Optical characteristics of sol-gel derived $M_3SiO_5:Eu^{3+} \ (M = Sr, Ca and Mg)$ nanophosphors for display device technology

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Optical characteristics of sol-gel derived M₃SiO₅:Eu³⁺ (M = Sr, Ca and Mg) nanophosphors for display device technology

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Abstract: A series of trivalent europium-doped M₃SiO₅ (M = Sr, Ca and Mg) phosphors were synthesized using sol–gel process at 950°C. Samples were further reheated at high temperature to study the effect of reheating on crystal structure and optical characteristics. X-ray diffraction measurement of these materials was carried out to know the crystal structure. Diffraction pattern showed monoclinic structure having space group Cm for Ca₃SiO₅ materials. However, tetragonal phase with space group P4/ncc was observed for Sr₃SiO₅ materials. Mg₃SiO₅ material show mixed diffraction peaks at 950 and 1,150°C. Transmission electron microscopic analysis was used to estimate the particle size of silicates. Photoluminescence emission spectra were recorded to check the luminescence properties of prepared materials. These phosphors exhibited a strong orange-red light under excitation at 395 nm. The prepared phosphors exhibited most intense peak in 610–620 nm region due to the 5D₀ → 7F₂ transition of europium (III) ion available in lattice. To overcome the deficiency of red silicates, M₃SiO₅ materials were explored and they might be integrated with ultraviolet LEDs to generate light which may be suitable for display applications.

Subjects: Materials Chemistry; Materials Science; Optoelectronics; Chemical Physics

Keywords: M₃SiO₅; europium; sol-gel; luminescence; crystal structure; study of matter at extreme conditions

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PUBLIC INTEREST STATEMENT
Research and development of nanophosphor is a part of rapidly growing nanoscience and nanotechnology. Silicate-based phosphors have attracted significant interest owing to their excellent luminescence characteristics, high chemical and physical stability, and low synthetic cost. Doping of rare earth into the host matrix demonstrate an approach to develop highly efficient and stable nanophosphors for solid-state lighting and display technology. Specially, europium(III) is an effective dopant that emits strong red light due to ⁵D₀ → ⁷F₂ transitions generated under external energy excitation. Europium(III) doped phosphors have wide applicability in the field of color television display, flat panel display, plasma display panels, device indicators, lasers, automobile headlights, etc. and hence they are emerging as an important class of optical materials.
1. Introduction

Most of the commercial available phosphor materials belong to certain host lattices like oxides, silicates, aluminates, sulfides, oxy-sulfides, nitrides etc. Currently, silicate matrices have attracted a lot of interest to develop efficient luminescent materials as they exhibit excellent photoluminescence properties, good chemical stability, and relative ease of preparation than others (Singh et al., 2014). Silica-based materials can be explored at general synthetic conditions and their composition, shape, and size can be easily controlled to improve the photoluminescence performance of the phosphor materials (Kang et al., 2011).

