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Study of Radiophotoluminescence of Eu Doped $CaSO_4$ Phosphor for Gamma Dosimetric Applications

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ABSTRACT

$CaSO_4$:Eu phosphor is synthesised by acid distillation method with varying synthesis parameters for studying its Radiophotoluminescence (RPL) properties for gamma absorbed dose measurements. Five phosphor samples are prepared by varying quantity of solvent, distillation temperature and reaction time. XRD, SEM, particle size analysis and photoluminescence studies are carried out. The characterisation study shows polycrystalline luminescent particles of average size varying from 35 µm to 55 µm. Characteristic emission of Eu^{3+} is observed around 590, 615 and 620 nm at 242 nm excitation. Gamma dose response of maximum Eu^{3+} PL intensity sample is studied in the range 10 cGy to 1000 cGy using Co-60 source. Gamma radiation exposure induces conversion of Eu^{3+} to Eu^{2+} giving luminescence at 385 nm with 320 nm excitation. Repetitive measurements of gamma exposed samples are carried out and no significant fading is observed within one week of post-irradiation. The phosphor has the potential to be used for gamma dosimetry.

Keywords: Dosimetry; Phosphor synthesis; Radiophotoluminescence

1. INTRODUCTION

Exposure to ionising radiations can cause damage to human tissues or organs[1]. External exposure occurs when the whole or part of the human body is exposed to penetrating radiations from an external source. The extent of the damage increases with the increase in radiation absorbed doses in the human body. Radiation dosimetry of occupational radiation workers is carried out to control the radiation exposures within permissible limits[1-2]. Luminescence based passive dosimetry systems are in practice for assessment of radiation absorbed doses. The working principle of these systems is that the intensity of light emitted by the phosphor material is proportional to the radiation doses absorbed by the material and unknown doses of ionising radiations can be estimated by calibrating the system with the exposure of known doses. Thermal stimulated luminescence (TSL) and Radiophotoluminescence (RPL) based passive dosimeters are used for cumulative dose measurements. $CaSO_4$:Dy and LiF:Mg, Ti are being used as a Thermoluminescent Dosimeters (TLD) to monitor personal exposure to X-ray and gamma radiations. However, TLDs have the disadvantage of removal of stored information of radiation exposure during the readout procedure[3]. RPL is the luminescence stimulated by light from the irradiated phosphor material, the stimuli only excite and do not ionize the material. The luminescence output can be read multiple times from the irradiated material non-destructively. Ag^+ doped phosphate glass (RPL glass) is based on RPL technique for readout of radiation exposure. Radiation induced valence conversion of Ag^+ to Ag^{2+} is a dosimetric parameter. Estimation of absorbed doses is possible by using the intensity of orange luminescence. The glass has some limitations as it gives real absorbed dose measurements after several hours of exposure due to its build-up phenomenon[4-6]. RPL intensity of irradiated glass is found to be increased with elapsed time after irradiation. The build-up kinetics is found to be associated with the decrease in mobility of Ag^+ with the decrease of alkali contents in the glass. RPL properties are also reported for Sm doped fluorophosphate, fluoroaluminate glasses, BaF_2-Al_2O_3-B_2O_3 glass ceramics and Ag doped mixed phosphate glasses[7-9].

Certain rare earth doped, particularly Eu and Sm doped phosphors, also exhibit RPL. Preliminary measurements of RPL signal are reported on X-ray and UV irradiation of Eu and Sm doped $CaSO_4$ phosphor[10]. Eu can go into $CaSO_4$ matrix in divalent or trivalent form depending on the method adopted for phosphor preparation[11-13]. Radiation induced valency conversion of rare earth doped CaF_2 and alkali sulphates are also reported for high exposures[14]. Eu doped phosphors also find application in photo-, cathodo- and electro-luminescent, photoluminescence liquid crystal displays[15-16].

