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Physical and structural studies of Some Bismuth Borate Glasses doped with La^{3+}ions.

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Abstract
Bismuth borate glasses doped with La_2O_3 *is prepared by melt quenching technique with the chemical compositions: x* La_2O_3*–*$0.4Bi_2O_3$*-*$0.4B_2O_3$*-0.2CaO (where x = 0, 0.01, 0.02,0.03, 0.04,0.06, and 0.08 mol%). XRD, FTIR and optical absorption measurements have been applied to investigate the structural and optical properties. The amorphous nature of the prepared glasses has been assured using XRD pattern. The increase in density is assigned to the creation of BO4 units in the glass matrix. FTIR spectral measurements have been utilized to identify the network vibrations of* La_2O_3 *doped bismuth borate glasses. The direct and indirect allowed transitions of the optical bandgap and other related parameters such as band tailing B, refractive index and Urbach energy ΔE(ev) were determined from Tauc's plot. Smaller values in Urbach energy refer to the less disorder in the glasses. Some physical properties as the molar volume, internuclear distance* $r_i(A^0)$*, polaron radius* $r_p(A^0)$ *and field strength F* cm^{-2} *have been reported. A deconvoluted spectrum is used to get exact band positions in the FTIR spectra. FTIR spectra confirme the creation of BO4 units, the presence of BO3, BO4, BiO3, BiO4 structural units.*

Keywords: *Borate glasses, IR studies, Optical properties.*

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I. INTRODUCTION

Rare earth ions have a large cationic field strengths which effectively lower the crystallization of glass. Studying the spectroscopic countenance of rare earth (RE) ion-doped glasses are very important for their different implementations in the formulation of modern optical devices like glass lasers, light converters, …ect. Glasses doped with rare-earth ions $(RE)^{n+}$ treated as a luminescentmatter due to high emission efficiencies that corresponding to 4f-4f and 4f-5d electronic transitions in rare earth ions. The 4f-4f transition gives a distinctive severe fluorescence pattern from the ultraviolet to infrared region because of shielding effects of the external 5s and 5p orbits on the 4f electrons [1-3]. Lanthanum oxide (La_2O_3) is one of the most rare earth ions generally used because it has high glass transition temperature (T_g), hardness, lower cost among the remainder of rare earth oxides [4]. Among different glasses, borate glasses are very good stewared matrices due to boric oxide acts as a good glass former and a flux material. Addition of La_2O_3 to borate glass may strengthen the density and optical properties. Several invesigations have been on doping the rare earth ions with different oxide glasses. These could be use in different and important applications. Gautam et al. studied the optical features for (Ba,Sr) TiO_3 Borosilicate glasses doped with Lanthanum oxide. It was found that the IR and raman bands dependent on the synthesis and borate is amajor network former [5]. Doweidar et al. reported the effect of Lanthanum oxide on the structure of $PbO-B_2O_3$ glasses. They searched the the role of rare earth ions in altering the structure of $PbO-B_2O_3$ glasses [6]. Many researchers have studied the physical structural and nuclear shielding features for $B_2O_3–SiO_2–Bi_2O_3$ glasses [7]. The present work, mainly deal with the preparation of lanthanuim bismuth borate glasses and study the effect of La_2O_3 on density, refractive index and structural changes in the glass samples.

IR spectroscopic analysis is used to define the chemical active groups of the samples. IR spectroscopy is an important and common tool for structural elucidation and compound identification. UV-visible absorption spectroscopy is a very a valuable technique used to describe the optical and electronic properties of various substances such as glasses. Optical transitions are two sorts, direct and indirect. In these transitions, electrons in the valance band interact with the electromagnetic radiations and reaching them to the conduction band by gaining main band gap. These transitions take place in both crystalline and amorphous materials [5]. The structure of B_2O_3 consists of boroxyl rings random network also BO_3 triangles joint with B-O-B linkages. Also, additions of a modifier oxide cause a change of some BO_3 triangles to BO_4 tetrahedra and leads to build of different units such as di borate, tri borate or penta borate groups [8]. The changes in the structural units due to the chosen chemical compositions, including a diverse sort of modifiers and the preparation conditions of the glass. The heavy metal oxide Bi_2O_3 glass has a small rate of crystallization in addition to the density, refractive index, polarizability, optical basicity and susceptibilityare high. Bi^{3+} ion has a small field strength, so it cannot enable to comprise glass itself, but when doped with glass formers as B_2O_3 works as a glass former at high condensations and works as a network modifier at depressed concentrations [9].

