

The influence of different synthesis routes on thermoluminescence of CaSO₄: Dy, P, Ce phosphors

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ABSTRACT

CaSO₄: Dy, P is known for its dosimetric characteristics for a very long time now. In this work we have carried out a comparative study on TL intensity of CaSO₄: Dy, P, Ce phosphors using two different wet chemical synthesis routes. Nobody seems to have reported this kind of study on CaSO₄: Dy, P, Ce³⁺ phosphors and as a result it may be regarded as something new. Phosphors were characterized by scanning electron microscopy, photoluminescence and thermoluminescence techniques. The two methods were found to drastically affect the TL intensity of CaSO₄: Dy, P, Ce phosphors. The TL intensity of CaSO₄: Dy, P, Ce prepared by co-precipitation method is less by a factor of 204 than the CaSO₄: Dy, P, Ce phosphor prepared via acid evaporation method. Moreover, co-doping of P and Ce ions into CaSO₄:Dy was observed to enhance the thermoluminescence intensity by a factor of 1.11 than the standard phosphor in case of phosphor prepared via acid evaporation method. Surface morphology resulted through two different synthesis routes was also studied. Structures like broad metal slabs were observed for CaSO₄: Dy, P, Ce phosphors using acid evaporation method. We hope that this study may prove very helpful in selecting a preparation method for CaSO₄: Dy, RE phosphors especially when better TL properties and good crystal grain size are desired. We also think that acid evaporation method is always a better choice to prepare CaSO₄: Dy, RE phosphors than co-precipitation method. Copyright © 2013 VBRI press.

Keywords: Synthesis routes; thermoluminescence intensity; phosphor; dosimetry.



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Introduction

Considerable work has been done on singly doped CaSO_4 phosphors in the last few decades. Rare earths, especially Dy or Tm doped CaSO_4 thermoluminescent phosphors are highly attractive due to their desired dosimetric properties. [1-5]. CaSO_4 : Dy is a phosphor first prepared and introduced by Yamashita et al. for use in thermoluminescence dosimetry (TLD) of ionizing radiations [6]. This phosphor finds applications because of its high sensitivity and ease of preparation. Several efforts have been done to enhance the sensitivity of this phosphor by co-doping. However, it has been found a little difficult to find the impurity which would sensitize the thermoluminescence of CaSO_4 : Dy. Prockic is believed to have made a phosphor which is 20% more sensitive than CaSO_4 : Dy but he has not disclosed the information about the second dopant [7]. Mori and co-workers have investigated the thermoluminescence produced when calcium sulfate co-doped with Eu^{3+} and Pr^{3+} (CaSO_4 : Eu^{3+} , Pr^{3+}) is irradiated with X-rays [8]. Recently Moharil et.al, have reported solid state metathesis of Eu^{2+} doped CaSO_4 phosphor [9]. Since Dy acts as an emission centre in CaSO_4 : Dy, efforts for increasing the luminescence of Dy^{3+} ion may lead to a phosphor with increased TL sensitivity as well. Several ways of increasing the Dy^{3+} luminescence are known, e.g. Ce^{3+} is known to sensitize the luminescence of Dy^{3+} . Another way is to increase the concentration of Dy^{3+} itself. However, above 0.2 mol% Dy^{3+} luminescence in CaSO_4 , is concentration quenched. Bakshi et.al studied the effect of Bi^{3+} co-doping in CaSO_4 : Dy and they found that Bi^{3+} acts as quencher of dosimetric peak in CaSO_4 : Dy, Bi^{3+} phosphor [10]. By using a charge compensator like Li, Na or K, one may push this limit by dissolving more Dy^{3+} in the CaSO_4 lattice [11]. Incorporation of these impurities unfortunately causes unfavourable changes in the glow curves. An alternative way is to introduce a charge compensator like P substitutionally on S sites. We have made experiments along these lines and come up with a CaSO_4 : P, Dy phosphor which has sensitivity 50% more than that of CaSO_4 : Dy, and a glow curve and other characteristics similar to those of CaSO_4 : Dy [12].