$CaSO_4$:Eu phosphor material has been reported for TL and RPL measurements for UV, X-ray irradiations and high gamma doses in kGy ranges. Acute doses of up to ~ 0.1 Gy are believed to produce no functional impairment of tissues[1,17]. In this paper, authors reported investigation carried out on Eu doped $CaSO_4$ phosphor intending to develop RPL phosphor material for accident dosimetry applications in defence for gamma dose measurements in the range of few cGy to 1000

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cGy during nuclear or radiological emergency management. The study is restricted to the specific dose range keeping in view the onset of human tissue reactions (deterministic effects) and lethality dose due to acute radiation exposure. The effects of various synthesis parameters on Eu doped $CaSO_4$ phosphor samples for their suitability in the enhancement of radiation sensitivity have also been reported.

2. MATERIALS AND METHOD

The synthesis method used for preparation of $CaSO_4$:Eu RPL phosphor samples is the modification of co-precipitation method described by Lakshmanan[18], *et al.* The starting materials used were $CaCO_3$ (98.5%, Merck), HNO_3 (Min 69% GR, Merck), HCl about 35% (Merck), H_2SO_4 (about 98%, Merck) and Eu_2O_3 (99+, Merck Germany). $CaCO_3$ was dissolved in HNO_3 and continuously stirred to make a transparent solution. A stock solution of (Eu_2O_3+HCl) was prepared.

Sample-1 of $CaSO_4$:Eu phosphor was synthesised by adding, the mixture of ($CaCO_3$+ HNO_3) & (Eu_2O_3+HCl) drop by drop in hot un-distilled H_2SO_4 (~80 °C). The temperature of solvent was gradually raised to 150-180 °C. The acid was distilled out in a closed system in 5 hrs. The precipitate was collected and repeatedly washed with double distilled water to remove the traces of acid. Subsequently, the precipitate was dried in an oven at 80 °C for 30 min. Annealing of synthesised phosphor was further done in a furnace at 400 °C for 1 hr followed by quenching to room temperature on aluminium sheet.

Sample-2 was synthesised keeping the synthesis process same as of Sample-1. Purified H_2SO_4 (distilled) was used instead of un-distilled H_2SO_4 for preparing the sample in impurity free solvent. While preparing Sample-3 and Sample-4, the quantity of distilled H_2SO_4 was increased to 50 ml and 100 ml respectively from 15 ml. The temperature of the solution was gradually raised to 280-300 °C and the solution was kept at this temperature for 7 hr. The temperature of acid bath was continuously monitored with a thermometer placed in thermal pocket of distillation assembly. The solution was further kept undisturbed at room temperature for 15 hr and then the heating was resumed. The temperature of the solution was slowly raised to 340-350 °C and excess acid was distilled out in 5 hr. Except of the quantity of solvent and heating stages the other processing steps of the synthesis method were kept same.

While preparing the sample-5 phosphor, H_2SO_4 was added drop by drop to the mixture of ($CaCO_3$+ HNO_3) and (Eu_2O_3+HCl) solution to have a less exothermic reaction. The other processing steps were kept same as sample-3 and 4.

The doping concentration of Eu was used typically 0.02 mol%. Description of the synthesised phosphor samples is given in Table 1.

Powder X-ray diffractograms were recorded on Pan Analytical xpertpro XRD system using Cu K_α radiation of wavelength 1.5406 A° in the 2θ range of 20-80° with a step size of 0.05°/s. Surface morphology was investigated using scanning electron microscopy (ZEISS) after deposition of gold coating on phosphor samples. Particle size distribution analysis was done on Microtrac particle size analyser employing laser scattering and photoluminescence spectra were obtained using Jasco FP6500 spectrofluorometer with Xe-lamp (150W) as an excitation source. Excitation and emission spectra were recorded with a spectral slit of 1 and 3 nm respectively. The amount of phosphor sample was kept same in each measurement. Gamma irradiations of phosphor samples were performed in NABL accredited test and calibration facility using Co-60 source of activity 10.1 Ci. The calibration of the facility is being periodically carried out by BARC using standard reference ionisation chambers. The phosphor samples were exposed to different gamma doses by mounting them on a remotely operated moveable positioner system. Source to sample distance was kept at 34.5 cm to expose the samples at a dose rate of 100 cGy/h.

