

MICROWAVE-SYNTHESIZED $\text{Sr}_2\text{ZnMoO}_6$: Eu_2O_3 PHOSPHORS IN TeO_2 : ZnO : BaO GLASS: STRUCTURAL, OPTICAL, AND LUMINESCENCE PROPERTIES FOR POTENTIAL REDDISH-ORANGE SSL APPLICATIONS

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Abstract

The $\text{Sr}_2\text{ZnMoO}_6$ phosphor was incorporated into TeO_2 : ZnO : BaO glass using the microwave synthesis, with different amount of phosphors (0.00, 2.50, 5.00, 7.50, and 10.00 wt%). The samples were characterized through density, refractive index, XRD, FTIR, absorption spectra, luminescence properties, and lifetime analysis. The crystalline structure was analyzed by using X-ray diffractometer and compared with the standard $\text{Sr}_2\text{ZnMoO}_6$ crystal structure. Absorption spectra analysis showed four peaks associated with the transitions of Eu^{3+} ion, while photoluminescence was observed between 550 and 725 nm when excited at 465 nm. The emission intensity increased as the concentrations of $\text{Sr}_2\text{ZnMoO}_6$ phosphor increased up to 7.50 wt%, after which it decreased. These samples exhibited a high photoluminescence quantum yield of 56.49%, with the high-intensity peak observed at 614 nm, corresponding to the $^5\text{D}_2 \rightarrow ^7\text{F}_0$ (reddish orange) transitions of Eu^{3+} ion. The luminescence lifetime values from the $^5\text{D}_2 \rightarrow ^7\text{F}_0$ state decreased (0.891, 0.886, 0.880, and 0.874 ms) by adding a concentration of phosphors, The results indicate that these samples have the potential to be used in solid-state lighting applications.

Keywords: Phosphors-in-glass; Microwave synthesis; Photoluminescence; Eu^{3+} ion

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Introduction

Solid-state lighting (SSL) has been receiving a lot of interest as an alternative to traditional fluorescent and incandescent bulbs in recent years. Due to their high energy efficiency, small size, fast response, and longer lifespan than traditional bulbs. However, extended use of these bulbs can still lead to issues. The problem arises from the degradation of the resin used to cover the bulb, causing a decrease in light output and alterations in color. This deterioration of the resin is caused by heat from continuous use over a long time. To solve these problems, materials such as transparent ceramics, single crystals, and phosphor-in-glass (PIG) have been developed as replacements (Yie *et al.*, 2017). The complex synthesis process of single crystals and transparent ceramics also poses problems for commercial applications, including the long synthesis time and high production costs. Therefore, phosphor-in-glass synthesis is an attractive option because of its simple synthesis process, short synthesis time, low production costs, and no size limitations to synthesis. To synthesize efficient light emitters, materials typically combine an activator with a stable host. Among potential host materials, $\text{Sr}_2\text{ZnMoO}_6$ phosphors stand out for their desirable optical properties, inexpensive, low-temperature preparation, and compatibility with rare earth element doping (Cao *et al.*, 2016). Rare earth (RE) doped phosphors are indeed a promising class of materials for solid-state lighting applications. Their unique properties, stemming from the trivalent state of their 4f electrons, make them attractive for solid-state lighting due to their desirable optical characteristics, chemical stability, and excellent electronic properties. One of the rare earth elements that has received a lot of attention is europium (Eu^{3+} ion). The unique luminescence properties of Eu^{3+} ion from electron transitions within their f-electron orbitals (4F6 configuration). These transitions occur when the ion is excited (5D0 level) and then relaxes to lower energy levels (7Fj, where $j = 0, 1, 2, 3, 4$). The most intense emission, appearing around ~615 nm, corresponds to the 5D0 to 7F2 transition (Tariwong *et al.*, 2020), which generates bright red light and is suitable for solid-state lighting applications. Glass is a material synthesized from inorganic compounds at high temperatures. Glass has high transparency, resistance to temperature and humidity. There are several types of glass systems available such as silicate, bismuthate, phosphate, germanate, borate, and tellurite. Each type has distinct characteristics based on the ingredients that are used in their composition. One particularly interesting glass for

