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# Photoluminescence properties of europium doped di-strontium magnesium di-silicate phosphor by solid state reaction method

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## ABSTRACT

Europium doped di-strontium magnesium di-silicate phosphor namely (Sr<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>3+</sup>) was prepared by the traditional high temperature solid state reaction method. The phase structure of sintered phosphor was akermanite type structure which belongs to the tetragonal crystallography with space group P4<sub>2</sub>m, this structure is a member of the melilite group and forms a layered compound. The EDX and FTIR spectra confirm the present elements in Sr<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>3+</sup> phosphor. Photoluminescence measurements showed that the phosphor exhibited strong emission peak with good intensity, corresponding to <sup>5</sup>D<sub>0</sub> → <sup>7</sup>F<sub>2</sub> (613 nm) red emission and weak <sup>5</sup>D<sub>0</sub> → <sup>7</sup>F<sub>1</sub> (590 nm) orange emission. The excitation spectra monitored at 613 nm show broad band from 220 to 300 nm ascribed to O–Eu charge-transfer band (CTB) centered at about 269 nm, and the other peaks in the range of 300–400 nm originated from f–f transitions of Eu<sup>3+</sup> ions. The strongest band at 395 nm can be assigned to <sup>7</sup>F<sub>0</sub> / <sup>5</sup>L<sub>6</sub> transition of Eu<sup>3+</sup> ions due to the typical f–f transitions within Eu<sup>3+</sup> of 4f<sup>6</sup> configuration.

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## 1. Introduction

Light-emitting diodes (LEDs) have recently attracted attention as novel sources for illuminating light. LEDs have several advantages over fluorescent lights, including a longer operating life, better energy efficiency and a mercury-free composition (Schubert & Kim, 2005). Such LEDs, are in the early stages of development, and addressing the problem of their color rendering characteristics is particularly important. It is well known that white LEDs are mainly fabricated by combining

blue LEDs with a yellow-emitting phosphor (YAG:Ce<sup>3+</sup>). However, the white light obtained has a poor color rendering, because the yellow emission of YAG:Ce<sup>3+</sup> lacks any red and blue-green emissions (Hirosaki et al., 2005; Neeraj, Kijima, & Cheetham, 2004; Wu et al., 2005). Recently, one solution to this problem has been to fabricate a white LED with high color rendering by combining red, green and blue emitting tricolor phosphors with irradiation by a near UV LED. Therefore, the development of red and green phosphors show high emission intensities is desired (Kamei, Kojima, & Nishimiya, 2010).

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Melilites, which are generally formulated as  $M_2T(1)T(2)_2O_7$  (where M and T(1) are usually the alkaline-earths or transition metals [M = Sr, Ca, Ba; and T(1) = Mg, Zn], and T(2) is usually Al, Ga, Si or Ge), have been investigated widely as optical materials [10]. Due to their tetragonal and non-centrosymmetric crystal structure, lanthanides or transition metals can be accepted easily as constituents or dopants by the melilites, allowing the synthesis of high-quality doped single crystals (Kaminskii et al., 2008). The silicate matrix  $Sr_2MgSi_2O_7$  is a typical melilite. Phosphors based on this material are usually low-cost and are chemically and physically stable. They have been studied widely with  $Eu^{2+}$  doping, which shows that a blue emission and long persistent luminescence by co-doping with some other rare earth ions (Calson et al., 2009; Pan et al., 2008; Xu & Chen, 2008).  $Eu^{3+}$  ions were chosen as a luminescent species in many cases (Li, Guan, Li, Wen, & Yang, 2010; Nguyen, Mho, & Yeo, 2009; Srivastava, 2009; Volanti et al., 2009; Yang, Ren, Tao, Cui, & Yang, 2009) for the reasons of measurable lifetimes up to at least 1100 and simple luminescent spectra when compared to other rare earths (Chambers, Rousseve, & Clarke, 2009; Wan, Wang, Chen, Qian Li, & Mu, 2005). It is interesting to investigate the luminescent properties of  $Eu^{3+}$  in  $Sr_2MgSi_2O_7$  matrix with the purpose of synthesis of a low cost and high efficiency orange – red phosphor.