Nowadays, lighting and display devices are based mainly on white light-emitting diodes (WLEDs) as they are used in a number of applications like device indicators, general illumination, digital cameras, cell phones, LCDs, computer monitors, automobile headlights, etc. (Kang et al., 2005; Qiao, Zhang, Ye, Chen, & Guo, 2009). These applications are due to the small size, long service life, high efficiency, low voltage, light weight, fast response time, good stability, robustness, adjustable color and energy-saving characteristics of WLEDs (Hsu, Sheng, & Tsai, 2009; Jang et al., 2008). Silicate-based materials having composition BaSc₂Si₃O₁₀:Eu²⁺ (blue) and Ca₂MgSi₂O₇:Eu³⁺ (orange-red) are significant in the field of WLEDs (Soju, Bisen, & Brahme, 2015; Wang et al., 2015). Ultraviolet LED/phosphor combinatorial chemistry explored different phosphor materials to accomplish the purpose of white light generation (Kim, Jeon, Choi, & Park, 2005). Phosphors including Ba₃MgSi₂O₈:Eu²⁺ and Mg₃Y₄Si₃O₁₃:Eu³⁺ give blue and intense red color when excited with ultraviolet light source (Ci et al., 2015; Yonezaki & Takei, 2016). Recently, some silicate (Sr₂SiO₄:Eu⁺, Sr₃SiO₅:Eu⁺ and Sr₃SiO₅:Eu⁺, Ba⁺) phosphor materials are integrated with InGaN blue LED chip into a single package to generate white light (Park, Choi, Yeon, Lee, & Kim, 2006). NUV LED chip-excited tricolor luminescent materials may also be used to produce white light which have high efficiency, good coloration, and low thermal quenching (Li, Wang, Yang, Guo, & Li, 2009; Zhang, Xu, Qiu, & Yu, 2013). Therefore, it is quite logical to develop efficient red luminescent materials having such characteristics. Near ultraviolet (NUV) LED excited Ba₉Y₂Si₆O₂₄:Ce³⁺ phosphor material has been developed to emit blue-green light (Brgoch et al., 2013). Several silicates of Sr, Ca, Mg, and Ba absorb ultraviolet radiations (Park, Choi, Kim, & Kim, 2005) and generate red light when activated with europium (III) ion. Previously, silicate-based materials have been prepared using solid-state method (Kang et al., 2011; Sun, Zhang, Zhang, Luo, & Wang, 2008). But, materials synthesized using solid-state method require long reaction time and high temperature processing conditions, which correspondingly enhances the particle size of materials (Bisen & Sharma, 2016). Repeated milling and washing with chemicals tends to degrade the luminescence property of materials and gives deformed shaped crystals. Sol–gel is an efficient process for the synthesis of silicate phosphors due to homogenous mixing of initial precursors, low reaction temperature, and more homogenous products formation (Singh, Sheoran, Tanwar, & Bhagwan, 2016).

In this paper, we report the optical characterization of silicate materials having composition Mₓ₃Eu₃SiO₅ (where M = Sr, Ca and Mg and x = 0.03 mol) prepared using sol–gel method (Singh & Sheoran, 2016). Different techniques (PL, XRD, and TEM) were used to study the characteristics of these luminescent materials. Effect of temperature on luminescence properties of these materials has been studied using photoluminescence analysis. Crystal structure of prepared luminescent materials was analyzed using X-ray diffraction study and transmission electron microscopic analysis estimated the particle size of these materials.

2. Experimental details

2.1. Synthesis of nanophosphors

Eu⁺ doped M₃SiO₅ (M = Sr, Ca and Mg) luminescent materials were synthesized using sol–gel technique at 950°C. The chemicals used were mainly Eu(NO₃)₃·6H₂O, Sr(NO₃)₂, Mg(NO₃)₂·6H₂O, Ca(NO₃)₂·4H₂O, and silica powder. The reagents used were of analytical grade (AR) and purchased from chemical drug house (CDH) and HiMedia. Initially, silica gel was prepared by dissolving a mole of silica powder in concentrated nitric acid solution in a round-bottom flask (RBF). Then, allowed the
contents of RBF to stir for the formation of silica gel which occurred nearly in 10 min. On the other behalf, a stochiometric amount of europium nitrate and corresponding metal nitrates of Sr, Ca, and Mg were mixed homogenously in a silica crucible using distilled water. Then, nitrate mixture of silica crucible was slowly added into the flask containing gel and this mixed solution was again stirred for nearly 30 min. Stirring was done to acquire uniform mixing of precursors. Precursors of the flask were transferred again into the silica crucible which was further warmed on a heating plate for semi-solid paste formation. This semi-solid paste was now put in the furnace at 950°C (as prepared) for 1 h to produce white powders of silicate materials. The resulting white powders were then reheated at a temperature of 1,050 and 1,150°C to check the effect of reheating on the crystal structure and photoluminescence properties of materials. Photographs of as-prepared europium doped silicate materials were taken in the presence and absence of ultraviolet light and shown in the Figure 1. The synthetic procedure used for material synthesis was also explained with the help of flow chart as given in Figure 2. Probable chemical reaction between reactants is shown below that gives rise to the final products.

