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Optical spectroscopic analysis of sodium zinc phosphate glass doped cadmium oxide used for laser window protection

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Abstract

The phosphate glasses possess chemical composition of $40P_2O_5$-34ZnO-(21-x) Na_2O-5CuO- xCdO where (x = 1.2.3 to 6) have been prepared using the melt quenching technique. XRD analysis has been executed on the investigated samples in order for confirming the amorphous nature. The density was measured by the conventional Archimedes method and the molar volume was calculated. The density and the molar volume were found to be increased by increasing CdO content. The optical absorption has been measured from 200 to 2500 nm. While absorption is observed only in the UV range for the base glass, as well defined absorption band has been observed in the visible region, the width of which increases by increasing CdO content. No sharp edges were found in the optical spectra, which verify the amorphous nature of these glasses. The optical absorption and transmission spectra were recorded at room temperature using UV/vis absorption (JASCO V570) spectrophotometer in the wavelength range 2500-200 nm. The refractive index has been appreciated according to the Clausius–Mossotti method using the ionic refraction. The optical band gap energies for these glasses were found to be in the range 3.42–3.14eV. The absorption coefficient is observed to show an exponential dependence on the photon energy. The obtained values of refractive index have been used in estimating some characteristic features such as elasto-optic coefficient and susceptibility. The results reveal the importance of such glass composition in optical filter applications.

Keywords: Optical spectroscopy, Phosphate glass, Bandpass filter, Laser safety.

1. Introduction

Oxide glasses are classically qualified as a network collected by building entities such as SiO_2, B_2O_3, P_2O_5, TeO_2,CdO and modifiers such as alkaline oxides: Li_2O, Na_2O, K_2O, ZnO or alkaline earth oxides: CaO, MgO, SrO . In such glasses, the oxygen from the metal oxide becomes part of the covalent glass network, creating new structural units. The cations of the modifier oxide are generally present in the neighborhood of the non- bridging oxygen (NBO) in the glass structure. The extent of the network modification obviously depends on the concentration of the modifier oxide present in the glass. A glass network affects various physical properties such as density, molar volume, glass transition temperature, polarization, etc. Phosphate glasses, compared with silicate glasses, have lower thermal conductivity, mechanical strength, and higher thermal expansion coefficient, poor chemical durability limits their uses in many applications. The glass former most used nowadays is phosphate due to its excellent properties like low melting temperature, low dispersion, and high refractive index [1-2]. Chemical durability of the phosphate glass was greatly improved by the addition of ZnO because Zn ion acts as anionic cross linker between different phosphate anions, inhibiting the hydration reaction Incorporation of Cu is expected to improve the physico-chemical behavior of phosphate glasses. Therefore, it is interested to study the effect of simultaneous incorporation of Cu and Zn in phosphate glasses. Sodium phosphate glasses doped by transition metals are very important in photons and biological applications, Zinc oxide, ZnO which acts as a good modifier has been added to improve the chemical durability since Zn ion acts as a strong ionic cross linker between different phosphate anions, preventing hydration reaction, addition of transition metal ions like copper to phosphate glasses gives them interesting electrical, optical, and magnetic properties. Cadmium-phosphate-based glasses have been used as matrices for increase cadmium slender quantum dots, but little is known of the structures of binary cadmium phosphate. There are many types of laser eye-protection glasses in the market. Some of them can protect eyes against two or many wavelength laser beams and transmission of one kind of laser beam is 99.9%. The design of laser-protective eye-wear is to reduce any laser radiation reaching one’s eye to a level below the MPE (Maximum permissible exposure), meaning to an exposure level that will not cause injury. At least three output parameters of the laser must be known: maximum exposure duration, wavelength, and output power (or output irradiance, radiant exposure, or energy) as well as the applicable safe corneal radiant exposure. A YAG laser with a wavelength of 1064 nm is used in many applications like material processing, medicine, spectroscopy, and metrology, etc. The development of a window against this type of wavelength is very important, due to the speedy development of the technology of this type of laser system. Humans need two types of protection against the high power Nd: YAG lasers, the room window, and the eye goggles. The YAG window must have a good transmission wavelength in the visible spectrum, and an excellent protection against the specified wavelength. As well, we want to reduce the light transmission during the laser material processing in the laser room to protect the human eye, skin. The laser protection window must be very cheap, and have a long lifetime and depend on the absorption phenomenon, rather than reflection. The manufacturing of this glass will be applicable and producible by the optical glass industry, different other techniques used before in YAG laser protection. The copper doped phosphate glass is excellent for this type of application, the changing of doping can increase to the near infrared absorption band, which can cover the YAG laser band at 1064 nm and some other lasers wavelengths [3].Hence, glasses containing copper ions in different oxidation states are highly useful for consideration as band pass filters for different applications such as sun glass technology and laser protection eye-wear [4-7]. This paper developed the design and fabricate protection glass window against laser scattering in the field of laser working. This study is individual for this type of glass and its application in YAG laser window protection. Glass fabrication by the traditional method with an oxide glass former like silicate, borate, and phosphate, are used in many applications depending on the doping materials.

