifier to boric oxide glass can create structural changes and modify the properties of glass matrix. The modifier oxide changes a part of the BO_3 triangles to BO_4 tetrahedral thus cracking bridging oxygen bonds to create non bridging oxygen (NBOs) [11-13]. Doping transition metals ions improve the physical, electrical [14] and optical properties of the glass [15]. ZnO has dual role in borate glass

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that it acts either as modifier or network former [16, 17]. When it acts as a network modifier, it is coordinated in an octahedral structure [18], and thus converting bridging oxygen to non-bridging oxygen in glass specimens. While when it acts as a glass former, it enters the network with ZnO_4 structural units [19,20] The change in color of the glasses and the change in the magnetic moment depend not only on the content of alkali oxide present but also on the nature of the transition metal and the alkali-metal oxide used [21]. Glass containing cobalt ions have intense blue color [15, 21, 22] and used as optical filter. Glass scientists interpreted the optical spectra of glass containing cobalt ions where a balance between tetrahedral and octahedral coordination states of cobalt ions exist in different glasses [23]. Combination of both octahedral and tetrahedral states explains the small difference between the ligand field stabilization energies of the two coordination symmetries [23].

The bandpass filter transmits a region of light and prevent another [17- 21, 24], i.e. some filters are selective absorption and transmittance [24]. Absorption glass filter is used to prevent UV [17]. In this work, different samples (S0 -S5) of sodium zinc borate glass will be prepared by cobalt oxide as doping material with different ratios. The physical properties of this borate glass; such as density, molar volume, structure XRD, transmission, absorption, and refractive index, will be studied to examine the effect of cobalt oxide concentration. The application of such new glass samples as absorption filter will be examined.

Experimental Method

Glass preparation:

In this work, the conventional melt quenching technique was used to prepare different samples of sodium zinc borate glass doped with cobalt oxide having the general composition: 60 B_2O_3 , 30 ZnO , (10-x) Na_2O ,x Co_3O_4 ,(where x= 0(S0), 0.1(S1) , 0.2(S2) , 0.3(S3) , 0.4(S4) , 0.5 (S5) mol%). The samples were prepared from pure grade chemicals in the powder form. B_2O_3 is started as (H_3BO_4), sodium oxide (Na_2O) as anhydrous sodium carbonate (Na_2CO_3), zinc oxide (ZnO) as zinc oxide powder, and cobalt oxide as (Co_3O_4). Pure chemical components were mixed and grinded using mortar for 30 min then heated in porcelain crucible using muffle furnace for about 1 hour at 330 C^0 to get rid of gases such as H_2O, NH_3, NO_2, and CO_2 .After that the obtained mixtures were put into a melting furnace for 1 h at 1000 C^0, the molten products were shaken many times to confirm high homogeneity and to remove bubbles of gases [25-27]. Finally, the casting was quenched and annealed at 250 C^0 for 30 min using stainless steel mold with pressing plate to get thin disks with small thickness in millimeter unit then samples left to be cooled at room temperature. Annealing technique is essential step to remove the internal stress that stilled in glass during quenching.

Glass Characterization

X – Ray diffraction (XRD) was used to identify the crystallite phase of transparent samples, using a Brucker axis D8 diffract meter with crystallographic data software Topas 2, using CuKα (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°.

Density measurements were carried out at room temperature using Archimedes method with ethanol as immersion liquid (density ρ_o= 0.888g/cm3). The density and molar volume Vm of the samples were calculated according to Eqs. (1, 2), respectively [27].

$$\rho = W1/(W1\text{-}W2)\ \rho_o \qquad (1)$$

Where W1, W2 are weights of sample in air and ethanol, respectively.

$$Vm = Mw/\rho \qquad (2)$$

Where Mw is a molar mass

The relative error in the density and molar volume measurements were ±0.0002 g/cm3 and ±0.0023 cm3/mole, respectively.

Oxygen packing density OPD is a measure of tightening of packing of oxide network and can be determined by using the following formula [28, 29]

$$OPD = (n_o*\rho)/Mw \qquad (3)$$

Where n_o is the number of oxygen atoms per formula unit.

The optical absorption and transmission spectra were recorded at room temperature using UV/VIS absorption (JASCO V570) spectrophotometer in the range of wavelength (190–2500) nm.

Results and Discussion

Different samples of sodium zinc borate

glass doped with cobalt oxide were prepared by quenching melting technique as mentioned above. The resulted undoped glass sample (S0) was colorless and transparent but after doping by cobalt oxide, the borate glass specimens (S1- S5) were blue color and less transparent than undoped glass sample (S0). Also, it was observed that as increasing of doped cobalt oxide content the intensity of blue color increased while the glass transparency decreased.

XRD technique

Fig.1. shows the XRD spectra of the prepared glass samples. The XRD pattern of the studied samples consisted of fluctuations and no sharp peaks were found indicating that these specimens had an amorphous structure of all samples. Therefore, no change in its nature before and after sequential addition of transition metal oxide (Co_3O_4).

Density, oxygen packing density and molar volume

The density and corresponding molar volume of glass samples were calculated as shown before and the results were given in table1. The results indicated that the density of undoped glass (S0) is equal to 3.063 g cm^{-3} while that of glass samples of doped cobalt oxide slightly increased almost at rate +0.002 with increasing of Co_3O_4 content , Fig .2. This due to the difference in atomic weights between Na and Co. Furthermore, molar volumes of doped glass samples were found to be increased step wisely more than undoped with small percentage, almost +0.025, with increasing Co_3O_4 concentration as shown in fig.2. Therefore, it was clear that the density and molar volume of the samples increased with each other as increasing concentration of Co_3O_4, this observation is opposite to what is well known about the relation between density and molar volume [30-32]. Two factors controlling this observation; first is Co ion is heavier than Na ion, the other factor is that the number of non-bridging oxygen (NBO) atoms increases leading to open glass network structure and thus increase molar volume. Table 1 showed that oxygen packing density (OPD) values of doped cobalt oxide samples S1-S5 are less than that of S0 sample (93.12) and the difference was inversely proportional to the amount of doping Co_3O_4. As a result, the structure of doped glass network became less tightly packed and the degree of disorder increased by the presence of Co_3O_4, i.e. formation of open structure which interpreted the observation of increasing molar volume.

