6.0 Introduction
SrS:Eu and SrS:Ce are wildly studied phosphors to get white light from blue LED, when excited with 450-470nm light, these phosphors emit Luminescence in the range of 550-775nm peaking around 600nm. The present chapter Strontium Sulfate phosphor doped with Eu (0.5 mol %), Ce (0.5 mol %), keeping constant and adding Ga, Tb, Dy and La as co-dopants (0.5 mol %) are prepared using the conventional solid state reaction method. Strontium Carbonate and powder Sulfur excesses along with Rare Earth Oxides are added in appropriate staciometric portion and grounded in mortar and pestle in 30 minuets, the received powder is loded in the alumina crucible, and carbon black powder is used as reducing agent. These materials are heated at 900°C for 90 minuets. The received powder phosphor looks pale orange, which is in fine powder form. The Photoluminescence (PL) emission and excitation studies are carried and the results are follows.

The following phosphors are synthesized and studied for their PL and other characterization and presented in this chapter-6

<table>
<thead>
<tr>
<th>S.No</th>
<th>Sample Name</th>
<th>Doping Concentrations (mol %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>SrS:Eu²⁺,Ce³⁺</td>
<td>0.5, 0.5.</td>
</tr>
<tr>
<td>2</td>
<td>SrS:Eu²⁺,Ce³⁺,Ga³⁺</td>
<td>0.5, 0.5, 0.5.</td>
</tr>
<tr>
<td>3</td>
<td>SrS:Eu²⁺,Ce³⁺,Tb³⁺</td>
<td>0.5, 0.5, 0.5.</td>
</tr>
<tr>
<td>4</td>
<td>SrS:Eu²⁺,Ce³⁺,Dy³⁺</td>
<td>0.5, 0.5, 0.5.</td>
</tr>
<tr>
<td>5</td>
<td>SrS:Eu²⁺,Ce³⁺,La³⁺</td>
<td>0.5, 0.5, 0.5.</td>
</tr>
</tbody>
</table>

6.1 Results and discussions of SrS: Eu, Ce phosphor

6.1.1. Photoluminescence Study of SrS: Eu, Ce phosphor

Fig-6.1A, 1B and 1C are the emission and excitation of the phosphor SrS:Eu(0.5 mol %), Ce(0.5 mol %). Fig-6.1A is the excitation spectrum monitoring at 600nm. From the figure it is found there are two excitation bands starting from 225 - 325nm, peaking around 265nm and second is from 400 - 570nm. The first band is UV region and second band is perfect blue green visible region, there is also another small absorption band of 365nm, the 365nm band is due to charge transfer transition of Ce³⁺ from 4f to conduction band. The excitation bands at 254nm and 460nm can be
assigned to the $e_g$ to $2t_{2g}$ 5d bands of Ce$^{3+}$. The broad excitation bands of the 600 nm are found at 265 nm and 540 nm, which can be attributed to the $(4f^7) e_g$ and $4f^6$ 5d$(t_{2g})$ field splitting 5d bands of Eu$^{2+}$ respectively.

Fig-6.1A: Excitation spectrum of SrS:Eu,Ce phosphor monitored at 600 nm

Fig-6.1B is the emission and excitation of SrS:Eu (0.5 mol %), Ce (0.5 mol %) phosphor, when excited with 254nm, 262nm, and 274nm, emissions are found at 365nm, 395nm and 467nm with less intensity along with a broad emission peaking at 600nm. The emission peak is found at 600 nm and the excitation bands for this emission observed at 290 nm and 460 nm. The excitation profile is modified from doped SrS: Eu$^{2+}$, Ce$^{3+}$ system. As excitation wavelength is increases 254nm and 274nm the emission intensity at 600nm increases from 129 to 320 units, which is 2.5 times more. Since energy of 254nm to 274nm, the energy transfer occurs between
Ce$^{3+}$ to Eu$^{2+}$, when excitation is from 450nm to 540nm, the emission intensity increased from 255 to 350 units, an increase of 40%. It is found from fig-6.1A, two absorption bands one is around 460nm to 480nm and another is 530nm to 540nm, this is due to dipole-dipole interaction and higher energy transition rate between Ce$^{3+}$ to Eu$^{2+}$. Emission intensity of 600nm peak verses excitation wavelength is presented in fig-6.1D, The 600nm emission corresponding to the $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition of Eu$^{2+}$, is broad emission range from 550-675nm. From the emission spectrum it was observed that the sample shows 600nm emission and the maximum emission intensity for 600nm peak is found for 540nm excitation wavelength.
Table 6.1

<table>
<thead>
<tr>
<th>S.No</th>
<th>Excitation Wavelength (nm)</th>
<th>600nm (2.066eV) emission intensity (arb.u)</th>
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<td>262</td>
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</tr>
<tr>
<td>8</td>
<td>540</td>
<td>349</td>
</tr>
</tbody>
</table>

Table 6.1 is the emission intensity of 600nm peak with different excitation wavelengths for SrS: Eu, Ce phosphor.
6.1.2 XRD analysis of SrS:Eu, Ce phosphor

The crystalline structure of the SrS: Eu$^{2+}$, Ce$^{3+}$ phosphor was analyzed by X-ray powder diffraction studies (XRD). Fig-6.1E is the XRD pattern of SrS: Eu$^{2+}$, Ce$^{3+}$ (0.5 mol %) phosphors of 20 range from 20° to 50°. The crystallite size is calculated using Scherrer’s formula d=K.λ/ β cosθ, where ‘K’ is the Scherer’s constant (0.94), ‘λ’ the wavelength of the X-ray (1.54060 Å), ‘β’ the full-width at half maxima (FWHM) (0.1178), ‘θ’ the Bragg angle of the XRD big peak, is found **69.13nm**.
6.1.3 SEM study of SrS: Eu, Ce phosphor