II. EXPERIMENTAL PROCEDURE

Table (1): Apotograph of bismuth borate glass doped with La_2O_3 oxide, (x La_2O_3 – Bi_2O_3 - $0.4B_2O_3$ - $0.2CaO$)


X=0 (LBBC)


X=0.01 (0.01LBBC)


X=0.02 (0.02LBBC)


X=0.03 (0.03LBBC)


X=0.04 (0.04LBBC)


X=0.06 (0.06LBBC)


X=0.08 (0.08LBBC)

In the present work La^{3+} doped glasses have been intended by the conventional melt quenching technique. The starting materials Bi_2O_3, B_2O_3, CaO and La_2O_3 applied in this study were being analytical grade (99% purity). The batch composition (in wt%) of bismuth borate glasses doped with different contents of La_2O_3 oxide obtained as indicated below. about 30 g of the batches of the compositions were taken in a porcelain crucible which thoroughly mixed to obtain a homogenous mixture and then melted at 1100^{o}c by an electrical furnace for 2 h. After prefect melting, the melting was quickly poured into a preheated stainless-steel mold and annealed at 400 ^{o}c for 2 hours before cooling down to room temperature to avoid the figuration of cracks and air bubbles. The annealed glasses were polished to take optical and density measurements. Powder X-ray diffraction (XRD) measurements were executed using Japan diffractometer employing CuKα radiation to prove the amorphous nature of the prepared samples. Fourier transforme infrared spectrum of glass matrix (FTIR) was recorded using Jasco FT/ IR-4600spectrometer at room temperature with a resolution of 4 cm^{-1} in the amplitude 400-4000 cm^{-1}. The optical absorption spectra were registered using Jasco Spectrometer in the region 200-450 nm with a resolution of 4nm.

III. RESULTS AND DISCUSSION

3.1. X-Ray Diffraction (XRD)

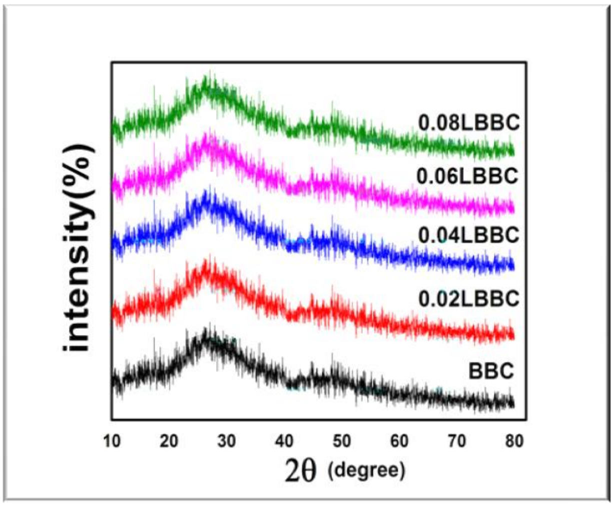

Fig. (1): XRD pattern of some glass samples.

X-ray diffraction model of the bismuth borate glasses doped with La^{3+} was recorded in the range of 10° ≤ Ө≤ 80°. As the fgure (1) shows, there are no discrete or sharp peaks appear in the XRD pattern of the all samples, only two broad humps around 29° and 51°. Such humps are characteristic and manifested on only the amorphous materials. Therefore it can be stated that all prepared samples are a good glasses. The excitance of two humps may refers to presence of two nucleating against through the glass matrecies.