Measurements of UV-VIS spectroscopy

In this part, optical transmission/absorption spectra of the prepared glass were investigated to reveal several optical properties and study the effect of doped Co_3O_4 concentration on these properties.

Fig.3. illustrates the optical absorption spectra of the prepared glass samples in the whole range of wavelength (190-2500 nm). The spectra Co^{2+} ions doped glass samples manifested characteristic in UV-VIS region according to ligand field theory. Fig.3. showed two absorption bands the first broad band placed between (415-746 nm) can be assigned energy transition: $^4A_2$ (^4F) $\rightarrow ^4T_1$ (^4P) transitions. The second broad band located between (850-1945 nm) can be attributed to $^4A_2 \rightarrow ^4T_{1g}$ (^4F) transitions. The sample observed blue color may be indicated tetrahedral sites [15, 21, 33-35]. Transitions observed are considered as multi absorption. This interpreted broadening of bands. As concentration of Co_3O_4 increased the absorbance and intensity of blue color samples glass increased.

The transmission spectra were consistent with absorption data. Fig.4. the optical transmittance spectra of glass samples in wave length range (190– 2500 nm) for different concentrations of Co_3O_4. As concentration of cobalt oxide increased and concentration of sodium oxide decreased the transmission decreased and intensity of blue color increased of glass composition. The transmission height ranged from 53.13 to 23.3 % as shown in fig. 4, the obtained optical filter can be perfect for UV preventing application.

UV cut-off region of the studied borate glass samples given in figure 5 and table 2, increased from 320nm (S0) to 370 nm (S1-S5) with increasing Co_3O_4 contents. This increment with increasing of Co_3O_4 concentrations can be attributed to conversion of bridging oxygen atoms to non-bridging oxygen atoms. So the negative charges present on the non-bridging oxygen atoms led to the electron excitations with higher wavelengths [36]. The conventional network modifiers contained in glass system are generally colorless in the visible region of the spectrum. This colorless glass changed to color due to the existence of transition metal element such as Co, Cu, Fe, V, Ti and Ni. This phenomena can be explained by the Ligand – field theory of Hartmann. This theory explained the coloration of glass by transition elements (3d) that is due to electronic transitions between energy levels of the electron – degenerate d. The coloration of glass is explained by transition metals '3d' due to electronic transitions between energy levels of electron degenerate d. Increasing of concentration

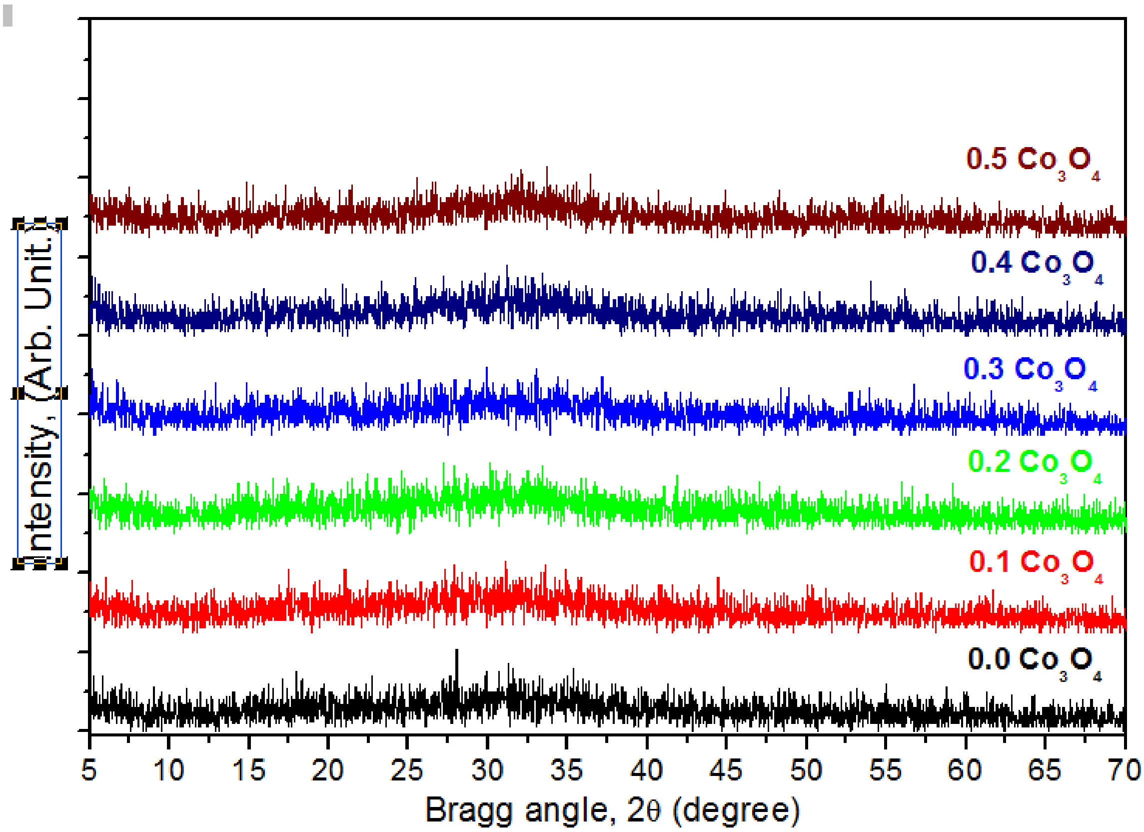

Fig. 1. XRD pattern for the prepared glass samples.