Fig-6.1F1 and 6.1F2 are SEM micrographs of the SrS: Eu$^{2+}$(0.5 mol %), Ce$^{3+}$ (0.5 mol %) phosphor for different magnifications. The particles looks agglomerated and irregular shapes having size of 1 micron to 6 micron are observed from the SEM graphs.
Fig-6.1F1:- SEM image of SrS:Eu, Ce (10.4 KX)

Fig-6.1F2:- SEM image of SrS:Eu, Ce (9.07 KX)
6.1.4 Particle size analysis of SrS: Eu, Ce (0.5 mol % of each)

The Particle size distribution histograms of the SrS:Eu$^{2+}$, Ce$^{3+}$ (0.5 mol %) phosphor particles as shown in the Fig-6.1G. The prepared phosphor specimen particle size was measured by using laser based system Malvern Instrument U.K. From the figure it is found the average particle size is 4.5 $\mu$m, the average surface area 1.341 M$^2$/gm.

![Particle size histogram of SrS:Eu(0.5 mol %), Ce(0.5 mol %)](image)

Fig-6.1G: Particle size histogram of SrS:Eu(0.5 mol %), Ce(0.5 mol %)
6.1.5 FTIR study of SrS:Eu, Ce phosphor

In order to determine the chemical bonds in a molecule FTIR analysis was carried out. Fig-6.1H is the FTIR of the SrS:Eu$^{2+}$ Ce$^{3+}$ (0.5 mol %) phosphor, the main absorption around 3400 are assumed H-O-H stretching followed by other bonds of Sr-S stretching, Sr-O stretching and CO-OH stretching. CO-OH and H-O-H stretching are due to absorbed CO$_2$, H$_2$O molecules from atmosphere.

![Fig-6.1H: FTIR spectrum of SrS:Eu, Ce Phosphor](image-url)

- Wavenumber (cm$^{-1}$): 4500, 4000, 3500, 3000, 2500, 2000, 1500, 1000, 500
- Transmittance (%): 110, 100, 90, 80, 70, 60, 50, 40, 30, 20, 10, 0
- Peaks: 4254.97, 3989.71, 3462.22, 2873.94, 2482.39, 2123.63, 1768.72, 1427.32, 1138, 962.84, 859.58, 705.95
6.2 Results and discussions of SrS: Eu, Ce, Ga phosphor

6.2.1 Photoluminescence Study of SrS: Eu, Ce, Ga phosphor

Fig-6.2A, 6.2B and 6.2C are the emission and excitation of the SrS:Eu(0.5 mol %), Ce(0.5 mol %), Ga(0.5 mol%) phosphor. Fig-6.2A is the excitation spectrum monitoring at 600nm. From the figure it is found there are two excitation bands starting from 225-325nm, peaking around 265nm and second is from 400 - 570nm. The first band is in UV region and second band is perfect blue green visible region, there is also another small absorption band of 365nm, the 365nm band is due to charge transfer transition of Ce$^{3+}$ from 4f to conduction band. The excitation bands at 254nm and 460nm can be assigned to the $e_g$ to $2t_g$ 5d bands of Ce$^{3+}$. The broad excitation bands of the 600 nm are found at 265 nm and 540 nm, which can be attributed to the $(4f^7) e_g$ and $4f^6 5d^1(t_{2g})$ field splitting 5d bands of Eu$^{2+}$ respectively.
Fig-6.2B is the emission and excitation of the SrS:Eu(0.5 mol %), Ce(0.5 mol %), Ga(0.5 mol%) phosphor, when the phosphor is excited with 254nm, 262nm, and 274nm, the emissions are found at 467nm with less intensity along with a broad emission peaking at 600nm. The emission peak is found at 600 nm and the excitation bands for this emission are observed at 290 nm and 460 nm. The excitation profile is modified from doped SrS: Eu^{2+}, Ce^{3+}, Ga^{3+} system.

As excitation wavelength is increased from 254 - 274nm, the emission intensity of 600nm peak increase from 105 to 215 units, which is around 2 times more. Since energy of 254nm is more when compared to 274nm, the resonance energy transfer occurs between Ce^{3+} to Eu^{2+}. When the excitation is from 450nm to 540nm, the emission intensity increases from 286 to 469 units, an increase of 80%. Two absorption bands are observed, one is around 460nm to 490nm and another is 530nm to 545nm, this is due to dipole-dipole interaction and resonance energy transition rate between Ce^{3+} to Eu^{2+}. Emission intensity of 600nm peak verses various excitation wavelengths are presented in fig-6.2D and table-6.2 for better comparison.

From figure 6.2D and table it is found that the emission intensity of 600nm peak gradually increases as increase in excitation wavelength. The 600nm emission corresponding to the $^5D_0 \rightarrow ^7F_2$ transition of Eu^{2+}, which is broad emission range from 550-675nm. From the emission spectrum it was observed the maximum emission intensity for 600nm peak is found for 540nm excitation wavelength. Full width at half-maximum (FWHM) is around 60nm.
Fig-6.2B:- Excitation spectrum & Emission spectrum of SrS:Eu(0.5),Ce(0.5),Ga(0.5) phosphor

- 1: Excitation sp monitoring at 600nm
- 2: Emission under 254nm Ex
- 3: Emission under 262nm Ex
- 4: Emission under 274nm Ex
- 5: Emission under 450nm Ex
- 6: Emission under 470nm Ex
- 7: Emission under 490nm Ex
- 8: Emission under 540nm Ex
Table-6.2

<table>
<thead>
<tr>
<th>S.No</th>
<th>Excitation Wavelength (nm)</th>
<th>600nm (2.066eV) emission intensity (arb u)</th>
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<tbody>
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<td>1</td>
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<td>372</td>
</tr>
<tr>
<td>7</td>
<td>540</td>
<td>469</td>
</tr>
</tbody>
</table>

