Studies on the photoluminescence and thermoluminescence properties of CaZrO$_3$:xEu$^{3+}$ phosphor for dosimetric applications

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Abstract
Series of CaZrO$_3$:xEu$^{3+}$ (x = 0.01, 0.02, 0.03, 0.04 and 0.05) phosphors have been prepared by low temperature sol–gel auto combustion method. The structure and morphology of the samples were investigated by X-ray diffraction (XRD) and field-emission scanning electron microscope (FE-SEM). The energy-dispersive X-ray spectroscopy (EDXS) was employed to analyze the elemental composition of the phosphor. The XRD patterns indicated that the sample was single phase at 350 °C with a perovskite structure. The optimum temperature for the single-phase and crystalline phosphors of CaZrO$_3$:xEu$^{3+}$ was 700 °C. Study of photoluminescence (PL) at room temperature showed that the phosphors can be excited by light with a wavelength of 391 nm. The results of emission spectrum showed that the red luminescence of CaZrO$_3$:xEu$^{3+}$ due to electric dipole transition of $^5D_0\rightarrow^7F_2$ was dominant at wavelength of 615 nm and weaker transition at wavelength of 590 nm which was due to magnetic dipole transition of $^5D_0\rightarrow^7F_1$. For the thermoluminescence (TL) study the prepared sample irradiated by X-ray lamp, the TL curve was then recorded at fixed heating rate of 2 °C/s. The TL glow curve showed well single peak at a temperature of 165 °C. The effect of Eu$^{3+}$ concentration at fixed X-ray exposure time was studied and maximum TL occurred at x = 0.02. Also the variation of TL intensity with X-ray time (5 to 15 min) showed linear response with dose. The TL glow peak shows more stability and less fading in prepared phosphor which is suitable for TL dosimetry.

Keywords Photoluminescence · Thermoluminescence · CaZrO$_3$:xEu$^{3+}$ · Phosphor · Dosimetric
1 Introduction

The main group of compounds are oxides with $\text{ABO}_3$ formula with perovskite structure. Solar cell, energy harvesting device, solid electrolyte, anode material in solid oxide fuel cell, photocatalysts, sensors, fillers, satellite broadcasting, crystalline host for phosphor materials and multilayer capacitors are some applications of these oxides (Noh and Lee 2015; Kunti et al. 2021; Tang and Zhang 2021; Fukushima et al. 2020; Tripathi et al. 2018). One of the most common perovskite compounds is calcium zirconate ($\text{CaZrO}_3$) with a high melting point ($2600^\circ\text{C}$) and band gap of 5.6 eV which can be used as host phosphor (Stoch et al. 2012; Ianoș and Barvinschi 2010; Gupta et al. 2015).

Rare earth (RE) ions such as Eu, Tm, Ce, Sm, Gd, Er and Pr can be doped in $\text{CaZrO}_3$ host and improve the luminescence properties (Zhang et al. 2008a). A luminescence material commonly emits in the visible region of the electromagnetic spectrum but they can also emit in the ultraviolet (UV) and infrared (IR) regions depending upon the activator ion doped (Gupta et al. 2015). The physical and chemical properties of this material can be greatly enhanced by doping of luminescent center induced by interaction of host and the dopant ion (Taxak et al. 2014).

$\text{CaZrO}_3$:Tm showed blue emission (Zhang et al. 2008b); $\text{CaZrO}_3$:Pr showed green emission (Pinel et al. 2004), $\text{CaZrO}_3$:Ce displayed violet and blue emission (Evangeline et al. 2017); $\text{CaZrO}_3$:Sm, Gd indicated yellow–orange emission (Qingqing et al. 2012); $\text{CaZrO}_3$:Er and $\text{CaZrO}_3$:Er, Ce revealed green photoluminescence (PL) emission (Du et al. 2012).

Europium ion is popular as an activator dopant for many different in organic lattices producing red light emission (Kunti et al. 2021).

The use of thermoluminescence (TL) for dosimetry in medical, personal and environmental fields is now very popular. Among the most important properties that can be considered for an ideal dosimetry are: low sensitivity and fading, high stability, linear dosimetry in a dose range and having the ability to use repeatedly without any changing in the sensitivity of the luminescence properties (Zahedifar and Sadeghi 2013; Salah et al. 2015; Zahedifar et al. 2017).