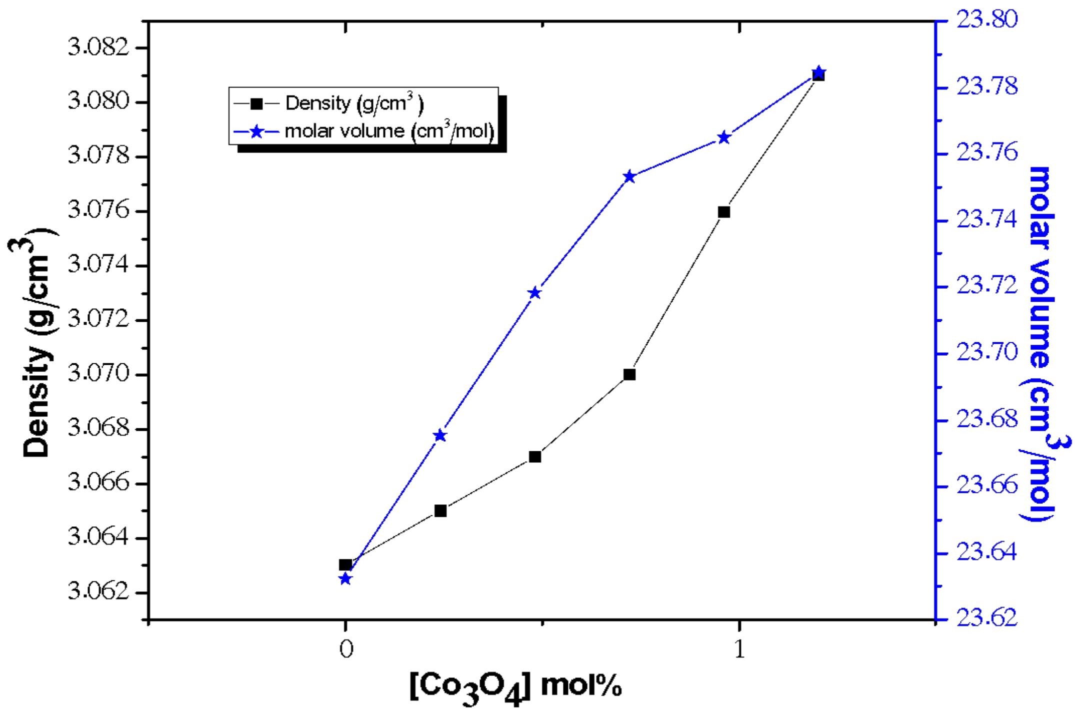

Fig.2. Effect of Co_3O_4 concentration on density and molar volume of the prepared glass samples.

TABLE 1. Composition, density (ρ), oxygen packing density (OPD) and molar volume (Vm) of the studied glass samples.

Samples	Glass composition (mol %)				Molar mass (Mw)	ρ (g cm^{-3})	Vm (cm^{-3}mol^{-1})	OPD
	B_2O_3	ZnO	Na_2O	Co_3O_4				
S0	60	30	10	0.0	72.386	3.063	23.632	93.12
S1	60	30	9.9	0.1	72.565	3.065	23.675	93.09
S2	60	30	9.8	0.2	72.747	3.067	23.718	93.07
S3	60	30	9.7	0.3	72.922	3.070	23.753	93.05
S4	60	30	9.6	0.4	73.101	3.076	23.765	93.00
S5	60	30	9.5	0.5	73.280	3.081	23.785	92.99

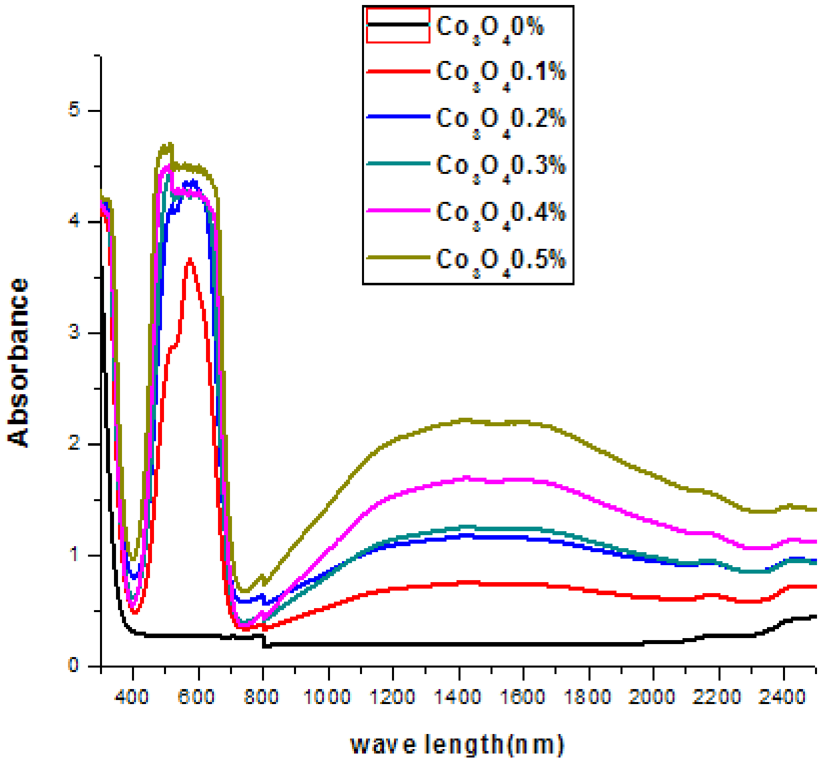

Fig.3. Absorption spectra for the glass samples with different Co_3O_4 contents.