Table-6.2 is the different excitation wavelengths verses intensity of corresponding emission spectrum of 600nm peak
From figures 6.2A, 6.2B and 6.2C the excitation bands around 240nm to 300nm and 400nm to 560nm are looks like the excitation bands of SrS:Eu$^{2+}$, Ce$^{3+}$, the only difference is the intensity of first band is 250 units and second band is 450 units. When excitation is varied from 254nm to 540nm of the SrS:Eu$^{2+}$, Ce$^{3+}$,Ga$^{3+}$ phosphor, the emission at 600nm peak intensity linearly grows which can be seen in figure 6.2C. The effect of Ga enhances the photoluminescence (PL) intensity of the present phosphor by 30% when compared to SrS:Eu$^{2+}$, Ce$^{3+}$ phosphor, when excited with 540nm. However Ga emission could not be isolated and it is observed seen due to overlapping of the peaks 550nm to 660nm.
Gallium added more luminescence to 600nm peak leads to enhancement in the intensity of SrS:Eu$^{2+}$, Ce$^{3+}$,Ga$^{3+}$ phosphor. It may be also noted that Ga did not act as sensitizer it only added more luminescence to the present emission band in this phosphor.

Table 6.2 is the excitation wavelengths and emission intensity of SrS:Eu$^{2+}$, Ce$^{3+}$,Ga$^{3+}$ phosphor, the same is presented in fig-6.2D, it is found from figure 6.2D, the emission intensity steeply increases from 254nm to 275nm and linearly increase from 275nm to 400nm and 450nm to 600nm steep increasing of 600nm peak intensity is observed.

**6.2.2 XRD analysis of SrS:Eu, Ce,Ga phosphor**

The crystalline structure of the SrS:Eu$^{2+}$, Ce$^{3+}$, Ga$^{3+}$ phosphor was analyzed by X-ray powder diffraction studies (XRD). Fig-6.2E is the XRD pattern of SrS:Eu$^{2+}$, Ce$^{3+}$,Ga$^{3+}$ (each 0.5 mol %) phosphor. The crystallite size is calculated using Scherrer’s formula $d=\frac{K\lambda}{\beta \cos\theta}$, where ‘K’ is the Scherer’s constant (0.94), ‘$\lambda$’ the wavelength of the X-ray (1.54060 Å), ‘$\beta$’ the full-width at half maxima (FWHM), ‘$\theta$’ the Bragg angle of the XRD big peak, from the figure more XRD peaks are observed when compared to SrS:Eu$^{2+}$, Ce$^{3+}$ phosphor (Fig-6.1E), the main peak is at 25.26° and the second peak at 29.74° followed by many other Bragg’s reflections. The crystallite size is calculated for 25.26° and 29.74° peaks from scherrer’s formula. The average crystallite size from both the peaks are 63.68nm and 51.39nm, from XRD study it appears SrS:Eu$^{2+}$, Ce$^{3+}$, Ga$^{3+}$ might have formed more than one phase.
6.2.3 SEM study of SrS:Eu, Ce, Ga phosphor

Fig-6.2F1 and 6.2F2 are SEM micrographs of the SrS:Eu$^{2+}$, Ce$^{3+}$, Ga$^{3+}$ phosphor. The particles look agglomerated having the size of few sub microns to 12 microns are observed from the SEM graphs.
Fig-6.2F1:- SEM image of Sr:S:Eu, Ce, Ga (9.09 KX)

Fig-6.2F2:- SEM image of Sr:S:Eu, Ce, Ga (9.12 KX)
6.2.4 Particle size analysis of SrS:Eu, Ce, Ga phosphor

The Particle size distribution histograms of the SrS:Eu$^{2+}$, Ce$^{3+}$,Ga$^{3+}$ (each 0.5 mol %) phosphor particles as shown in the Fig-6.2G. The prepared phosphor specimen particle size was measured by using laser based system Malvern Instrument U.K. From the figure it is found there are two peaks one is at 10 $\mu$m and another is at 50 $\mu$m are observed, and the average surface area is found to be 1.3974 M$^2$/gm.

From XRD study we presume two phases of SrS:Eu$^{2+}$, Ce$^{3+}$,Ga$^{3+}$ phosphor and particle size distribution study two major distributions are observed which are around 10 $\mu$m and 50 $\mu$m, this also suggest the presence of two phases. SEM study suggests presence of sub microns to few microns particles of the phosphor is also suggested more than one phase. From overall discussion SrS:Eu$^{2+}$, Ce$^{3+}$,Ga$^{3+}$ phosphor one can normally concluded that presence of Ga emission of PL intensity by 30% increase. The preference of more than one phase but did not influence the PL emission peak shape of 600nm.
6.2.5 FTIR study of SrS:Eu, Ce, Ga phosphor

In order to determine the chemical bonds of the phosphor, the FTIR analysis was carried out. Fig-6.2H is the FTIR of the SrS:Eu$^{2+}$, Ce$^{3+}$, Ga$^{3+}$ (each 0.5 mol %) phosphor, the main absorption around 3400 are assigned to H-O-H stretching followed by other bonds of Sr-S stretching, Sr-O stretching, Sr-S stretching, Eu-O stretching and CO-OH stretching. CO-OH and H-O-H stretchings are due to absorbed CO$_2$, H$_2$O molecules from atmosphere.
6.3. Results and discussions of SrS: Eu, Ce, Tb phosphor