In recent years, the use of nanomaterials in various fields has grown a lot. As the particle size decreases, the surface to volume ratio of these materials increases. This makes various properties such as optical and dosimetric of nanomaterials to their mass state, and this is because they have a high ability to create trapping with high surface centers. Today, several methods are used to produce powders with nanometer dimensions. These methods are: solid state (Sahu et al. 2017), precipitation (Han et al. 2020), hydrothermal (Meetei and Singh 2014) and sol–gel method (Zhang et al. 2008a; Fan et al. 2020). $\text{CaZrO}_3$:xEu$^{3+}$ was widely synthesized by solid state reaction method (Yajima et al. 1991; Wang et al. 1997; Dubey and Tiwari 2016; Katayyan and Agrawal 2017a; Huang et al. 2011; Xu et al. 2011; Maurya et al. 2018). It includes disadvantages such as; long calcification time, high temperature, large particle size, wide grain size distribution, low surface area, high agglomeration degree, lack of chemical homogeneity and etc. In order to overcome the disadvantages of ceramic method, synthesis of $\text{CaZrO}_3$ powders is a subject of wide interest and new chemical preparation methods have been reported. One of the simple and accessible methods for preparing nano phosphors is sol–gel combustion method, that the type of fuel and the temperature at which combustion occurs affects the PL properties. Zhang et al. (2008) synthesized $\text{CaZrO}_3$:xEu$^{3+}$ red phosphor at 1000 °C by the polymerization complex method based on the Pochini-type reaction. Taxak et al. (2014) synthesized $\text{CaZrO}_3$:xEu$^{3+}$ red phosphor by sol–gel combustion method based on metal nitrates with tartaric acid as
fuel at 700 °C. Tiwari et al. (2015) reported the preparation of CaZrO$_3$:xEu$^{3+}$ phosphor by sol–gel combustion method with urea as fuel at 550 °C. Du et al. (2013) prepared CaZrO$_3$:xEu$^{3+}$ phosphor with Li$^+$ doping by sol–gel combustion method with the reaction temperature at 700 °C based on nitrates and metal acetates with citric acid as fuel.

In this study, CaZrO$_3$:xEu$^{3+}$ phosphors were produced by sol–gel auto combustion method based on metal nitrates using citric acid as fuel at a relatively lower combustion temperature (350 °C) compared to other reported work. The combustion process at a lower temperature is more economical, environmental friendly and finally improve the PL properties. Since all the reported articles have studied the PL properties of CaZrO$_3$:xEu$^{3+}$ phosphor and only one article has reported the TL properties under UV radiation (Tiwari et al. 2014), so after studying the PL properties of CaZrO$_3$:xEu$^{3+}$ phosphors, the present paper reports the TL glow curve and calculation of kinetic parameters of Eu$^{3+}$ doped CaZrO$_3$ phosphor. The TL glow curve of prepared sample with the variation of X-ray dose (5 to 15 min) show linear response with dose.

2 Experimental details

The starting materials are high purity Ca(NO$_3$)$_2$ · 4H$_2$O, ZrN$_2$O$_7$, Eu(NO$_3$)$_3$ · 6H$_2$O and C$_6$H$_8$O$_7$·H$_2$O (citric acid) as fuel. Ca$_{1-x}$ZrO$_3$:xEu$^{3+}$ is synthesized by auto combustion sol–gel method and the chemical equation for the reaction is:

$$(1 - x)\text{Ca(NO}_3\text{)}_2 \cdot 4\text{H}_2\text{O} + x\text{Eu(NO}_3\text{)}_3 \cdot 6\text{H}_2\text{O} + \text{ZrN}_2\text{O}_7 + 1.2\text{C}_6\text{H}_8\text{O}_7 \longrightarrow \text{Ca}_{1-x}\text{ZrO}_3 : x\text{Eu}^{3+}(s) + \text{gaseous products.} \quad (1)$$