3.2.Fourier Transform Infrared (FTIR) spectra

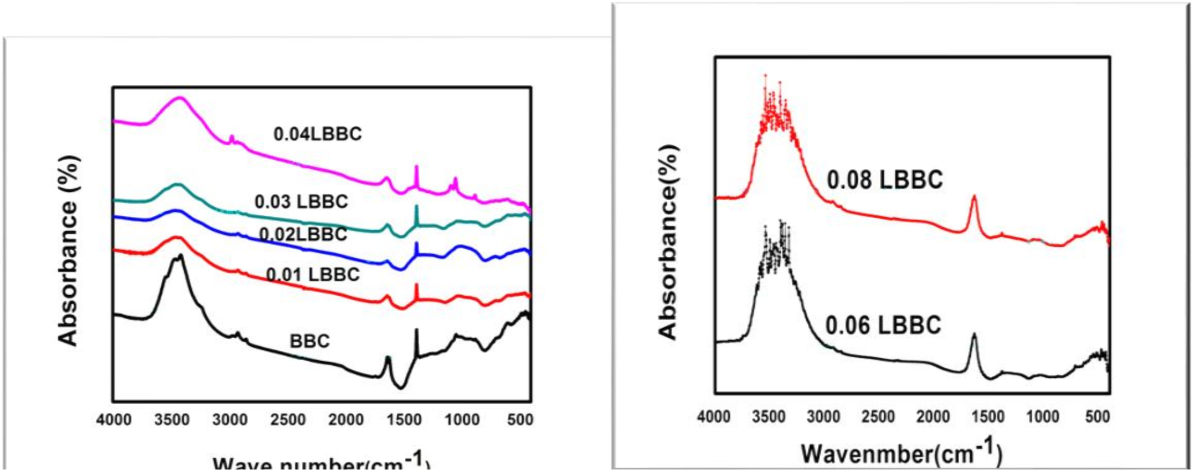

Fig. (2): FTIR spectra for the glass system.

FTIR absorption spectra of the glass systems have been listed between 400 and 4000 cm^{-1} to obtain main notification revealing on the ordering the fundamental structural units.

From Fig.(2), it is observed that the major absorption frequencies can be grouped into two regions.

1- Mid-region expanded from (2000 – 400) cm^{-1} and is featured by the presence of the special absorption bands of the network forming groups.

2- Near (IR) region expanding from (2000-4000) cm^{-1} includes the absorption bands owing to the vibrations of water and hydroxyl (OH).

Generally, in bismuth borate-based glasses, IR vibrations are effective in 450-1700 cm^{-1} region and the boron atom has a small mass contrasted with another network forming elements such as Bi and La. The base vibrational modes with the borate network show above 500cm^{-1}. In bismuth-based glasses, a continuous network of octahedral BiO_6 structural units connected through an oxygen atom and the high polarizability of Bi^{3+} cations lead to boost in the covalent bonding between Bi and O. Also, it is quickly obtained that, the IR absorption bands in the frequency region smaller than 500 cm^{-1} are generated due to the vibrations of Bi-O polyhedral structures [9]. The undoped (basic) glass reveals various bands enlarged from 400-4000 cm^{-1}. The band observed around 483 cm^{-1} is refered to the vibrations of Bi-O and Bi-O-Bi in distorted BiO_6 units, Bi-O^- stretching vibrations in distorted linked BiO_6 units [9], stretching vibrations of Bi-O- /Bi-O-Ca, isolated di-borate groups, di-borate units and the bending vibrations of BO_4 anions[10]. This band observed also in the range from 460 cm^{-1} to 475 cm^{-1} in all glass samples with different intensities which shifted to lower wavenumber with increasing La_2O_3 oxide due to lowering degree of distortion. The bands observed from nearly 600cm^{-1} up to 700 cm^{-1} in BBC, 0.01La_2O_3,0.02 La_2O_3, 0.03 La_2O_3, 0.04 La_2O_3, 0.06 La_2O_3, and 0.08 La_2O_3, glass systems are attributed to symmetric breathing vibrations of meta borate rings, penta borate groups, Bi-O-stretching vibrations in BiO_6units[9], B-O-B bending vibrations[11,12] and symmetrical stretching vibrations of Bi-O bond in BiO_3 units [13]. The decrease in intensities of some bands with increasing La_2O_3 content can be related to several possibilities, including the variation in some configuration of borate arrangement and configuration of bismuth glasses. The bands observed at 1043, 1027, 997 and 1039 cm^{-1} by the glass systems BBC, 0.01 La_2O_3, 0.02 La_2O_3, and 0.03 La_2O_3, with different intensities and with some broadening at 1027, 997, and 1039 cm^{-1} are attributed to normal vibrations of tri-, tetra-, and penta borate groups, B-O stretching vibrations of the BO_4 tetrahedral units and entity of di borate groups[9,11,14]. Adding 0.04La_2O_3, to the basic glass, the band at 1043cm^{-1} exhibits splitting into two component peaks at 1049 and 1088 cm^{-1} and shifted to higher wave number, these bands nearly disappear in the glass samples 0.06 La_2O_3, and 0.08 La_2O_3, Also the main prominent bands observed at 1383 cm^{-1} in all glass systems with different intensities are attributed to:

A- Stretching vibrations of B-O^- bonds and stretching vibrations of BO_3-O-BO_4 bonds in BO_3 pyramidal units.