In the present study we have adopted two wet chemical methods and attempted to study their influence on thermoluminescence intensity CaSO_4 : P, Dy, Ce phosphors. A marking difference in thermoluminescence (TL) intensity of CaSO_4 : Dy, P, Ce phosphors was observed using the two wet chemical methods. This can be seen from the figures 3 and 5 of CaSO_4 : Dy, P, Ce phosphors. Besides, co-doping P^{5+} and Ce^{3+} impurities into CaSO_4 : Dy^{3+} phosphors, an attempt was made to take a brief look of surface morphology as well. By co-doping P^{5+} and Ce^{3+} impurities together into CaSO_4 : Dy^{3+} phosphors, the thermoluminescence intensity was found to increase by a factor of 1.11 than the standard phosphor. Since thermoluminescence results were found to be better in case of acid evaporation method than the co-precipitation method. Hence it is always better to synthesize CaSO_4 : P, Dy, Ce phosphors through the acid evaporation method than the co-precipitation method. Nobody seems to have reported this kind of study on CaSO_4 : Dy, P, Ce^{3+} phosphors and as a result it may be regarded as something new.

Experimental

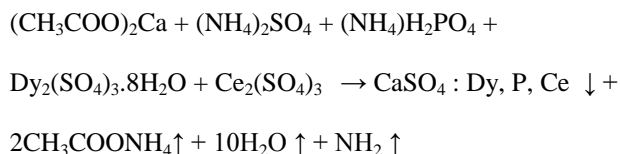
Materials and methods

The starting materials we used are $(\text{CH}_3\text{COO})_2\text{Ca}$ (Qualigens Excel R 98.5%), $(\text{NH}_4)_2\text{H}_2\text{PO}_4$ (Merck GR-99%), $(\text{NH}_4)_2\text{SO}_4$ (Merck GR-99.5%), $\text{Dy}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$ (Sigma Aldrich AR-99.99%), $\text{Ce}_2(\text{SO}_4)_3$ (Sigma Aldrich AR-99.99%), CeO_2 (S.D.Fine-99%), Dy_2O_3 (S.D.Fine-99%), Sulphuric Acid (S.D.Fine AR-98%) $\text{Ce}_2(\text{SO}_4)_3$ (Sigma Aldrich AR-99.99%) and Ethanol (Changshu Yanguan Chemical AR- 99.9%).

To study this phosphor, the characterizations we have carried out are Photoluminescence, Thermoluminescence and Scanning electronmicroscopy. The photoluminescence (PL) emission & excitation spectra of the samples were recorded by using a RF-5301PC SHIMADZU Spectrofluorophotometer. The same amount of sample was used in each case. Emission and excitation spectra were recorded using a spectral slit width of 1.5 nm. A γ -source of ^{60}Co was used for irradiation of the samples. TL measurements were carried out with the Nucleonix (TL – 1009) Reader. The same amount of sample in the form of fine powder was heated each time at a rate of 5°C/s . For comparison glow curve of commercially available CaSO_4 : Dy TLD phosphor was also recorded under identical conditions. SEM micrographs were obtained using JEOL, 6380A scanning electronmicroscope.

Co-precipitation method

For the preparation of CaSO_4 : P, Dy, Ce phosphors, all starting materials used were of analytical grade. Calcium acetate, dysprosium sulfate, cerium sulfate, ammonium dihydrogenphosphate and ammonium sulphate in appropriate quantities was dissolved separately in double distilled water. These individual solutions were stirred vigorously for 30 minutes on a magnetic stirrer so that homogenous solution was formed. Then these individual solutions were added, except ammonium sulphate solution, in one beaker to form a single solution. The solution was again stirred for half an hour on a magnetic stirrer. The solution was then mixed with ammonium sulfate solution in the presence of ethanol. The precipitate was filtered out and washed several times with double distilled water. The powder samples thus obtained were dried at 353K in an oven for few hours and further annealed at 973K for 1 h followed by quenching. In this way the phosphors were prepared. For doping with phosphorus, $\text{NH}_4\text{H}_2\text{PO}_4$ was used. The concentration of P used was 1 mol %. The concentration of Dy^{3+} used was 0.1 mol % but the concentration of other rare earth ion(s) was varied from 0.05 mol % to 0.5 mol %. The maximum thermoluminescence (TL) intensity was observed for the composition 1 mol % P, 0.1 mol % Dy^{3+} and 0.1 mol % Ce^{3+} . Chemical reaction is as follows,