solid-state lighting (SSL) applications is tellurite glass. This glass has low phonon energy, reducing the chances of non-radiative processes in rare-earth (RE) ions. As a result, tellurite glass offers a high refractive index, as well as thermal and chemical stability, efficient thermal conductivity, and a low melting point, making it highly interesting for SSL applications (Abdullahi *et al.*, 2019). In the synthesis of TeO_2 glasses is often a problem in forming glass because this type of glass is sensitive. The addition of other elements improves this type of glass (Kabalci *et al.*, 2011a). It has been reported that adding alkaline earth oxides to glass improves vitrification (glass formation) and the structural integrity of the glass (Wongwan *et al.*, 2024). Moreover, addition of BaO to glass host matrices produces different optoelectronic effects and also has high thermal neutron capture (Khan *et al.*, 2019). Another element that is popularly used in tellurite glass hosts is ZnO because the addition of ZnO improves the glass structure, forms a network, and has high-intensity optical and spectral properties (Boonin *et al.*, 2020). In addition, it has been reported that the presence of ZnO in tellurite glass increases the transparency of the glass (Kabalci *et al.*, 2011b). The current study employs microwave sintering to create phosphor within the glass, a technique valued for its rapid heat generation and shortened synthesis duration compared to traditional methods. Given the low boiling point of the glass host, prolonged firing risks compound evaporation, rendering microwave heating method is more suitable. Furthermore, the researchers analyzed the physical, structural, optical, and photoluminescence characteristics of samples produced through microwave melting at varying ratios to identify optimal proportions for potential use in solid-state lighting technology.

Experiment

The $\text{Sr}_{1.9}\text{ZnMoO}_6\text{:}_{0.1}\text{Eu}_2\text{O}_3$ phosphors in 50 TeO_2 :40 ZnO :10 BaO glass with different concentrations of phosphors (0.00, 2.50, 5.00, 7.50 and 10 wt%) were synthesized by microwave technique. There are two parts to the sample preparation process. The first part is the synthesis of phosphors, and the second part is mixing phosphors with glass system and synthesizing by using microwave technique. Initially, the stoichiometric amount of SrCO_3 , ZnO , MoO_3 , Eu_2O_3 materials weighed and mixed them with a ball mill at 300 rpm for 4 h. After that, the mixture calcined at 500°C for

4 h and sintered at 900°C for 4 h. The phase formation of the as-synthesized phosphors was confirmed by X-Ray diffraction (XRD) analysis. Furthermore, the as-synthesized phosphors mixed with 50TeO₂:40ZnO:10BaO glass matrix. The mixed samples were sintered using a microwave technique at 1000 watt for 14 min and poured into a graphite mold, after which they were annealed at 500°C for 3 h and cooled down to room temperature as shown in Figure 1.

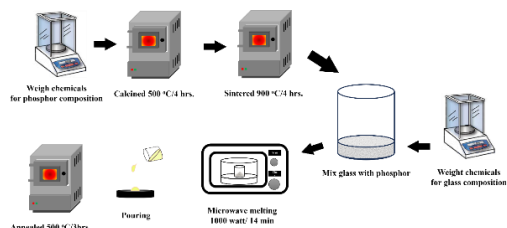


Figure 1. Schematic diagram of the Sr_{1.9}ZnMoO₆:0.1Eu₂O₃ phosphors in 50TeO₂:40ZnO:10BaO glass materials by microwave sintering.

Results and Discussion

Physical properties

Figure 2 shows the Sr_{1.9}ZnMoO₆:0.1 Eu₂O₃ phosphors in 50TeO₂:40ZnO:10BaO glass was cut and polished to 1 × 1.5 × 0.3 cm³. Transparency of the samples decreases due to the concentrations of phosphors increases. By using Archimedes' principle, the density of samples measured using Equation (1):

$$\rho_{samples} = \frac{W_A}{W_A - W_W} \times \rho_w \quad (1)$$

Where W_A is the weight of the glass in air, W_W is the weight of the glass in distilled water, and ρ_w is the density of distilled water (~1 g/cm³). The density of the sample was measured at least three times, and the average was calculated. It was found that the average density of the samples increased after the incorporation of phosphors increased as shown in Figure 3. The high molecular weight of phosphors mixed into the glass matrix is the reason for this increase in density. The molar volume of the samples in this study was analyzed using Equation (2):