B- **B-** Stretching vibrations of B-O-B bonds in pyramidal BO_3 units, B-O- stretching in meta borate rings and chains[8].

C- **C-** B-O stretching vibration of NBO trigonal BO^{-3}_3 units [12] and stable tetrahedral BO_4 units[14].

The observed bands centered at 1637 cm^{-1} with different intensities in BBC, 0.01 La_2O_3, 0.02 La_2O_3, 0.03 La_2O_3, and 0.04 La_2O_3, glass samples with some broadening also appear when the concentration of La_2O_3 dopant increase to 0.06 and 0.08 wt% at 1640 cm^{-1} confirming the fact that the nonbridging oxygen decease due to the alteration of BO_3 structural units into BO_4 units [11], asymmetric stretching relaxation of the B-O band of trigonal BO_3 units[15]. The results suggested that La_2O_3 not make large alterations in the structure of the glass system as suggested by Zhichao et al, only change at the position, broadening and intensities of some bands. Briefly, introduce of La oxide does not result in essential effects in FTIR spectra.

3.3.Physical properties

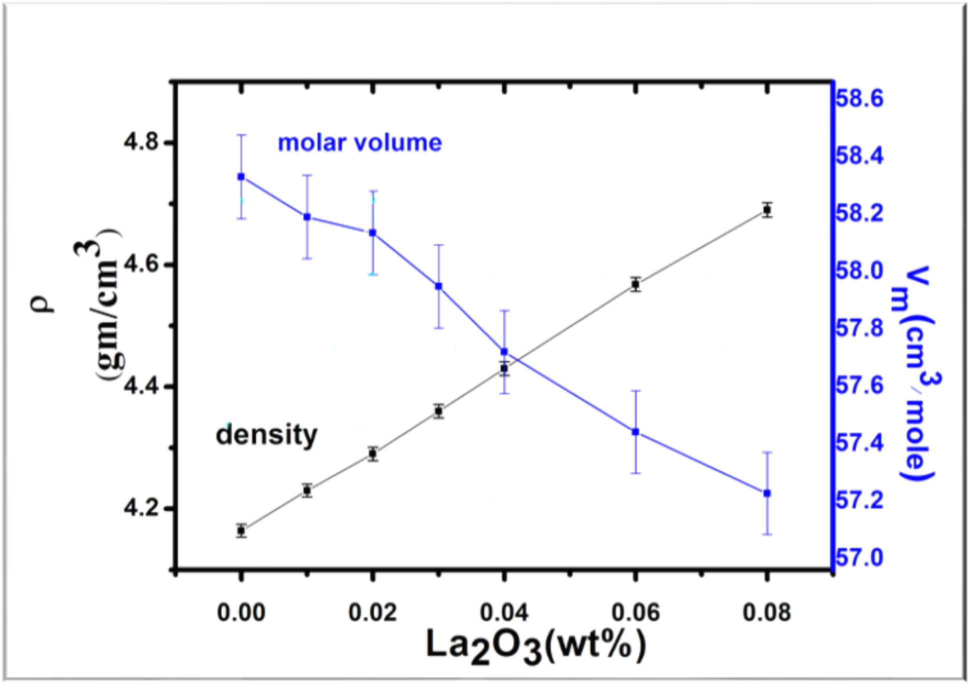

Fig. (3): Density and molar volume of glass samples.