6.3.1. Photoluminescence Study of SrS: Eu, Ce, Tb phosphor

Fig-6.3A, 6.3B and 6.3C are the emission and excitation of the SrS:Eu(0.5 mol %), Ce(0.5 mol %), Tb(0.5 mol%) phosphor. Fig-6.3A is the excitation spectrum monitoring at 600nm. From the figure it is found there are two excitation bands starting from 225- 325nm, peaking around 265nm and second is from 400 - 570nm. The first band is in UV region and second band is perfect blue green visible region. The excitation bands at 254nm and 460nm can be assigned to the e_g to 2t_g 5d bands of Ce^{3+}. The broad excitation bands of the 600 nm are found at 265 nm and 540 nm, which can be attributed to the (4f^7) e_g and 4f^6 5d^1(t_2g) field splitting 5d bands of Eu^{2+} respectively.
Fig-6.3B is the emission and excitation of the SrS:Eu(0.5 mol %), Ce(0.5 mol %), Tb(0.5 mol %) phosphor, when excited with 254nm, 262nm, and 274nm, emissions are found at 365nm, 395nm and 467nm with less intensity along with a broad emission peaking at 600nm. The emission peak is found at 600 nm and the excitation bands for this emission are observed at 290 nm and 460 nm.

As excitation wavelength is increases from 254 - 274nm, the emission intensity at 600nm increases from 124 to 293 units, which is around 2.3 times more. Since the energy of 254nm more when compare to 274nm. However the resonance energy transfer occurs between Ce$^{3+}$ to Eu$^{2+}$, when excitation is from 450 - 540nm.

The emission intensity increased from 317 to 465 units, an increase of 50%. The two absorption bands are due to dipole-dipole interaction and higher energy transition rate between Ce$^{3+}$ to Eu$^{2+}$. Emission intensity of 600nm peak verses excitation wavelength is presented in fig-6.3D and table-6.3 for better comparison, from figure 6.3D and table-6.3 it is found the emission intensity of 600nm peak gradually increases as increase excitation wavelength. The 600nm emission corresponding to the $^5D_0\rightarrow ^7F_2$ transition of Eu$^{2+}$, is broad emission range from 550-675nm. From the emission spectrum it was observed that the sample shows 600nm emission and the maximum emission intensity for 600nm peak is found for 540nm excitation wavelength.
Fig-6.3B: Excitation & Emission spectrum of SrS:Eu,Ce,Tb phosphor

1. Excitation spectrum
2. Emission for 254nm Ex
3. Emission for 262nm Ex
4. Emission for 274nm Ex
5. Emission for 450nm Ex
6. Emission for 470nm Ex
7. Emission for 490nm Ex
8. Emission for 520nm Ex
9. Emission for 540nm Ex

Wavelength (nm) vs Intensity (arb u)
Fig-6.3C:- Emission of SrS: Eu, Ce, Tb phosphor under different Excitation wavelengths

Table-6.3

<table>
<thead>
<tr>
<th>S.No</th>
<th>Excitation Wavelength (nm)</th>
<th>600nm (2.066eV) emission intensity (arb.u)</th>
</tr>
</thead>
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<td>1</td>
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<td>8</td>
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<td>465</td>
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</table>

Table-6.3 is the different excitation wavelengths verses intensity of corresponding emission spectrum of 600nm peak
From figures 6.3A, 6.3B and 6.3C the excitation bands around 240 - 300nm and 400 - 560nm are looks like the excitation bands of SrS:Eu\(^{2+}\), Ce\(^{3+}\), Ga\(^{3+}\), the only difference is the intensity of first band is 270 units and second band is 460 units. When excited from 254nm to 540nm of the SrS:Eu\(^{2+}\), Ce\(^{3+}\), Tb\(^{3+}\) phosphor, the emission at 600nm peak intensity linearly grown which can be seen in figure 6.3C. The effect Tb enhances the photoluminescence (PL) intensity of the present phosphor by 30% when compared to SrS:Eu\(^{2+}\), Ce\(^{3+}\) phosphor, when excited with 540nm, and is also found the intensity increased by 10% of each excitation i.e from 254 - 500nm when compared to
SrS:Eu$^{2+}$, Ce$^{3+}$, Ga$^{3+}$. However when excited with 540nm the PL emission intensity of Ga and Tb doped phosphor is same which is around 465 units. However Tb emission could not be isolated and it is observed due to overlapping of the peaks 550nm to 660nm. Terbium is added more luminescence to 600nm peak leads to enhancement in the intensity of SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$ phosphor. It may be also noted that Tb did not act as sensitizer it only added more luminescence to the present emission band in this phosphor.

Table-6.3 is the excitation wavelengths and emission intensity of SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$ phosphor, the same is presented in fig-6.3D, it is found from figure 6.3D, the emission intensity steeply increases from 254 - 275nm and linearly increase from 275 - 400nm and 450 - 600nm steep increasing of 600nm peak intensity is observing.

6.3.2 XRD analysis of SrS:Eu, Ce,Tb phosphor

The crystalline structure of the SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$ phosphor was analyzed by X-ray powder diffraction studies (XRD). Fig-6.3E is the XRD pattern of SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$ (each 0.5 mol %) phosphor. The crystallite size is calculated the two major peaks using Scherrer’s formula $d = K \lambda / \beta \cos \theta$, where ‘K’ is the Scherer’s constant (0.94), ‘$\lambda$’ the wavelength of the X-ray (1.54060 Å), ‘$\beta$’ the full-width at half maxima (FWHM), ‘$\theta$’ is the Bragg angle of the XRD big peak, from the figure more XRD peaks are observed when compared to SrS:Eu$^{2+}$, Ce$^{3+}$ phosphor (Fig-6.1E), the main peak is at 25.18° and the second peak at 29.66° followed by many other Bragg’s reflections. The crystallite size is calculated for 25.18° and 29.66° peaks from scherrer’s formula. The average crystallite size from both the peaks are 80.39nm and 64.28nm, from XRD study it appears SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$ might have formed more than one phase.
6.3.3 SEM study of SrS:Eu, Ce, Tb phosphor