$$V_m = \frac{M_t}{\rho} \quad (2)$$

Where V_m is the molar volume and M_t is the molecular weight of the samples. The molecular weight of the samples in this work is the sum of the proportional molecular weights of the glass and phosphors. Figure 3 shows the molar volume of the sample decreased significantly at the beginning and after that, the molar volume was slightly changed, which corresponded with the trend of density. Due to molar volume is the ratio between the molecular weight and the density. As the density increases rapidly during the initial phosphor increase, the molar volume decreases according to the equation.

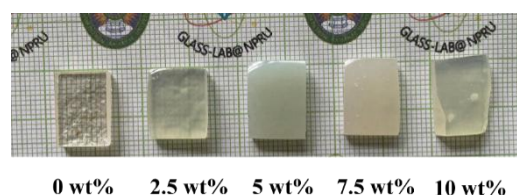


Figure 2. The Sr_{1.9}ZnMoO₆:0.1Eu₂O₃ phosphors in 50TeO₂:40ZnO:10BaO glass with different weights of phosphor.

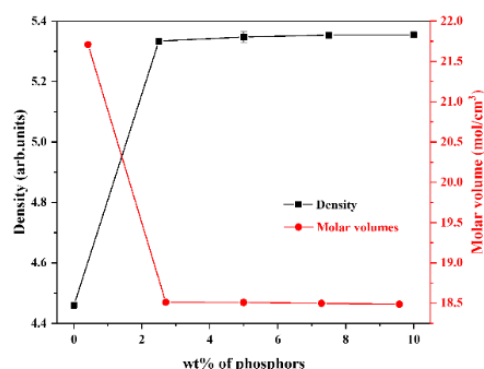


Figure 3. The density and molar volume of as-prepared samples

X-Ray diffraction analysis analysis (XRD)

Figure 4 shows the XRD pattern of the prepared sample in the range of 10 - 80 degrees at a voltage 40 kv and electric-current 30 mA by Shimadzu X-Rays diffractometer- 6100. The XRD pattern of the prepared sample revealed clearly the presence of crystalline peaks due to the concentrations of phosphors increased. the peak positions of the phosphors (10.00 wt%) incorporated into the glass sample was compared standard Sr₂ZnMoO₆ (PDF# 01-079-7469) card as shown in Figure 5. From the results, it was confirmed that the addition of phosphors maintained its crystalline structure in the glass matrix.

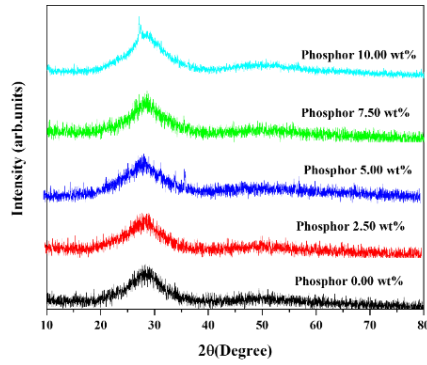


Figure 4. The XRD pattern of $\text{Sr}_{1.9}\text{ZnMoO}_6:1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass

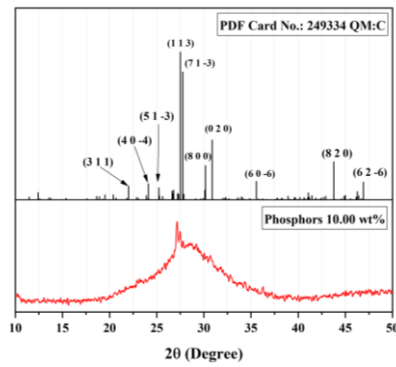


Figure 5. The XRD pattern of $\text{Sr}_{1.9}\text{ZnMoO}_6:1\text{Eu}_2\text{O}_3$ phosphors in glass compared with $\text{Sr}_2\text{ZnMoO}_6$ standard