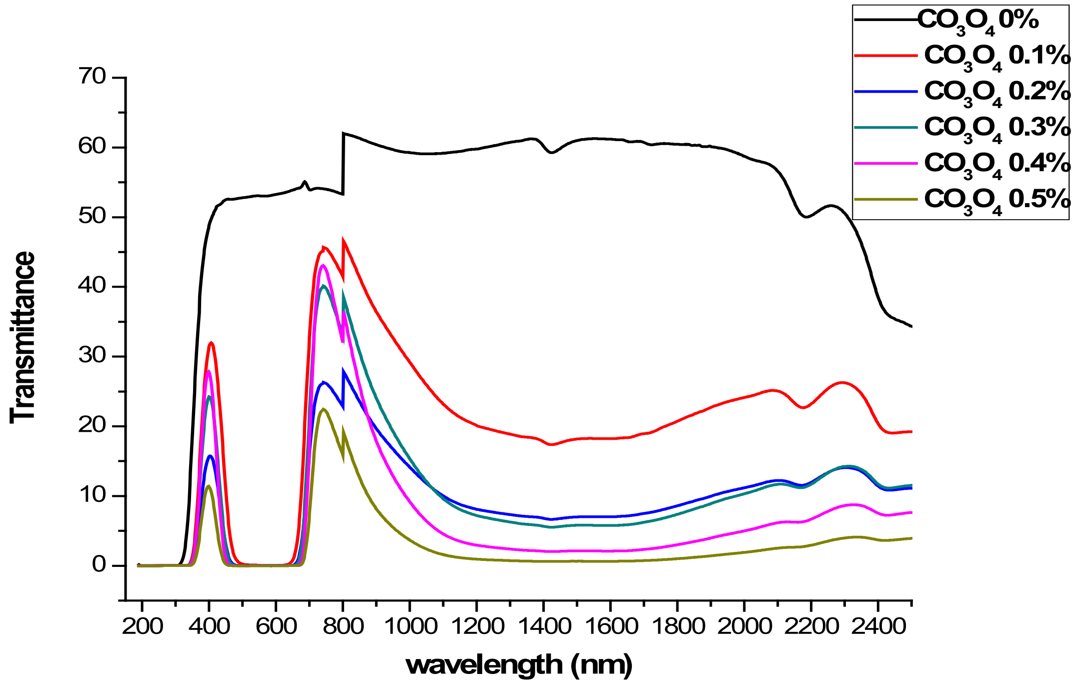

Fig.4. Transmission spectra for the glass samples with different Co_3O_4 content.

TABLE. 2. The cut-off for UV–Vis region for the glass samples with different Co_3O_4 content.

Co_3O_4 concentration	S0	S1	S2	S3	S4	S5
UV-Vis cut off λ (nm)	320	350	355	360	365	370

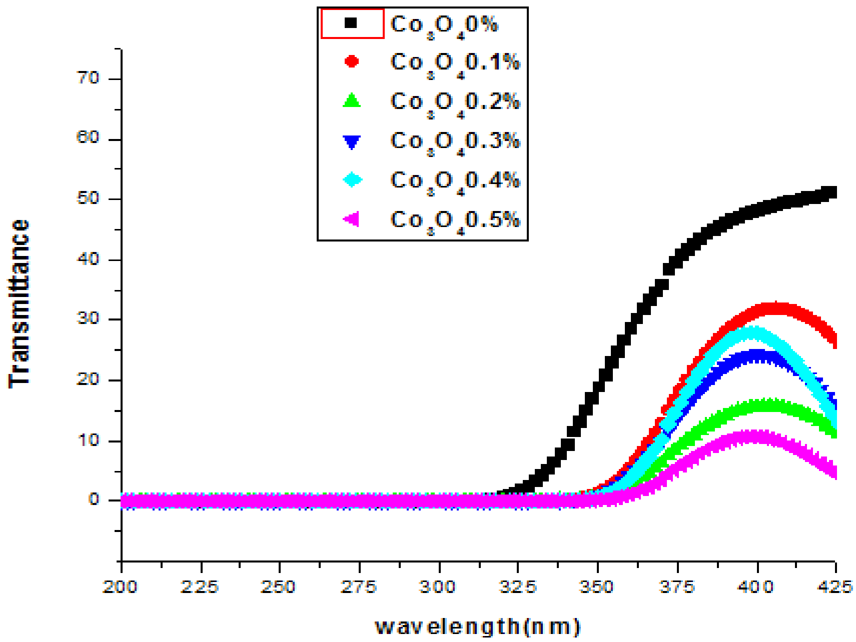

Fig.5.The UV–VIS cut-off versus wavelength of the glass samples.

of Co_3O_4 transition metal caused more absorption and consequently less transmission.

Study of refractive index (n) is very important for any optical material. In glass, the electronic polarization frequency is in UV, where it induced strong absorption. While molecular polarization frequency was in infrared, where it caused multi photon absorption. Refractive index had weak frequency dependence, decreasing with increasing wavelength (normal dispersion). Refractive index depends on the chemical composition of glass samples and is a function of ion refraction was created from Clausius–Mossotti method [37, 38].

The refractive index at certain wavelength can be calculated using reflectance (R) by the following relation [40].

$$R= (n-1)/(n+1) \quad (4)$$

On the other hand, the reflectance (R) was numerated by using the following equation, [40-42]

$$A+R+T=1; \text{So} \quad R=1-A-T \quad (5)$$

Where, A: absorbance R: reflectance T: transmittance

Fig. 6.Gives the reflectance (R) was decreased by increasing concentration of doping materials.