In the present paper, we report the synthesis of europium doped di-strontium magnesium di-silicate ( $Sr_2MgSi_2O_7:Eu^{3+}$ ) phosphor by high temperature solid state reaction method. This paper reports the structural characterization on the basis of XRD, TEM, FESEM, EDX and FTIR analysis and studies of optical properties are also done on the basis of photoluminescence (PL).

## 2. Experimental

### 2.1. Synthesis

Europium doped di-strontium magnesium di-silicate  $Sr_2MgSi_2O_7:Eu^{3+}$  phosphor was prepared by the high temperature solid state reaction method. The raw materials are strontium carbonate [ $SrCO_3$  (99.90%)], magnesium oxide [ $MgO$  (99.90%)], silicon di-oxide [ $SiO_2$  (99.99%)] and europium oxide [ $Eu_2O_3$  (99.99%)], all of analytical grade (A.R.), were employed in this experiment. Boric acid ( $H_3BO_3$ ) was added as flux. Initially, the raw materials were weighed according to the nominal compositions of  $Sr_2MgSi_2O_7:Eu^{3+}$  phosphor. Then the powders were mixed and milled thoroughly for 2 h using mortar and pestle. The grinded sample was placed in an alumina crucible and subsequently fired at 1200 °C for 3 h. At last the nominal compounds were obtained after the cooling down of programmable furnace.

### 2.2. Characterization techniques

The powder X-ray diffraction (XRD) pattern has been obtained by Bruker D8 advanced X-Ray powder diffractometer using  $CuK\alpha$  radiation and the data were collected over the  $2\theta$  range 10–80°. Particle size of prepared  $Sr_2MgSi_2O_7:Eu^{3+}$  phosphor was determined by TEM using TECHNAI G2. The samples required for TEM analysis were prepared by dispersing the sintered phosphor in methanol using an ultrasound bath technique. A drop of this dispersed suspension was put onto 200-mesh carbon coated copper grid and then dried into the