Fig-6.3F1 and 6.3F2 are the SEM micrographs of the SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$ phosphor. The particles look agglomerated having the size of sub microns to 10 microns are observed from the SEM images.
Fig-6.3F1: SEM image of SrS:Eu, Ce, Tb (8.84 KX)

Fig-6.3F2: SEM image of SrS:Eu, Ce, Tb (8.93 KX)
6.3.4 Particle size analysis of SrS:Eu, Ce, Tb phosphor

The Particle size distribution histograms of the SrS:Eu^{2+}, Ce^{3+}, Tb^{3+} (each 0.5 mol %) phosphor particles as shown in the Fig-6.3G. The prepared phosphor specimen particle size was measured by using laser based system Malvern Instrument U.K. From the figure it is found there are two peaks one is at 5 μm and another is at 25 μm are observed, and the average surface area is found to be 1.4287 M²/gm.

From XRD study we presume two phases of SrS:Eu^{2+}, Ce^{3+}, Tb^{3+} phosphor and particle size distribution study two major distributions are form around 5 μm and 25 μm, this also suggest the presence of two phases, from SEM study presence of sub microns to few microns particles of the phosphor is also suggested more than one phase. From overall discussion SrS:Eu^{2+}, Ce^{3+}, Tb^{3+} phosphor one can normally concluded that presence of Tb enhances PL emission intensity by 30%. However the presence of more than one phase but did not influence the PL emission peak shape of 600nm.

Fig-6.3G :-Particle size histogram of SrS:Eu, Ce, Tb
6.3.5 FTIR study of SrS:Eu, Ce, Tb phosphor

In order to determine the chemical bonds of the phosphor the FTIR analysis was carried out. Fig-6.3H is the FTIR of the SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$ (each 0.5 mol %) phosphor, the main absorption around 3400 are assigned to H-O-H stretching followed by other bonds of Sr-S stretching, Sr-O stretching, Sr-S stretching, Eu-O stretching and CO-OH stretching. CO-OH and H-O-H stretchings are due to absorbed CO$_2$, H$_2$O molecules from atmosphere.

![FTIR spectrum of SrS:Eu, Ce, Tb phosphor](image-url)
6.4 Results and discussions of SrS: Eu, Ce, Dy phosphor

6.4.1. Photoluminescence Study of SrS: Eu, Ce, Dy phosphor

Fig-6.4A, 6.4B and 6.4C are the emission and excitation of the SrS:Eu(0.5 mol %), Ce(0.5 mol %), Dy(0.5 mol%) phosphor. Fig-6.4A is the excitation spectrum monitoring at 600nm. From the figure it is found there are two excitation bands starting from 225-325nm, peaking around 265nm and second are from 400-570nm. The first band is in UV region and second band is in perfect blue green visible region. The excitation bands at 254nm and 460nm can be assigned to the $e_g$ to $2t_g$ 5d bands of Ce$^{3+}$. The broad excitation bands of the 600 nm are found at 265 nm and 540 nm, which can be attributed to the $(4f^7) e_g$ and $4f^6 5d^1(t_{2g})$ field splitting 5d bands of Eu$^{2+}$ respectively.
Fig-6.4B is the emission and excitation of the SrS:Eu(0.5 mol %), Ce(0.5 mol %), Dy(0.5 mol%) phosphor, when excited with 254nm, 262nm, and 274nm, emissions are found at 365nm, 395nm and 467nm with less intensity along with a broad emission peaking at 600nm. The emission peak is found at 600 nm and the excitation bands for this emission are observed at 290 nm and 460 nm.
As excitation wavelength is increased from 254 - 274nm, the emission intensity of 600nm peak increases from 119 to 309 units, which is around 2.6 times more when compared to 254nm with 274nm, the energy of 254nm more than when compared to 274nm. However, the resonance energy transfer occurs between Ce$^{3+}$ to Eu$^{2+}$, when excitation is from 450 - 540nm. The emission intensity increased from 337 to 525 units, an increase of 60%. The two absorption bands are due to dipole-dipole interaction and higher energy transition rate between Ce$^{3+}$ to Eu$^{2+}$. Emission intensity of 600nm peak versus excitation wavelength is presented in fig-6.4D and table-6.4 for better comparison.

From figure 6.4D and table-6.4 it is found the emission intensity of 600nm peak gradually increases as increase in the excitation wavelength. The 600nm emission is corresponding to the $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition of Eu$^{2+}$, which is a broad emission from 550-675nm. From the emission spectrum it was observed that the phosphor shows 600nm emission and the maximum emission intensity for 600nm peak is found for 540nm excitation wavelength. Full width at half-maximum (FWHM) is around 60nm.
Fig-6.4C: Emission of SrS:Eu,Ce,Dy phosphor under different Excitation wavelength

Table-6.4

<table>
<thead>
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<th>S.No</th>
<th>Excitation Wavelength (nm)</th>
<th>600nm (2.066eV) emission intensity (arb.u)</th>
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Table-6.4 is the different excitation wavelengths verses intensity of corresponding emission spectrum of 600nm peak
From figures 6.4A, 6.4B and 6.4C the excitation bands around 240 - 300nm and 400 - 560nm are looks like the excitation bands of SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$, the only difference is the intensity of first band is 310 units and second band is 490 units. When excited from 254nm to 540nm of the SrS:Eu$^{2+}$, Ce$^{3+}$, Dy$^{3+}$ phosphor, the emission at 600nm peak intensity linearly grown which can be seen in figure 6.4C. The effect Dy enhances the photoluminescence (PL) intensity of the present phosphor by 40% when compared to SrS:Eu$^{2+}$, Ce$^{3+}$ phosphor, when excited with 540nm, and is also found the
intensity increased by 12% of each excitation i.e from 254 - 500nm when compared to SrS:Eu$^{2+}$, Ce$^{3+}$, Ga$^{3+}$, and also found the intensity increased by 10% of each excitation i.e from 254 - 500nm when compared to SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$. However when excited with 540nm the PL emission intensity of Ga and Tb doped phosphor is same which is around 465 units. However 'Dy' emission could not be isolated and it is observed due to overlapping of the peak which is 550 - 660nm. Addition of 'Dy' added more luminescence by way of enhance the photoluminescence (PL) intensity of 600nm peak in the SrS:Eu$^{2+}$, Ce$^{3+}$,Dy$^{3+}$ phosphor. It may be also noted that Dy did not act as sensitizer it only added more luminescence to the present emission band of this phosphor.