2. Experimental techniques

2.1. Preparation of Glasses

Glass systems of chemical composition $40P_2O_5$-34ZnO-(21-x)Na_2O-5CuO- x CdO where (x=1.2.3to6), was prepared using the melt quenching technique. The starting materials used are $(NH_4)_2HPO_4$, ZnO, Na_2CO_3, CuO and CdO .The chemical composition of each sample was mixed and grinded using mortar for 20 min. Hence, the samples in porcelain crucible were melted at 1000 °C in muffle furnace for one hour. then shaking in clockwise to ensure the material in high homogeneity, the molten materials were quenched at 300 °C in stainless steel mold and left to be annealed for one hour to relies the gases from the chemicals like CO_2 and NH_3 . The resulting samples take thin disk form using a pressing plate at the moment of quenching for studying the optical properties .The samples were annealed to avoid the internal stresses produced during quenching.

3 Results and Discussion

3.1. X-ray Diffraction

X-ray diffraction pattern of the material gives the identification of the constituent phases present in the material. It gives the information about the state of the combination of the chemical elements present in the mixture and also about the formation of compound if any, as a result of the individual constituents. From the X-ray diffraction patterns of all the samples under study were recorded as amorphous in nature. As shown in Figure 1 (XRD) technique on a Brucker axis D8 diffractometer with crystallographic data software Topas 2, using Cu-Ka (k = 1.5406 A°) 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.

3.2. Density

The density measurement has a vital role for the prediction of the structure change caused in the glass network by the replacement of oxides. The density of glass is greatly dependent on the structure compactness, coordination number, geometrical configuration change, dimension of interstitial spaces and cross-link density. The densities of the inspected samples were calculated according to Archimedes method using toluene as an immersion liquid (ρ_o = 0.8669 g/cm^3) by the following relation:

$$\rho = \frac{\mathrm{Wair}}{\mathrm{Wair} - \mathrm{Wl}}\ \rho_o$$

where W_{air} and W_l are the weight of sample in air and toluene respectively [8].

3.3. Molar weight calculation, molar volume

Step I– Calculation of wt/mole weight/mole = molar weight of the constituents * mol% / 100
Step II – Calculation of molecular weight of sample (M) The molecular weight of the sample (M) is nothing but the summations of wt/mole of its constituents.
Step III – Calculation of Molar volume (V_m) Using molecular weight and density calculated as from above, the molar volume of the glass samples can be calculated from following expression:

$$\mathrm{Vm} = \frac{M}{\rho}$$

Where, V_m is molar volume, ρ is the density of the sample and M is the molecular weight of the sample [9].

Average molecular weight, Density, Molar volume, Parameters values are presented in Table 1. From Table 1, and Figure 2 the density is found to be increased as CdO content is increased. This can be attributed to the replacement of Na_2O by CdO and the change in density is most likely related to the difference in atomic weights of Na and Cd. Moreover, the molar volume is found to be increased proportionally to CdO content. The molar mass of cadmium oxide (128.41 g/mol) is heavier than the molar mass of sodium oxide (61.978 g/mol). The glass matrix with higher contents with cadmium oxide is denser. In addition, the increase of molar volume is due to the atomic radius of cadmium (161 pm) less than sodium (190 pm).As usual, the molar volume and density was changed with inverse direction of each other direction, but these unusual results. The low density sodium metal oxide (Na_2O) is replaced by the high density glass modifier CdO , Which may also cause increase in the mass density.

3.4. Ion concentration, Inter ionic distance and Oxygen packing density

The oxygen packing density of the glass samples were calculated using the following relation

$$\mathrm{O} = \mathrm{n}\,\frac{\rho}{\mathrm{M}}$$

where ρ, the density of desired glass samples, M, molecular weight of the sample and n, the number of oxygen atoms in the composition [10].

The ionic concentrations of the glass samples are determined using the following relation [11],

$$\mathrm{N} = \frac{\mathrm{NA} * \rho * \mathrm{P}}{\mathrm{Mw}}$$

NA is the Avogadro’s number, P= nx , x is the mole fraction in glass composition 'n' is the number of atoms of element ions in given oxide , ie n=1 for oxide like CdO , ZnO etc and n=2 for oxides like Li_2O , Na_2O etc , Mw is average molecular weight .

Inter ionic distance (R) of the glass samples is given as [12]

$$\mathrm{R} = \left(\frac{1}{\mathrm{N}}\right)^{1/3}$$

Were N = ionic concentrations

Oxygen packing density, Ionic concentrations and Inter ionic distance, Parameters values are presented in table.1. From table .1. is oxygen packing density (OPD) which governs the tightness in packing of oxide network, the values of OPD are found to decrease with increase in the content of CdO which strongly related with increase in the values of density. In this respect, the structure of glass network became less tightly packed and the degree of disorder is increased by increasing CdO contents, i.e. formation of open structure, which explains the observed results of molar volume. The molar volume, inter ionic distance increasing; while oxygen packing density, ionic concentration decreases, which suggests the increased free space within the glass structure, [13-14], it means that the glass structure becomes loosely packed [15].