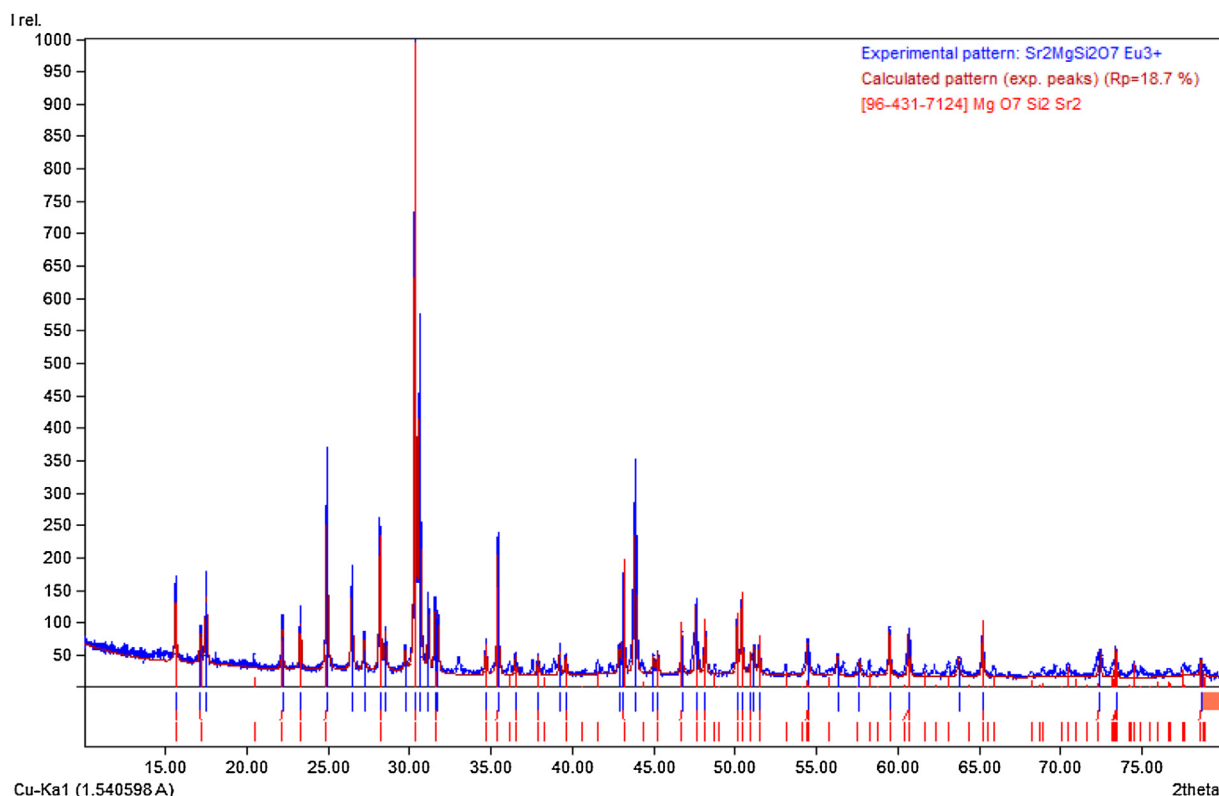


Fig. 1 – X-ray diffraction patterns of  $Sr_2MgSi_2O_7:Eu^{3+}$  phosphor.

air. The surface morphology of prepared  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor was determined by the FESEM (ZIESS Ulta Plus-55) operated at the acceleration voltage of 5 kV. The samples were coated with a thin layer of gold (Au) and then surface morphology of prepared phosphor was observed. Energy dispersive spectroscopy (EDX) was used for the elemental (quantitative and qualitative) analysis of the prepared phosphor. FTIR spectra were recorded with the help of IR Prestige-21 by SHIMADZU for investigating the functional group region ( $4000\text{--}1400\text{ cm}^{-1}$ ) as well as the finger print region ( $1400\text{--}400\text{ cm}^{-1}$ ) of sintered phosphor in middle infrared region ( $4000\text{--}4000\text{ cm}^{-1}$ ) by mixing the sample with potassium bromide (KBr, IR grade). The PL excitation and emission spectra were measured by a spectrofluorophotometer (SHIMADZU, RF-5301 PC). All measurements were carried out at the room temperature.

### 3. Results and discussions

#### 3.1. XRD analysis

In order to determine the phase structure, powder XRD analysis has been carried out. The typical XRD patterns of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor with the standard XRD pattern is shown in Fig. 1. The position and intensity of diffraction peaks of the prepared  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor were matched and found to be consistent with the standard XRD pattern (COD card No. 96-431-7124) by MATCH 2 software. The figure of merit (FOM) while matching these was 0.9671 (96%) which illustrates that the phase of the prepared sample agrees with the standard pattern COD card No. 96-431-7124. In Fig. 1, it can be concluded that prepared samples are chemically and structurally  $\text{Sr}_2\text{MgSi}_2\text{O}_7$  phosphor. The crystalline phase of the prepared  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor is nearly same ( $a = b = 7.9948\text{ \AA}$  and  $c = 5.1636\text{ \AA}$ ) with the standard lattice parameters ( $a = b = 7.9957\text{ \AA}$  and  $c = 5.1521\text{ \AA}$ ). The phase structure of the  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor is akermanite type structure which belongs to the tetragonal crystallography with space group  $P4_21m$  (113 space number and  $D_{3d}^{32}$  space group), this structure is a member of the melilite group and forms a layered compound. The average crystallite size was calculated from the XRD pattern using Debye Scherrer relation

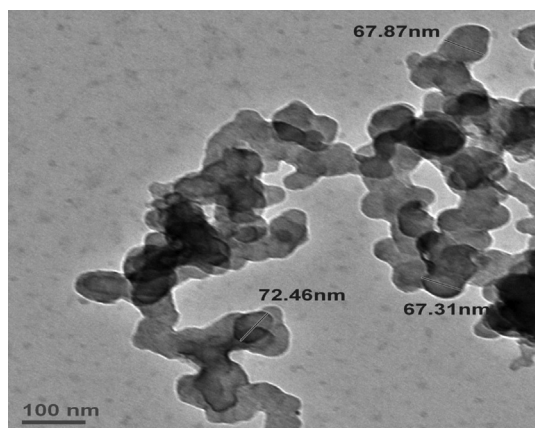


Fig. 2 – TEM image of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor.

