Novel Er$^{3+}$ doped heavy metals-oxyfluorophosphate glass as a blue emitter

Aly Saeed · S. Sobaih · W. A. Abu-raia · A. Abdelghany · Sh. Heikal

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Abstract
Developed P$_2$O$_5$-ZnO-PbO-WO$_3$-NaF glasses containing Er$^{3+}$ ions were prepared by the melt/casting procedures. X-ray diffraction patterns confirmed the amorphicity of the prepared materials. The density and Fourier Transform Infrared FTIR spectroscopy showed that Er$^{3+}$ ions play a modifier role and the studied glasses have low phonon energy. The observed decrease in the measured glass transition temperature indicates that the decrease in the bonding strength of the studied glass structure. The studied glass has high thermal stability. Measured UV–Vis absorption spectra exhibited several bands in the ultraviolet and visible regions. The studied glass has a high refractive index. The metallization criterion showed that the studied glasses have an insulating behavior. The metallization criterion values of the present glasses are in the range of nonlinear optical materials. Under 320 nm excitation wavelength, the studied glass generates three blue bands at 446, 457, and 473 nm. The CIE-1931 chromaticity diagram coordinates confirmed the blue emission of the prepared glass. According to the obtained results, the produced glasses have a high potential to be used as efficient luminescence materials for photonic devices in the blue region.

Keywords Photonic glass · Blue emission · Heavy metals-oxyfluorophosphates glass · Er$^{3+}$ ion

1 Introduction
Recently, photonic materials play a tremendous role in several modern devices. A high-broad category of optical instruments relies on the efficiency of light emission in a certain region of the electromagnetic spectrum (Shi et al. 2018; Li et al. 2018). The generation and control in the four fundamental colors of white light, blue, green, yellow, and red using photonic materials have been investigated extensively (Annapoorani

Aly Saeed
aly-saeed@eru.edu.eg

1 Mathematical and Natural Sciences Department, Faculty of Engineering, Egyptian-Russian University, Cairo, Egypt

2 Basic Science Department, Institute of Aviation Engineering and Technology, Giza, Egypt

3 Physics Department, Faculty of Science (Girls Branch), Al-Azhar University, Cairo, Egypt

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et al. 2015; Xia et al. 2016; Basavapoornima et al. 2020). Several photonic materials have been studied for various optical and electro-optical devices, among them glass materials. Glassy materials emerged and occupied a topping rank in this field due to their various advantages like ease of fabrication, translucence, the solubility of high concentration of rare-earth ions, and cost-effective production (Romaniuk 2007; Zhang et al. 2020). Phosphate is considered a superior host matrix that has good thermal stability, high transparency over the UV and visible wavelength range, and especially good solubility of rare-earth ions. Due to their properties, phosphate glasses were regarded as better hosts compared to other host glass networks (Jiang et al. 2014; Ming et al. 2012; Marzouk et al. 2017). The poor chemical durability of phosphate glass is always treated by the addition of suitable oxides such as ZnO, MoO3, and Al2O3. On the other hand, inlaying the phosphate network with ZnO improves the optical and electronic properties of phosphate glass (Kuwik et al. 2020; Galleani 2018). Fluoride host materials have a high quantum yield of luminescence due to ionic bonds and low phonon energy of fluoride (Deepa et al. 2019; Naftaly and Jha 2000). The heavy metals-rich glass has a combination of desirable optical, mechanical, and thermal properties of its constituents. The inclusion of heavy metal ions in the glass matrices is of interest to the realization of more efficient photonic behavior in which their presence improves the efficiency of fluorescence. The addition of heavy metal oxides minimizes the host phonon energy and thereby suppressing the non-radiative losses (Aly Saeed et al. 2018; Bosca et al. 2009). In view of the foregoing, heavy metal oxyfluorophosphate glass is an excellent selection as host materials for several active optical applications. Trivalent erbium ion Er3+ is used as a dopant in various host glasses due to its unique optical properties. The erbium ion is an ideal candidate to be used widely in photonic applications due to its rich energy levels in the near-infrared, visible, and ultraviolet ranges (Pugliese et al. 2016; Sun et al. 2005; Patwari and Eraiah 2019).