3.5. Optical properties

The absorption spectra is good important tool to find out various information like optical band gap, cut-off wavelength, refractive index and polarizibility ,etc .The optical absorption and transmission spectra of polished glasses at room temperature in the region 200-2500 nm is recorded. Figure 3 shows the optical absorption spectra of the glass samples over the wavelength range (200–2500 nm) for different ratios CdO. It can be noticed that the absorption increase with increasing the contents of CdO. 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.

Figure 4 shows the optical transmission spectra of the glass samples in the wavelength range 200–2500 nm for different ratios of CdO. The transmission spectra are consistent with the absorption data .The band pass in optics is a technique that allows us to pass a band of spectral lines through a filter. The obtained results reveal a band pass filter in the visible range with band stop in the ultra violet range. It is clearly observed that these glasses possess large optical transmission window and no sharp absorption edge is observed which confirms the glassy nature also confirmed by XRD. As shown in Figure 5 and listed in Table 4, the UV cut off is found to be increased from to nm by increasing CdO content. 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 [16]. The decrease in transmission height with increasing CdO content can be attributed to the replacement of Na_2O by CdO which results in attenuation of light in the transmission band .The area and the center of the band pass are calculated as shown in Table 4. This technique is very cheap for use as in laser protection window, and the final finishing needs no extra effort in the product.

The optical band gap, Eg of the glass samples was determined using the relation:

$$\alpha h\upsilon = \mathrm{B}\ (h\upsilon - Eg)^2$$

where, B is constant and α is the absorption coefficient that was calculated using the relation

$$\alpha(\omega) = 2.303\ (\frac{\mathrm{A}}{\mathrm{d}})$$

deduced from the Lambert–Beer law

$$\mathrm{I} = \mathrm{I}_{\circ}\mathrm{e}^{-\alpha(\omega)\mathrm{d}}$$

where d is the thickness of the sample and A is the absorption ,I_o and I are the intensities of the incident and transmitted beams respectively ,The thickness of the glass specimens was measured using a digital micro meter gauge [17] .The value of Eg has been determined from the linear region of curves by extrapolating them to meet the hν axis at $(\alpha h\upsilon)^{1/2} = 0$, As shown in Fig.6. As listed in Table.3.The optical energy gap is found to be decreased from 4.09 to 3.78 by increasing the CdO content .The variation as clearly observed from the table is nonlinear which shows that dual role of network former as well as network modifier is played by CdO in glass matrix, However there is overall decrease in values of Eg. This may be due to structural changes occurs in glass matrix on addition of CdO [18]. This decrease can be explained in terms of increase in concentration of non-bridging oxygen (NBOs) [19-21] .These non-bridging oxygen are very energetic and contribute more in the valence band and as a result of this valence band shifts a bit upward which in turn reduces the optical band gap.[22-24]

The absorption coefficient, α, is related to the extinction coefficient, k, by the following formula

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

where λ is the wavelength. If λ is in nm, multiply by 10^7 to get the absorption coefficient in the units of cm^{-1}.

the real and imaginary parts of dielectric constants ε' and ε" an also be calculated if the refractive index (n) is known using the following relation $\varepsilon' = (n^2 - k^2)$ and $\varepsilon'' = 2nk$ [25] .The optical conductivity was determined using the relation:

$$\sigma = \frac{\alpha nc}{4\pi}$$

where c is the velocity of light

The values of refractive index description of how to light slows down as it passes through an optical material, n is one of the fundamental tools describing the optical properties of the materials which are related to the electronic polarization of ions and the local field of the material [26],The refractive index is calculated using the following equation**:**

$$n = \frac{1+\sqrt{R}}{1-\sqrt{R}}$$

where R is the reflectance and can be estimated in term of absorbance A and transmittance T by

$$R = 1 - A - T$$

The calculated value of linear refractive index of all the glass samples is given in Table 2, the refractive index is a function of ion refraction as produced by Clausius–Mossotti method [27-29].

It is observed that 'n' decrease with increase in the CdO content in the present glass system and lies in the range from 1.355 - 1.318. This can be attributed to the replacement of Na_2O by CdO and the change in refractive index is the most likely related to the difference in ion refraction of Na and Cd. The refractive index of the studied glasses increases when the density increases with a variation in the composition of the glass system. The presented data on refractive indices with respect to the density have been compared with the already reported results for sodium containing glasses [30]. It is found that these results are also in good agreement with the already existing information about the refractive index and molar volume of glasses [31].

Molar refraction (R_m) a quantity calculated from a function of the refractive index of a substance multiplied by its molecular weight and divided by its density, which can be taken as a measure of the volume occupied by a molecule of the substance.

The molar refractivity R_m for each glass was evaluated using [32]

$$Rm = \frac{M}{\rho}\,\frac{n^2-1}{n^2-2}$$

where M is the average molecular weight and ρ is the density in g/cm^3.This equation gives the average molar refraction of isotropic substances, i.e., for liquids, glasses and cubic crystals.