Table-6.4 is the excitation wavelengths and emission intensity of SrS:Eu$^{2+}$, Ce$^{3+}$,Dy$^{3+}$ phosphor, the same is presented in fig-6.4D. It is found from figure 6.4D, the emission intensity of 600nm peak increases steeply from 254 - 275nm excitation and linearly increase from 275 - 400nm excitation and 450 - 540nm excitation steep increase of 600nm peak intensity is observed.

6.4.2 XRD analysis of SrS:Eu, Ce, Dy phosphor
The crystalline structure of the SrS:Eu$^{2+}$, Ce$^{3+}$, Dy$^{3+}$ phosphor was analyzed by X-ray powder diffraction studies (XRD). Fig-6.4E is the XRD pattern of SrS:Eu$^{2+}$, Ce$^{3+}$,Dy$^{3+}$ (each 0.5 mol %) phosphor. The crystallite size is calculated for two major peaks using Scherrer’s formula $d = \frac{K\lambda}{\beta \cos \theta}$, where ‘K’ is the Scherer’s constant (0.94), ‘λ’ the wavelength of the X-ray (1.54060 Å), ‘β’ the full-width at half maxima (FWHM), ‘θ’ is the Bragg angle of the XRD big peak. From the figure more XRD peaks are observed when compared to SrS:Eu$^{2+}$, Ce$^{3+}$ phosphor (Fig-6.1E), the main peak is at 25.21° and the second peak at 29.66° followed by many other Bragg’s reflections. The crystallite size is calculated for 25.18° and 29.66° peaks using scherrer’s formula. The average crystallite sizes from both the peaks are 63.62nm and 60.48nm. From XRD study it appears SrS: Eu$^{2+}$, Ce$^{3+}$, Dy$^{3+}$ might have formed more than one phase.
6.4.3 SEM study of SrS:Eu, Ce, Dy phosphor

Fig-6.4F1 and 6.4F2 are the SEM micrographs of the SrS: Eu$^{2+}$, Ce$^{3+}$, Dy$^{3+}$ phosphor. The particles look agglomerated having the size of sub microns to 5 microns are observed from the SEM images.
Fig-6.4F1: SEM image of SrS:Eu, Ce, Dy (8.92 KX)

Fig-6.4F2: SEM image of SrS:Eu, Ce, Dy (9.81 KX)
6.4.4 Particle size analysis of SrS:Eu, Ce, Dy phosphor

The Particle size distribution histogram of the SrS:Eu$^{2+}$, Ce$^{3+}$,Dy$^{3+}$ (each 0.5 mol %) phosphor is shown in the Fig-6.4G. The prepared phosphor particle size was measured by using laser based system Malvern Instrument U.K. From the figure it is found there are two peaks one is at 0.5 $\mu$m and another is at 10 $\mu$m are observed, and the average surface area is found to be 2.0148 M$^2$/gm.

![Particle size histogram of SrS:Eu, Ce, Dy phosphor](image)

Fig-6.4G:-Particle size histogram of SrS:Eu, Ce, Dy phosphor

From XRD study we presume two phases of SrS:Eu$^{2+}$, Ce$^{3+}$,Dy$^{3+}$ phosphor and particle size distribution study two major distributions are found around 0.5 $\mu$m and 10 $\mu$m, this also suggest the presence of two phases , from SEM study presence of sub microns to few microns particles of the phosphor is also suggested more than one phase. From overall discussion SrS:Eu$^{2+}$, Ce$^{3+}$,Dy$^{3+}$ phosphor one can normally concluded that presence of Dy enhances PL emission intensity by 40%. However the presence of more than one phase but did not influence the PL emission peak shape of 600nm.
6.4.5 FTIR study of SrS:Eu, Ce, Dy phosphor

In order to determine the chemical bonds of the phosphor, the FTIR analysis was carried out. Fig-6.4H is the FTIR of the SrS:Eu\textsuperscript{2+}, Ce\textsuperscript{3+}, Dy\textsuperscript{3+} (each 0.5 mol %) phosphor, the main absorption around 3400 are assigned to H-O-H stretching followed by other bonds of Sr-S stretching, Sr-O stretching, Sr-S stretching, Eu-O stretching and CO-OH stretching. CO-OH and H-O-H stretchings are due to absorbed CO\textsubscript{2}, H\textsubscript{2}O molecules from atmosphere.
6.5 Results and discussions of SrS: Eu, Ce, La phosphor