3. RESULTS AND DISCUSSION

3.1 Powder XRD

The X-ray diffraction patterns of the synthesised $CaSO_4$:Eu phosphor samples along with the standard JCPDS are shown in Fig. 1. The powder XRD results show diffracted peaks indicating crystalline structures. The obtained diffraction data of $CaSO_4$ phosphor samples are in good agreement with the standard data of JCPDS card No. 74-1782. It confirms the orthorhombic structure for all the samples and products obtained are characteristic of $CaSO_4$. Since ionic sizes of Ca^{2+} (114 pm) and Eu^{3+} (108.7 pm) are approximately same, the doped ions may have occupied the primal Ca^{2+} sites of $CaSO_4$ lattice. Further, due to lesser dopant concentration, no significant shift in XRD peaks is observed. The average crystallite size calculated using the Scherrer equation for powder phosphor Samples 1-5 is 32.72 nm, 33.83 nm, 51.42 nm, 49.91 nm and 43.93 nm respectively. The increase in average crystallite size may be attributed to the change in the solvent quantity, distillation temperature and time during the synthesis of phosphors.

Table 1. Description of synthesised phosphor samples

Sample	Quantity of solvent H_2SO_4	Distillation temp. (°C)	Distillation time (h)	Remarks
1	15 ml (undistilled)	Single heating stage @150-180	5	Distillation of excess sulphuric acid was done in one attempt
2	15 ml (distilled)			
3	50 ml (distilled)	1st heating stage @ 280-300 2nd heating stage @ 340-350	12	The solution was kept undisturbed at room temperature for 15 hr after heating @280-300 °C
4	100 ml (distilled)			
5	50 ml (distilled)			Addition of sulphuric acid to aqueous solution and solution was kept undisturbed at room temperature for 15 hr after heating @280-300 °C

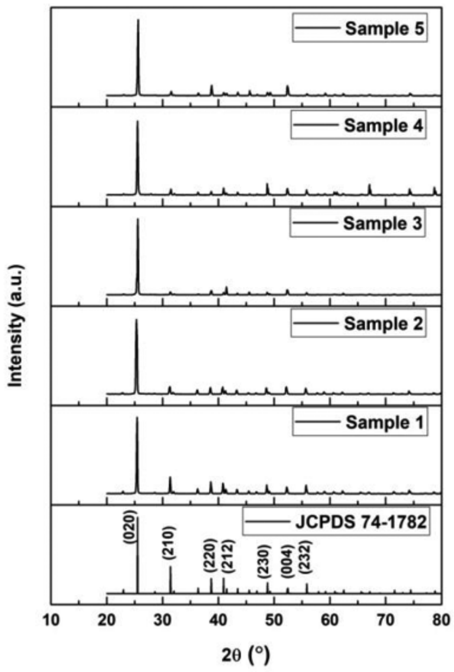


Figure 1. XRD of phosphor samples.

3.2 SEM Micrographs

The SEM micrographs of the phosphor Samples 1-5 in as-prepared condition are shown in Fig. 2. SEM micrograph of Sample-1 shows the granular structure and non-uniform morphology with smaller particles sizes. A similar morphology is observed in Sample-2 micrograph with the increase in particle sizes. No significant difference in morphology of Sample 1 and 2 is observed because the synthesis parameters for preparation of these samples are same. However, uniform morphology is obtained in Samples 3-5 with larger particle sizes, this may again be due to the change in distillation time, temperature and solvent quantity. The increase in these three synthesis parameters leads to change in particle size and it is further confirmed through particle size analysis study.

