Research Article

Influence of Rare Earth Doping on Microstructure and Luminescence Behaviour of Sodium Sulphate

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Na$_2$SO$_4$, Na$_2$SO$_4$:Li, and Na$_2$SO$_4$:Li, Eu, Dy phosphors were prepared by using slow evaporation technique followed by subsequent calcination at 400°C for 4 h. Doping with Li$^+$ ion stabilized the thenardite phase of host matrix, while codoping with RE$^{3+}$ stabilized the phase transformation from stable thenardite to metastable mirabilite crystal structure. The microstructure and morphology were studied by using scanning electron microscopy and transmission electron microscopy. The thermoluminescence studies revealed that isovalent doping of Li$^+$ served as a quencher and addition of codopant introduces the additional trap sites in the host matrix. The room temperature emission spectra of Li-doped, RE$^{3+}$-codoped, and undoped Na$_2$SO$_4$ were studied under ultraviolet radiation. For pure Na$_2$SO$_4$, the two peaks which appeared are at 364 and 702 nm, respectively. The emission intensities of RE$^{3+}$-codoped samples increase with increase in dopant concentration.

1. Introduction

Alkali sulphates have been known for a long time as versatile and excellent phosphor materials. These sulphates have attracted the attention of many workers in view of their potential applications in radiation dosimetry, TV screens, cathode ray tubes, and so forth. A variety of defect centres are likely to be formed in sulphate based phosphors [1–6]. Sulphate based radiation dosimeter materials doped with rare earth (RE) ions have been extensively investigated due to their high luminescence sensitivity [7]. Significant advancements have been made in thermoluminescence (TL) and photoluminescence (PL) experiments during the last couple of decades [8–10]. Up to date, sodium sulphate is extensively investigated from the prospect of phosphor material attributed to its simple chemical composition and defect rich crystal. Na$_2$SO$_4$ exhibits a variety of phase transitions between its five anhydrous polymorphs (labelled I–V). The phase transformation sequence among the Na$_2$SO$_4$ polymorphs can be described as

\[ \text{V} \xrightleftharpoons{473 \text{ K}} \text{ III} \xrightleftharpoons{503 \text{ K}} \text{ II} \xrightleftharpoons{510 \text{ K}} \text{ I} \xrightleftharpoons{1156 \text{ K}} \text{ Melt (I)} \]

Na$_2$SO$_4$ forms two naturally occurring minerals: mirabilite (Na$_2$SO$_4$·10H$_2$O) and thenardite (Na$_2$SO$_4$). Both are in thermodynamic equilibrium at 32°C which may be lowered to 18°C in the presence of foreign ions [II]. At room temperature, phase V (thenardite) is reported to be stable while phase III is metastable. Phases I and II are high-temperature polymorphs; however, phase II is reported to have a narrow stability zone. Phase IV is considered to be metastable and its phase relation and structure have yet to be well established [12–15]. Correcher et al. [16] observed the spectra of infrared-stimulated luminescence (IRSL), radioluminescence (RL), and TL of thenardite. Sidike et al. [17] studied the photoluminescence (PL) spectra, excitation spectra, and decay curves of natural, heat-treated, and γ-irradiated thenardite from Ai-Diang salt lake and concluded that crystal defects were responsible for observed luminescence. Reliable studies on the PL and TL properties of thenardite are very few till to date, to the best of our knowledge. In order to develop new dosimetric materials and to obtain a better understanding of the physical mechanism of radiation effects, Na$_2$SO$_4$ is doped and codoped with activators.