\[
\begin{align*}
3\text{Sr(NO}_3\text{)}_2(s) + \text{SiO}_2 & \rightarrow \text{Sr}_3\text{SiO}_5 + 6\text{NO}_2 + 3/2\text{O}_2 \\
3\text{Ca(NO}_3\text{)}_2 \cdot 4\text{H}_2\text{O}(s) + \text{SiO}_2 & \rightarrow \text{Ca}_3\text{SiO}_5 + 6\text{NO}_2 + 4\text{H}_2\text{O} + 3/2\text{O}_2 \\
3\text{Mg(NO}_3\text{)}_2 \cdot 6\text{H}_2\text{O}(s) + \text{SiO}_2 & \rightarrow \text{Mg}_3\text{SiO}_5 + 6\text{NO}_2 + 6\text{H}_2\text{O} + 3/2\text{O}_2
\end{align*}
\]

2.2. Instrumentation
Crystal structure of phosphor materials was examined from X-ray diffraction pattern recorded using Rigaku Mini Flex 600 diffractometer. CuKα radiation was used for recording the spectrum having a wavelength of 1.540 Å. Diffraction pattern was recorded from 10 to 70° with a step interval of 0.02° having a scanning speed of 4°/min. Photoluminescence properties were measured at room temperature from 400 to 700 nm using Horiba Jobin YVON Fluorolog Model FL-3–11 Spectrophotometer equipped with Xe-lamp as the source of excitation. Transmission electron micrographs were taken with the help of Hitachi F-7,500 to estimate the particle size of these powdered materials.

Figure 1. Photographs showing powders of europium (III) doped (a) Sr$_3$SiO$_5$, (b) Ca$_3$SiO$_5$, and (c) Mg$_3$SiO$_5$ silicates in the presence and absence of ultraviolet light.
3. Results and discussion

3.1. Optical properties
Photoluminescence emission spectra of trivalent europium-doped $M_xSiO_5$ ($M = Sr, Ca, Mg$ and $x = 0.03$ mol) phosphor materials were recorded with the excitation of 395 nm. Luminescence in phosphor materials is generated due to $4f \rightarrow 4f$ electronic transition of europium (III) ion. Optical properties of phosphor materials get influenced by concentration of dopant ion and temperature employed for reheating of materials. Luminescence intensity of Sr$_3$SiO$_5$ materials enhances with europium concentration up to a limit (3 mol %) but, after that, it starts decreasing due to concentration quenching effect as seen in Figures 3 and 4. This effect originated due to increase in nonradiative energy transfer between Eu$^{3+}$ ions which correspondingly decreases the luminescence intensity as the concentration of the Eu$^{3+}$ ion further increased. Although size and ionic charge issues exists wherever a bivalent metal ion is substituted by trivalent europium ion but, each time variation of metal ion (Sr$^{2+}$, Ca$^{2+}$ and Mg$^{2+}$ having ionic size 1.12 Å, 0.99 Å and 0.72 Å, respectively) in the host lattice, gives rise to a distinct emission spectra as seen in Figure 5 having several peaks originated due to different transitions of Eu$^{3+}$ (0.95 Å) ion available in the lattice. The origin of these transitions depends upon the location of activator ion in lattice and type of transition is determined by selection rules. These transitions were illustrated by drawing energy level diagram of europium (III) ion as shown in Figure 6. These phosphor materials exhibited sharp spectral peaks in 400–700 nm region due to $^3D_0 \rightarrow ^7F_J$ ($J = 1–3$) transitions of Eu$^{3+}$ ion. $^3D_0 \rightarrow ^7F_2$ is the most intense and electric dipole allowed transition located at 616 nm in Sr$_3$SiO$_5$ (Li et al., 2009) and Ca$_3$SiO$_5$ phosphors. In Mg$_3$SiO$_5$ material, this transition is centered at 614 nm which is dominant over $^3D_0 \rightarrow ^7F_1$ (magnetic dipole transition). Peak positions and corresponding color of these silicate phosphors are reported in Table 1. Further reheated samples at 1,050 and 1,150°C were used to study the effect of temperature on luminescence intensity of these silicates materials. Remarkable enhancement in luminescence intensity was observed for these silicates with reheating temperature as clear from Figure 5.
emission spectrum was also used to compare the luminescence intensity between the prepared series of materials as in Figure 7. From the luminescence results, it is clear that highest photoluminescence intensity is observed in case of red emitting Mg$_3$SiO$_5$ material. Emission characteristics of the materials are determined in terms of color coordinate ($x$ and $y$) values. In phosphor materials, chromaticity coordinates play an important role in the white light-emitting diode applications. Color coordinates of the M$_x$SiO$_5$:Eu$^{3+}$ (M = Sr, Ca, Mg and $x = 0.03$ mol) phosphors are calculated using chromaticity calculator and reported in Table 2. Values corresponding to these color coordinates are represented in CIE (Commission Internationale de l’Eclairage) triangle that indicates that these materials are emitting light in the orange-red region as shown in Figure 8.
Figure 5. Photoluminescence emission spectra of europium (III) doped (a) Sr$_3$SiO$_5$, (b) Ca$_3$SiO$_5$, and (c) Mg$_3$SiO$_5$ materials at different temperatures.
Figure 6. Energy level diagram of europium (III) ion showing different transitions in prepared materials.