$D = k\lambda/\beta\cos\theta$ , where  $D$  is the crystallite size for the  $(hkl)$  plane,  $\lambda$  is the wavelength of the incident X-ray radiation [ $\text{CuK}\alpha$  ( $0.154\text{ nm}$ )],  $\beta$  is the full width at half maximum (FWHM) in radiations, and  $\theta$  is the corresponding angle of Bragg diffraction. Sharper and isolated diffraction peaks such as  $2\theta = 30.48$  were chosen for calculation of the crystallite size. Based on the Debye-Scherrer's formula, the average crystallite size of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  is  $\sim 67$ .

#### 3.2. Transmission electron microscopy (TEM)

The particle size of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  is shown in Fig. 2. The TEM images confirm the particle size of the prepared phosphor. From the TEM image, it can be observed that the prepared sample consists of grain with different size distribution. Moreover, the agglomeration of powder particles was also observed due to high temperature. The transmission electron microscopy results are in good correlation with the XRD result.

#### 3.3. Field emission scanning electron microscopy (FESEM)

The surface morphology of the  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor is shown in Fig. 3(a,b) with different magnification. The surface morphology of the particles was not uniform and they aggregated tightly with each other due to the high temperature heat treatment. Moreover, the agglomeration of powder particles was also observed. From the FESEM image, it can be observed that the prepared sample consists of particles with different size distribution. The scanning electron microscopy results are in good correlation with the TEM result.

#### 3.4. Energy dispersive X-Ray spectroscopy (EDX)

The composition of the powder sample has been measured using EDX. Energy dispersive x-ray spectroscopy (EDX) is a standard procedure for identifying and quantifying elemental composition of sample area as small as a few nanometers.

The existence of europium (Eu) in prepared phosphor is clear in their corresponding EDX spectra. Their appeared no other emission apart from strontium (Sr), magnesium (Mg), silicon (Si) and oxygen (O) in  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  EDX spectra of the samples. In the EDX spectrum, the presence of Sr, Mg, Si, O and Eu, intense peak are present which preliminary indicates the formation of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor in Fig. 4.

#### 3.5. Fourier transform infrared spectra (FTIR)

The Fourier Transform Infrared (FTIR) spectra have been widely used for the identification of organic and inorganic compounds. Fig. 5 shows the FTIR spectra of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor. In observed IR spectrum, the absorption bands of silicate groups are clearly evident. An intense band centred at  $974.14\text{ cm}^{-1}$  is assigned due to Si–O–Si asymmetric stretch, bands at  $874.98$  and  $631.89\text{ cm}^{-1}$  to Si–O symmetric stretch. Bands at  $513.54$  and  $467.17\text{ cm}^{-1}$  are assigned to Si–O–Si vibrational mode of bending. Furthermore, in keeping with the absorption bands, posited at  $1089.29$ ,  $924.56$ ,  $778.62$ ,  $716.65$

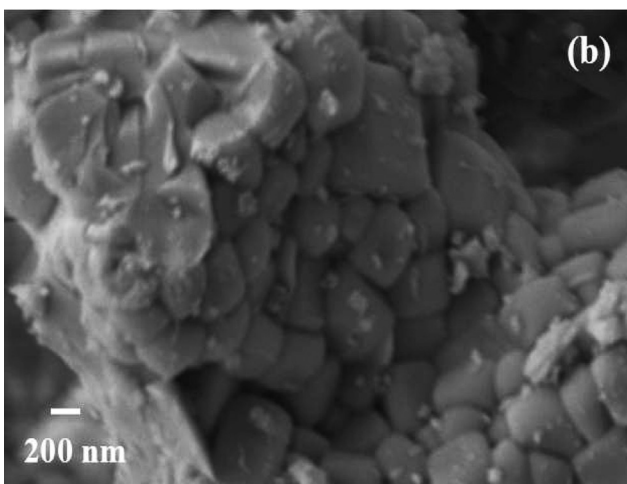
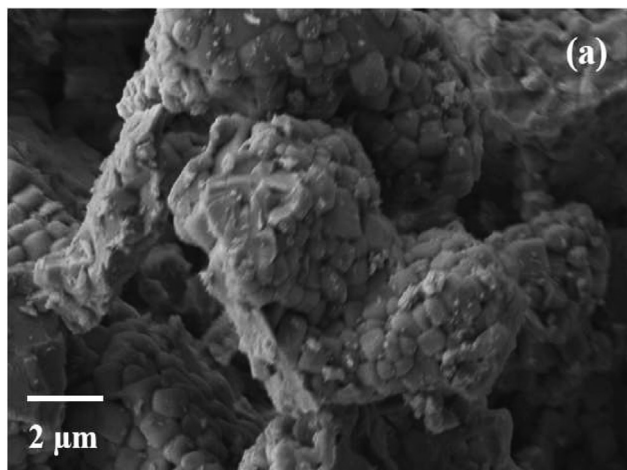