The density boost with the rise in La_2O_3 contents from 4.164 to 4.69g cm^{-3}, owing to their high relative molecular mass. The modulation in molar volume based on the rates of fluctuation of both density and molecular weight. However, the rate of boosting molecular weight is higher than the rate of rising in density. This would be accompanied by a diminution in molar volume as shown from the table. The molar volume of the glass system decreases with increasing in La_2O_3 contents from 58.369 to 57.266 cm^3 as shown in Fig.(3), which refered to the drop in the number of non- bridging oxygen (NBOs). It may be supposed that the enlargement in La_2O_3 concentration causes the glass system closed glass network structures. The La^{3+} ion concentrations (N) were calculated utilizing the formula.

$$\mathbf{N(ions/cm^3)= x(N_A)(\rho)/M_T} \qquad \mathbf{(3\text{-}1)}$$

Where x is the mole fraction of La_2O_3 oxide, N_A is the Avogadro's number, ρ is the density and M_T is the average molecular weight. It is observed from the table (1) that the concentrations increase with increasing La_2O_3 contents in the glass composition system. The observed values of N are used to calculate the polaron radius (r_p) and inter nuclear distance (r_i) according to the relations:

$$\mathbf{r_p(A^o)=(1/2)(\pi/6N)^{1/3}} \qquad \mathbf{(3\text{-}2)}$$

$$\mathbf{r_i(A^o)=(1/N)^{1/3}} \qquad \mathbf{(3\text{-}3)}$$

The decrease of r_p with increasing La_2O_3 content in the glass systemwas most likely linked to the increased value of (N) for La^{3+} which caused stronger field strength around La^{3+} ions. The RE ions are situated between the layers and thus the average RE-oxygen distance decrease, as a result of that, the La-O bond strength increases, leading to a stronger field around the La^{3+} ions. The field strength (F) around La^{3+} ions are measured by using the equation:

$$\mathbf{F(cm^{-2})= \{Z/r_p{}^2\}} \qquad \mathbf{(3\text{-}4)}$$

Where Z is the atomic number of La ions in the glass system.

3.4.Optical properties

In the amorphous system, the optical bandgap is coming back to the energy gap between the valence band and the conduction band. The optical band gap is a very important device to recognize the electric transitions in both amorphous and crystalline materials. The absorption coefficient **α(υ)**close the spectral edge can be studied using the relation:

$$\mathbf{\alpha(\upsilon)=(1/d)\ Ln(I_t/I_0)} \qquad \mathbf{(3\text{-}5)}$$

Where **d** is the thickness of the sample, **Ln** $(\mathbf{I_t/I_0})$agreewith the optical density **(A)**, where $\mathbf{I_o}$ and $\mathbf{I_t}$ are the intensities of the incident and transmitted light respectively.

Absorption coefficient **α(υ)** as a function of photon energy **(hυ)** for direct and indirect allowed transitions can be expressed using the relation:

$$\mathbf{\alpha(\upsilon)= B(h\upsilon- E_g)^n/h\upsilon} \qquad \mathbf{(3\text{-}6)}$$

where $\mathbf{E_g}$ is the optical band gap energy, **h** is the plank's constant, **υ** is the frequency of the photon, **n** is the index number which used to obtain the allowed transitions (indirect or direct) with the values 1/2 and 2 respectively, **B** is the band tailing parameter which studied from the slope of the Tauc's plot. The ($\mathbf{E_g}$) values have been gained by extrapolating the linear region to the zero absorption at $\mathbf{(\alpha h\upsilon)^{1/2}=0}$ and$\mathbf{(\alpha h\upsilon)^2=0}$ for indirect and direct allowed transitions, respectively and the (B) values also get from the slope of the curves[16]. Tauc's plot of the $\mathbf{(\alpha h\upsilon)^2}$ as a function of photon energy for the direct allowed transitions of the prepared La^{3+}doped bismuth borate glasses are obviousin Fig. (4)**.**

