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Effect of doping some lanthanide oxides on optical and radiation shielding properties of cadmium borate glasses

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Abstract
Ce_2O_3, La_2O_3 and Nd_2O_3 and Sm_2O_3 doped in the cadmium borate host glass were prepared. Some optical and physical properties such as density, molar volume, optical band gap energy, the width of the band tails (Urbach energy) and radiation shielding properties were measured. In addition, the attenuation parameters, are calculated at different photon energies using the XCOM program and practical before and after irradiation. A correlation between the results from density, UV–Vis and FTIR measurements indicates that CdO plays a role as a network modifier and alter within the atomic structure is attributable to the formation of BO_4 units. The attenuation parameters are calculated at different photon energies using the XCOM program and measured in experiments. These values show that there is a correlation between theoretical and experimental values. Hence, these results indicate that the samples have gamma-ray shielding in this energy range.

Keywords Cadmium borate glass · Lanthanide oxides · Optical spectra · Shielding properties

1 Introduction

Glass containing lanthanide oxides have grabbed attention and interest because of numerous applications such as fiber amplifier, up-converters, laser, electro-optics devices and optical fiber lasers (Mierzejewski et al. 1988; Sharma et al. 2015)

Radiation protection has been the focus of numerous research works. Since glass could be a strong and simple material, researchers keep on creating distinctive sorts of glass to be used as shielding materials that have optical and basic properties not extremely influenced by radiation (Sharma et al. 2015).

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Including one of the lanthanide oxides (LO) to the glass makes it a decent contender for gamma radiation protecting because of their high density and high atomic number. As known, adding a few lanthanide oxides to lead glass beats the poisonous quality and keeps up the straightforwardness after exposure to radiation where the color remains unchanged (Sharma et al. 2012). High energy ionizing radiations as gamma-rays can actuate electronic and modification harms in solids and especially can cause various imperfections in glasses (Fayad et al. 2017). This conduct is thought to be connected with the catching of freed electrons and positive gaps including some photochemical responses or it could be because of the impediment of the entry of electrons or positive inside the glass organize amid the illumination procedure. Therefore, the investigation of deformity focuses in glasses to investigate their suitability for atomic protecting purposes or radiation dosimetry applications (Saudi 2013).

The behavior of lanthanide ions is similar to its behavior in inorganic precious stones in law symmetry. Their 4f orbital is much protected from cooperation with outside powers by overlaying 5S2 5P6 shells. Accordingly, the state coming about because of the changed 4f design is just marginally influenced by encompassing particles and proceeds for all intents and purposes invariant for a given particle in various mixes. Along these lines, for the most part, the uncommon earth particles do not influence the bright retention edge of the base glass (Sharma et al. 1996).

The main objective of the present work is the preparation and optical characterization of cadmium borate glass doped with uncommon earth (RE) oxides, (LO = La, Ce, Nd, Sm) and concentrating the impact of doped oxides on the optical properties, microhardness and light protecting properties of arranging glasses at low doses.

This work focuses on studying the attenuation parameter of low sources of gamma beside the effect high doses of gamma radiation up to 80 kGy on optical properties of the prepared glass system.

This is in addition to evaluating the ability of these systems in immobilizing the high level nuclear wastes.

2 Experimental details

The glasses of the composition $55B_2O_3$–45CdO–0.5LO were prepared from chemically pure and fine-grained grade materials, utilizing $H_3BO_3$from B_2O_3, $CdCO_3$ from CdO and (LO = La_2O_3, Ce_2O_5, Nd_2O_3, Sm_2O_3) for each uncommon earth oxide (99.99% purity, Alfa). The weighed batches were melted in platinum pots at 1100 °C for 2 h. The liquefying was pivoted a few times to accomplish a worthy homogeneity. The homogenous melts were thrown into preheated hardened steel molds of the required measurements. The readied tests were instantly moved into a stifle heater managed at 420 °C for strengthening The mute was turned off following 1 h and left to cool to room temperature at a rate of 30 °C/h.

The samples have been obtained in rectangle shape of 2 cm*1 cm. Glass density was measured at room temperature utilizing the standard Archimedes technique, with toluene as the inundation liquid of stable density (0.866 g/cm^3).