In the present work, fading TL and PL behaviour of pure and RE$^{3+}$-codoped Na$_2$SO$_4$ matrix are explained in...
Table 1: The crystallite size and stress factor of pure, doped, and codoped Na₂SO₄ samples.

| Phosphor                  | Crystallite size (nm) | FWHM values (rad) | d spacing (Å) | 2θ (degrees) | Stress factor (ε) x10⁻¹ |
|---------------------------|-----------------------|-------------------|---------------|--------------|------------------------|
| Na₂SO₄                   | 55                    | —                 | 0.29          | 1.43         | — 1.2                  |
| Na₂SO₄ : Li₀.₅%           | 55                    | 0.37              | 1.43          | 32.4         | 2                      |
| Na₂SO₄ : Li₀.₅%, Eu₀.₅%, Dy₀.₂% | 59                   | 0.14              | 0.083         | 2.77         | 2.1                    |
| Na₂SO₄ : Li₀.₅%, Eu₀.₅%, Dy₀.₅% | 39                 | 0.21              | 0.100         | 2.77         | 1.8                    |

T and M correspond to the thenardite and mirabilite content of Na₂SO₄.

detail. The host matrix shows the existence of birefringent phase after codoping with hypervalent ion. The codoped and pure Na₂SO₄ samples have been well characterized by powder X-ray diffraction (PXRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) techniques.

2. Experimental

2.1. Materials Preparation. Na₂SO₄ and Li₂SO₄ (0.5 mol%) were stoichiometrically dissolved in double-distilled deionized water (A). A known quantity of RE₂O₃ (RE = Eu and Dy) was dissolved in concentrated H₂SO₄ (B) and added to solution (A) to get RE/Na mole ratio in the concentration range of 0.08 to 0.5 mol%. The solution is then dried in an oven. The sample thus obtained in powder form was crushed and calcined at 400°C for 4 h in furnace. After natural cooling to RT, it is crushed to fine powder and pressed into pellets (80 kg/cm²).

2.2. Material Characterization. The PXRD pattern of sample is obtained using Philips PW/1050/70/76 X-ray diffractometer which was operated at 30 KV and 20 mA using CuKα radiation as the source with a nickel filter at a scan rate of 2/°min. The size, shape, and distribution of the grains were examined by SEM analysis using Quanta 200 FEI SEM. TEM measurement was carried out using a Philips CM 200 microscope having 2.4 Å resolutions.

TL glow curves were measured with system in the temperature range from 25 to 300°C operating with linear heating rates of 5 Ks⁻¹. Prior to the TL measurements, samples (pellets of 1 mm thickness with 5 mm in diameter are used for TL measurements) were exposed to γ irradiation from a ⁶⁰Co source to a total dose of 0.02 to 2 kGy at the sample cavity. Photoluminescence emission and excitation spectra were registered in identical experimental conditions, using Yvon Fluorometer, Jobsspectrometer with Xenon lamp source at an excitation wavelength of 340 nm at SAIF, IIT, Chennai, India.

3. Results and Discussion

3.1. Powder X-ray Diffraction Analysis. Figure 1 shows the PXRD of Na₂SO₄, Na₂SO₄ : Li₀.₅%, and Na₂SO₄ : Li₀.₅%, Eu₀.₅%, Dyₓₐ₅% (x = 0.2, 0.5). The undoped and Li⁺-doped Na₂SO₄ exhibited stable thenardite, while codoping of RE³⁺ to Na₂SO₄ resulted in the phase transformation from stable thenardite to metastable mirabilite phase. The mirabilite content in the sample showed critical dependence on RE³⁺ concentration.

The average crystallite size (D) and matrix distortion (ε) are calculated from FWHM of the most intense PXRD peak using the following, respectively [18, 19]:

\[ D = \frac{0.9 \lambda}{\beta \cos \theta}, \quad (2) \]
\[ \beta = 4 \epsilon \tan \theta, \quad (3) \]

where “D” is the average grain size of the crystallites, “λ” is the incident wavelength, “θ” is the Bragg angle, “ε” is the stress factor, and “β” is the diffracted full width at half maximum (in radians) caused by the crystallites.