Acid evaporation method

In order to prepare CaSO₄: P, Dy, Ce phosphors all starting materials used were of analytical grade. CaSO₄: P, Dy, Ce samples were prepared by dissolving the desired starting materials in hot H₂SO₄. In addition to distilling out acid vapours were subjected to condensation process for some time. After the completion of process, the solution was allowed to cool to room temperature. Finally the cooled solution was subjected to centrifuge. The sample phosphors thus prepared were repeatedly washed with double distilled water to remove the traces of acid. The sample phosphors were then dried at 353 K in an oven for few hours. In this way the phosphors were prepared. Some samples were annealed at various temperatures up to 1073K for 1 h following this preparation. However, annealing at 973 K was found to be suitable for obtaining high thermoluminescence (TL) intensities. These were used in the further experiments. For doping with phosphorus, NH₄H₂PO₄ was added to the sulphuric acid. The concentration of P used was 1 mol %. The concentration of Dy³⁺ used was 0.1 mol % but the concentration of other rare earth ion(s) was varied from 0.05 mol % to 0.5 mol %. The maximum thermoluminescence (TL) intensity was observed for the composition 1 mol % P, 0.1 mol % Dy³⁺ and 0.1 mol % Ce³⁺. Chemical reaction is as follows,

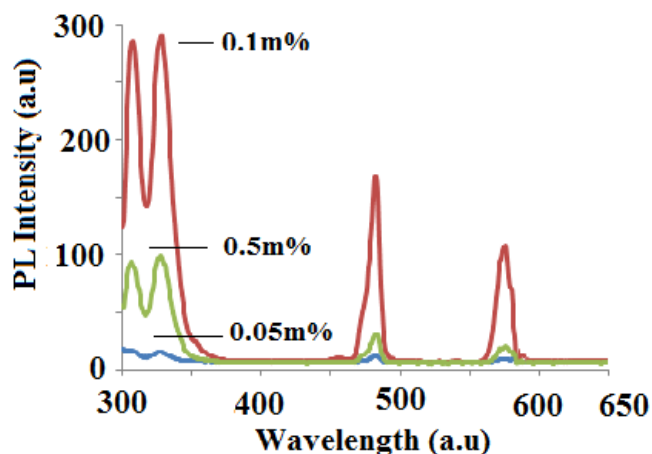
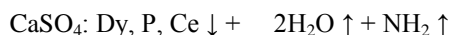
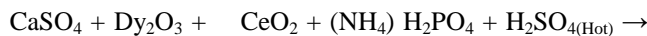


Fig. 1. PL emission spectra of CaSO₄: Dy, P,Ce (P=1 mol%;Dy=0.1 mol%; Ce=0.05,0.1,0.5 mol %) prepared via modified acid distillation method; emission wavelengths 309 nm,329 nm 482 nm & 574 nm under excitation wavelength 295nm.

Result and discussion*Photoluminescence (PL) characteristics*

PL measurements were carried out with RF-5301PC SHIMADZU Spectrofluorophotometer. At excitation wavelength 348 nm PL emission spectrum of CaSO₄: Dy, P, Ce, phosphors show the characteristic Dy³⁺ emission in the form of bands around 481 and 573 nm corresponding to transitions ⁴F_{9/2} → ⁶H_{15/2} and ⁴F_{9/2} → ⁶H_{13/2} respectively. Also under excitation wavelength 295 nm which is a characteristic of Ce³⁺ again we see the characteristics of Dy³⁺ but this time it is coupled with emission of Ce³⁺. This

means that there is a good overlapping between emission of Ce³⁺ and excitation of Dy³⁺ which in turn implies that certain level of energy transfer does take place when this kind of system is synthesized (**Fig. 1**). It is seen that the PL intensity has increased considerably when co-doped with Ce³⁺. Dy³⁺ excitation generally lies between 300 nm and 400 nm. The Ce³⁺ emission is around 309 and 329 nm which overlaps fairly well with the Dy³⁺ excitation. Ce³⁺ thus sensitizes [13-15] the Dy³⁺ emission in CaSO₄: Dy, P, Ce phosphors (**Fig. 1**).