Attenuation coefficients of the proposed glass system were measured in tight pillar transmission geometry by utilizing a 2*2 NaI (TI) crystal detector of an energy resolution of 12.5% at the 662 keV related to a multi-channel analyzer (MCA). Radioactive sources ^{60}Co and ^{137}Cs of various photon energies were utilized. Occurrence and transmitted forces

of photons were estimated on MCA for settling preset time for each sample by choosing a thin district symmetrical as for the centroid of the top photograph

The infrared absorption spectra were estimated at room temperature in the range 4000–400 cm^{-1} by a Fourier Transform infrared spectrometer (type VERTEX 70, FT/IR-430, Japan was used in measuring. Glass samples were measured before and after being subjected to 30, 50 and 80 kGy of gamma radiation.

The samples were pounded into fine powder. The IR absorption spectra were measured immediately after preparing the discs. Additionally, the IR spectra were measured after subjecting the prepared glasses to predetermined gamma-ray.

The optical estimations were considered by utilizing an UV–VIS spectrometer (Perkin–Elmer, Lambda 950), together with a double light source fit for yielding bright in addition noticeable light. The rate ingestion spectra were taken in the wavelength run 200–900 nm which utilizes air as a kind of perspective. Optical estimations were taken previously and quickly after light measurements.

The hardness is characterized as the proportion of the connected test load to the anticipated zone of the resultant deliberate impression (Gong et al. 2001). Vickers hardness, H_v, was estimated by utilizing a microhardness analyzer (Leco AMH 100, USA) for sample indentation. Microhardness can be calculated from the following relation: $H_v = 1.8p/d^2$, where p is the indentation load and d is the diagonal length impression.

A Co^{60} gamma cell (2000 Ci) was used as a gamma ray source with a dose rate of 1.5 Gy/s at 30 °C. The glass samples were located into gamma cell in means that each sample was exposed to the required dose.

3 Results and discussion

3.1 Density (ρ) and molar volume

The results show that the density increases with doping lanthanide oxides content from CeO_2 to Sm_2O_3 (Fig. 1). The molar mass of $Sm_2O_3 = 348.7182$ g/mol is heavier than the molar mass of $CeO_2 = 172.1148$ g/mol and so, the glass matrix becomes denser. This behavior may be explained by the addition of each one element of lanthanide oxides to the glass network which may cause some changes in the structure such as, rearrangement of

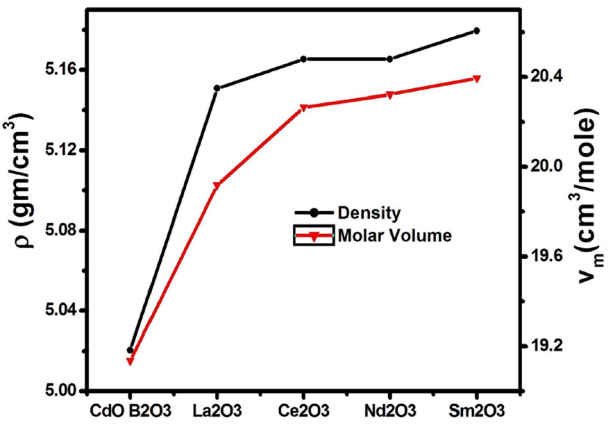

Fig. 1 Dependence of the density and the molar volume for different lanthanide oxide contents

the atoms, changes in geometrical configuration, coordination numbers of the constituents, cross-link density and dimension of interstitial spaces of the glass. Hence, it affected the density in a direct manner (Marzouk et al. 2014).

Several authors explained that the density of the glass changes within the inverse direction of the molar volume. However, the opposite trend is observed in the glasses prepared in this study. In agreement with previous studies, the molar volume of the prepared glass increases with increasing the lanthanide oxide concentrations this is due to the larger atomic radii of Ce_2O_3 compared to those of B_2O_3, and thus results in the extension of free volume (Saddeek et al. 2008).

3.2 Attenuation parameters

The attenuation method is incredibly necessary. Radiation is reduced in intensity once passing through some material (Holloway 1973; Pfaender 2012; Singh et al. 2002, 2003). The absorbent graphing curve gives the experimental γ ray a linear attenuation coefficient of the different lanthanide oxides with different thicknesses and different energies in terms of cm^{-1}. The linear attenuation is better in lower energy than above, as shown in the (Fig. 2).

The mass attenuation coefficients were calculated from the intensity of the measured incident gamma-rays and the intensity of transmittal gamma-rays, in addition, from WinXCom program can be calculated in theoretical curves.

Figure 3 shows the experimental and theoretical results of the mass attenuation coefficients of the glass samples of various energies as a function of the various lanthanide oxides. There was a convenient correlation between experimental and theoretical values.