From data shown in fig.7, it was noted that the refractive index (n) was increased by increasing Co_3O_4 content. This is attributed to the difference in ion refraction of Na and Co ions, where the ion refraction of Co is greater than Na which interpreted increasing of refractive index by increasing doped cobalt oxide concentration.

The permittivity (ε), the electric susceptibility (χ) and the polarizability (γ) of the glass samples were calculated using the following equations [40, 42]

$$\varepsilon=n^2 \quad (6)$$

$$\chi = (\varepsilon-1)/4\pi \quad (7)$$

$$\gamma = (3(\varepsilon-1))/(4\pi N\varepsilon+2) (8)$$

The extinction coefficient (k) was calculated by using the following relation and the values are shown in Fig.8 [25, 40- 43]

$$K = \alpha\lambda/4\pi \quad (9)$$

Where α is absorption coefficient, λ is wavelength.

The real and imaginary parts of the dielectric constants (έ and ὲ) were calculated by using the following relations [25, 40- 43]

$$\acute{\varepsilon} = n2 - K2 \quad (10)$$

$$\grave{\varepsilon} = 2nK \quad (11)$$

The calculated values of permittivity , extinction coefficient , dielectric constants , electric susceptibility , polarizability and its wavelength and doping concentration dependence were showed in figs 9, 8 ,10 , 11 , 12 , 13. All the above calculated values were found to increase with Co_3O_4 contents, as marked according to Clausius–Mossotti. This is connected to increase ion refraction due to variation in chemical composition of glass samples [44- 46].

Finally, the optical band gap, Eg, of the borate glass was calculated using the relation [47, 48]

$$ah\upsilon=B(h\upsilon-Eg)2 \quad (12)$$

Where, B is constant and α is the absorption coefficient that was calculated by using the relation [25, 43, 49]

$$(\alpha\upsilon)=(1/d)\ln(lo/l)=2.303(A/d) \quad (13)$$

Where I_0 and I are intensities of incident and transmittance beam respectively, A is absorbance and d is the thickness of glass samples. For amorphous material the plot of $(h\upsilon\alpha)^{1/2}$ as function of photon energy (hυ) is used to determine optical band gap as shown in fig.14. Increasing Co_3O_4 concentration in glass decreased the optical band gap from 3.25 to 3.03 eV with as shown in figure .14, which was attributed to increase in number of non-bridging oxygen (NBO) atoms.

Conclusions

This work illustrated the effect of Co_3O_4 content on chemical composition and optical properties of borate glass system: 60 B_2O_3 , 30 ZnO , (10-x) Na_2O ,x Co_3O_4 , (where x= 0, 0.1 , 0.2 , 0.3 , 0.4 , 0.5 mol%). The undoped glass sample S0 was colorless and highly transparent while doped cobalt oxide (S1- S5) samples were blue color and less transparent than (S0). XRD proved the amorphous nature of the prepared glass systems and the absence of any crystalline phases. The study showed that the density and molar volume increased with increasing Co_3O_4 .The same trend was also found UV-VIS, cut-off values increased with increasing concentration of doped material. From practical experiment turned out that the undoped glass sample was colorless and high transparent but coloration of glass, intensity of blue color increased and transparency decreased with doping concentration. The optical energy gap decreased from 3.25 to 2.03 eV due to increase in number of non – bridging oxygen (NBO). The results also showed increasing in refractive index, permittivity, extinction coefficient, dielectric constants, electric susceptibility, and polarizability with increasing

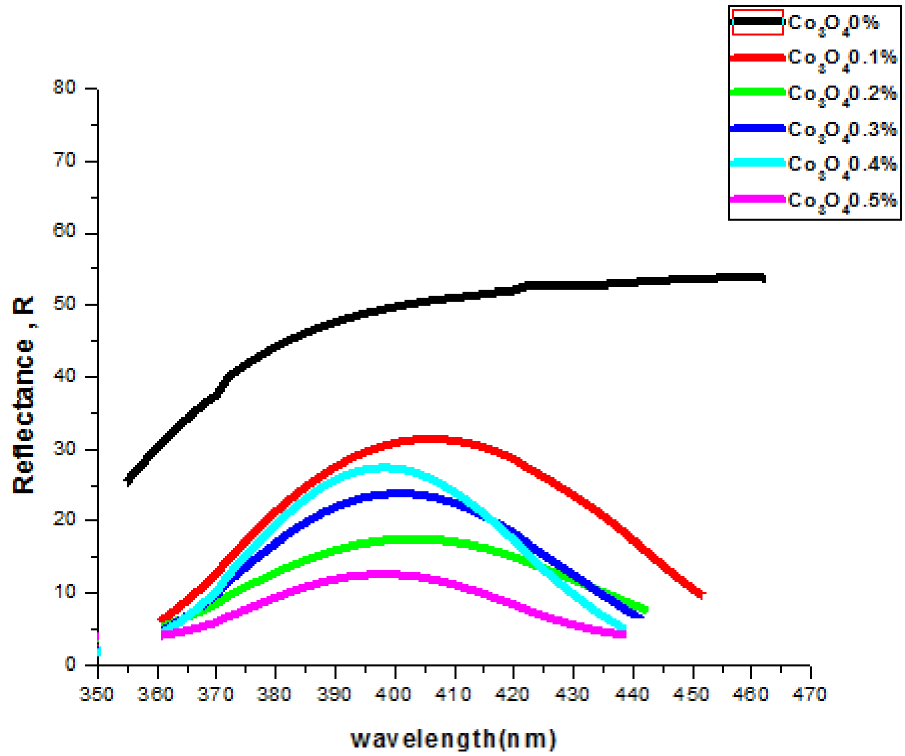

Fig.6.The calculated reflectance, R, versus wavelength for glass samples.