The motivation of the present work is to develop a low phonon energy glass system incorporated with Er3+ ions for photonic applications. X-ray diffraction XRD, density, FTIR, DSC, optical absorption were measured. Many structural, thermal, and optical parameters such as mean phosphor–phosphor separation, glass stability, optical band gap, refractive index.

2 Experimental techniques

2.1 Materials synthesis

Proper amounts of high purity oxides and fluoride were synthesized and melted to get heavy metal oxyfluorophosphate glass containing Er3+ ion in the chemical formula 45P2O5-25ZnO-(20-x)PbO-5WO3-5NaF-xEr2O3, where x in mol% is equal to 0, 1, and 2. The raw materials in the powder form were mixed thoroughly in the agate mortar and placed in a porcelain crucible for melting it in an electric furnace at 1100 °C for 2 h to obtain a homogenous bubble-free liquid. During the melting period, the molten was intermittent stirring at regular intervals then quenched on a preheated stainless steel mold. The obtained glasses were annealed below the glass transition temperature to eliminate internal thermal stresses.
2.2 Measurements

The structural phase of the prepared materials was examined using X-ray diffraction mechanism [Shimadzu XD3A radiation with CuKα = 1.54056 Å]. Bulk density was measured at room temperature using Archimedes principle by xylene as the buoyancy liquid (0.86 g/cm³) using the relation (Aly Saeed et al. 2018; El-Batala et al. 2021)

\[ \rho = \frac{W_a \rho_x}{W_a - W_b} \]  

where, \( W_a \) is the glass piece weight in air, \( W_b \) is its weight in xylene liquid, \( W_a - W_b \) is the buoyancy and \( \rho_x \) is the density of xylene. The measured values have been conducted three times and the average was taken.

The structural units of the obtained glasses were studied by a computerized FTIR spectrophotometer [JASCO FT-IR-300] using KBr as reference material in the spectral range 400–4000 cm⁻¹. Differential scanning calorimeter DSC measurements were carried out by TA Instruments, SDT Q600 in an open platinum pan at heating rates of 10 °C/min up to higher than 600 °C in a high-purity nitrogen atmosphere and at a flow rate 15 Psi. The test was performed to identify the characteristic temperatures such as the glass transition and onset of crystallization with accuracy ± 3 (K) under non-isochronal conditions. About 2 mm glass sample thickness had been polished for UV–visible absorption spectra measurements in the range 190–1100 nm using Jenway 6405 UV/Vis Spectrophotometer. The photoluminescence spectra were measured using a SPEX spectrofluorimeter (Model Fluorolog-II, SPEX CertiPrep, Metuchen, NJ) in the wavelength region of 300–500 nm under 320 nm excitation wavelength with ± 1.5 wavelength accuracy emitted from 150 W Xenon lamp. The PL measurements were performed on the flat and smooth surface specimens.

3 Results and discussion

3.1 Powder X-ray diffraction (XRD)

The obtained results of the x-ray diffraction patterns are exhibited in Fig. 1. The two beaks and high noise signal in the diffraction patterns reflect the feature of the non-periodic arrangement of atoms in the obtained solid materials. The showed spectra confirm the amorphous structure of the studied materials. The X-ray diffractograms have notable broadband due to the interplanar spacing in the glass structure.

3.2 Density, Molar Volume, and mean phosphor–phosphor separation

The alteration of density \( \rho \) and molar volume \( V_m \) with the increase of Er \(^{3+}\) ions are shown in Fig. 2. The Molar volume \( V_m \) (cm\(^3\)/mol) was calculated using the molecular weight \( M \) (g/mol) and the experimental density \( \rho_{\text{exp}} \) (g/cm\(^3\)) according to the formula (Aly Saeed et al. 2018; El-Batala et al. 2021).

\[ V_m = \frac{M}{\rho} \]
The results revealed diminution in density and growth in molar volume with Er\(^{3+}\) ions increment. The noticeable decrease in the density and augmentation in molar volume indicate that the inclusion of Er\(^{3+}\) ions to the proposed glass matrix create more interstitial space in the glass network. This space occurs due to the formation of non-bridging oxygens NBOs. The tangible augment of molar volume refers to Er\(^{3+}\) ions occupy the interstitial space in the present glass network, which means that the Er\(^{3+}\) ions enter as a modifier in the studied glass network. This behavior of Er\(^{3+}\) is responsible for creating the NBOs inside the present glass structure.