Fig. 3 – (a, b) FESEM image of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor.

and  $667.08\text{ cm}^{-1}$  respectively can be ascribed to the presence of  $\text{SiO}_4$  group (Gou, Chang, & Zhai, 2005).

The FTIR spectrum of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor contains clearly exhibited bands in the region ( $3421.05\text{ cm}^{-1}$ ) of hydroxyl group show the stretching vibration of O–H groups. The hydroxyl group in sintered phosphor is might be due to presence of moisture through environment. The asymmetric stretching of ( $\text{CO}_3^{2-}$ ) carbonates can be observed in the range of  $1900\text{--}1700\text{ cm}^{-1}$ . Two weak shoulders, which corresponds to the out of plane bending of appears at approximately  $1980.58$ ,

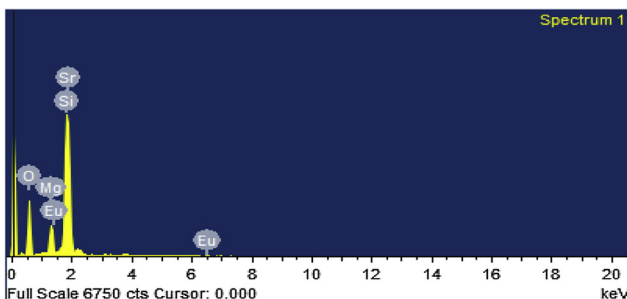


Fig. 4 – EDX spectra of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor.

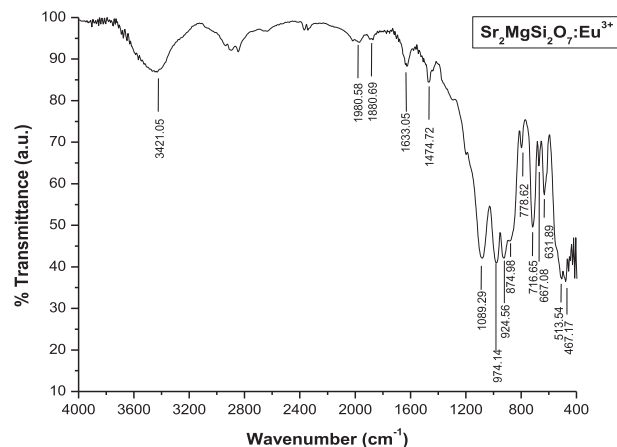


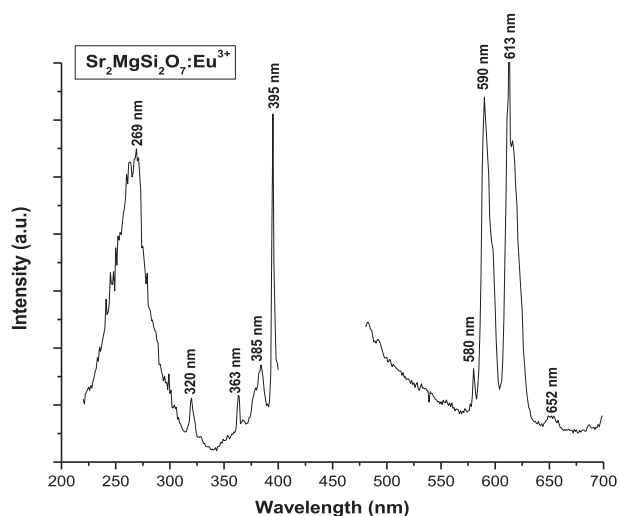
Fig. 5 – FTIR Spectra of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor.