6.5.1. Photoluminescence Study of SrS: Eu, Ce, La phosphor
Fig-6.5A, 6.5B and 6.5C are the emission and excitation of the SrS:Eu(0.5 mol %), Ce(0.5 mol %), La(0.5 mol%) phosphor. Fig-6.5A is the excitation spectrum monitoring at 600nm. From the figure it is found there are two excitation bands starting from 225- 325nm, peaking around 265nm and second is from 400 - 570nm. The first band is in UV region and second band is perfect blue green visible region. The excitation bands at 265nm and 460nm can be assigned to the $e_g$ to $2t_g$ 5d bands of Ce$^{3+}$. The broad excitation bands of the 600 nm are found at 265 nm and 540 nm, which can be attributed to the $(4f^7) e_g$ and $4f^6 5d^1(t_{2g})$ field splitting 5d bands of Eu$^{2+}$ respectively.
Fig-6.5B is the emission and excitation of the SrS:Eu(0.5 mol %), Ce(0.5 mol %), La(0.5 mol %) phosphor, when excited with 254nm, 262nm, and 274nm, emissions are found at 365nm, 395nm and 467nm with less intensity along with a broad emission peaking at 600nm. The emission peak is found at 600 nm and the excitation bands for this emission are observed at 290 nm and 460 nm.
As excitation wavelength is increases from 254 - 274nm, the emission intensity at 600nm increases from 78 to 173 units, which is around 2 times more. Since the energy of 254nm more when compare to 274nm. However the resonance energy transfer occurs between Ce$^{3+}$ to Eu$^{2+}$, when excitation is from 450 - 540nm.

The emission intensity increased from 215 to 361 units, an increase of 60%. The two absorption bands are due to dipole-dipole interaction and higher energy transition rate between Ce$^{3+}$ to Eu$^{2+}$. Emission intensity of 600nm peak verses excitation wavelength is presented in fig-6.5D and table-6.5 for better comparison, from figure-6.5D and table-6.5 it is found the emission intensity of 600nm peak gradually increases as increase excitation wavelength. The 600nm emission corresponding to the $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition of Eu$^{2+}$, is broad emission range from 550-675nm. From the emission spectrum it was observed that the sample shows 600nm emission and the maximum emission intensity for 600nm peak is found for 540nm excitation wavelength. Full width at half-maximum (FWHM) is around 60.4nm.

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<td>8</td>
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Table-6.5 is the different excitation wavelengths verses intensity of corresponding emission spectrum of 600nm peak
From figures 6.5A, 6.5B and 6.5C the excitation bands around 240 - 300nm and 400 - 560nm are looks like the excitation bands of SrS:Eu$^{2+}$, Ce$^{3+}$, Tb$^{3+}$, the only difference is the intensity of first band is 170 units and second band is 350 units. When excited from 254nm to 540nm of the SrS:Eu$^{2+}$, Ce$^{3+}$, La$^{3+}$ phosphor, the emission at 600nm peak intensity linearly grown which can be seen in figure 6.5C.

The effect La decreases the photoluminescence (PL) intensity of the present phosphor by 45% of each excitation i.e from 254 to 520nm, however when excited with 540nm the PL emission intensity of SrS: Eu$^{2+}$, Ce$^{3+}$,La$^{3+}$ phosphor is raised by 4% when compared to SrS:Eu$^{2+}$, Ce$^{3+}$ phosphor. However La emission could not be isolated and...
it is observed due to overlapping of the peaks 550nm to 660nm. Lanthanum is added more luminescence to 600nm peak for 540nm excitation leads to enhancement in the intensity of SrS:Eu$^{2+}$, Ce$^{3+}$,La$^{3+}$ phosphor. It may be also noted that La did not act as sensitizer it only added more luminescence to the present emission band in this phosphor.

Fig-6.5D:-Excitation wavelength verses Emission intensity of 600nm peak for SrS:Eu,Ce,La phosphor
Table-6.5 is the excitation wavelengths and emission intensity of SrS:Eu$^{2+}$, Ce$^{3+}$ ,La$^{3+}$ phosphor, the same is presented in fig-6.5D, it is found from figure 6.5D, the emission intensity steeply increases from 254 - 275nm and linearly increase from 275 - 400nm and 450 - 600nm steep increasing of 600nm peak intensity is observing. It is interesting to note the emission intensity of 600nm peak intensity is less by 50% in SrS:Eu$^{2+}$, Ce$^{3+}$ ,La$^{3+}$ for the excitation band 254 -275nm excitation, since the 254 - 275nm excitation band of absorption intensity is less by 50%, when compared to SrS:Eu$^{2+}$, Ce$^{3+}$ absorption band and intensity of 600nm peak.

6.5.2 XRD analysis of SrS:Eu, Ce, La phosphor

The crystalline structure of the SrS: Eu$^{2+}$, Ce$^{3+}$, La$^{3+}$ phosphor was analyzed by X-ray powder diffraction studies (XRD). Fig-6.5E is the XRD pattern of SrS:Eu$^{2+}$, Ce$^{3+}$,La$^{3+}$(each 0.5 mol %) phosphor. The crystallite size is calculated the two major peaks using Scherrer’s formula $d=K\lambda/\beta\cos\theta$, where ‘$K$’ is the Scherer’s constant (0.94), ‘$\lambda$’ the wavelength of the X-ray (1.54060 Å), ‘$\beta$’ the full-width at half maxima (FWHM), ‘$\theta$’ is the Bragg angle of the XRD big peak, from the figure more XRD peaks are observed when compared to SrS:Eu$^{2+}$, Ce$^{3+}$ phosphor (Fig-6.1E), the main peak is at 25.24° and the second peak at 29.68° followed by many other Bragg’s reflections.