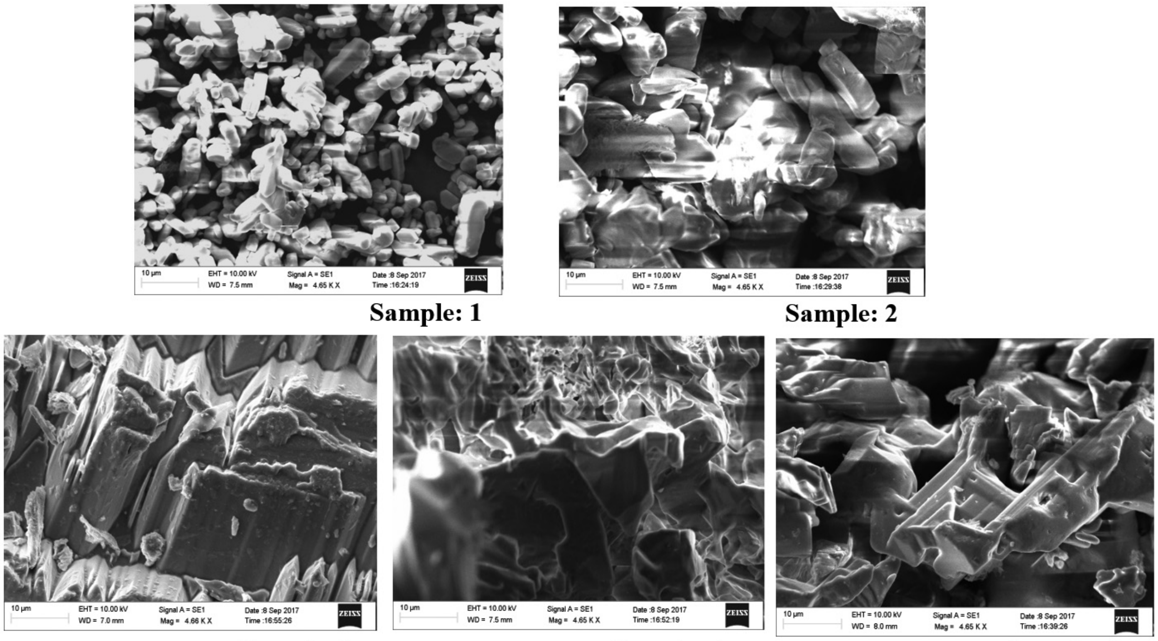
3.3 Particle Size Analysis

Particle size distribution graphs of different phosphor samples are shown in Fig. 3. The average particle size of phosphor samples 1-5 are 33.94 µm, 37.21 µm, 55.82 µm, 45.73 µm and 41.73 µm respectively. This indicates that the phosphor samples synthesised with increase in acid bath temperature, quantity of solvent and reaction time have larger average particle size and results are in agreement with SEM observations.

3.4 Photoluminescence (PL)

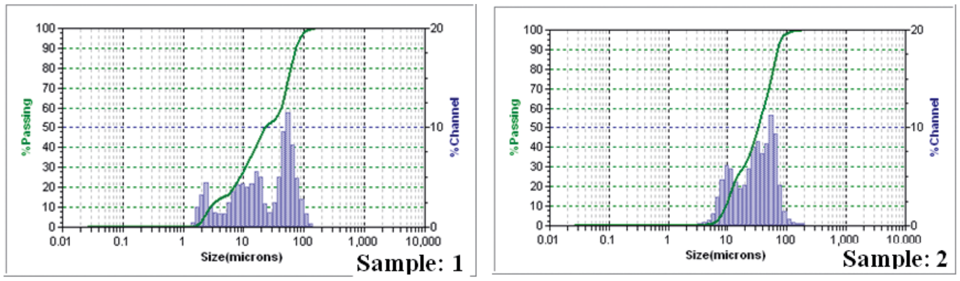
The typical excitation and emission spectra of Eu^{3+} and Eu^{2+} in $CaSO_4$:Eu phosphor sample are obtained and shown in Fig. 4 (a,b). Eu^{3+} gives emission in the form of sharp lines around 590 nm, 615 nm and 620 nm corresponding to f-f transitions[19]. The excitation spectrum of Eu^{3+} for 615 nm emission shows excitation at 242 nm (Fig. 4 (a)). Eu^{2+} gives emission around 385 nm in the form of a narrow band corresponding to d-f transition[19]. Several excitation peaks of Eu^{2+} are observed at 244, 250 nm, 270 nm, 320 nm and 340 nm for 385 nm emission as shown in Fig. 4(b).