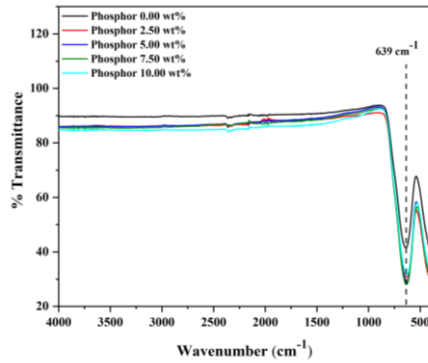


Figure 6. The FTIR of $\text{Sr}_{1.9}\text{ZnMoO}_6:1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass

Fourier Transform Infrared (FTIR)

Fourier Transform Infrared (FTIR) was carried out in the wave number range from 400 to 4000 cm^{-1} . The vibrational modes found at 639 cm^{-1} which corresponds to the stretching vibration of the $\text{TeO}_3/\text{TeO}_4$ units bending (Selvi *et al.*, 2017) as shown in Figure 6 and Table 1. The vibration of the OH group oscillations can be confirmed by the peak at 2000 cm^{-1} (Selvi *et al.*, 2017). The difference in

the FTIR spectrum explains the formation of the network bond chains or average network chain length (Rani *et al.*, 2009). This work found that when phosphor was mixed to the glass matrix, the transmittance at the vibrational peak of Te-O bond decreased. The presence of phosphor may have led to enhanced bonding in the sample.

Table 1. Assignments of vibrational bonds of phosphors in the glass system

Wavenumber (cm^{-1})	Assignment	Reference
639	Stretching vibration of the $\text{TeO}_3/\text{TeO}_4$	(Selvi <i>et al.</i> , 2017)
> 2000	Vibration of the OH group	

Absorption spectra

The absorption spectra of the $\text{Sr}_{1.9}\text{ZnMoO}_6:1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass was recorded at room temperature in the wavelength range of 400 - 2300 nm as shown in Figure 7. Absorption spectra of all samples were found at 465, 534, 2088 and 2203 nm, which corresponds to $^7\text{F}_0 \rightarrow ^5\text{D}_2$, $^7\text{F}_0 \rightarrow ^5\text{D}_1$, $^7\text{F}_0 \rightarrow ^7\text{F}_6$ and $^7\text{F}_1 \rightarrow ^7\text{F}_6$ transitions, respectively (Pawar *et al.*, 2018). Moreover, the absorption strength of sample increases with the concentrations of phosphors in the glass matrix due to the shielding effect of outermost electrons on 4f electrons of RE ions (Rani *et al.*, 2020). The absorption spectra has a relatively low intensity, some noise will be observed. In addition, the peak observed at 830 nm is caused by the detector switching from Visible to NIR.

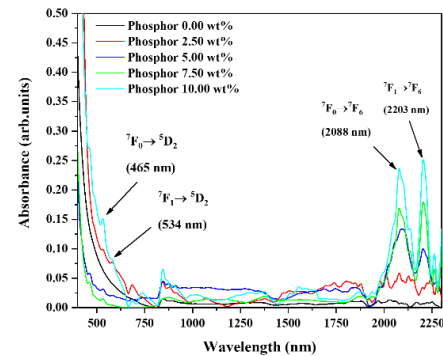


Figure 7. The absorption spectra of $\text{Sr}_{1.9}\text{ZnMoO}_6:0.1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass.

Photoluminescence spectra and lifetime

Excitation spectra were employed in order to analyze the energy levels of Eu^{3+} ions and to determine the suitable wavelength for PL spectra of the samples. The excitation spectra of the samples