The mean phosphor–phosphor separation \(d_{P-P}\) was calculated using the standard formula (El-Batala et al. 2021).

\[
d_{P-P} = \left( \frac{V^P_M}{N_A} \right)^{\frac{1}{3}} \\
V^P_M = \frac{V_M}{2(1 - X_P)}
\]  

(3)
where the volume $V_P^M$ corresponds to the volume that contains one mole of phosphor within the given structure and $X_P$ molar fraction of $P_2O_5$, and $N_A$ Avogadro’s number.

The observed increase in mean phosphor–phosphor separation as shown in Fig. 3 asserts that the expansion in the glass network i.e., the insertion of $\text{Er}^{3+}$ opens the glass network.

### 3.3 FTIR characterization

Four clear bands were observed in the free $\text{Er}^{3+}$ glass sample as shown in Fig. 4. Two intense bands located at 649 and 1480 cm$^{-1}$, and two low intense bands centered at 785 cm$^{-1}$ and 960 cm$^{-1}$ are determined.

The high intense band located at 649 cm$^{-1}$ is attributed to the symmetric stretching modes ($P$–$O$–$P$)$_s$ linkages of $Q^1$ (Kuwik et al. 2020; Deepa et al. 2019; El-Batala et al. 2021). The 649 cm$^{-1}$ band is the highest and hence it is corresponding to the phonon energy. The present glasses have low phonon energy ranged from 649 to 656 cm$^{-1}$ compared to the other glasses such as silicate, borate, and germanate (Möncke and Eckert 2019;
Yoshimoto et al. 2017). The located low intense band at 785 cm\(^{-1}\) due to the asymmetric stretching modes (P–O–P\(_{as}\)) linkages. The low intense band at 960 cm\(^{-1}\) indicates the existence of asymmetric stretching vibrations of \(PO_{4}^{3-}\) tetrahedra (P–O – ionic group). The presence of this band suggests the ionic character of all studied glasses. The broadband at 1480 cm\(^{-1}\) is assigned to the asymmetric stretching of double-bonded P=O modes (Kuwik et al. 2020; Deepa et al. 2019; El-Batala et al. 2021; Morshidy and Sadeq 2019; Sadeq and Morshidy 2019). With the inclusion of Er\(^{3+}\), all the peaks exhibit a slight variation to longer wavenumber and increase in intensity. No bands were observed in the Er\(^{3+}\) doped glasses. These observations confirm the formation of NBOs due to the incorporation of Er\(^{3+}\) ions into the matrix, the increase of the degree of polymerization of the glasses, and the modifier role of the Er\(^{3+}\) ions in the studied glass network (Kuwik et al. 2020; Deepa et al. 2019; El-Batala et al. 2021; Möncke and Eckert 2019; Yoshimoto et al. 2017).

3.4 Thermal properties

The obtained values of the glass transition temperature \(T_g\), onset crystallization temperature \(T_c\), and thermal stability \(\Delta S\) that evaluated via the temperature gap between \(T_c\) and \(T_g\) \((T_c - T_g)\) (Deepa et al. 2019) are shown in Fig. 5. The observed decreases in \(T_g\) and increases in \(T_c\) indicate a decrease in the bonding strength of the glass structure, which in turn means the glass network somewhat less rigid. The change in \(T_g\) and \(T_c\) is also linked to the number of bridging and non-bridging oxygen in the glass network. The decrease in \(T_g\) and increase in \(T_c\) mean an increase in the number of non-bridging oxygen, which indicates that the structure becomes more open. These results support the density and FTIR results. The diminution of \(T_g\) with the increasing of Er\(^{3+}\) concentration implies Er\(^{3+}\) enriches the glass-forming ability. The thermal stability of the glass is an important factor in photonic devices, in which high values of thermal stability are desirable. Obviously, the addition of Er\(^{3+}\) improves the stability of the prepared glass. The value of \(\Delta S > 100^\circ C\), which indicates that the present glasses exhibit suitable thermal stability and could be useful for photonics applications.