The PL emission spectra of Samples 1-5 obtained at excitation wavelength of 242 nm to observe the PL intensity of characteristic peaks of Eu^{3+} for phosphor Samples 1-5 are shown in Fig. 4 (c). It is evident that Sample 1 and 2 has similar Eu^{3+} PL intensity due to their identical preparation and low sensitivity compared to other samples which may be attributed to smaller particle sizes as observed in SEM micrographs and particle size analysis. Eu^{3+} PL intensity of Sample 3 and 4 is found nearly same due to the larger particle size phosphors



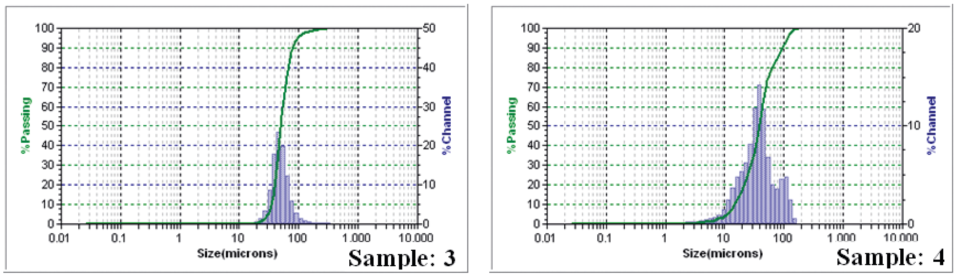
Sample: 1
Sample: 2
Sample: 3
Sample: 4
Sample: 5

Figure 2. SEM micrographs of phosphor samples.



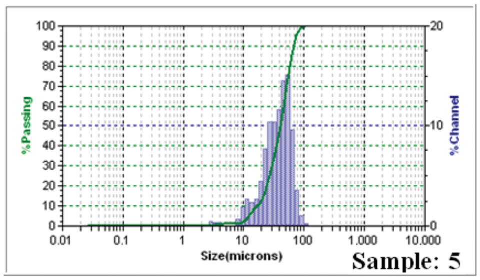
1.63 – 124.5 µm (Average 33.94 µm)

3.89 – 176 µm (Average 37.21 µm)



18.5 – 296 µm (Average 55.82 µm)

2.75 – 148 µm (Average 45.73 µm)



3.27 – 104.7 µm (Average 41.47 µm)

Figure 3. Particle size distribution of phosphor samples.

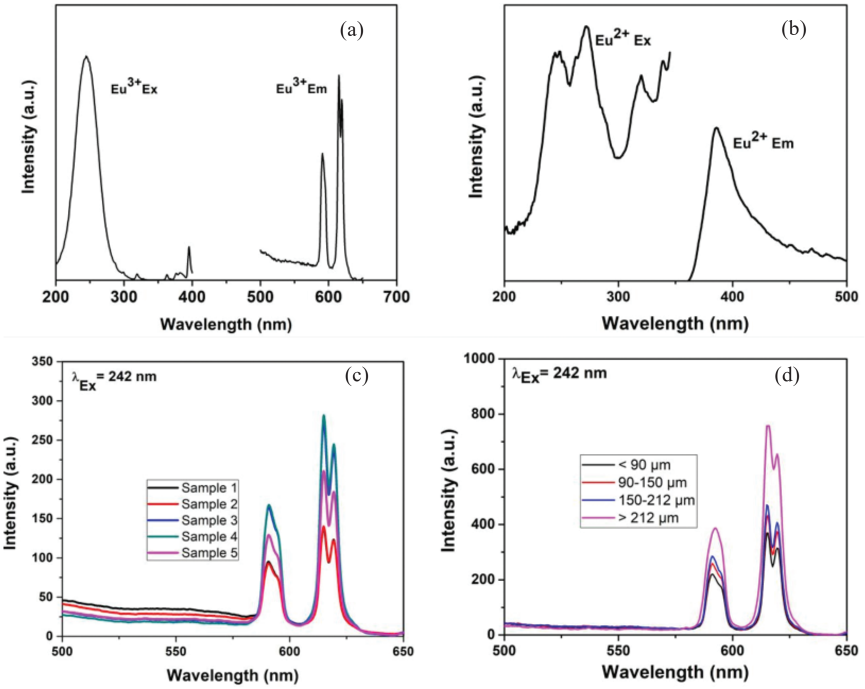


Figure 4. (a) Eu^{3+} emission (for 242 nm excitation) and excitation (for 615 nm emission), (b) Eu^{2+} emission (for 320 nm excitation) and excitation (for 385 nm emission), (c) PL spectra of Eu^{3+} emission for phosphor Samples 1-5, and (d) PL spectra of Eu^{3+} emission of samples with different particle size.