were recorded with emission wavelength at 614 nm as shown in Figure 8. The spectra consist of seven excitation peaks with positions at 363, 382, 394, 414, 465, 526 and 533 nm. The peaks were assigned to the transitions from the ground state 7F_0 to the excited state 5D_4 , 5L_7 , 5L_6 , 5D_3 , 5D_2 , 5D_1 and ${}^7F_1 \rightarrow {}^5D_1$ transitions, respectively. The peak at 465 nm, which corresponds to the ${}^7F_0 \rightarrow {}^5D_2$ transition, has the highest intensity among all the excitation peaks. This peak is chosen for the measurement of emission spectra in the prepared samples. Figure 9 shows the emission spectra recorded obtained from excitation at 465 nm, revealing four peaks at wavelength 590, 614, 653, and 702 nm which corresponding to 7F_1 , 7F_2 , 7F_3 , and 7F_5 transitions respectively. The emission intensity of the samples that were prepared rises as the concentration of phosphors increases in weight, reaching its peak at 7.50 wt%. Following this, the PL intensity decreases. (Wang *et al.*, 2014). Figure 10 shows the energy levels diagram of the Eu^{3+} ion and the luminescence mechanism. Luminescence occurs when Eu^{3+} ion absorbs the excitation energy (in this

work is excited at 465 nm), and the ions transfer from the ground state (usually 7F_0) to a higher state (5D_2). After excitation, the ion undergoes non-radiative relaxation, where it loses some energy through vibrations and drops to a lower excited state (5D_0), this process does not emit light. Next, the ion returns to the ground state again. During this transition, the energy difference is released as light, producing luminescence. The specific transitions determine the color and intensity of the emitted light. Figure 11 shows the PL lifetime of the $\text{Sr}_{1.9}\text{ZnMoO}_6: 0.1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass for the most intense emission peak at 614 nm (${}^5D_0 \rightarrow {}^7F_2$) of Eu^{3+} ions under excited at 465 nm. The average lifetime values are obtained for various concentrations (2.50, 5.00, 7.50 and 10.00 wt%) of phosphors in glass systems are 0.891, 0.886, 0.880 and 0.874 ms, respectively. The luminescence lifetime values decreases with increasing concentraions of phosphors which results in an increase in the energy transfer process via the relaxation. (Kiwsakunkran *et al.*, 2022).

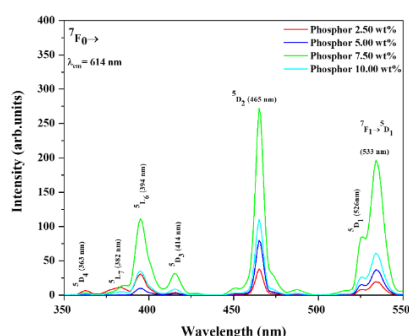


Figure 8. The excitation spectra of $\text{Sr}_{1.9}\text{ZnMoO}_6: 0.1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass

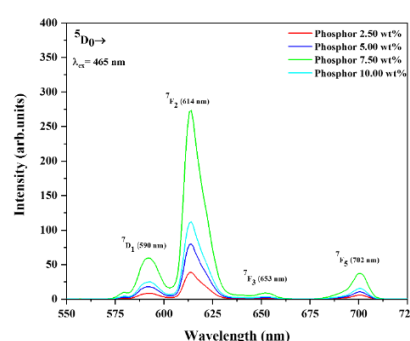


Figure 9. The emission spectra of $\text{Sr}_{1.9}\text{ZnMoO}_6: 0.1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass

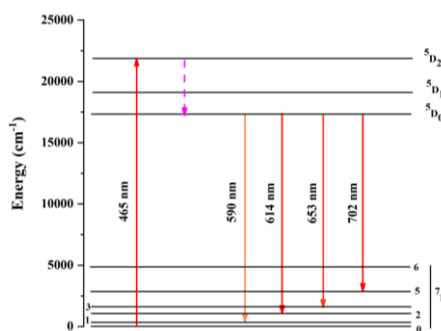


Figure 10. Energy level diagram representing the luminescence mechanism

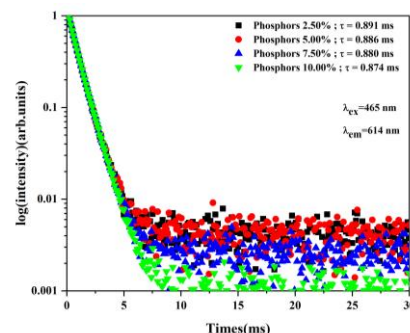


Figure 11. The luminescence lifetime of $\text{Sr}_{1.9}\text{ZnMoO}_6: 0.1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass

Quantum yield and CIE 1931

Table 2 shows the results of the quantum yield measurement of $\text{Sr}_{1.9}\text{ZnMoO}_6: 0.1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass at different concentrations of phosphors. The quantum yield is defined as the ratio of the number of emitted photons to the number of absorbed photons (Kindrat and Padlyak, 2018). The samples external quantum yield ranges from 18 to 56.49%, and it rises as the phosphor concentrations increase, but decreases once it reaches the maximum concentration of 7.50 wt%. The comparison is made between the quantum yield of the sample that has been synthesized and the quantum yield of works that were studied before. Figure 12 shows a CIE 1931 diagram of $\text{Sr}_{1.9}\text{ZnMoO}_6: 0.1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass at a concentration of 7.50 wt%. The color coordinates ($x = 0.656$, $y = 0.343$) of the sample which is located in the reddish-orange region. The results suggest that $\text{Sr}_{1.9}\text{ZnMoO}_6: 0.1\text{Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2:40\text{ZnO}:10\text{BaO}$ glass system suitable for use in solid-state lighting.

Table 2. External quantum yield (QY) of as-synthesized samples and some other Eu^{3+} -doped compounds

Compositions	Quantum yield (%)	References
Phosphor 2.50 wt%	18.07	This work
Phosphor 5.00 wt%	41.35	This work
Phosphor 7.50 wt%	56.49	This work
Phosphor 10.00 wt%	45.12	This work
$\text{Gd}_{0.93}\text{VO}_4:0.07\text{Eu}^{3+}$ phosphor	64.00	(Tang <i>et al.</i> , 2012)
$\text{LBB}:0.05\text{Ce}^{3+}, 0.05\text{Eu}^{2+}$	43.70	(Li <i>et al.</i> , 2017)
$\text{Li}_2\text{B}_4\text{O}_7 (\text{Li}_2\text{O B}_2\text{O}_3):\text{Eu}$	48	(Kindrat and Padlyak, 2018)
(0.5 mol. %) glass		
$\text{BaF}_2 \text{SrF}_2 \text{AlF}_3 \text{YF}_3$	45	(Kindrat and Padlyak, 2018)
$\text{Al}(\text{PO}_3)_3$ glass		

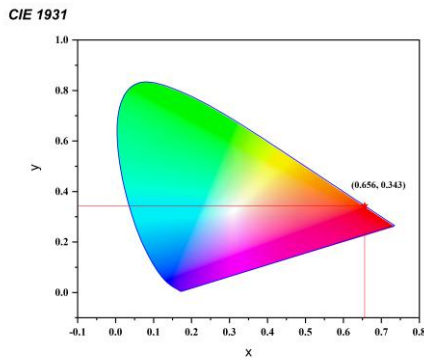


Figure 12. CIE 1931 chromaticity color coordinate diagram of phosphors in glass

X-ray induced luminescence

The $\text{Sr}_2\text{ZnMoO}_6:\text{Eu}_2\text{O}_3$ phosphors were incorporated into glasses containing $\text{TeO}_2: \text{ZnO}: \text{BaO}$ using microwave synthesis. These glasses were then radiated by X-rays with 50 kV and 30 mA to observe the PL spectra as depicted in Figure 13. The intensity of the peak becomes stronger as the concentration of phosphors increases in the range of 500-800 nm. The most intense samples were cut and polished to a dimension of $1 \times 1.5 \times 0.3 \text{ cm}^3$ for comparison with BGO crystals. The method to calculate the integral efficiency of the samples in this work is to calculate the area under the curve of the samples and compare it with the BGO crystals as shown on Figure 14. The integral scintillation efficiency of phosphors (7.50 wt%) in the glass system was 11.26%. The $\text{Sr}_2\text{ZnMoO}_6:\text{Eu}_2\text{O}_3$ phosphors incorporated in $\text{TeO}_2: \text{ZnO}: \text{BaO}$ glass has the potential to be used in X-ray detection.