![Energy level diagram](image)

Table 1. Representing different transitions of europium (III) ion and their corresponding color in prepared lattices

| Composition     | Peak position of different transitions and corresponding color |
|-----------------|---------------------------------------------------------------|
| Sr$_3$SiO$_5$:Eu$^{3+}$ | $^5$D$_0$$\rightarrow$$^7$F$_1$ | 589 | 616 | 648, 661 | Orange-red |
| Ca$_3$SiO$_5$:Eu$^{3+}$ | $^5$D$_0$$\rightarrow$$^7$F$_2$ | 590 | 616 | 660 | Orange-red |
| Mg$_3$SiO$_5$:Eu$^{3+}$ | $^5$D$_0$$\rightarrow$$^7$F$_3$ | 590 | 614 | 652 | Red |

Figure 7. Relative emission spectrum of europium (III) doped silicate materials at 950°C.

![Relative emission spectrum](image)

Table 2. Showing color coordinates of the Eu$^{3+}$ doped silicate materials

| Temperature (°C) | Sr$_3$SiO$_5$:Eu$^{3+}$ | Ca$_3$SiO$_5$:Eu$^{3+}$ | Mg$_3$SiO$_5$:Eu$^{3+}$ |
|------------------|-------------------------|-------------------------|-------------------------|
|                  | x           | y           | x           | y           | x           | y           |
| 950              | 0.40        | 0.32        | 0.42        | 0.33        | 0.46        | 0.34        |
| 1,050            | 0.44        | 0.33        | 0.45        | 0.34        | 0.48        | 0.35        |
| 1,150            | 0.46        | 0.34        | 0.49        | 0.35        | 0.54        | 0.36        |
3.2. Structure determination using X-ray diffraction analysis