The electronic polarizability , αe was calculated using the formula

$$\alpha e = \frac{3}{4\pi N}\,\frac{n^2-1}{n^2+2}$$

Where N the number of atomic concentration, $\varepsilon = n^2$ the dielectric constant [33]. The Permittivity "ε" and Elasto optic coefficient "p" can be calculated from the refractive index "n" that determined from Clausius–Mossotti equations as shown below:

$$\varepsilon = n^2$$

$$P \approx \frac{(1-B)(1+2B)}{3}$$

$$B = \frac{1}{\varepsilon}$$

Where N is the atomic concentration and B is an estimated value [34]. The atomic concentration was calculating using the following relation

$$N = \frac{\rho\ NR}{M}\ NA$$

where ρ is the density, M is the average molecular weight of prepared glass samples, N_R is the number of moles of rare earth ions and N_A is the Avogadro's number [35]. The electric susceptibility can be calculated knowing the estimated values of permittivity ε through the equation [34]

$$\chi = \frac{\varepsilon - 1}{4\pi}$$

The evaluated values are given in Table 2 and 3, show in Figures 7, 8, 9 to 17, it is observed that the average molecular weight and the theoretical density varies linearly where the other parameters varies non-linearly with CdO content. According to Clausius–Mossotti, this can be attributed to the increase ion refraction due to the change in chemical composition of the glass material [36]. In present work, these parameters have studied to explain structural features.

Conclusion

The existent work is true to study the influence of CdO in a glass system of chemical composition $40P_2O_5$-34ZnO-(21-x)Na_2O-5CuO- x CdO where (x=1.2.3 to 6). XRD pattern confirm the glass formability of the prepared samples. Both the density and molar volume of are found to be increased by increasing CdO content .The variations observed in various physical parameters like density , crystalline volume etc, shows that CdO has a strong influence on glass matrix. The refractive index has been estimated according to the Clausius–Mossotti method. The obtained values of refractive index have been used in the calculations of elasto-optic coefficient and susceptibility. By increasing CdO content, the UV cut off is increased from 315 to 327 nm. The optical energy gap is found to be decreased from 3.42 to 3.14 eV by increasing CdO content. The results assure advanced increase in number of non-bridging oxygen (NBO) atoms. The optical transmission is as well studied and presents the influence of Cd ions on the cut-off for the UV and infrared bands. The practicality of using this type of glass as band pass filters is specified by all glass samples showing a band stop in the UV band stop and infrared bands, which is an excellent protection window for laser safety against a YAG laser. Overall, this study opens promising avenue to improve an excellent protection eyewear for laser safety against YAG laser.

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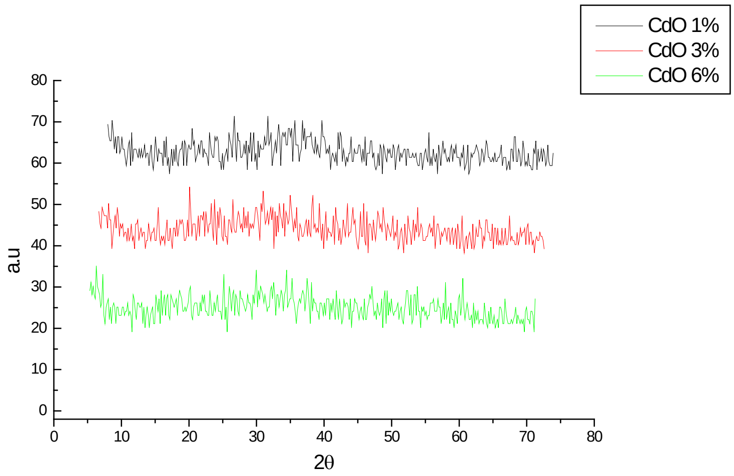