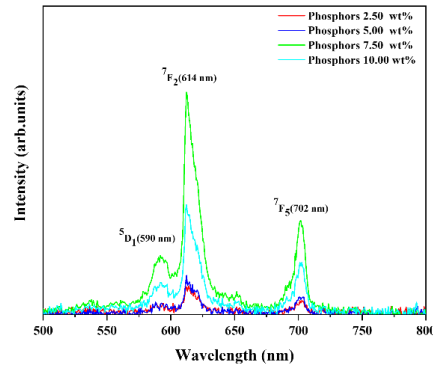


Figure 13. The X-ray-induced luminescence of the $\text{Sr}_2\text{ZnMoO}_6:\text{Eu}_2\text{O}_3$ phosphors-in $\text{TeO}_2: \text{ZnO}: \text{BaO}$ glasses at different weights of phosphors

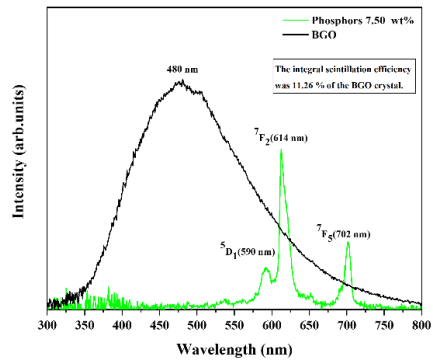


Figure 14. The X-rays induced optical luminescence spectra of phosphor (7.50 wt%) sample compared with BGO scintillator

Conclusion

The $\text{Sr}_2\text{ZnMoO}_6\text{:Eu}_2\text{O}_3$ phosphor-in $\text{TeO}_2\text{:ZnO:BaO}$ glass was prepared by microwave synthesis technique at various concentrations of phosphors (0.00, 2.50, 5.00, 7.50 and 10 wt%). The XRD pattern of the prepared sample revealed clearly the presence of crystalline peaks as the concentrations of the phosphor increased and the position of the peak was consistent with the standard (PDF# 01-079-7469) card of $\text{Sr}_2\text{ZnMoO}_6$. The Fourier transform infrared analysis revealed that a vibration at 639 cm^{-1} corresponds to the stretching vibration of the $\text{TeO}_3/\text{TeO}_4$ units bending and vibration of the OH group occurred after the wavenumber 2000 cm^{-1} . Absorption spectra of all samples were found at 464, 534, 2088, and 2203 nm, which corresponds to $^7\text{F}_0 \rightarrow ^5\text{D}_2$, $^7\text{F}_0 \rightarrow ^5\text{D}_1$, $^7\text{F}_0 \rightarrow ^7\text{F}_6$ and $^7\text{F}_1 \rightarrow ^7\text{F}_6$ respectively. The absorption strength of sample increases with the concentrations of phosphors in the glass matrix. The emission spectra show four peaks at wavelength 590, 614, 653, and 702 nm which corresponds to $^7\text{F}_1$, $^7\text{F}_2$, $^7\text{F}_3$, and $^7\text{F}_5$ transitions, respectively. The emission intensity of prepared samples under excited at 465 nm increases with increase of phosphors concentrations of 7.5 wt%. But decreases after reaching a concentration of 7.5 wt%. The average lifetime measured for the prepared samples are 0.891, 0.886, 0.880 and 0.874 ms, respectively. The external quantum yield of samples ranges from 18 - 56.49 % and it increases with increased phosphors concentrations. However, it decreases after reaching 7.5 wt% of phosphor sample. When compared with other works, this sample was found to have higher efficiency than glass but still lower than pure phosphor which is suitable for solid-state lighting applications. According to the results, $\text{Sr}_{1.9}\text{ZnMoO}_6\text{:0.1Eu}_2\text{O}_3$ phosphors in $50\text{TeO}_2\text{:40ZnO:10BaO}$ glass represent a high quality with a sufficient level of effectiveness for use in solid-state lighting applications. Furthermore, the samples were subjected to X-ray emissivity analysis and compared with commercial BGO crystals to study their possible application as a scintillator material. The integral scintillation efficiency of the sample is determined to be 11.26% when compared with BGO crystal. The phosphor (7.50 wt%) incorporated into the glass sample has the potential to be used in the development of a scintillator material.

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