X-ray diffraction pattern of the silicate materials prepared using sol–gel technique is presented in the Figure 9. Sr$_3$SiO$_5$ material synthesized at 950°C has tetragonal form with a space group of $P4/ncc$. Diffraction pattern of this sample is closely matched with JCPDS No. 26-0984 (Li et al., 2009; Zhang et al., 2013). Diffraction profile of this material at 1,150°C also exhibits similar polymorphic form with significant enhancement in peak intensity. Dopant ion does not influence the crystal structure of prepared materials. Ca$_3$SiO$_5$ material is known to exhibit seven different polymorphic forms (Song, Lee, Cho, & Ok, 2012; Staněk & Sulovský, 2002; Taylor & Aldridge, 1993). Some notations like TI, TII, TIII, MI, MII, MIII, and R were used for triclinic, monoclinic, and rhombohedral forms of tricalcium silicate (De La Torre, Bruque, Campo, & Aranda, 2002). Diffraction peaks of sol–gel-derived Ca$_3$SiO$_5$ material are well consistent with PDF No. 42-551 exhibiting monoclinic (M3) form (Jadhav & Debnath, 2011). Although the peaks are intense and more distinct in the phosphor materials prepared using sol–gel method. Eu$^{3+}$ doped Ca$_3$SiO$_5$ material crystallizes in non-centro symmetric (NCS) monoclinic form possessing a space group of $Cm$ (No. 8) (Song et al., 2012). Further reheating at 1,150°C correspondingly enhances the peak intensity of the material. Particle size of these materials is calculated with the help of Debye–Scherrer’s equation:

$$D = \frac{0.89\lambda}{\beta\cos\theta}$$

where $D$ is the particle size, $\lambda$ is the wavelength of X-ray used, $\beta$ is the full width at half maximum, and $\theta$ is the angle of incidence. Particle size has been calculated using most intense peak in XRD pattern of these phosphors which is summarized in Table 3. Diffraction profile of europium (III) doped Mg$_3$SiO$_5$ material at 950 and 1,150°C is shown in Figure 9. Literature data were not available before this report to confirm about the crystal structure of Mg$_3$SiO$_5$ material. X-ray pattern shows mixed diffraction peaks of Mg$_3$SiO$_5$ and MgO JCPDS No. 01-1235 (Li, Xu, Li, Liu, & Jia, 2014) phosphor.

3.3. Transmission electron microscopic analysis

The morphological features including shape and size of prepared phosphor materials are estimated using transmission electron microscopic analysis. Phosphors particles exhibited nearly spherical shape and they are also aggregated as seen in Figure 10. TEM micrographs confirmed the size of
Figure 9. X-ray diffraction pattern of europium (III) doped (a) Sr$_3$SiO$_5$, (b) Ca$_3$SiO$_5$, and (c) Mg$_3$SiO$_5$ materials.
prepared phosphors in nano-range. The measured particle size of as-prepared silicate materials is reported in Table 3 which is in accordance with particle size calculation from XRD analysis. These luminescent materials are found to be in nano (13–75 nm) range which have great applicability in the field of lighting and display technology.

4. Conclusion
Nanosized $M_3SiO_5$ ($M = Sr, Ca, Mg$ and $x = 0.03$ mol) silicate materials were successfully explored using sol–gel method and their characteristics were studied using different techniques. Emission spectra of these materials exhibited intense peaks in 400–700 nm region due to the different transitions of trivalent europium ion. XRD pattern gave sharp peaks indicating the crystalline structure of prepared materials. Particle size of these materials was calculated from diffraction analysis using Scherrer’s equation. TEM micrographs confirmed the existence of particles size in nano-range. $M_3SiO_5$ silicates showed better luminescence properties with doping of europium (III) ion in crystal lattice which emitted red light under 395 nm excitation. Hence, these nanophosphors might be integrated with LED chip to generate red light which could be suitable for lighting applications.

Table 3. Representing particle size of as prepared europium-doped silicate materials

| Type of lattice | $Sr_3SiO_5:Eu^{3+}$ | $Ca_3SiO_5:Eu^{3+}$ | $Mg_3SiO_5:Eu^{3+}$ |
|----------------|---------------------|---------------------|---------------------|
| Particle size (TEM analysis) | 74.88 | 13.71 | 43.17 |
| Particle size (XRD analysis) | 72.08 | 21.12 | 39.38 |

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