Figure 1: The XRD analysis for the glasses samples

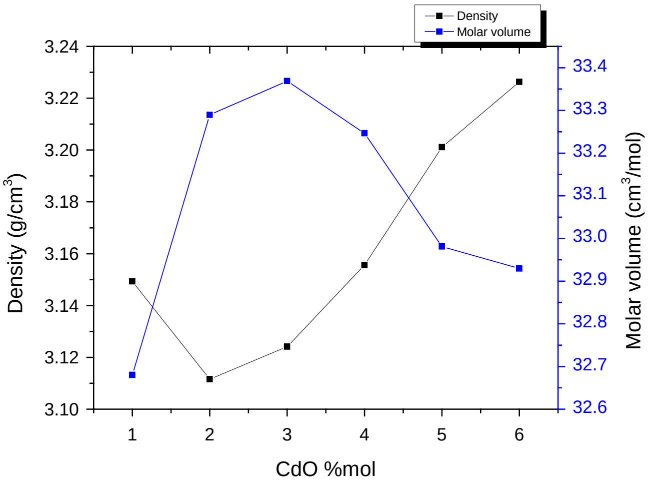

Figure 2: Effect of CdO content on the density and molar volume

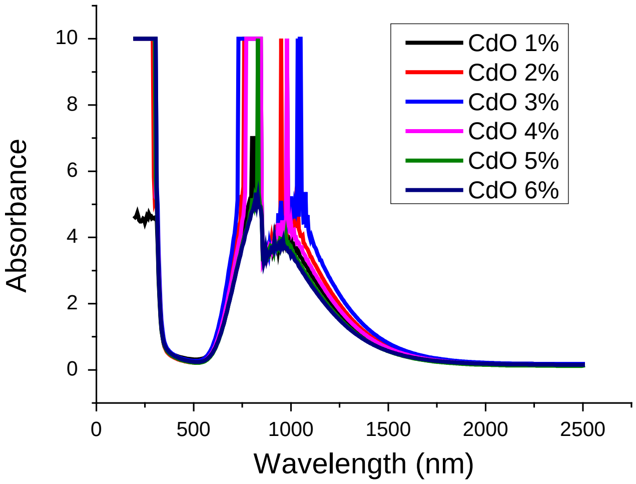

Figure 3: Absorption spectra for the glass samples with different CdO content

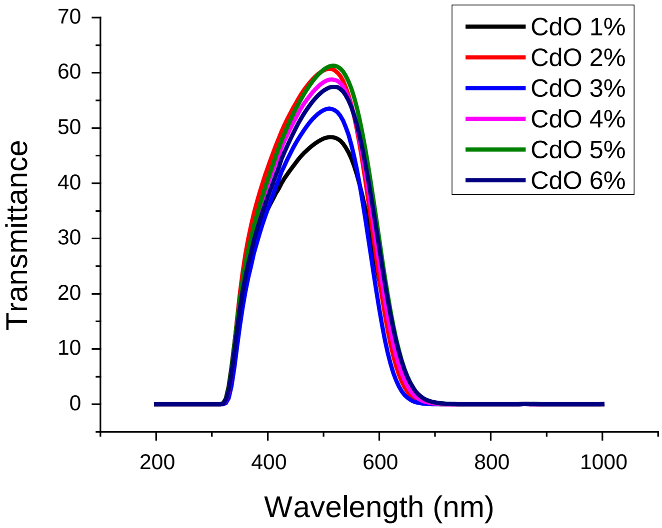

Figure 4: Transmission spectra for the glass samples with different CdO content

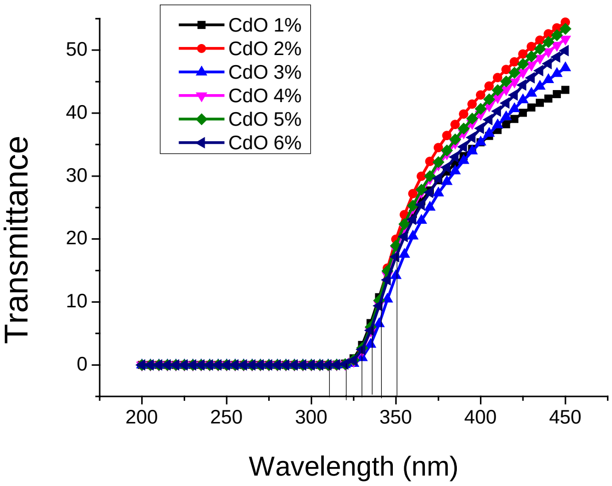

Figure 5: UV band stop for glass samples with different CdO ratios

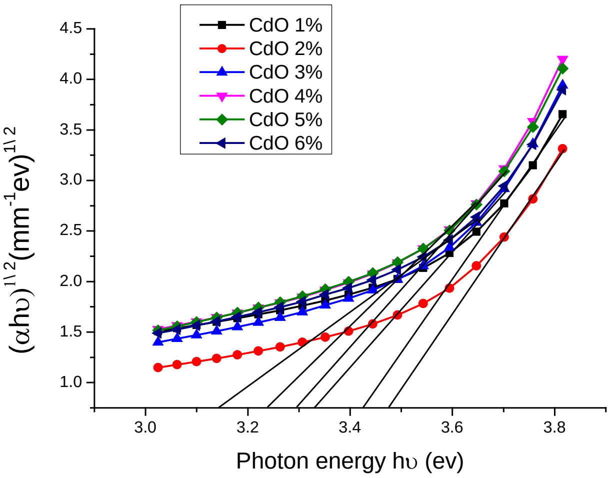

Figure 6: Determination of the optical band gap

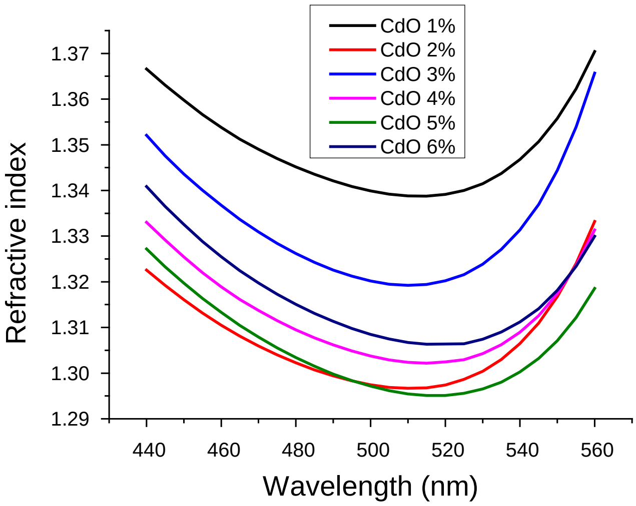

Figure 7: Wavelength dependence of the refractive index with different CdO content