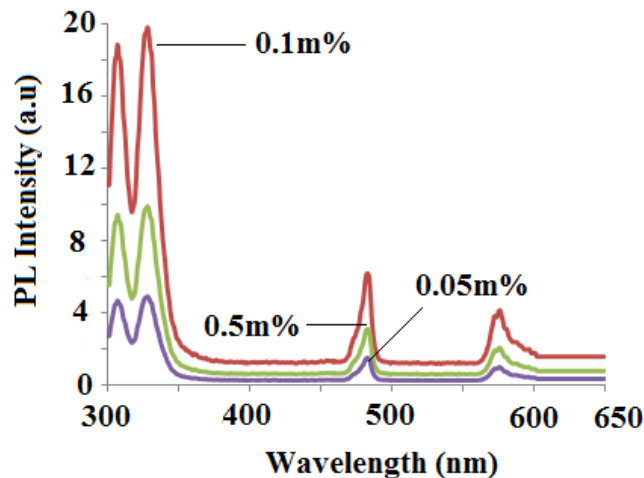


Fig. 2. PL emission spectra of CaSO₄: Dy, P,Ce(P=1mol%;Dy=0.1mol%;Ce=0.05,0.1,0.5mol%) prepared via chemical precipitation method; emission wavelengths 309 nm, 329 nm, 482 nm & 574 nm were observed under excitation wavelength 295 nm.

It thus shows that the incorporation of the impurities is in the desired i.e. Dy³⁺ and Ce³⁺ forms though the phosphor materials were prepared by two different techniques. It is to be noted here that photoluminescence (PL) intensity is poor in case of phosphor prepared via co-precipitation method (**Fig. 2**) as compared to phosphor prepared via modified acid evaporation method. There can be number of reasons why this trend occurs here. But the obvious one seems that it is method dependent. Hence it may be predicted here that dopants get incorporated with quite ease in CaSO₄: Dy, P, Ce phosphors through acid evaporation method than the co-precipitation method. Addition of trivalent ions such as Dy³⁺ and Ce³⁺ results in the introduction of additional charge which must be compensated, e.g. by creation of cation vacancies. Alternatively, phosphorus which replaces hexavalent S ions can be used as a charge compensator [13]. Concentration of phosphorus used was 1 mol%. Since for 1mol% phosphorus results were found to be better so we used only this concentration in our present experiments.

Thermoluminescence (TL) characteristics

Fig. 3 and **4** show the TL glow curves of the two phosphor materials exposed to gamma radiations from ⁶⁰Co source at room temperature. Thermoluminescence (TL) glow curves of the two phosphor materials was recorded using Nucleonix TLD reader. The TL characteristics of the two as prepared phosphors are slightly different from each other. For example high temperature peaks such as around 220 - 250 °C and the one above 300 °C are totally suppressed in the phosphor prepared via co-precipitation method. Also in

the case of phosphor prepared via modified acid evaporation method the temperature peak above 300 °C is absent completely. The TL intensity in the case of phosphor prepared via co-precipitation method is very less as compared with standard CaSO₄: Dy TLD phosphor.

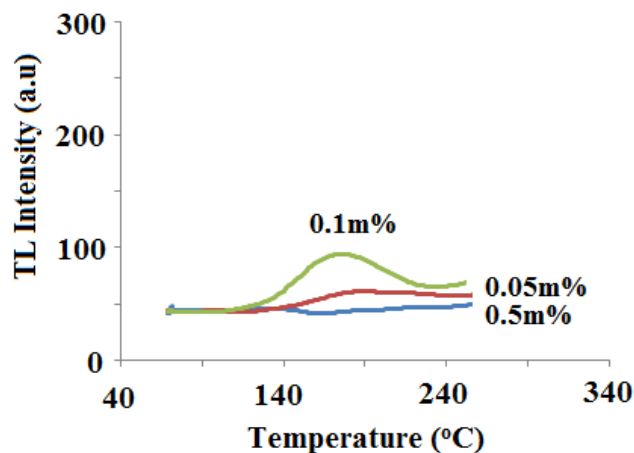


Fig. 3. TL glow curves of CaSO₄:Dy,P,Ce (P=1mol%;Dy=0.1mol%;Ce=0.05,0.1,0.5mol%) exposed to gamma radiations of ⁶⁰Co source prepared via chemical precipitation method.