The higher ionic radius of the RE³⁺ ion can possibly act as an interstitial impurity in the host matrix. The introduction of substitutional dopant metal ions with a higher ionic size would induce cationic vacancies at the surface of thenardite grains, which favours the bond rupture, ionic rearrangement, and structure reorganization for the formation of mirabilite phase. The thenardite to mirabilite phase is generally considered as a nucleation growth process during which the mirabilite nuclei are formed within the thenardite phase. The thenardite grain size decreases with an increase in the Dy³⁺ concentration (Table 1).
Table 2: Comparison of lattice parameters and cell volume of pure, Li-doped, and RE$^{3+}$-codoped Na$_2$SO$_4$ samples.

| Phosphor                      | $a$ (Å) | $b$ (Å) | $c$ (Å) | $V$ (Å$^3$) |
|-------------------------------|--------|--------|--------|-----------|
| Na$_2$SO$_4$                 | 5.80   | —      | 12.20  | 7.60      | 538       |
| Na$_2$SO$_4$:Li$_{0.5\%}$    | 5.86   | —      | 12.32  | 7.66      | 553       |
| Na$_2$SO$_4$:Li$_{0.5\%}$, Eu$_{0.5\%}$, Dy$_{0.2\%}$ | 10.7   | 9.51   | 10.43  | 15.6      | 6.08      | 3.452     | 679       | 512       |
| Na$_2$SO$_4$:Li$_{0.5\%}$, Eu$_{0.5\%}$, Dy$_{0.5\%}$ | 11.0   | 9.55   | 10.47  | 15.8      | 6.12      | 3.481     | 705       | 525       |

T and M correspond to thenardite and mirabilite content of Na$_2$SO$_4$.

With an increase in the Dy$^{3+}$ content, the variation in the lattice parameter was reflected in the elongation of the $c$-axis (Table 2). Since only the dimension changes while $a$-axis and $b$-axis remain almost constant for the range of dopant concentration, it can be concluded that with increase in Dy$^{3+}$ concentration the crystal growth proceeds along $c$-axis. The extent of matrix distortion was nearly ~200 times higher for doped samples compared to bare samples due to the higher ionic radius of RE$^{3+}$ compared to host Na$^+$. It is well known that the incorporation of RE$^{3+}$ ions within the host crystal introduces lattice strains as a result of size mismatch.

Due to smaller ionic radius, the stress associated with Li$^+$ is dominated by lattice contraction. It is interesting to note that stress factor associated with Li$^+$ doping (lattice contraction) is larger compared to RE$^{3+}$ doping (lattice expansion).

3.2. Scanning Electron Microscopy Studies. The surface morphological features of the Na$_2$SO$_4$, Na$_2$SO$_4$: Li$_{0.5\%}$, and Na$_2$SO$_4$: Li$_{0.5\%}$, Eu$_{0.5\%}$, Dy$_{0.5\%}$ samples are shown in Figure 2. The pure, Li$^+$-doped, and RE$^{3+}$-codoped Na$_2$SO$_4$ showed the uniform distribution of generalized hexagonal habit of grains corresponding to orthorhombic prisms and pyramids without any agglomeration. The doping leads to noticeable changes on the grain size of the sample.

3.3. Transmission Electron Microscopy Studies. The shape and size of these particles were also determined by TEM (Figures 3 and 4). TEM images and selected area electron diffraction (SAED) of Na$_2$SO$_4$ and Na$_2$SO$_4$: Li$_{0.5\%}$ showed that the crystals are hexagonal-shaped particles with almost no amorphous constituents and are weakly aggregated. SAED patterns of these samples were neither regular diffraction
Figure 3: (a) TEM image and (b) selected area electron diffractions of Na$_2$SO$_4$.

spots nor whole diffraction rings, indicating that the number of polycrystalline grains in the selected area was finite.

Results revealed that, in pure Na$_2$SO$_4$, these spots might belong to the $d$-values of 3.636, 3.18, 3.07, and 2.78 Å which corresponds to (200), (131), (040), and (311) planes of thenardite. In Na$_2$SO$_4$: Li$^+$, Eu$^{3+}$, Dy$^{3+}$ these spots correspond to $d$-values 4.166, 3.77, 2.85, and 2.01 Å which correspond to (112), (220), (−402), and (151) planes. Among them (220) and (151) belong to thenardite and (112) and (−402) belong to mirabilite content of Na$_2$SO$_4$.