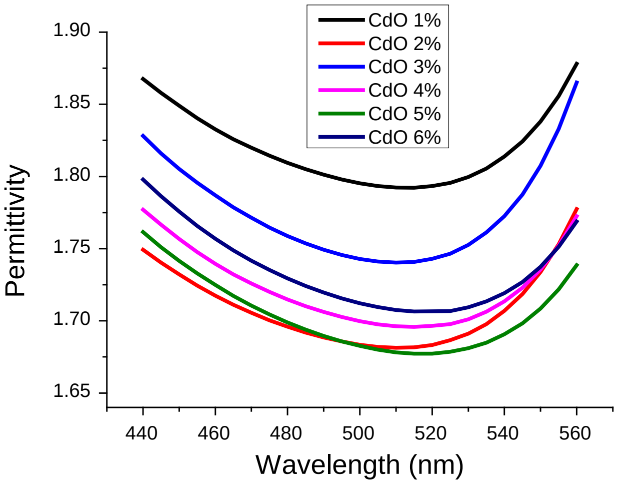

Figure 8: Wavelength dependence of the permittivity with different CdO content

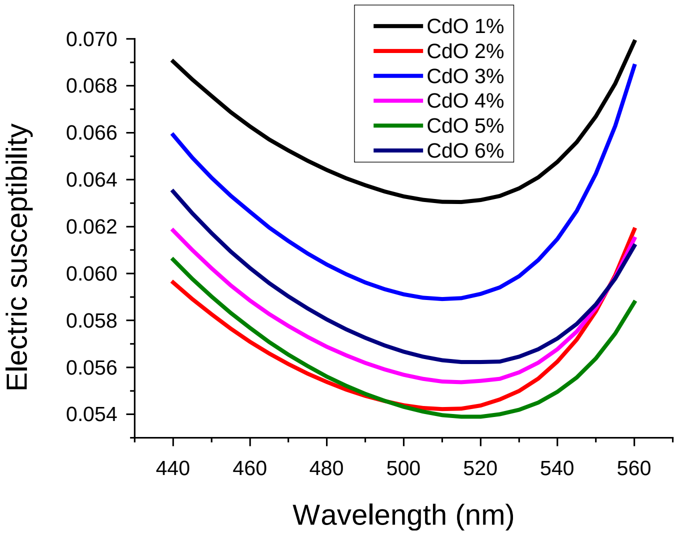

Figure 9: Wavelength dependence of the electric susceptibility with different CdO content

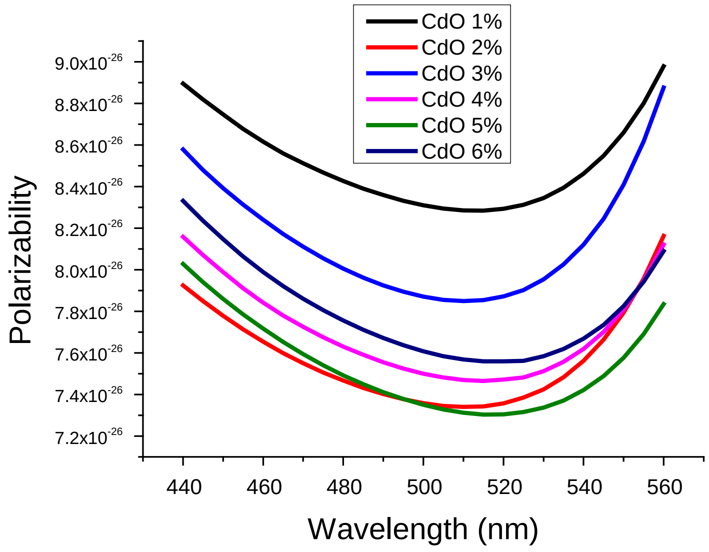

Figure 10: Wavelength dependence of the polarizability with different CdO content

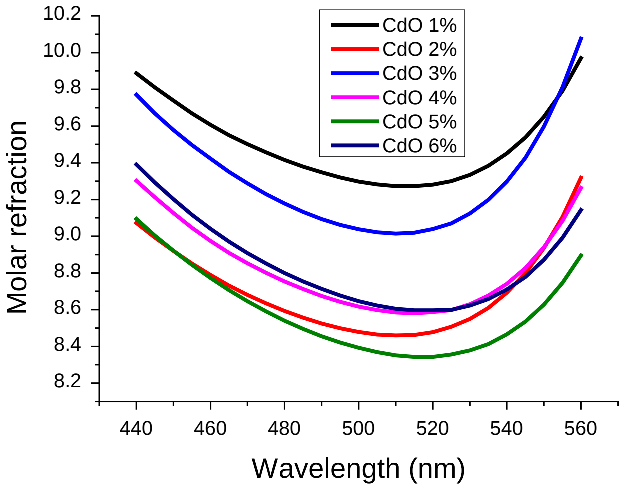

Figure 11: Wavelength dependence of the molar refraction with different CdO content