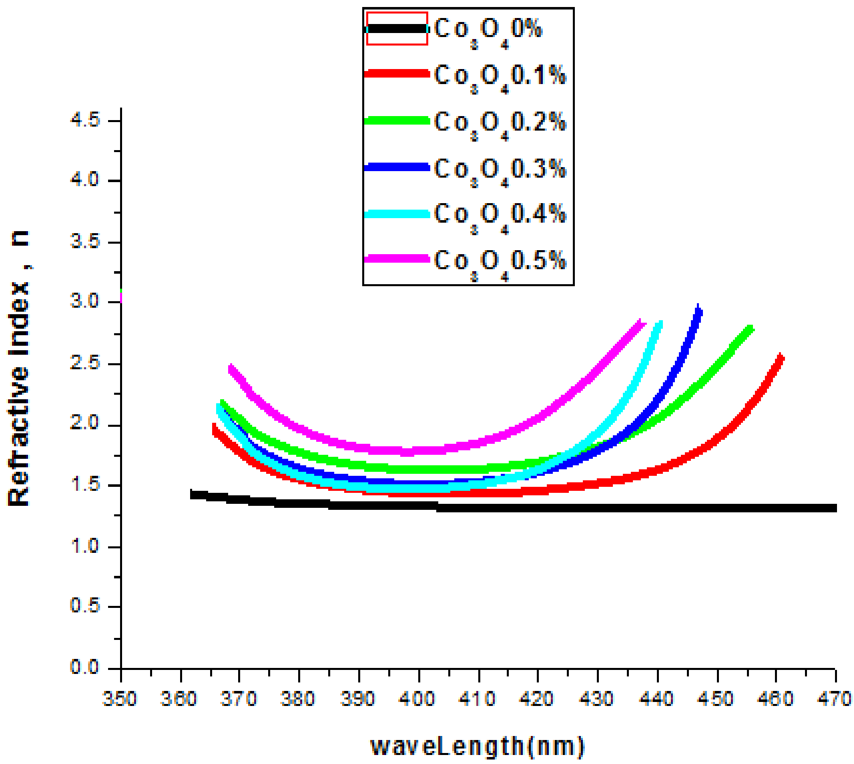

Fig.7.The refractive index, n, versus wavelength for glass samples.

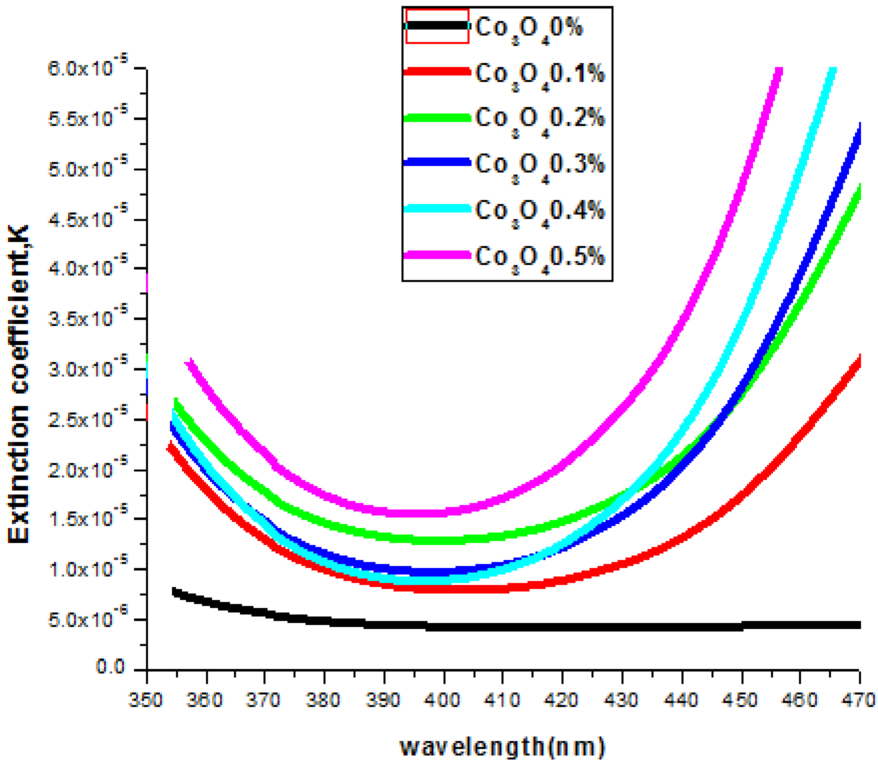

Fig.8.The extinction coefficient, K versus wavelength for present samples.

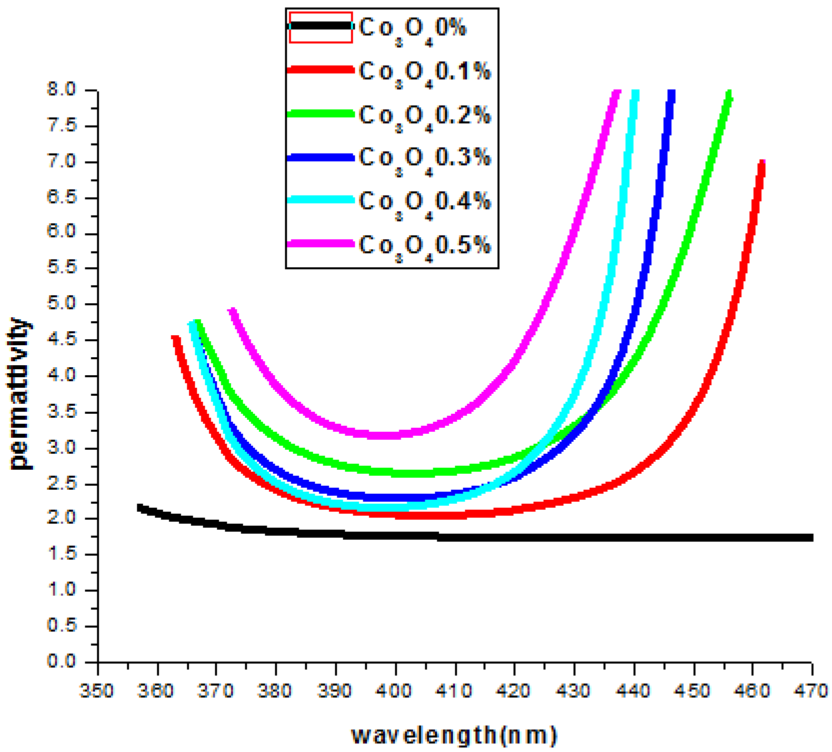

Fig.9.The permittivity versus wavelength for present samples.