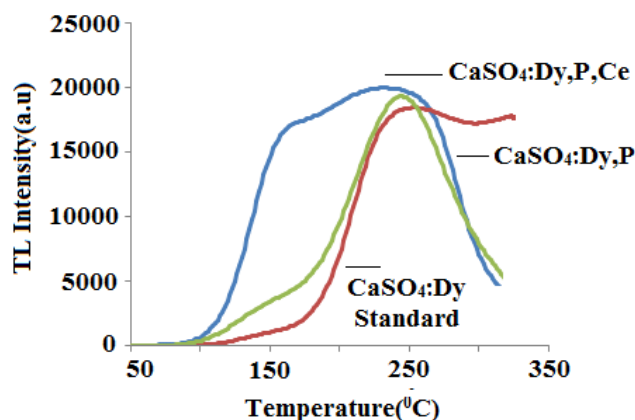


Fig. 4. TL glow curves of CaSO₄:Dy,P,Ce (P=1mol%;Dy=0.1mol%;Ce=0.05,0.1,0.5mol%) phosphors.

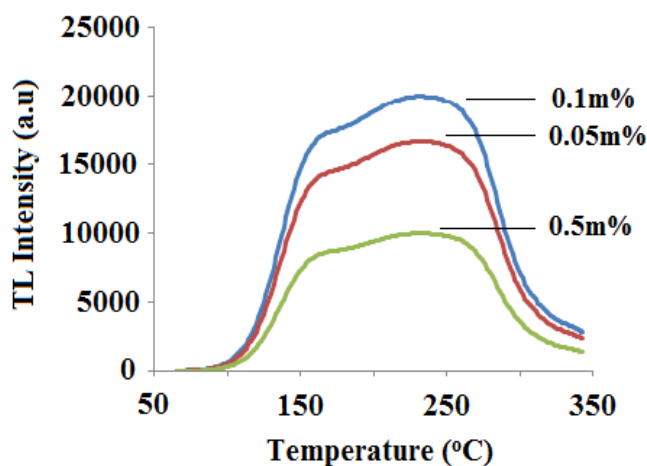


Fig. 5. Comparison for glow curves of standard CaSO₄:Dy TLD phosphor & CaSO₄:Dy,P;CaSO₄:Dy,P,Ce (P=1mol%; Dy=0.1mol%; Ce=0.1mol%) phosphors prepared via modified acid distillation method. The three phosphor materials were given gamma exposure of ⁶⁰Co source.

Moreover, the TL intensity of the phosphor material prepared by modified acid evaporation method is 1.11 times greater than the standard CaSO₄: Dy TLD phosphor (Fig. 5). This is really significant. It is well known that contribution to thermoluminescence (TL) from intrinsic defects present in the CaSO₄ lattice is enhanced by the addition of rare-earths. But some recent literature suggests that change in the surface morphology of the bulk material does affect the thermoluminescence (TL) [16-21] of the phosphor material. However, the phenomenon of thermoluminescence is quite perplexed and therefore a great deal of study is required to understand this process.

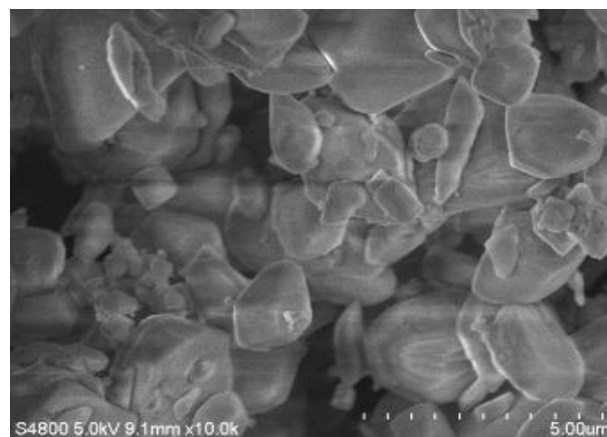


Fig. 6. SEM images of CaSO₄: Dy, P, Ce phosphor prepared via chemical precipitation method.