The mass attenuation coefficients of the different doping lanthanide oxides are almost constant with slight changes, this may be due to the convergence of their density values and the atomic number. Also, a high percentage of CdO in this glass system that participates in the structural chains of the glass network has helped absorbing the gamma-rays compared to other systems (Saudi et al. 2014).

Half value layer (HVL) is considered as the thickness of the shield matter at that the intensity of the incident radiation is reduced to 1/2 of its initial value.

Figure 4 revealed that the half value layers of the studied glass systems at various energies decrease with increasing the atomic number of the doped lanthanide oxides and increase with increasing the energy. That could be attributed to the increase in the mass attenuation coefficient and relates to the higher density of the glass samples. Therefore, it could be concluded that the prepared cadmium borate host glass, doped with the appropriate content of the mentioned lanthanide oxides, has better shielding properties than some standard known glass systems.

3.3 Micro-indentation before and after irradiation

The Vickers hardness was determined from the mean value of 10 indents, so the errors in the measured values correspond to the standard deviation (2%). Figure 5 shows that the highest hardness values are for the unirradiated glasses, while the Hv decreases with radiation at 50 and 80 kGy up to a fixed value, i.e. there is no significant difference in their values (approximately the same values) for these doses corresponding to the response of the irradiated region. The increase of microhardness of different lanthanide oxides is due to the low flow mechanism in the glass containing oxides. The decrease in flow movement is expected by adding the lanthanide oxides from La_2O_3 to Sm_2O_3 respectively, as the atomic