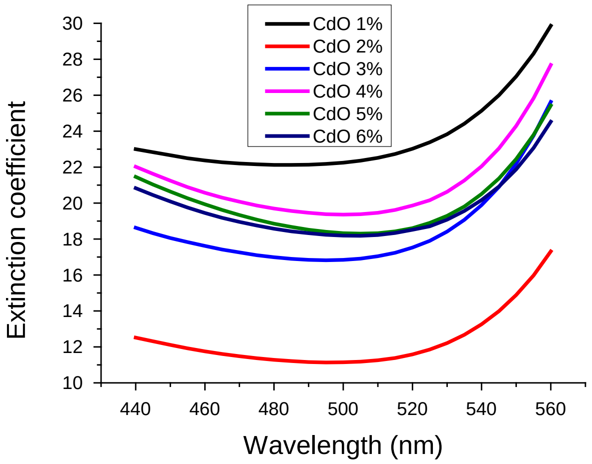

Figure 12: Wavelength dependence of the extinction coefficient with different CdO content

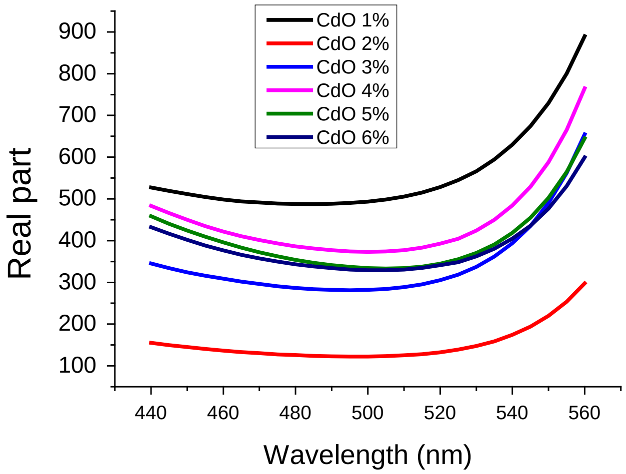

Figure 13: Wavelength dependence of real part with different CdO content

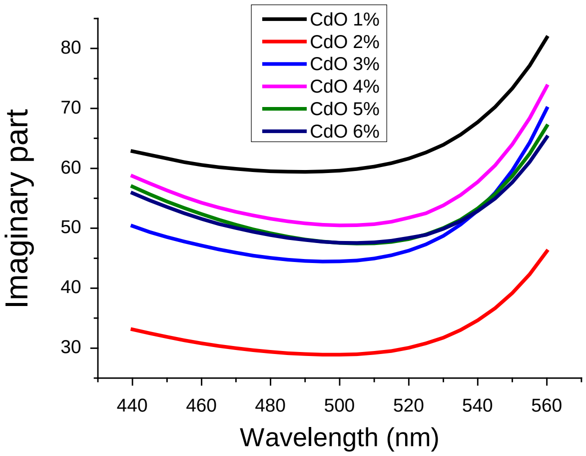

Figure 14: Wavelength dependence of imaginary part with different CdO content

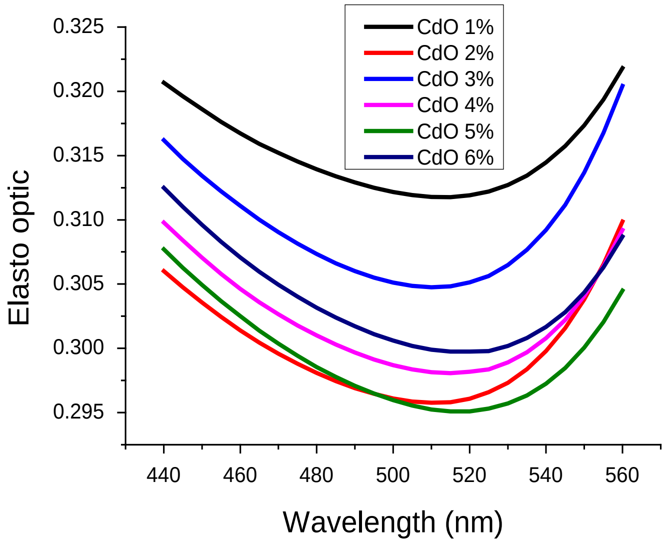

Figure 15: Wavelength dependence of elasto optic with different CdO content

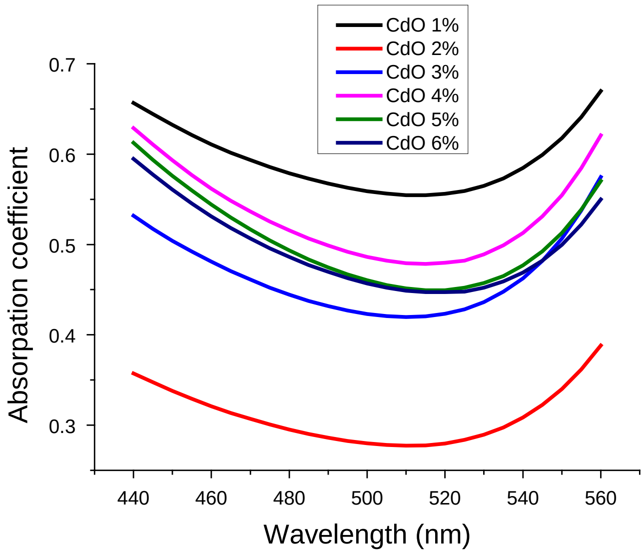

Figure 16: Wavelength dependence of absorption coefficient with different CdO content