3.5 Optical properties

The absorption coefficient spectra of Er\(^{3+}\) free and co-doped studied glasses are displayed in Fig. 6. Nine absorption bands in the 1 mol% Er\(^{3+}\) doped samples are observed due to the various transitions in Er\(^{3+}\) ions.

![Fig. 5 \(T_g\), \(T_c\), and \(\Delta S\) of the studied glasses](image-url)
The absorption bands corresponding to the transitions between the Er\(^{3+}\) ion ground-state, \(4I_{15/2}\), and its various excited states belonging to the \(4f^2\) configuration. The observed peaks are centered at 363, 376, 407, 455, 486, 519, 542, 650, and 980 nm, these peaks were attributed to the transition from the ground state \(2I_{15/2}\) to \(4G_{9/2}, 4G_{11/2}, 4P_{3/2}, 4F_{5/2}, 4F_{7/2}, 4H_{11/2}, 4S_{3/2}, 4F_{9/2},\) and \(4I_{11/2}\) and transitions of \(\text{Er}^{3+}\) ions, respectively. Besides these bands, an additional band is observed at 2 mol\% \(\text{Er}^{3+}\) and centered at 800 nm, which is attributed to the transition to \(4I_{9/2}\) in \(\text{Er}^{3+}\). The longer shift in the absorption spectrum of 2 mol\% indicates the glass becomes less rigid and the increase of non-bridging oxygen atoms (Pugliese et al. 2016; Sun et al. 2005).

### 3.5.1 Optical parameters

For indirect band gap \(E_g\), photon absorption stimulates phonon absorption or/and emission. The photon absorption matches to photon energy \(E_g - E_{ph}\) represents the phonon absorption with energy \(E_{ph} = h\omega\) (where \(\omega\) is the phonon frequency). While, the photon energy corresponding to \(E_g + E_{ph}\) represents that the photon absorption is happened by phonon emission, for the absorption coefficient \(\alpha\) is larger than that for phonon absorption. The value of \(E_g\) for indirect allowed transition is deduced through solving \(E_g - E_{ph}\) and \(E_g + E_{ph}\) (Morshidy and Sadeq 2019; Sadeqa and Morshidy 2019). Figure 7 shows the relation between of \((ahv)^{1/2}\) versus photon energy \(h\nu\) for the sample \(\text{Er}^{3+}\) free sample as a representative figure, the other samples show the same trend.

The obtained values of optical band gaps versus different concentrations of \(\text{Er}^{3+}\) ions are depicted in Fig. 8. The remarked shrank in \(E_g\) with \(\text{Er}^{3+}\) ions addition is accredited to the disruption of the bridging oxygen atoms. The inclusion of \(\text{Er}^{3+}\) creates more non-bridging oxygen through the glass network, which augmented the disorder in the localized states of electrons; thereby decreasing the donor centers causing reducing the optical band gap.

The Urbach energy \(E_u\) was calculated relying on the Urbach and Tauc’s model via the relation (El-Batala et al. 2021)
where, $\alpha_o$ is a constant. The Urbach energy is deduced through plot $\ln(\alpha) v^{1/2}$ against $h\nu$ and by taking the slope of the straight line of the plotted curve. The augmentation of $E_u$ as shown in Fig. 8 indicates the increase of the disorder in the studied glass network accredited to the imperfections formation as anomalous bond with variations in bond angle deformation, dangling bonds, or NBOs in the glasses, wrong bonds besides color centers.