compared to other phosphor samples. Sample-5 also shows Eu^{3+} PL intensity lower than Sample 3 & 4. To examine particle size dependence of Eu^{3+} PL intensity, the phosphor sample is further sieved to get the phosphor of different particle size range < 90 µm, 90-150 µm, 150-212 µm and > 212 µm. It is observed from the PL spectra (Fig. 4(d)) that PL intensity of Eu^{3+} increases with the increase in particle size, which supports lower Eu^{3+} PL intensity of Sample 1-2 due to lower average particle size. Increase in PL intensity with increase in particle size may be attributed to the enhancement of reflections from each side of the particle. Considering the better PL intensity of Eu^{3+} and lesser solvent quantity requirement i.e. 50 ml H_2SO_4 compared to Sample-4 (100 ml) preparation, Sample-3 is chosen for gamma dose response study.

3.5 Gamma Dose Response of Phosphor

The phosphor Sample-3 having particle size in the range 90-212 µm is selected to study the gamma dose response in cGy ranges. Particle size < 90 µm is left out due to their lesser Eu^{3+} PL intensity and > 212 nm particle size is not taken due to difficulty in preparation of powder disc sample for the study. Phosphor sample-3 with a fixed quantity of phosphor (~ 200 mg) in the disc form (Dia 15 mm x 1 mm) is exposed to different known gamma doses to investigate the gamma radiation detection capability of synthesised phosphor through Eu^{2+} RPL after radiation induced valence conversion of rare earth ion Eu^{3+}. The amount of doses imparted to the sample is 10, 50, 200 and 1000 cGy. Emission spectra of the un-irradiated and gamma irradiated sample recorded at an excitation wavelength of 320 nm to observe changes in Eu^{2+} characteristic peak are shown in Fig. 5. The excitation wavelength at 320 nm is chosen as it contributes only to Eu^{2+} emission and does not lead to Eu^{3+} emissions.

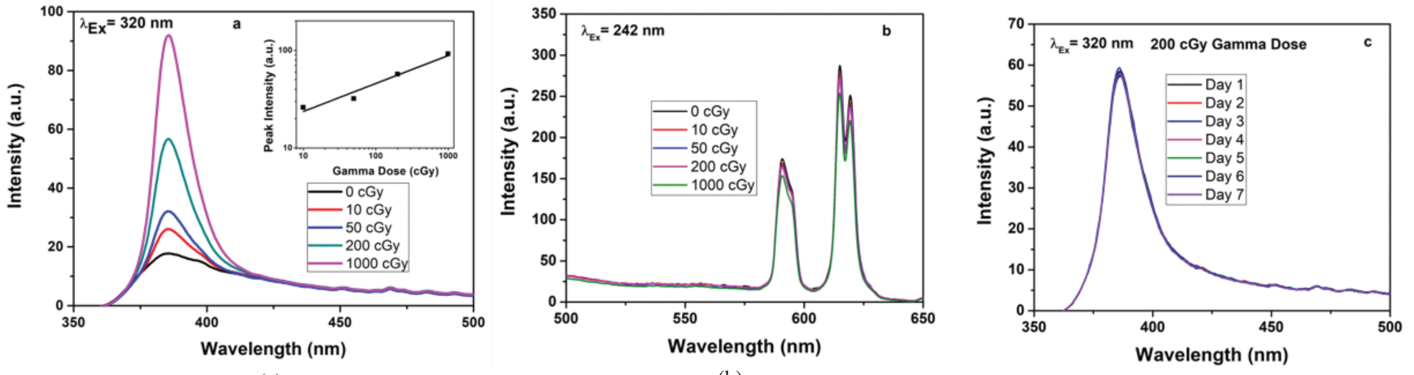
Europium enters primarily in Eu^{3+} form in the $CaSO_4$ lattice and presence of Eu^{2+} in the unirradiated sample in emission spectra (Fig. 5 (a)) shows background signal. It is evident from Fig. 5 (a) that there is a significant increase in the characteristic peak of Eu^{2+} with the gamma doses. This indicates gamma radiation induced valence conversion of Eu^{3+} to Eu^{2+} has taken place in $CaSO_4$ lattice. The characteristic peak of Eu^{2+} at 385 nm in $CaSO_4$ host matrix is due to $4f^65d^1 \rightarrow 4f^7$ transitions. With the increase in cumulative gamma doses absorbed by the material, the intensity of characteristic peak of Eu^{2+} increases. The Sample-3 phosphor is found sensitive to gamma doses in the range 10 cGy to 1000 cGy. The improvement in radiation sensitivity of phosphor is attributed to increase in the average particle size and enhancement of PL intensity of Eu^{3+} in $CaSO_4$ lattice. The linear increase in peak intensity of Eu^{2+} due to radiation induced conversion of Eu^{3+} to Eu^{2+} with gamma doses is shown as an inset in Fig. 5. The minimum RPL detectable gamma absorbed dose is found to be greater than those reported for X-irradiation by Calvert & Danby because of predominance of photoelectric effect by low energy X-rays for high atomic number materials[10].