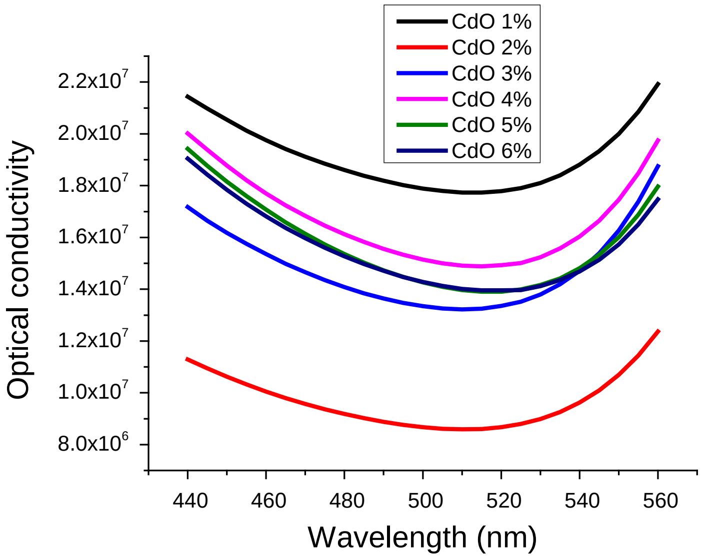

Figure 17: Wavelength dependence of optical conductivity with different CdO content

Table 1. Average molecular weight, Density, Molar volume, Oxygen packing density Ionic concentrations and Inter-ionic distance for $40P_2O_5$-34ZnO-(21-x)Na_2O-xCdO glass system

Name of Sample	Average molecular weight M (gm/mol)	Density (gm/cm^3)	Molar volume Vm (cm^3 /mol)	Oxygen packing density O (cm^3 /mol)	Ionic oncentrations N	Inter-ionic distance
S1	102.9193	3.1493	32.680	7.9253	1.843×10^{22}	1.597×10^{-67}
S2	103.5836	3.1116	33.289	7.7802	3.618×10^{22}	2.110×10^{-68}
S3	104.2479	3.1241	33.369	7.7616	5.414×10^{22}	6.298×10^{-69}
S4	104.9122	3.1556	33.246	7.7903	7.246×10^{22}	2.627×10^{-69}
S5	105.5765	3.1011	32.981	7.8529	9.130×10^{22}	1.313×10^{-69}
S6	106.2408	3.2263	32.929	7.8652	1.097×10^{23}	7.566×10^{-70}

Table 2. Refractive index, Dielectric constant, Molar refractivity, Electronic polarizability , Elasto optical, Electric susceptibility for $40P_2O_5$-34ZnO-(21-x)Na_2O-xCdO glass system

Refractive index (n)	Dielectric constant (ε)	Molar Refractivity Rm (cm³)	Electronic polarizability (αe)	Elasto optic	Electric susceptibility
1.35575	1.83806	9.65027	8.6593×10^{-26}	0.31735	0.06669
1.31675	1.73375	8.93508	7.7933×10^{-26}	0.30381	0.05839
1.34438	1.80738	9.59671	8.4095×10^{-26}	0.31367	0.06425
1.31741	1.73558	8.93981	7.8089×10^{-26}	0.30407	0.05853
1.30711	1.70855	8.62785	7.5768×10^{-26}	0.30005	0.05638
1.31813	1.73747	8.87119	7.8250×10^{-26}	0.30434	0.05868

Table 3. Absorpation coefficient, Optical conductivity, Extinction coefficient, Energy gap for $40P_2O_5$-34ZnO-(21-x)Na_2O-xCdO glass system

absorption coefficient	optical conductivity	Extinction coefficient	Energy gap
0.6176	2.0001×10^{8}	27.04702	3.42
0.3399	1.0691×10^{8}	14.88554	3.47
0.5066	1.6268×10^{8}	22.18566	3.33
0.5546	1.7452×10^{8}	24.28745	3.29
0.5127	1.6008×10^{8}	22.45264	3.23
0.4996	1.5729×10^{8}	21.87741	3.14

Table 4. The peak analysis of the band pass filter

CdO (mol%)	Area	Center (nm)	Width (nm)	Height	UV cut off (nm)
CdO 1%	11627	486.34	176.4	52.591	190-310
CdO 2%	13779	484.74	167.45	65.622	190-320
CdO 3%	11505	485.74	160.09	57.340	190-329
CdO 4%	13451	488.95	169.71	63.238	190-335
CdO 5%	14228	492.01	172.80	65.698	190-341
CdO 6%	13296	492.92	172.36	61.549	190-350