The refractive index $n$ based optical band gap energy $E_g$ was calculated from Eq. 5 (Wang et al. 2009; Aly Saeed and Elbashar 2016). The obtained values of refractive indices are shown in Fig. 9. The higher values of the refractive indices are attributed to the creation of non-bridging oxygen in the glass network. The non-bridging oxygen is much more polarizable than bridging oxygen. The glasses with a more fragmented network exhibit higher refractive indices.

$$
\alpha(v) = \beta \exp \left( \frac{h\nu}{E_u} \right) \tag{4}
$$
The solid behavior i.e. metallic or insulator was identified through the values of the metallization criterion. The metallization criterion can be valued from the relation suggested by Dimitrov and Komatsu (Wang et al. 2009; Aly Saeed and Elbashar 2016)

\[
\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{\frac{E_g}{20}}
\] (5)

In the present glasses, the values of the metallization criterion changed from 0.415 to 0.406 as shown in Fig. 9. The values of the metallization criterion of the studied glasses are less than one. Therefore, the studied glasses have insulating nature. The studied glasses possess a metallization criterion in the 0.415–0.406, which are a good basis for nonlinear optical materials (Wang et al. 2009; Aly Saeed and Elbashar 2016).

### 3.6 Emission spectral analysis

Figure 10a displays the emission spectra of the studied Er\(^{3+}\) doped glasses after pumping by 320 nm. Three emission bands are observed in the blue area of the spectra and located at 446, 457, and 473 nm. The observed peaks are assigned to \(4F_{3/2} \rightarrow 4I_{15/2}\) (446 nm), \(2F_{5/2} \rightarrow 4I_{15/2}\) (457 nm), and \(4F_{7/2} \rightarrow 4I_{15/2}\) (473 nm) transitions. The emission intensity of 2 mol% Er\(^{3+}\) is less than 1 mol% Er\(^{3+}\) due to the concentration-quenching effect. To get more understanding for the energy transfer mechanisms involved between different energy levels of Er\(^{3+}\), the energy schematic diagram is disciplined in Fig. 10b. The ground state electrons \(4I_{15/2}\) of Er\(^{3+}\) ions are excited to the \(2P_{3/2}\) using 320 nm excitation wavelength. The Er\(^{3+}\) ions populated at \(2P_{3/2}\) energy level then decay nonradiatively to a long-lived \(4F_{3/2}, 4F_{5/2},\) and \(4F_{7/2}\) levels, which is due to the multiphonon relaxation process MRP process. The electrons at \(4F_{3/2}\) are decay rapidly through radiative
relaxation to the ground state $^4I_{15/2}$ level and produce blue emission peaks at 446 nm. The electrons at $^4F_{3/2}$ are also decayed through the nonradiative relaxation NR process to populate long-living $^4F_{5/2}$ level.

The emission band at 457 nm is produced by radiative relaxation from $^4F_{5/2}$ level to the ground state $^4I_{15/2}$ level. The emission peak at 473 nm is produced through rapid decay of radiative relaxation from the $^4F_{7/2}$ level to the ground state $^4I_{15/2}$. The CIE chromaticity coordinates for 1 and 2 mol% of Er$^{3+}$ ion are (0.135, 0.045) and (0.133, 0.047), respectively as shown in Fig. 11. These coordinates correspond to the blue region as shown in Fig. 11.

**Fig. 10**  
(a) The emission spectra and (b) Transition mechanisms of Er$^{3+}$ in the studied glass network.
4 Conclusion

Developed heavy metals oxyfluorophosphate glasses inlaid by 1 and 2 mol% of Er$^{3+}$ ions were prepared. The obtained results of density, FTIR, thermal properties, optical band gap, Urbach energy, and refractive index are in perfect tune with each other. The structural properties showed that Er$^{3+}$ ions act as a modifier role in the present glass network. The studied glass network is an open structure and many non-bridging oxygen atoms were created in the glass network. The studied glasses have low phonon energy ranging from 649 to 656 cm$^{-1}$. The present glasses have high thermal stability. Under 320 nm excitation wavelength, three bands in the blue region centered at 446, 457, and 473 nm were generated. Hence, the reported glasses are potentially usable as photonic materials, especially as a blue emitter.

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