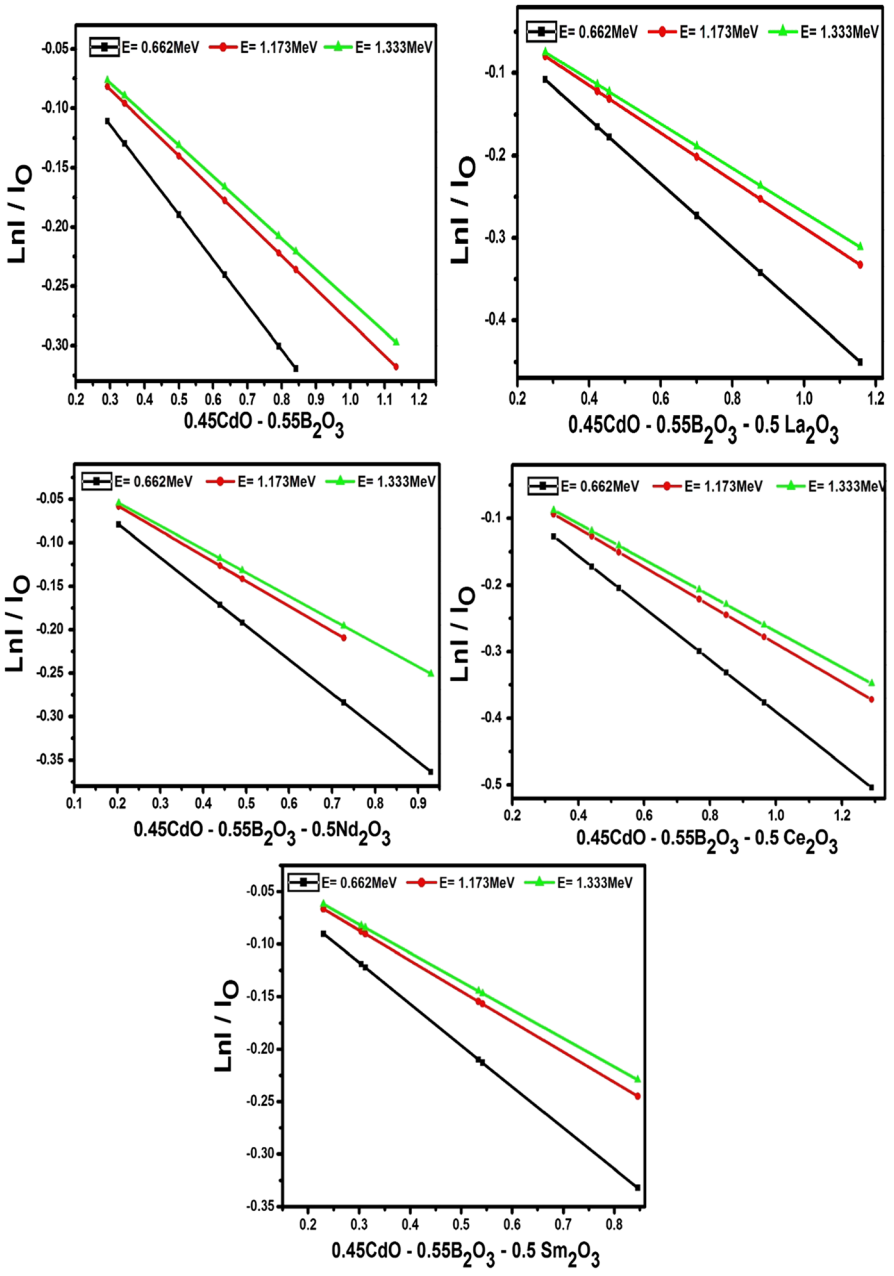

Fig. 2 Gamma-ray attenuation graph for different thicknesses of the different lanthanide oxides concentration

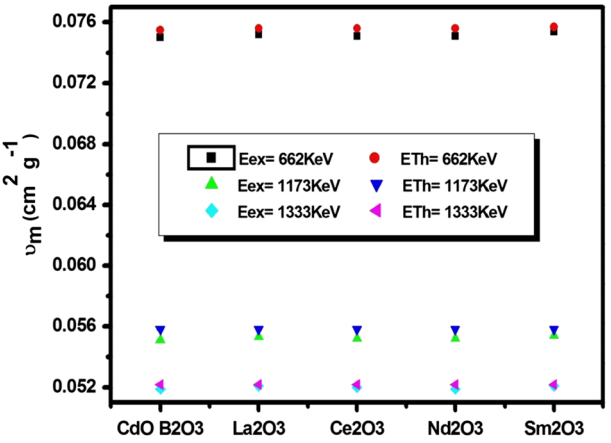

Fig. 3 Experimental and theoretical values of mass attenuation coefficients (μ_m) as function of the different lanthanide oxides concentration

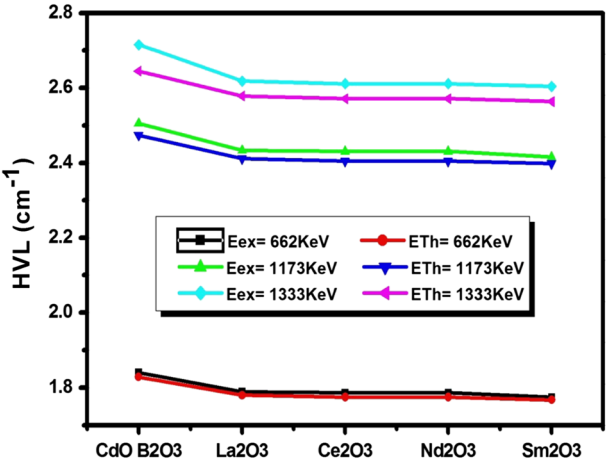

Fig. 4 Experimental and theoretical values of half value layer (HVL) as function of the different lanthanide oxides concentration

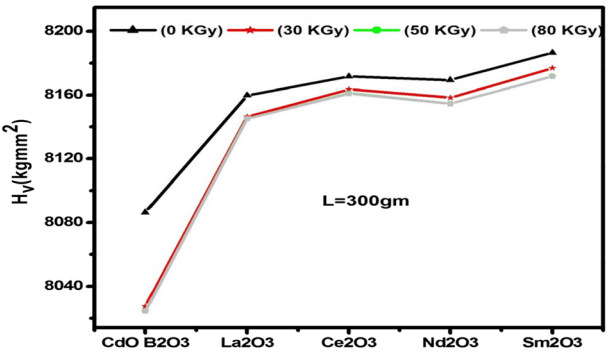

Fig. 5 The Vickers hardness for glass samples at 300 gm

mass increases, the density increases and consequently increasing the hardness (Sharma et al. 2012).

Furthermore, adding a content 0.5% of lanthanide ions to the present glass network interstitially causes some types of modification of B–O–B linkages. Besides, the conversion of BO_3 units into BO_4 ones results in an increase in the density of the glass network and the rigidity increases which in turn contributes to increasing the microhardness. This behavior indicates that adding lanthanide oxides to the studied cadmium borate glass improves the mechanical properties and the strength of the cross-links between chains of the cadmium borate glasses.