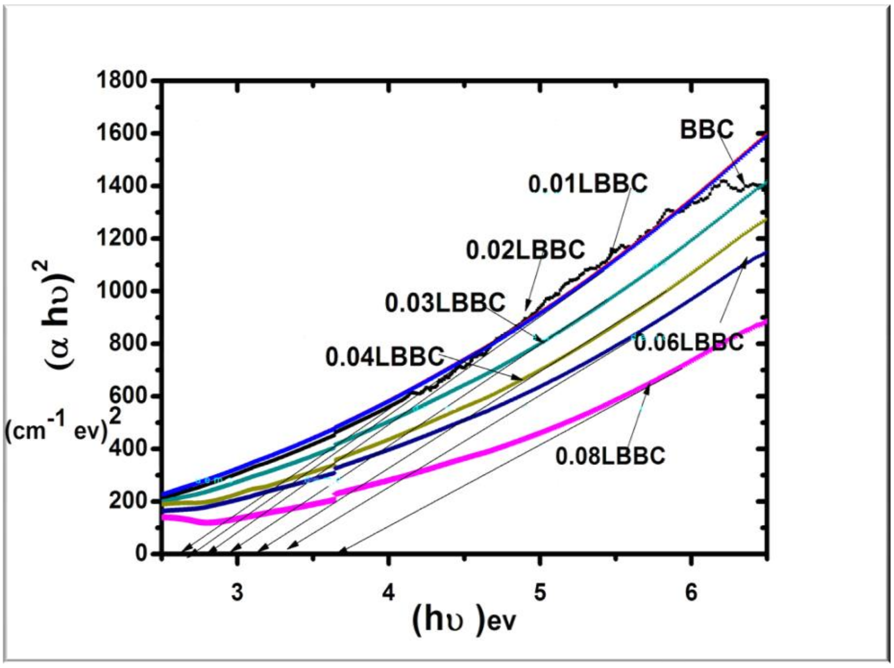

Fig. (4): Tauc's plot for the La^{3+} doped bismuth borate glasses.

It is observed from the table (2) that the bandgap values are increase with increase the concentrations of La_2O_3 oxide. The direct and indirect band-gap amount boost with the increases of La^{3+} ion contents. Such excess is due to the bonding defect and the construction of bridging oxygen in the title glasses. The ratio between BO_4 and BO_3 units decreases when the concentration of La^{3+} ion increases in the prepared glasses so the non-bridging oxygen is decreased in the present glass network and the same is emphasized through the vibrational spectralanalysis[11]. The absorption coefficient $\alpha(\upsilon)$ close to the band edge has been examined by using Urbach's energy relation:

$$\alpha(\upsilon)=\alpha_0 \exp(h\upsilon/\Delta E) \qquad (3\text{-}7)$$

Where α_0 is a constant, **h** is the plank's constant, υ is the frequency of the photon and ΔE is the Urbach energy which used to recognize the width of the localized states to get the degree of troublein the amorphous and crystalline materials[11].

It is observed from the table that, the Urbach energy values are nearly boosted with the excess in La^{3+}concentratios and is found to be in the range 0.0081 to 0.0288eV. Band tailing parameter values is found to vary between 0.806-0.532($cm^{-1/2}$ $eV^{1/2}$) and vary between 461.66- 323.7 (cm^{-2} ev^2)for indirect and direct allowed transitions. The same trend between the optical bandgap and Urbach energy values of the La^{3+}: BBC glasses are owing to the lower defects produced in the localized states.

The prepared La^{3+} doped bismuth borate glasses are exhibit lower Urbach's energy values which submit the fact that the present glass materials have long-range order which arising with minimum defects.

The refractive indices (n) of different glass samples are studied by using the relation:

$$n^2-1/n^2+1=1-\sqrt{(Eg/20)} \qquad (3\text{-}8)$$

The molar refraction (R_M) is evaluated by the relation derived by Volf and Lorentz is given by:

$$R_M = (n^2-1/n^2+1)V_m \qquad (3\text{-}9)$$

Where V_m is the molar volume of the glass system [16,17]. The molar polarizability (α_m) is evaluated using the following equation,: where N_A is Avogadro's number

$$R_M = 4\pi\, \alpha_m N_A/3 \qquad (3\text{-}10)$$

The reflection loss of normal incident electromagnetic waves at the absorbersurface and dielectric constant (ε) aregivenbythe relations[17].

$$\varepsilon = n^2 \qquad (3\text{-}11)$$

$$R=(n-1/n+1)^2 \qquad (3\text{-}12)$$

Direct and indirect band gap energies, refractive index and other related optical properties of La^{3+} doped bismuth borate glass samples for different La_2O_3 concentrations are illustrated in Table (2).