According to nominal composition of Ca$_{1-x}$ZrO$_3$:xEu$^{3+}$ (x = 0.01, 0.02, 0.03, 0.04 and 0.05), a stoichiometric amount of metal nitrates were dissolved separately in minimum quantity of deionized water, then citric acid was added in this solution with molar ratio of citric acid to nitrates based on total oxidizing and reducing valencies of oxidizer and fuel (citric acid) according to concept used in propellant chemistry (Taxak et al. 2014). During the heating, pH of the solution was set to 7 (Evangelie and Azeem 2016) using ammonia solution and the solution was placed on a magnetic stirrer for 20 min at 80 °C until a resin solution was obtained, then transferred into a preheated furnace maintained at 350 °C. The material underwent rapid dehydration and foaming followed by decomposition, generating combustible gases. Finally the soft gray powder was obtained. In order to form the final phases and clear the existing carbons, the powder will be placed in the electric furnace at various temperature ranging from 550 to 1000 °C for 3 h to increase the brightness. An X-ray diffractometer (Philips Expert) was employed using a Cu K$\alpha$ (\(\lambda=1.5406\) Å) radiation in the range of 2$\theta$=20$^\circ$–80$^\circ$ as a source for characterizing the samples. Then the microstructure of samples was analyzed using FE-SEM; models-4160. Also, the compositions were examined by EDXS in the SEM. PL spectrum was recorded using a Perkin-Elmer spectrometer model LS55 with photo multiplier tube and Xenon lamp at room temperature. Finally, the TL response was recorded by a ASENWARE company, Xj 10-60N model based on TL reader. A heating rate of 2 °C/s was used for recording glow curves.
3 Results and discussion

The structure and phase purity of the powders can be examined by XRD. The XRD patterns of CaZrO$_3$ powders calcined at various temperatures before and after annealing at different temperatures ranging from 550 to 1000 °C are shown in Fig. 1a. Below 550 °C, the diffract patterns show weak crystalline structure of perovskite, which can be matched to orthorhombic CaZrO$_3$ phase (JCPDS No. 35-0790). By increasing the temperature up to 550 °C, the additional phases gradually diminish. The diffraction peaks considerably intensify at calcination temperatures above 700 °C, indicating improvement of the structure with an annealing process. To study the effect of europium doping on the structural parameters (with the space group of Pbnm) of the perovskite CaZrO$_3$ powder, variable concentration of Eu (0.01–0.05) was added and a same calcination was conducted. The corresponding XRD patterns are shown in Fig. 1b. Since the radius of Eu$^{3+}$ ion (0.095 nm) and Ca$^{2+}$ ion (0.099 nm) are close to each other, europium is expected to substitute calcium, not zirconium with radius of 0.072 nm (Huang et al. 2011). XRD patterns in Fig. 1b show no considerable variation due to doping, indicating the negligible effect in the used Eu range on the lattice parameters.

The size of the Ca$_{0.98}$Eu$_{0.02}$ZrO$_3$ crystallite was calculated using the Debye–Scherrer formula (Tiwari et al. 2015):

$$D = 0.9 \frac{\lambda}{\beta \cos \theta},$$

where $D$ is particle size, $\beta$ is FWHM (full width half maximum), $\lambda$ is the wavelength of X-ray source (0.154 nm) and $\theta$ is the angle of diffraction. The structural information and particle size calculation is present in Table 1, which shows the crystallite size across the most intense peak i.e. (121) direction is 24 nm.

FE-SEM and EDXS pictures are shown in Figs. 2 and 3, respectively. Figure 2a, b reveal agglomerates with 130 nm and 123 nm average size that are constructed from small particles with spherical and polyhedron shapes. The EDXS spectrum of CaZrO$_3$ in Fig. 3a indicates the presence of calcium, zirconium and oxygen with concentration as shown inside. In Fig. 3b, in addition to the elements mentioned, europium is also visible.

The PL excitation spectrum of Ca$_{0.98}$Eu$_{0.02}$ZrO$_3$ calcined at 700 °C is shown in Fig. 4. The spectrum includes two parts; (1) broad band region (200–300 nm) peaking at 268 nm is called charge transfer (CT) state band that can be attributed to CT transition from 2p orbital of O$^{2-}$ ions to an empty 4f orbital of Eu$^{3+}$ ions (O$^{2-}$ → Eu$^{3+}$) (Sahu et al. 2017), (2) several sharp lines in the range of 320 to 500 nm are related to the configurational 4f-4f