3.4 Infrared absorption spectra of the studied glasses before irradiation

Infrared spectroscopy is a potent technique for the structural studies of glasses that provides different vibrational modes correlate with the structural chains, which constitute the glass network (Pavani et al. 2011). The IR analysis (Fig. 6) shows strong and broad absorption bands which indicate not only the amorphous nature of glass samples, but also the ability of the studied high percent of CdO to share in the structural chains of the glass network besides the forming configuration units, together with the trigonal BO_3 and tetrahedral BO_4 groups. The low intensity little broadness curve centered at 565 cm^{-1} is assigned to the borate deformation modes, such as the in-plane bending in B-O triangles overlapped with CdO (Kamitsos et al. 1987), or from the BO_4 units in anionic rings as confirmed by Malhapatra et al. who studied the network structure of thermal seal glass (Kaur et al. 2013). A distinguished band positioned at 669 cm^{-1} is attributed to the meta borate units (Kaur et al. 2013). The strong high intensity wide broad band covering the range 780–1150 cm^{-1} representing the BO_4 units with center at about 916 cm^{-1} that can be assigned to stretching vibration mode of the B–O chains (Marzouk and Ezz-Eldin 2008). In addition, a small curvature in the region 1000–1150 cm^{-1} is associated with the vibration of BO_4 units with non-bridging oxygens (Dousti et al. 2013). The last broad band extending in the range 1200–1500 cm^{-1} represents the

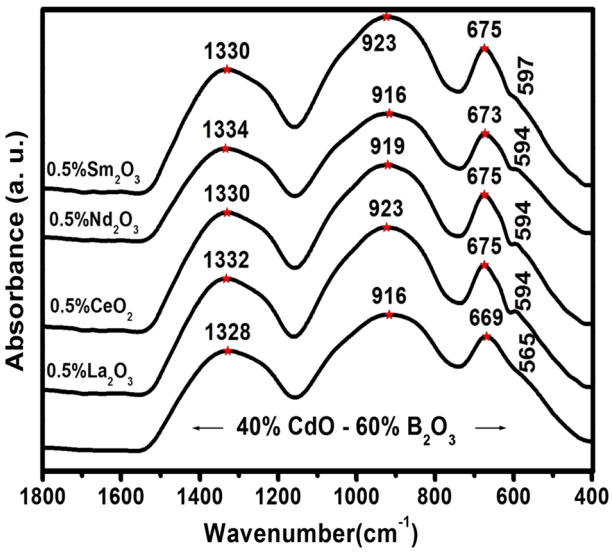

Fig. 6 FTIR spectra of undoped and doped glass systems before gamma irradiation

stretching vibrations of B-O chains in the polymerized $(BO_3)^{3-}$ units in meta-, pyro, and ortho-borates (Mierzejewski et al. 1988). It contains a small curvature centered at 1240, which is correlated to the asymmetric stretching vibration of B-O bond in isolated groups of BO_3 (Van Uitert et al. 1987).

The addition of the different rare earth ions causes slight changes in the intensity of the main absorption bands as shown in Fig. 6. The curvature at 565 cm^{-1} is shifted to narrower kink at around 594 cm^{-1}. This can be attributed to the rare earth oxides that tend to occupy the modifier network positions and in the presence of the dual function heavy metal CdO which tend to be a modifier and network former that affect the percentages of borate groups into glass network (Kaur et al. 2016).