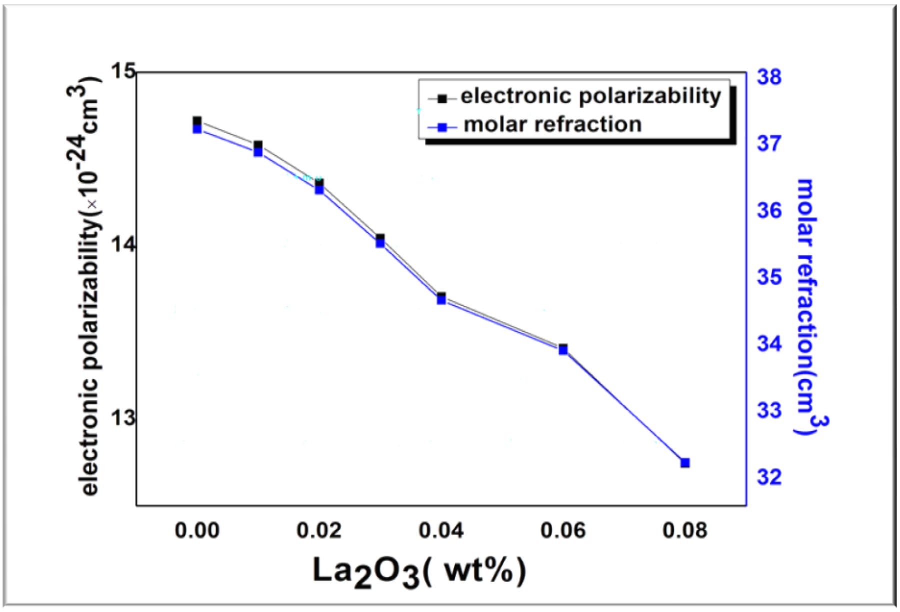

Fig.(5): Molar refraction and electronic polarizability of the glass system.

Table (2): The physical and Optical properties of La $^{3+}$doped bismuth borate glasses.

Physical and Optical properties	BBC	0.01LBBC	0.02LBBC	0.03LBBC	0.04LBBC	0.06LBBC	0.08LBBC
Average molecular weight(M_T)g	243.051	246.309	249.567	252.826	256.084	262.599	269.116
Density ρ (gm⁄cm^3)	4.164	4.23	4.29	4.36	4.43	4.568	4.69
Molar volume V_m(cm^3)	58.369	58.229	58.174	57.987	57.759	57.48	57.266
La^{3+} ion concentration(N$_\times$ $_{10}{}^{22}$)ions/cm^3	---	0.01034	0.02071	0.03116	0.04168	0.06134	0.08397
Polaron radius r_p(A^o)	---	8.586	6.811	5.944	5.395	4.743	4.272
Inter-nuclear distance r_i(A^o)	---	9.889	7.845	6.846	6.214	5.463	4.919
Field strength F(×10^{16}) (cm^{-2})	---	0.773	1.229	1.613	1.958	2.534	3.123
Molar polarizability ×10^{-24}cm^3)	14.72	14.58	14.361	14.043	13.706	13.407	12.743
Direct band gap energy(ev)	2.631	2.703	2.846	3.007	3.185	3.346	3.705
Band tailing parameter B (cm^{-1}ev)^2 at n=2	461.66	461.6	451.17	378.58	369.3	359.75	323.7
Indirect band gap energy(ev)	1.55	1.55	1.63	1.69	1.75	2.01	2.5
Band tailing parameter B (cm^{-1}ev)$^{1/2}$ at n=1⁄2	0.806	0.806	0.757	0.623	0.604	0.586	0.532
Urbach energy(ΔE)(ev)	0.0081	0.0151	0.0152	0.013	0.0399	0.0278	0.0288
Refractive index (n)	2.5	2.48	2.44	2.39	2.34	2.3	2.2
Molar refractivity Rm(cm^3)	37.144	36.796	36.23	35.43	34.579	33.826	32.149
Reflection loss R (%)	0.184	0.181	0.175	0.168	0.161	0.155	0.141
Dielectric loss(ε)	6.25	6.15	5.95	5.71	5.47	5.29	4.84

IV. CONCLUSIONS:

La^{3+} doped bismuth borate glasses were prepared with different doping concentrations of La_2O_3 in the glass system and characterized for their structural, physical and optical properties. The glasses FTIR spectra noted only shift in the peak position, small broadening and variation in the intensity of some bands owing to that La^{3+} ions are coordinated by NBO in bismuth borate glasses, and no new bands were observed. The physical properties of the glass system were discussed. From the optical measurements, it was observed that the direct and indirect band gap values boost with the excess of La^{3+} ion contents such elevation is because of the bonding defect and the formation of bridging oxygen, which confirmed by the diminish in the molar volume results byincreasing La^{3+}contents. Also, the optical bandgap shows slight dependence on compositions.

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