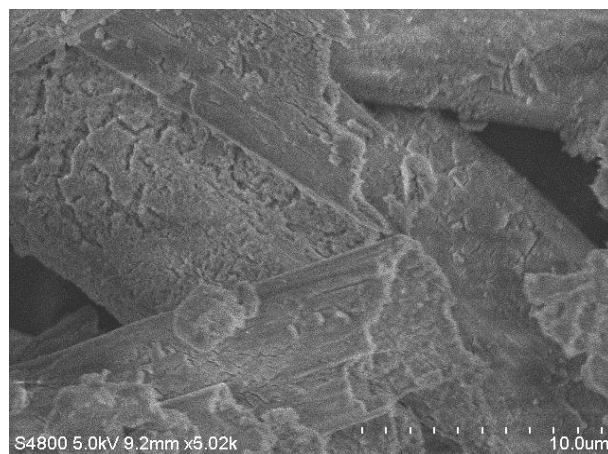


Fig. 7. SEM images of CaSO₄: Dy, P, Ce prepared via modified acid distillation method.

SEM patterns of CaSO₄: Dy, P, Ce prepared using different synthesis routes

As per the literature survey, the role of morphology and particle size in TL is important. It is known that phosphor having a particle size in the micron range is regarded as a good TL material. No doubt TL phosphors with particle size in the micron range are very sensitive and have a linear dose-response at low doses but they have a drawback of dose saturation at high doses. This is contrary to the case in nano TL phosphors, which are likely insensitive at low doses but show a good linear dose response at high doses

(i.e., dose saturation limit is higher than microcrystalline TL phosphors) [16-21]. However, the TL phenomenon is quite a complicated process and a great deal of study is further needed to understand this process. From the micrograph (Fig. 6) it can be seen that particles have shaped into triangular and prismatic forms in the case of phosphor $\text{CaSO}_4: \text{Dy}, \text{P}, \text{Ce}$ prepared by co-precipitation method. Also it is obvious from the images that each large particle is surrounded by a number of small particles indicating that there is likely some regular coordination amongst atoms in the material. However, in the case of phosphor $\text{CaSO}_4: \text{Dy}, \text{P}, \text{Ce}$ synthesized through modified acid evaporation method particles have grown like broad metal slabs with appreciable diameter (Fig. 7). This is presumably different from the morphological characteristics of $\text{CaSO}_4: \text{Dy}, \text{P}, \text{Ce}$ phosphor prepared via co-precipitation method. The particle size in each case falls in the micrometer range which is considered very favourable for thermoluminescence characteristics of a phosphor material. Nano size materials also find other applications [22-24].

Conclusion

Adoption of two methods not only influences the thermoluminescence (TL) intensity but also causes change in position of dosimetric peaks. For example suppression of high temperature peaks takes place preferentially in $\text{CaSO}_4: \text{Dy}, \text{P}, \text{Ce}$ phosphor prepared via co-precipitation method. Likewise, suppression of temperature peak above 300 °C occurs in $\text{CaSO}_4: \text{Dy}, \text{P}, \text{Ce}$ phosphor prepared via modified acid evaporation method. Slab like structures of $\text{CaSO}_4: \text{Dy}, \text{P}, \text{Ce}$ can be obtained by employing modified acid evaporation method. Co-doping of Ce^{3+} in $\text{CaSO}_4: \text{Dy}, \text{P}$ phosphor increases with the TL intensity by a factor of approximately 1.11 and PL intensity by a factor of 3. These results have been attributed to the energy transfer from Ce^{3+} to Dy^{3+} ion. With the co-precipitation method, even though, both PL and TL were observed but were not as good as obtained by modified acid evaporation method.

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