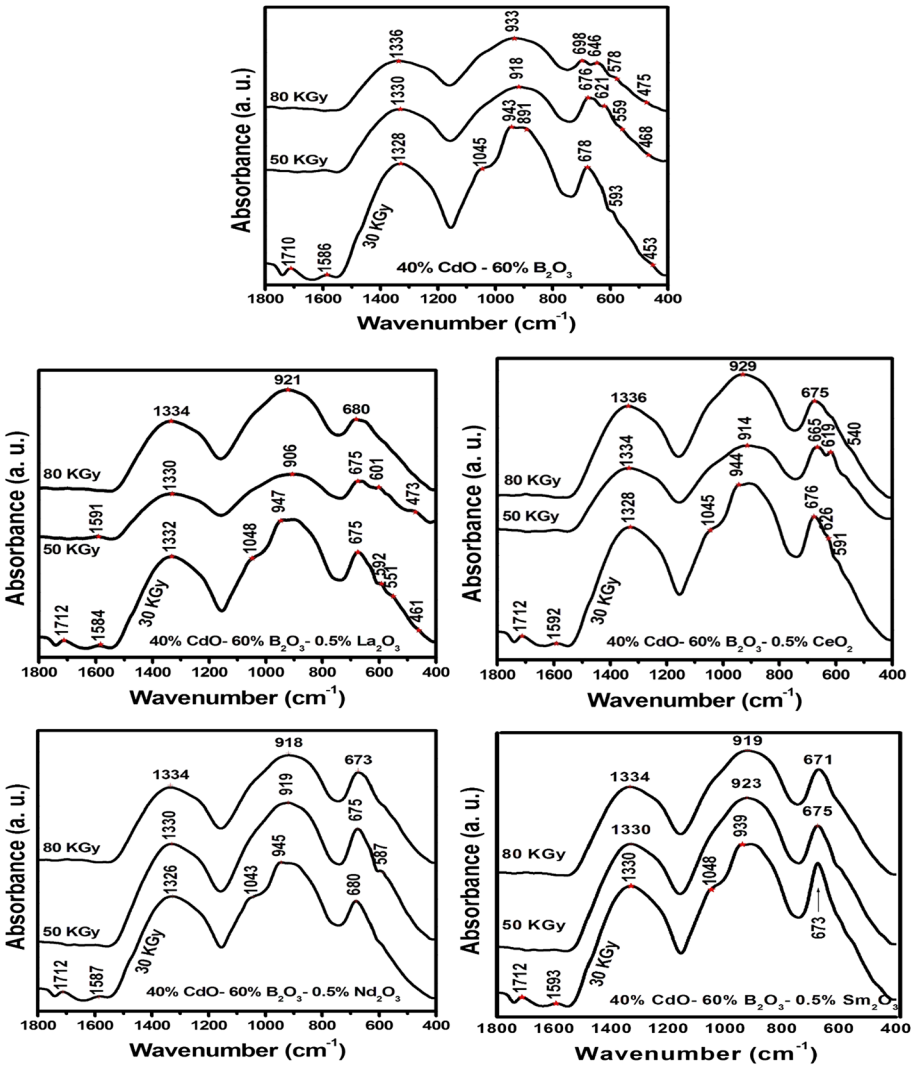

Fig. 7 FTIR spectra of undoped and doped glass systems after gamma irradiation

3.5 Infrared absorption spectra of the studied glasses after irradiation

It is obvious from the IR spectral analysis (Fig. 7) that the base cadmium borate glass and the glasses with lanthanide oxide contents show some changes in IR spectra with the first gamma dose of 30 KGy represented in an increase in the intensity of the bands and shifting in position peaks to higher or lower wavenumber. The IR peak at 453 cm^{-1} is assumed to indicate the vibration of the modifier Cd cations (Pavani et al. 2011). The BO_4 broad IR band becomes well defined by showing three distinct peaks positioned at 891, 943 and 1045 cm^{-1} in which the two former peaks are indicated to stretching vibrations of B–O bond in BO_4 units (Kamitsos et al. 1987), while the last one is assigned to stretching vibration of B–O–Cd linkages. The appearance of the two peaks located at 1586 and 1710 cm^{-1} is assumed to be due to the generation of super structure units with non-bridging oxygen after irradiation (Kaur et al. 2013). There is an observed decrease in the intensity of the IR bands with the higher doses of gamma 50, 80 KGy. These IR features are attributed to the assumption that the glass network chains were affected by the high energy of gamma rays which cause interruption of vacant randomness in structural units and unsymmetrical arrangement of the groups leading to weakening of the network grouping vibrations (Kaur et al. 2013).

The IR spectra of the irradiated 0.5% (La_2O_3, CeO_2) glass systems show almost the same spectral curves as the irradiated undoped glass system in each gamma ray dose. A marked decrease in the intensity at 50 KGy gamma dose was observed, then the intensity increases again at 80 KGy. This could be attributed to the high energy of the gamma radiation which causes the radiation annealing effect, an actual annealing and removal of defect flaws (Marzouk and Ezz-Eldin 2008). On the other hands, the IR spectral curves of the irradiated 0.5% (Nd_2O_3, Sm_2O_3) glass systems show a similarity with the IR spectral curves of the unirradiated glass systems especially at higher doses of 50, 80 KGy. Thus, these studied glass systems can be considered radiation hard at 50 KGy and 80 KGy.