3.4. Thermoluminescence Studies

3.4.1. TL Studies of Na$_2$SO$_4$ and Na$_2$SO$_4$: Li$^+$, Eu$^{3+}$, Dy$^{3+}$. TL property of host sample was investigated at different γ-ray dose levels. The pristine orthorhombic phosphor has only one glow main peak at ~180°C. The intensity of these glow peaks increases with irradiation and shifts towards higher temperature region (Figure 5).

In Na$_2$SO$_4$: Li$_{0.5}$%, Li$^+$ quenches the TL intensity of Na$_2$SO$_4$ and shifts the TL glow curves position towards lower temperature (Figure 6).

3.4.2. TL Studies of Na$_2$SO$_4$: Li$^+$, Eu$^{3+}$, Dy$^{3+}$. The glow curve of Na$_2$SO$_4$: Li$_{0.5}$%, Eu$_{3.5}$%, Dy$_{3.5}$% consists of a single glow peak ranging from 382 to 424 K (Figure 7). The TL intensity increases with increase in the γ-ray exposure and lies between that of Na$_2$SO$_4$: Li$^{3+}$%, Eu$_{3.5}$% and that of Na$_2$SO$_4$: Li$_{0.5}$%, Dy$_{3.5}$%. This suggests that an increasing number of traps responsible for these glow peaks were getting filled with an increase in radiation dose. These traps release the charge carriers on thermal stimulation to finally recombine with their counterparts, thus giving rise to different glow peaks. However, the TL intensity still remains higher compared to host sample.

3.4.3. Calculation of Kinetic Parameters. The trap parameters, such as activation energy $E$ and order of kinetics $b$, were calculated for the different concentration TL glow peak of the Na$_2$SO$_4$: Li$_{0.5}$% and Na$_2$SO$_4$: Li$_{3.5}$%, Eu$_{3.5}$%, Dy$_{3.5}$% ($x = 0.1, 0.2,$ and $0.5$) phosphor irradiated with a gamma dose of 2 kGy at RT using Chen’s set of empirical formulae for the shape of glow curve (Figure 8) [20].

In the present study, geometrical form factor value is very close to 0.52, suggesting that the TL emission involves retrapping of charges. Table 3 lists the peak parameters as obtained for deconvoluted peaks for different RE$^{3+}$ concentration

$$E_\alpha = C_\alpha \left( \frac{kT_m^2}{\alpha} \right) - b_\alpha \left( 2kT_m \right),$$

where

$\alpha = \omega, \tau, \delta, \omega = T_2 - T_1, \delta = T_2 - T_m, \tau = T_m - T_1,$

$C_\omega = 1.51 + 3.0 \left( \mu_g - 0.42 \right)$;

$b_\omega = 1.58 + 4.2 \left( \mu_g - 0.42 \right)$;

$C_\delta = 0.976 + 7.3 \left( \mu_g - 0.42 \right)$;

$b_\delta = 0$;

$C_\tau = 2.52 + 10.2 \left( \mu_g - 0.42 \right)$;

$b_\tau = 1$.

$$s = \left( \frac{\beta E}{kT_m^2} \right) \left( \frac{\exp \left( E/kT_m \right)}{1 + (b - 1) \left( 2kT_m/E \right)} \right),$$

where $k$ is the Boltzmann constant $= 8.6 \times 10^{-5}$ eVK$^{-1}$.

3.4.4. TL Fading. The effect of fading was studied up to 90 days by irradiating samples with a gamma dose of 2 kGy. The extent of fading was maximum for Na$_2$SO$_4$ compared to codoped Na$_2$SO$_4$, suggesting that introduced by addition trap sites favouring the intensification of TL signal (Figure 9). About 1.4%, 1.16%, and 1.19% of fading were observed for Na$_2$SO$_4$, Na$_2$SO$_4$: Li$_{3.5}$%, and Na$_2$SO$_4$: Li$_{0.5}$%, Eu$_{0.5}$%, Dy$_{0.5}$%, respectively, during the first five days, but thereafter the intensity remained almost constant up to 20 days; afterwards fading becomes maximum. However, it is suggested that the irradiation of nanocrystalline phosphor at high doses results in the formation of deep traps which resulted in the fading of TL signal.
Figure 4: (a) TEM images and (b) selected area electron diffraction of Na$_2$SO$_4$: Li, Eu, Dy.