It is also observed that there is no relative reduction in Eu^{3+} characteristic peaks with the increase in Eu^{2+} characteristic peak in the studied gamma dose range, as shown in Fig. 5(b). This effect is due to the greater fluorescence efficiency of the Eu^{2+} than Eu^{3+}. The Eu^{2+} characteristic emission at 385 nm arises due to transitions $4f^65d^1$ electronic state to the ground state $4f^7$. These are parity allowed transitions and give intense electric dipole radiation. Whereas, Eu^{3+} characteristic sharp emission lines are due to transitions within the $4f^6$ electronic configuration, which are parity forbidden. Thus Eu^{3+} emission is due to the weaker magnetic dipole transition and a small quantity of electric dipole radiation which is allowed because of the state mixing produced when the Eu^{3+} is incorporated in a low symmetry $CaSO_4$ crystal lattice[10,19].

PL spectra of radiation induced signal Eu^{2+} of a typical phosphor sample exposed to 200 cGy gamma dose are obtained multiple times and shown in Fig. 5 (c). No loss in PL intensity of Eu^{2+} characteristic peak is detected. This shows no build-up and fading of PL intensity of Eu^{2+} characteristic peak of irradiated phosphor within a week of post-irradiation, that confirms stability of radiation induced signal and radiophotoluminescence characteristic of phosphor. Fig. 5 (c) also represents the advantage of RPL dosimetry as the absorbed dose information is not lost during readout process, this enables non-destructive readouts.

4. CONCLUSIONS

Eu doped $CaSO_4$ phosphors are synthesised by acid distillation method for gamma dose measurements through its RPL properties. XRD shows the prepared phosphor is



(a) (b) (c)

Figure 5. (a) PL spectra of Eu^{2+} emission for gamma irradiated Sample-3, (b) PL spectra of Eu^{3+} emission for gamma irradiated Sample-3, and (c) Fading response of phosphor Sample-3.

polycrystalline with an orthorhombic phase. SEM micrographs show the larger particle sizes of phosphor samples prepared with the increase in the solvent quantity, distillation temperature and time. Particle size distribution analysis shows the strong dependence of average particle size of phosphor on the route of synthesis. Eu^{3+} PL intensity is found dependent on the particle sizes. Radiation induced valency conversion of europium ion is observed and synthesised $CaSO_4$:Eu phosphor with favourable synthesis parameters is found to be sensitive in gamma dose range 10 cGy - 1000 cGy, which would be practical for accident dosimetry applications. No post-irradiation fading is observed in the investigated period of one week. Radiation induced signal is found non-destructive which enables multiple readouts of gamma irradiated sample. Hence, the synthesised phosphor has the potential to be used for gamma radiation dosimetry.

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His contributions in the current study are in the establishment of phosphor synthesis facility, phosphor synthesis, material characterisation, result analysis and documentation of paper.

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His contributions in the current study are in overall guidance and result analysis.