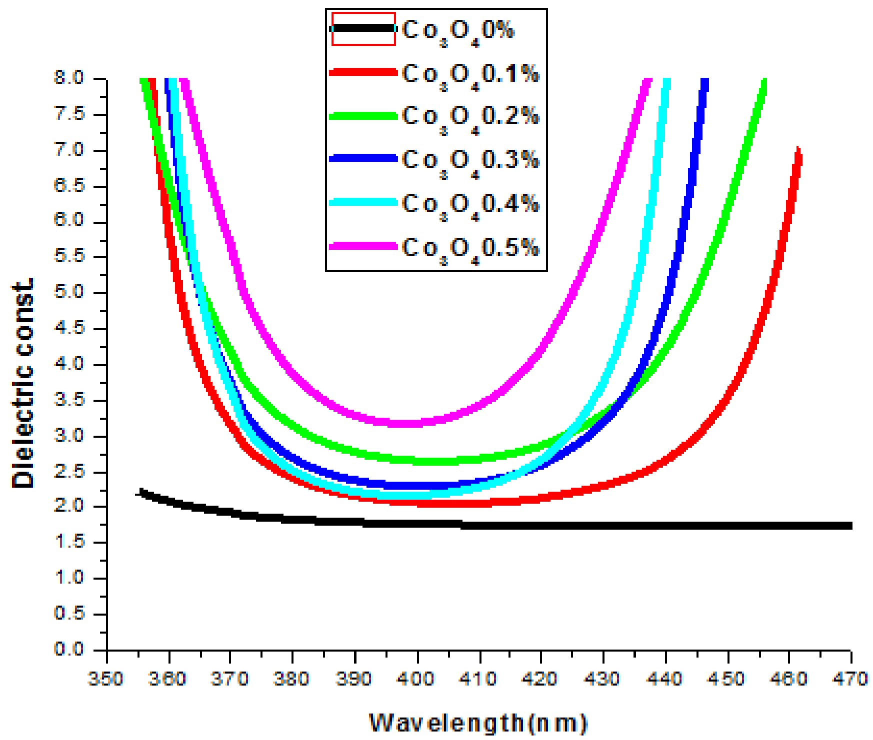

Fig.10.The dielectric constant, ὲ versus wavelength for present samples.

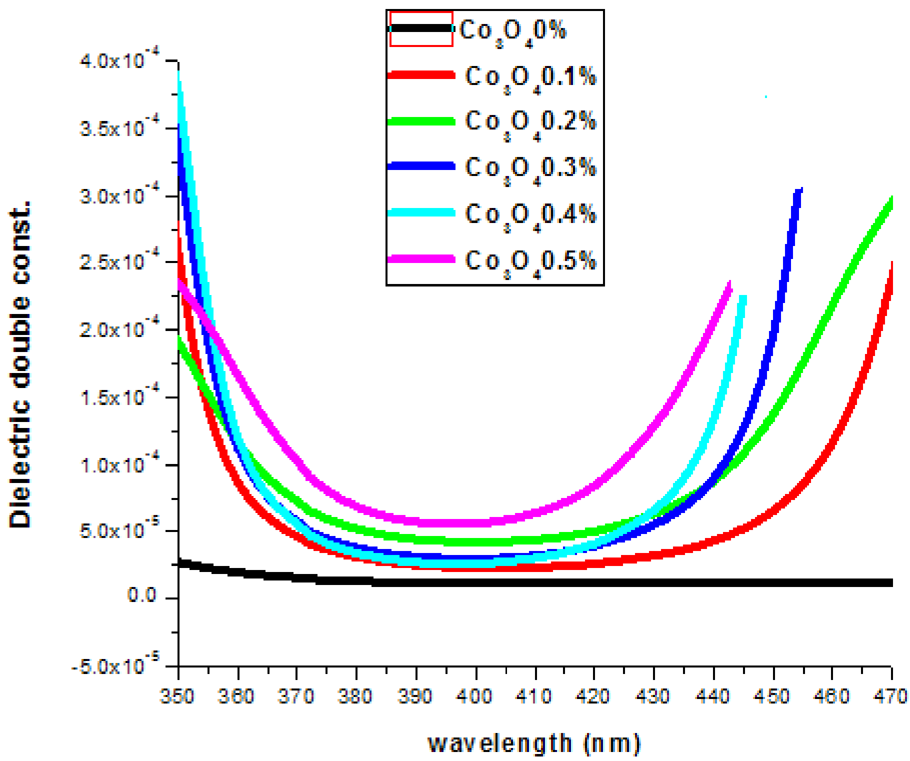

Fig.11.The dielectric double constant, ὲ̀ versus wavelength for present samples.