| Table 1 | Structural analysis with particle size by XRD technique for Ca$_{0.98}$Eu$_{0.02}$ZrO$_3$ phosphor |
|---|---|---|---|---|
| S. Nos. | hkl | $\theta$ | FWHM | D (nm) |
| 1 | (101) | 22.15 | 0.28 | 28 |
| 2 | (121) | 31.55 | 0.34 | 24 |
| 3 | (202) | 45.25 | 0.33 | 26 |
| 4 | (042) | 55.65 | 0.69 | 13 |
| 5 | (123) | 56.7 | 0.62 | 15 |
| 6 | (004) | 65.85 | 0.48 | 19 |
| 7 | (203) | 74.8 | 0.65 | 17 |
transitions of Eu$^{3+}$ in host lattices. The strongest sharp peak located at 391 nm corresponds to $^7F_0 \rightarrow ^5L_6$ transition of Eu$^{3+}$ ions. The weak peaks can be attributed to transitions of $^7F_0 \rightarrow ^5D_{4,4}$, $^7F_0 \rightarrow ^5G_{J,4}$, $^7F_0 \rightarrow ^5L_{7,7}$, $^7F_0 \rightarrow ^5D_3$ and $^7F_0 \rightarrow ^5D_2$ at wavelengths of 353, 368, 376, 411 and 465 nm, respectively (Navami et al. 2018). The shape and position of the PL spectrum...
do not change by different excitation wavelengths and only the intensity of emission spectrum changes (Xu et al. 2011). The PL emission spectrum of Ca$_{1-x}$ZrO$_3$:xEu$^{3+}$ (x = 0.01, 0.02, 0.03, 0.04 and 0.05) under excitation wavelength of 391 nm are shown in Fig. 5. This spectrum of phosphor consist of peaks due to 4f configuration of Eu$^{3+}$ transitions $^{5}D_0 \rightarrow ^{7}F_J$ (J = 0–4) at wavelengths of 578, 590, 615, 651 and 697 nm, respectively.
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The orange emission at wavelengths of 590 nm belonging to $^5D_0 \rightarrow ^7F_1$ magnetic dipole transition which is insensitive to site symmetry of Eu$^{3+}$ ions, and the transition varies with the crystal field strength. The red emission at wavelengths of 615 nm ascribes to the $^5D_0 \rightarrow ^7F_2$ electric dipole transition which is sensitive to local symmetry of Eu$^{3+}$ and depends on the symmetry of the crystal field (Sahu et al. 2017). PL intensity of Ca$_{1-x}$ZrO$_3$:xEu$^{3+}$ (x = 0.01–0.05) increases with increasing Eu$^{3+}$ concentration, reaching a maximum value at x = 0.02 and thereafter decreases with further increase in impurity concentration. The maximum Eu$^{3+}$ concentration in CaZrO$_3$:xEu$^{3+}$ led to the quenching of luminescence due to energy non-radiative transition induced by the cross relaxation between adjacent Eu$^{3+}$ ions (Du et al. 2012). As the Eu$^{3+}$ concentration increases, no change in the shape and position intensity of PL peaks is observed (Fig. 5).

Figure 6 shows the TL curve for samples CaZrO$_3$:xEu$^{3+}$ (x = 0.01–0.05) at heating rate of 2 °C/s. The sensitivity of TL materials strongly depends on the type and concentration of impurities added to the host material. To find the optimal TL radiation in terms of

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**Fig. 5** PL emission spectra of Ca$_{1-x}$ZrO$_3$:xEu$^{3+}$ (0.01–0.05)

**Fig. 6** TL glow curve of Ca$_{1-x}$ZrO$_3$:xEu$^{3+}$ (0.01–0.05) with the constant of X-ray time
impurity concentration, the X-ray radiation dose is kept constant while the Eu concentra-
tion is changed. According to the Fig. 6 the shape of the curve for all concentration does
not change and the TL curve for Ca$_{0.98}$Eu$_{0.02}$ZrO$_3$ displays a single radiative peak at 164 $^\circ$C
with highest intensity.

Essential properties of good TL dosimeter is the stability and repeatability. To inves-
tigate the fading of Ca$_{1-x}$ZrO$_3$:xEu$^{3+}$, irradiated phosphors before reading kept in a dark
room for different days. Figure 7a shows that the fading is negligible and this sample is sta-
ble. In order to investigate the repeatability, the sample is first annealed at 500 $^\circ$C for one
hour, then irradiated, and finally read. This process was repeated five times. As shown in
Fig. 7b, the sensitivity of the sample has changed by approximately less than 4% compared
to the initial state. During this study, glow curve of the Ca$_{0.98}$Eu$_{0.02}$ZrO$_3$ sample was inves-
tigated for different X-ray irradiation times for 5, 9 and 15 min (Fig. 8). The TL intensity
increases with the X-ray time exposer and has linear response (at the inset).