Table 3: Comparison of kinetic parameters for pure, Li-doped, and RE$^{3+}$-codoped Na$_2$SO$_4$ samples.

| Phosphor                        | Activation energy (eV) | Frequency factor ($s^{-1}$) |
|---------------------------------|------------------------|-----------------------------|
| Na$_2$SO$_4$                    | 0.15                   | $4.59 \times 10^9$          |
| Na$_2$SO$_4$: Li$_{0.5\%}$     | 0.35                   | $5.51 \times 10^{13}$       |
| Na$_2$SO$_4$: Li$_{0.5\%}$, Eu$_{0.5\%}$, Dy$_{0.1\%}$ | 0.18                   | $1.41 \times 10^{11}$       |
| Na$_2$SO$_4$: Li$_{0.5\%}$, Eu$_{0.5\%}$, Dy$_{0.2\%}$ | 0.19                   | $1.20 \times 10^{11}$       |
| Na$_2$SO$_4$: Li$_{0.5\%}$, Eu$_{0.5\%}$, Dy$_{0.5\%}$ | 0.20                   | $1.51 \times 10^{12}$       |

Figure 5: TL glow curves of $\gamma$-irradiated Na$_2$SO$_4$ calcined at 400°C.

3.5. Photoluminescence Studies

3.5.1. PL Studies of Na$_2$SO$_4$ and Na$_2$SO$_4$: Li$^{+}$. The PL emission spectra of host sample consist of a strong peak at 364 nm for pure thenardite and are attributed to band-to-band PL phenomenon due to the band gap excitation (Figure 10).

In addition, low intense peak at $\sim$702 nm is attributed to excitonic PL arising from intrinsic defects of thenardite Na$_2$SO$_4$ powders. The emission band with the most intense peak at approximately 702 nm showed a distinct vibronic structure. The vibronic structure characteristic of S$^{2-}$ centre was observed in the red region.

The $\gamma$-ray irradiated thenardite did not change the feature of PL but reduced the luminescence efficiency. This suggests that $\gamma$-ray irradiation of thenardite may generate a killer centre for luminescence in thenardite. We could not find any dip due to absorption by the killer centre. It seems that $\gamma$-ray irradiation has an annealing effect although mechanism was not yet clear.
The PL emission spectra of Na$_2$SO$_4$: Li$_{0.5\%}$ are the same as that of the host sample (Figure 11).

3.5.2. PL Studies of Na$_2$SO$_4$: Li$^\text{+}$, Eu$^{3\text{+}}$. The PL emission spectra of Na$_2$SO$_4$: Li$^\text{+}$, Eu$^{3\text{+}}$, Dy$^{3\text{+}}$ for 340 nm excitation showed bands at 481, 490, 575, and 711 nm. The first three peaks are assigned to Dy$^{3\text{+}}$ and the peak arising at 711 nm was assigned to Eu$^{3\text{+}}$ emission (Figure 12).

4. Conclusions

Pure and codoped phosphors have been prepared at RT by slow evaporation technique. PXRD pattern confirmed the phase transformation from stable thenardite to metastable mirabilite after codoping with RE$^{3\text{+}}$ ions. SEM studies showed the presence of uniform distribution of prisms and pyramids. The codopant introduces additional trap sites favouring the intensification of TL signal. Therefore, up to a given irradiation dose, this phosphor has potential candidate for use in radiation dosimetry. The PL intensities of codoped Na$_2$SO$_4$ samples were found to be dependent on the RE$^{3\text{+}}$ concentration.
Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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