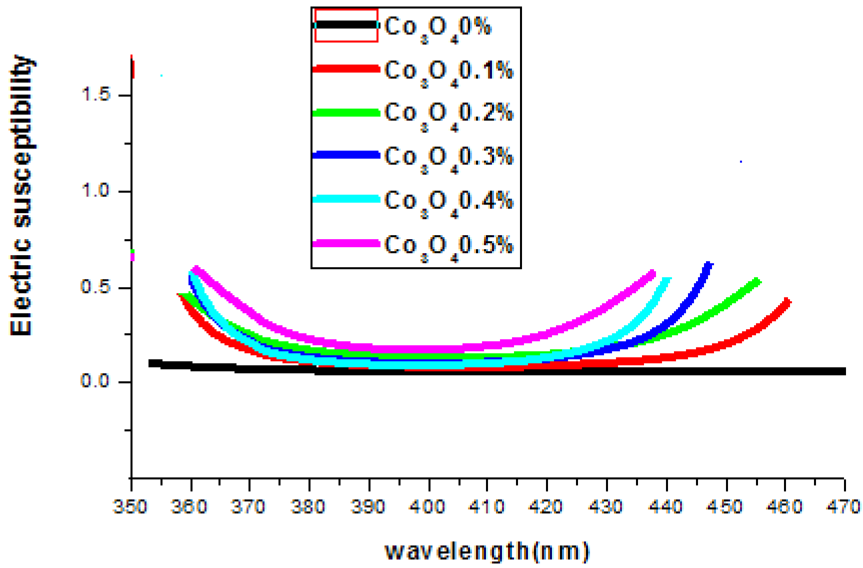

Fig.12.The electric susceptibility versus wavelength for present samples.

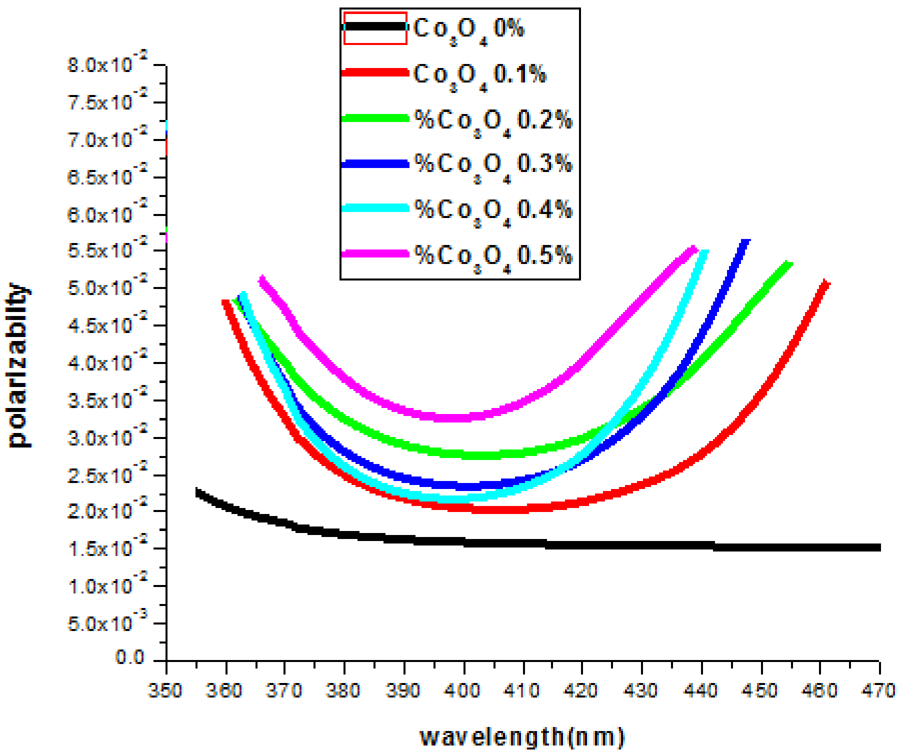

Fig.13.The polarizability versus wave length for present sample.

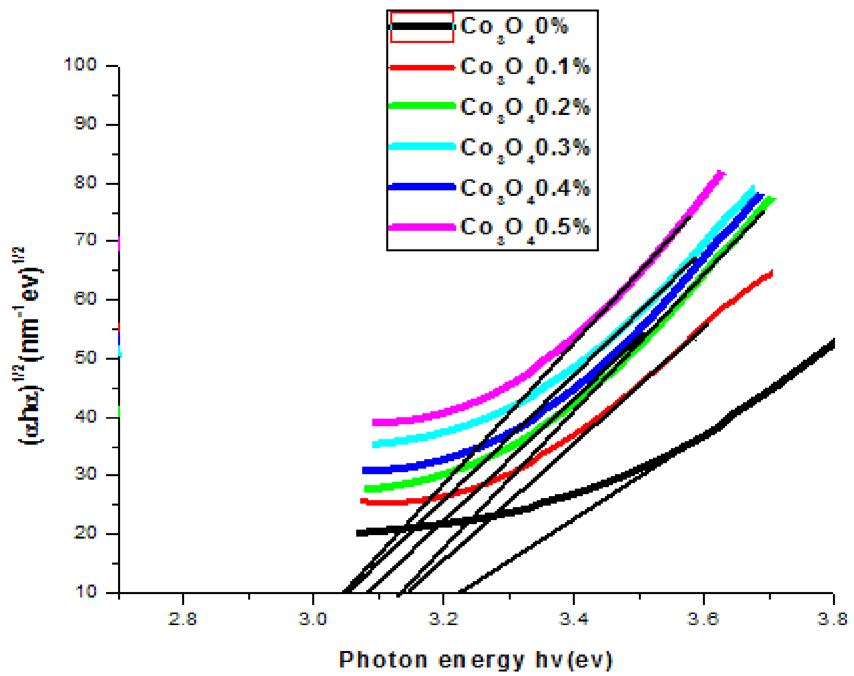

Fig.14.The optical energy gap for present glass samples.

Co_3O_4 content. Cobalt ions produced intense blue color in glass and used as optical filter.

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