$1880.69\text{ cm}^{-1}$ . These bands are due to a slight carbonation of the samples preparation [ $\text{SrCO}_3$  (raw material)]. The free  $\text{CO}_3^{2-}$  ions has a  $D3h$  symmetry (trigonal planar) and its spectrum is dominated by the band at  $1900\text{--}1700\text{ cm}^{-1}$ . The vibration band of  $1633.05\text{ cm}^{-1}$  are assigned due to the  $\text{Mg}^{2+}$  and bending of the sharp peaks in the region of  $1474.72\text{ cm}^{-1}$  are assigned due to  $\text{Sr}^{2+}$ . When  $\text{Eu}^{3+}$  enters the lattice, it will replace the  $\text{Sr}^{2+}$  in the  $\text{Sr}_2\text{MgSi}_2\text{O}_7$  host and occupy  $\text{Sr}^{2+}$  lattice sites due to distortion in the  $\text{Sr}_2\text{MgSi}_2\text{O}_7$  host crystal lattice. Original position of  $\text{Sr}^{2+}$  was replaced by  $\text{Eu}^{3+}$  and the original of  $\text{Sr}^{2+}$  located at somewhere (Sahu, Bisen, & Brahme, 2014a, 2014b, 2014c). Therefore the vibration mode of  $\text{Sr}^{2+}$  at  $1474.72\text{ cm}^{-1}$  is clearly observed from  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor. Another fact that supports that the radius of  $\text{Eu}^{3+}$  ( $1.07\text{ \AA}$ ) are very close to that of  $\text{Sr}^{2+}$  (about  $1.12\text{ \AA}$ ) rather than  $\text{Mg}^{2+}$  ( $0.65\text{ \AA}$ ) and  $\text{Si}^{4+}$  ( $0.41\text{ \AA}$ ). Therefore, the  $\text{Eu}^{3+}$  ions are expected to occupy the  $\text{Sr}^{2+}$  sites in the  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor (Chandruppa, Ghosh, & Patil, 1999; Fei, Chang, & Mao, 2005; Salim et al., 2009).

### 3.6. Photoluminescence (PL)

Fig. 6 shows the PL spectrum of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor. The sintered phosphor would excited by  $395\text{ nm}$ . As shown in Fig. 1, the emission spectrum is mainly composed of two strong emission peaks. The emission spectrum exhibits four typical emission peaks in the range of  $560\text{--}700\text{ nm}$ , which result from  $^5\text{D}_0 \rightarrow ^7\text{F}_j$  ( $J = 0, 1, 2, 3$  and  $4$ ). The emission peak at  $613\text{ nm}$  is ascribed to the electric dipole transition from  $^5\text{D}_0 \rightarrow ^7\text{F}_2$  of  $\text{Eu}^{3+}$ , while the emission near  $593\text{ nm}$  is assigned to the magnetic dipole transition from the  $^5\text{D}_0 \rightarrow ^7\text{F}_1$  of  $\text{Eu}^{3+}$ . Moreover, there are two weak emission peaks located at  $580$  and  $652\text{ nm}$ , which are ascribed to  $^5\text{D}_0 \rightarrow ^7\text{F}_0$  and  $^5\text{D}_0 \rightarrow ^7\text{F}_3$  transitions of  $\text{Eu}^{3+}$  respectively.