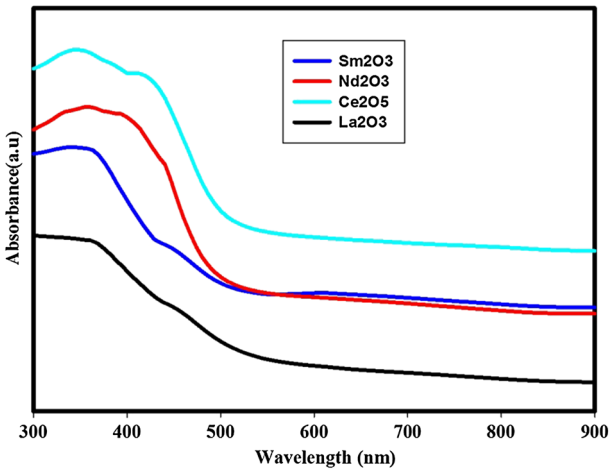

Fig. 8 Absorption spectra for all compositions of glass samples

3.6 Optical properties before and after gamma irradiation

The UV–Vis absorption spectra of the prepared glasses recorded in the range 300–900 nm is shown in Fig. 8. All the glass samples have high absorption intensity in the ultraviolet region. An absorption band peaked at 400 nm is observed. This absorption band is assigned to the electric dipole transition from the $6H^{5/2}$ ground state to the $6P^{3/2}$ excited state of LO^{3+} ions on the basis of its energy level diagram (Dousti et al. 2013; Mierzejewski et al. 1988)

Interaction of gamma-ray with lanthanide oxides doped in host cadmium borate glasses produces no induced bands, that may explain the stability of the glass containing a high percentage (45 wt%)of the significant (CdO) causing an obvious shielding behavior, However, a minor increase of the UV intensity with gamma irradiation is explained by the chemical reaction of few ferrous ions from iron impurities react with positive holes generated during the irradiation process. The net result is The ($\lambda_{cut\text{-}off}$), optical band gap values (E_g) and Urbach energy (ΔE) values of the current samples are given in Table 1. The conversion of some ferrous ions to additional ferric ions and hence this explains the slight increase of the intensity of the UV absorption. The stability of the spectral curve in the visible region after gamma irradiation that followed by the slightest decrease of the intensity in the UV region can be explained as follows:

(a) The presence of high content 45 mol% cadmium oxide is assumed to resist the penetration of the liberating electrons inside the network structure throughout the irradiation process.
(b) The minor decrease of UV intensity within the range of ferric iron absorption can be implicit to the photoreduction of few ferric ions to ferrous ions which have its main absorption at about 1100–1200 nm and hence the practical minor decrease of the UV absorption (Kaur et al. 2016). The optical energy gap can be calculated according to Davis and Mott (Davis and Mott 1970).

When lanthanide metal oxide is doping with the cadmium borate glasses, it may lead to some changes such as forming bridging oxygen or non- bridging oxygen. The last changes replicate directly on the absorption characters that consequently decrease or increase in the optical band gap (Fig. 9).

The obtained results as shown in Fig. (9) reveal that the optical band gap decreases when doped with Ce_2O_3, La_2O_3, and Nd_2O_3. However, the optical band gap slightly increases when doped with Sm_2O_3. According to previous studies (Sharma et al. 2015; Kaur et al. 2016), the optical band gap is influenced, not only by a chemical composition, but also by a structural arrangement of the sample matrix.

The partially occupied 4*f* levels close to the valence band edge decrease the band gap of cadmium borate glasses doping with Ce_2O_3, La_2O_3 and Nd_2O_3 leading to the increase of bonding defect and non-bridging oxygen and enhances the converting sp^3 tetrahedral BO_4 units into sp^2 planar trigonal BO_3 units with non-bridging oxygen as shown in Figs. 6 and 7.

The 4*f* energy level gradually becomes lower with the increase of their molar mass, finally lying in the valence band, which results in a monotonic increase of band gap energy in Sm_2O_3. Therefore, there is a reduction in the number of NBO

Table 1 Calculations of energy gap (E_g), refractive index (n), molar refraction (R_m),Urbach energy (E_{urbach}), cut-off wavelengths ($\lambda_{cut\text{-}off}$) before and after gamma irradiation

Parameters	Base				La_2O_3				CeO_2				Nd_2O_3				Sm_2O_3			
	0KGy	30KGY	50KGy	80KGY	0KGy	30KGY	50KGy	80KGY	0KGy	30KGY	50KGy	80KGY	0KGy	30KGY	50KGy	80KGY	0KGy	30KGY	50KGy	80KGY
E_g	2.033	1.812	1.793	1.701	2.217	2.131	2.071	1.839	2.311	2.866	2,399	1.884	2.459	2.646	2.625	2.581	2.126	2.082	2.081	2.067
n	1.36	1.21	1.2	1.141	1.48	1.43	1.39	1.23	1.55	1.92	1.61	1.62	1.65	1.77	1.76	1.73	1.42	1.39	1.39	1.38
R_m (cm^3/mole)	11.38	10.14	10.04	9.52	12.41	11.93	11.59	10.29	12.94	16.04	13.43	10.54	13.76	14.81	14.69	14.4	11.9	11.65	11.65	11.57
E_{urbach}	0.351	0.313	0.309	0.293	0.383	0.368	0.357	0.317	0.399	0.495	0.414	0.325	0.422	0.456	0.453	0.445	0.367	0.359	0.359	0.357
λ_{cutoff}	146	130	129	122	159	153	148	132	166	205	172	135	176	190	188	185	152	149	149	148