Given that TL curves are a suitable tool for obtaining information about trapping
parameters and full description of the TL characteristics of a TL material; it is very
necessary to calculate the trapping parameters. The TL glow curves can be analyzed in a variety of methods, among which the peak shape method is the most common method for calculating the various kinetics parameters [i.e. activation energy (E), order of kinetics (b) and frequency factor (s)]. TL parameters of prepared phosphors were calculated by peak shape method with the variation of X-ray exposures. The relationship between the frequency factor, (s) and the activation energy, (E) is given by Eq. (3) (Sahu et al. 2017; Manam and Das 2009).

\[
s = \frac{\beta E}{KT_m^2} \cdot \frac{1}{1 + (b - 1)\Delta_m} \cdot \exp \frac{E}{KT_m},
\]

where K is Boltzmann constant, b is order of kinetics, \(T_m\) is maximum temperature and \(\beta\) is heating rate (\(=2^\circ\text{C/s}\)) in this work. The following kinetic parameters calculated using peak shape method (Katyayan and Agrawal 2020).

\[\sigma = T_2 - T_M, \quad \tau = T_M - T_1, \quad \omega = T_2 - T_1, \quad \mu = \sigma/\omega = (T_2 - T_M)/(T_2 - T_1).\]  

The general expression for activation energy or trap depth (E) that is obeyed for all orders of kinetics proposed by Tiwari et al. (2014).

\[E_\eta = C_\eta \frac{KT_m^2}{\eta} - b_\eta (2KT_m),\]  

where \(\eta\) stands for \(\tau\), \(\sigma\) and \(\omega\), respectively. \(C_\eta\) and \(b_\eta\) are obtained using the expressions given by Eqs. 6 and 7, respectively (Furetta 2003).

\[C_\tau = 1.51 + 3(\mu - 0.42), \quad C_\sigma = 0.976 + 7.3(\mu - 0.42), \quad C_\omega = 2.52 + 4.2(\mu - 0.42),\]  

\[b_\tau = 1.58 + 4.2(\mu - 0.42), \quad b_\sigma = 0, \quad b_\omega = 1.\]  

Chen formula provides a method that identify the kinetics order of one trap according to the shape of the TL band. \(\mu\) is the shape factor and the difference between first and second order TL glow peak. \(\mu = (0.39–0.42)\) for the first order kinetics, \((0.42–0.48)\) for the mixed order kinetics and \((0.49–0.52)\) for the second order kinetics, respectively (Pagonis et al. 2006). The TL emission intensity is recorded maximum for \(\text{Ca}_{0.98}\text{Eu}_{0.02}\text{ZrO}_3\) phosphor.

The TL kinetic parameters for the \(\text{Ca}_{0.98}\text{Eu}_{0.02}\text{ZrO}_3\) phosphor was calculated by the peak shape method (Table 2). The value of \(\mu\) varying from 0.45 to 0.48 show mixed order kinetics. The required energy for escaping one electron from trap center known as activation energy or trap depth (E) calculated by Chen formula presented in Table 3.

The low value of activation energy (from 0.50 to 0.69 eV) confirms the trapping of electrons in shallow trapping sites (Katyayan and Agrawal 2017b).

| X-ray time (min) | \(T_1\) (°C) | \(T_m\) (°C) | \(T_2\) (°C) | \(\tau\) | \(\delta\) | \(\omega\) | \(\mu\) | E (eV) | s |
|-----------------|-------------|-------------|-------------|--------|--------|--------|------|-------|---|
| 5               | 132.2       | 165.6       | 197.1       | 33.4   | 31.5   | 64.9   | 0.48 | 0.8   | \(3.3 \times 10^{10}\) |
| 9               | 133.3       | 163.8       | 191.5       | 30.5   | 27.7   | 58.2   | 0.48 | 0.91  | \(6.0 \times 10^{11}\) |
| 10              | 132         | 164         | 190         | 32     | 26     | 58     | 0.45 | 0.91  | \(6.2 \times 10^{11}\) |
4 Conclusion

In this study, CaZrO$_3$:xEu$^{3+}$ (x = 0.01–0.05) phosphors were synthesized by sol–gel auto combustion method. The phosphors excited by 391 nm and exhibit orange–red emission with dominate peaks at 590 and 615 nm due to the magnetic dipole $^5$D$_{0}$$rightarrow$^7$F$_{1}$ and electric dipole $^5$D$_{0}$$rightarrow$^7$F$_{2}$ transition of Eu$^{3+}$ ions respectively. The optimal doping concentration is determined to be 2% for Eu$^{3+}$ ions doped CaZrO$_3$ host. The best TL glow curve was observed for sample Ca$_{0.98}$Eu$_{0.02}$ZrO$_3$, which has a single peak at maximum temperature of 165 °C and shows a linear response with X-ray radiation exposure time. The TL kinetic parameter for this sample showed the mixed order kinetics with low activation energy.

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Declarations

Conflict of interest The authors have not disclosed any competing interests.

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