According to the parity selection rule, when the  $\text{Eu}^{3+}$  ions are located at the site with an inversion symmetric center, the  $^5\text{D}_0 \rightarrow ^7\text{F}_1$  magnetic dipole transition is permitted, which results in orange red emitting around  $590\text{ nm}$ . But if located at the site without an inversion symmetric center, because the opposite parity  $5d$  configuration is mixed into  $4f^n$

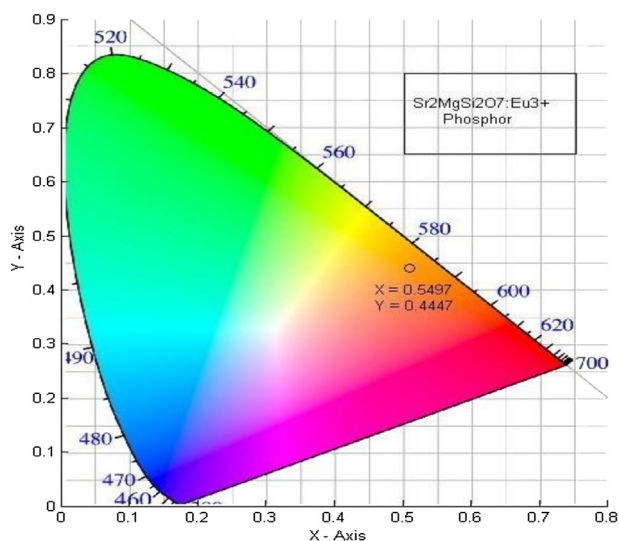


**Fig. 6 – Photoluminescence Spectra of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor.**

configuration, the parity selection rule is able to be lifted, and f–f forbidden transition is partially released, the hypersensitive  $^5\text{D}_0 \rightarrow ^7\text{F}_2$  electric dipole transition will be permitted, which results in red emitting around 613 nm. For the phosphor  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  prepared in our experiment, the strongest red emission peak is located at 613 nm will be dominated. It can be presumed that  $\text{Eu}^{3+}$  ions mainly occupy non-inversion symmetric center in host lattice (Sun, Qian, Liao, Wang, & Yan, 2001).

### 3.7. CIE chromaticity coordinate

In general, color of any phosphor material is represented by means of color coordinates. The luminescence color of the samples excited under 395 nm has been characterized by the CIE (Commission International de l'Éclairage) 1931 chromaticity diagram. The emission spectrum of the  $\text{Eu}^{3+}$  doped



**Fig. 7 – CIE diagram of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor.**

$\text{Sr}_2\text{MgSi}_2\text{O}_7$  phosphor was converted to the CIE, 1931 chromaticity using the photoluminescent data and the interactive CIE software (CIE coordinate calculator) diagram as shown in Fig. 7. Every natural color can be identified by (x, y) coordinates that are disposed inside the ‘chromatic shoe’ representing the saturated colors (Sahu et al., 2014a, 2014b, 2014c). Luminescence colors of  $\text{Eu}^{3+}$  doped  $\text{Sr}_2\text{MgSi}_2\text{O}_7$  phosphor are placed in the orange–red ( $X = 0.5497$ ,  $Y = 0.4447$ ) corners. The chromatic co-ordinates of the luminescence of this phosphor are measure and reached to orange–red luminescence.

## 4. Conclusion

In summary, we have successfully synthesized pure tetragonal phased  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor via traditional high temperature solid state reaction method. The phase structure of the  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor is consistent with standard tetragonal crystallography. From the XRD and TEM analysis, average particle size of  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor was ~67 nm. The radius of  $\text{Eu}^{3+}$  (1.07 Å) are very close to that of  $\text{Sr}^{2+}$  (about 1.12 Å) rather than  $\text{Mg}^{2+}$  (0.65 Å) and  $\text{Si}^{4+}$  (0.41 Å). Therefore, the  $\text{Eu}^{3+}$  ions are expected to occupy the  $\text{Sr}^{2+}$  sites in the  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor. The EDX and FTIR spectra confirm the present elements in  $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$  phosphor. Under the near ultraviolet (NUV) excitation (395 nm), photoluminescence measurements showed that the phosphor exhibited strong emission peak with good intensity corresponding to  $^5\text{D}_0 \rightarrow ^7\text{F}_2$  (613 nm) red emission and weak  $^5\text{D}_0 \rightarrow ^7\text{F}_1$  (590 nm) orange emission. The PL emission exhibited orange–red light which was confirmed from the calculated CIE coordinates. Thus, the solid state reaction method furnishes a simple method for preparing a silicate based phosphor.

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