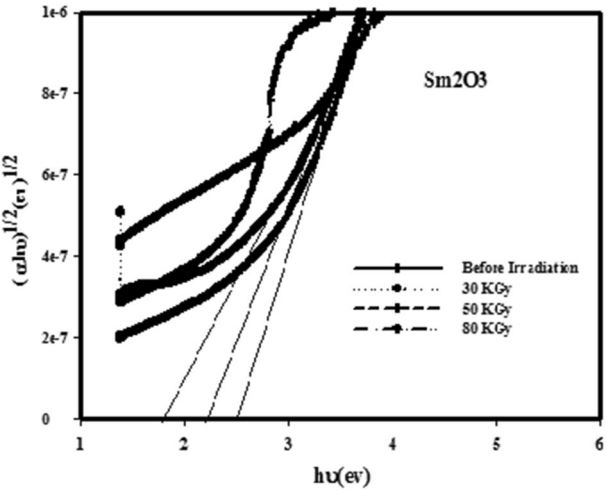

Fig. 9 Optical properties before and after gamma irradiation

(non-bridging oxygen) atoms. These observations can be explained by their molar mass Sm > Nd > La > Ce ions as mentioned before.

The ($\lambda_{cut\text{-}off}$), optical band gap values (E_g) and Urbach energy (ΔE) values of the current samples are given in Table 1. The refractive indices (n) of the samples are calculated from the optical band gap values (E_g) (Mierzejewski et al., 1988). According to data of result values, refractive index values exaggerated nearly linearly inside the molecular weight of the lanthanide oxide to cadmium borate in a glass matrix. The discovered variation in refractive index (n) values are small, indicating no important changes within the basic cadmium borate glass network with the replacement of lanthanide oxide. The slight increment of the refractive index values is attributable to the replacement of lanthanide oxide of increasing molar mass, density and molar volume.

The average molar refraction (R_m cm^3/mol) for the glasses and molar polarizability ($\alpha_m \times 10^{-24}$ cm^3) are given by the Lorentz–Lorentz equation (Saddeek et al. 2010).

The values of molar refraction (R_m) and molar polarizability (α_m) given in Table 1. The molar refraction exaggerated with the increase refractive index, that successively exaggerated oxide ion polarizability and electronic polarizability. The increase in molar refraction (R_m) and refractive index (n) accompany increases in polarizability. Hence refractive index of the current glasses not only depending on the density values, however additionally on the polarizability values of the glasses.

The calculation of optical band gap values after irradiating by different doses (30, 50 and 80) KGy and content of lanthanide oxides for all studied glass systems are in Table 1. The decrease in E_g values can be explained by the formation of defects such as NBO, displacements, and B–O bonds breaking there are inflicting the structure to point out in relaxation process and fill the large interstices in the interconnected network of boron and oxygen (Kaur et al. 2016). The limited slight decrease in E_g of Sm_2O_3 after different of high doses compared with the results of the mass attenuation coefficient is typically attributed to the Compton scattering method. This trend is observed in the glasses of Sm_2O_3concentrations (Kaur et al. 2016) therefore; that glass system can be considered a good candidate for gamma-ray shielding material.

Table 1 displays the variation of the optical band gap with irradiated doses and refractive index. Decreasing molar refraction (R_m) and refractive index (n) indicate a compaction of the glass network when irradiation.

4 Conclusions

In light of the results, we can say that: the density, microhardness and molar volume of glasses were increased with increasing of lanthanide oxides. UV–Vis and FTIR measurements showed that the CdO content in glass samples plays a role as a network modifier and change in the atomic structure is attributable to the formation of BO_4 units. It was found that there is agreement in the attenuation parameters at different photon energies are calculated using the XCOM program and measured in experiments. Therefore, the glasses have prepared may be used as radiation shielding materials with smart optical transparency and additional environmentally friendly. Thus, the cadmium borate glass doped with 0.5% Sm_2O_3 concentration glass may have a good gamma-ray attenuation comparison with either Ce_2O_3, La_2O_3, and Nd_2O_3, and it can be used as gamma-ray shielding during this range of energy.

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