The crystallite size is calculated for 25.24° and 29.68° peaks from scherrer’s formula. The average crystallite size from both the peaks are 63.65nm and 53.18nm, from XRD study it appears SrS:Eu$^{2+}$, Ce$^{3+}$, La$^{3+}$ might have formed more than one phase.
6.5.3 SEM study of SrS:Eu, Ce, La phosphor

Fig-6.5F1 and 6.5F2 are the SEM micrographs of the SrS:Eu$^{2+}$, Ce$^{3+}$, La$^{3+}$ phosphor. The particles look agglomerated having the size of sub microns to 8 microns are observed from the SEM images.
Fig-6.5F1:- SEM image of SrS:Eu, Ce, La (9.54 KX)

Fig-6.5F1:- SEM image of SrS:Eu, Ce, La (9.13 KX)
6.5.4 Particle size analysis of SrS:Eu, Ce, La phosphor

The Particle size distribution histograms of the SrS:Eu$^{2+}$, Ce$^{3+}$,La$^{3+}$ (each 0.5 mol %) phosphor particles as shown in the Fig-6.5G. The prepared phosphor specimen particle size was measured by using laser based system Malvern Instrument U.K. From the figure it is found there are two peaks one is at 5 μm and another is at 50 μm are observed, and the average surface area is found to be 1.2304 M$^2$/gm.

From XRD study we presume two phases of SrS:Eu$^{2+}$, Ce$^{3+}$,La$^{3+}$ phosphor and particle size distribution study two major distributions are form around 5 μm and 50 μm, this also suggest the presence of two phases , from SEM study presence of sub microns to few microns particles of the phosphor is also suggested more than one phase. From overall discussion SrS:Eu$^{2+}$, Ce$^{3+}$, La$^{3+}$ phosphor one can normally concluded that presence of La enhances PL emission intensity by 4%, when excited with 540nm. However the presence of more than one phase but did not influence the PL emission peak shape of 600nm.
6.5.5 FTIR study of SrS:Eu, Ce, La phosphor

In order to determine the chemical bonds of the phosphor, the FTIR analysis was carried out. Fig-6.4H is the FTIR of the SrS:Eu$^{2+}$, Ce$^{3+}$, La$^{3+}$ (each 0.5 mol %) phosphor, the main absorption around 3400 are assigned to H-O-H stretching followed by other bonds of Sr-S stretching, Sr-O stretching, Sr-S stretching, Eu-O stretching and CO-OH stretching. CO-OH and H-O-H stretchings are due to absorbed CO$_2$, H$_2$O molecules from atmosphere.
6.6 Discussions and Conclusions

SrS: Eu\textsuperscript{2+} is a very efficient phosphor with a red emission that can be excited with visible light. The following points can be considered:

1. Its emission is a single broad band peaked at 600 nm resulting from the 4f\textsuperscript{6}5d\textsuperscript{1} (t\textsubscript{2g}) to 4f\textsuperscript{7}(eg) transition.
2. The broad excitation bands of the 600nm peak are attributed to the e\textsubscript{g} and t\textsubscript{2g} field splitting 5d bands of Eu\textsuperscript{2+}, respectively. Different from Ce\textsuperscript{3+}, there is no charge transfer transition observed from Eu\textsuperscript{2+} ground state to the host conduction band, implying that the Eu\textsuperscript{2+} ground state is close to the host valence band or the lowest 5d band overlaps with the host conduction band. Similar to Ce\textsuperscript{3+}, the ligand field splitting of 5d level of Eu\textsuperscript{2+} also results in red shifts for both emission and excitation peaks from SrS host.
3. The broad band excitation peaks of 5d field splitting components e\textsubscript{g} and t\textsubscript{2g} are found, respectively, to have a red shift to 340 nm and a yellow-orange red shift to a region from 590 nm to 690 nm. This indicates a weaker field splitting of the Eu\textsuperscript{2+} 5d state due to a weaker ligand field generated by a larger lattice. SrS have similar lattice symmetry, making them easier to have a solid solution in order to adjust the positions of absorption and emission and to obtain better color rendering for white LED applications.
4. The emission spectrum can continuously be tuned from orange to red, to further extending the tailoring of the properties of these materials.
5. The SrS: Eu\textsuperscript{2+} sample heated at 900\textdegree C and cooled to room temperature and observed body colour of the soft cake is pale orange. This phosphor can be a color conversion material in LEDs due to their excitation and emission behavior.

With the solid state synthesis which is a green chemistry route, no post deposition annealing was necessary to incorporate the Eu\textsuperscript{2+} ions in the alkaline earth sulfide lattice. The formed particles are strongly luminescent when doped with Eu, with an emission band typical for Eu\textsuperscript{2+} in alkaline earth sulfides. The Eu\textsuperscript{2+} emission is due to allowed transitions between the excited state (t\textsubscript{2g}) of the 4f\textsuperscript{6} 5d configuration and the ground state (\textsuperscript{8}S\textsubscript{7/2}) of the 4f\textsuperscript{7} configuration.
The excitation spectrum of SrS:Eu:Ce(0.5Mol\%) crystallites co-doped with Ga, Tb, Dy and La monitored under 600nm wavelength. The broad excitation bands of the 600 nm emission are found at 285 nm and 540 nm along with a hump around 460nm which can be attributed to the \((4f^7)\) \(e_g\) and \(4f^6 5d^1(t_{2g})\) field splitting 5d bands of Eu\(^{2+}\) respectively. From the excitation spectrum we observed that this material has broad excitations like 254, 262, 274, 450, 470, 490, 520 and 540nm wavelengths and it was also observed that the excitation intensity of Eu,Ce,Dy (0.5Mol %) concentration shows high intensity under the above excitation wavelengths and the data is tabulated in table 6.4 and a graph is drawn between concentration Vs excitation intensity as shown in the figure 6.4D.

The as prepared SrS:Eu particles exhibit a broad PL emission band with an emission maximum at 600nm and a full width at half-maximum (FWHM) of 60nm at an excitation wavelengths of 254, 262, 274, 450, 470, 490, 520 and 540nm respectively. The 600nm emission is corresponding to the \(^{5}D_0\rightarrow ^{7}F_2\) transition of Eu\(^{2+}\), is a broad emission range from 550-675nm. From the emission spectrum it was observed that the sample shows 600nm emission and the intensity is highest under 540nm excitation wavelength.

The SEM micrographs are presented in figures 6.1F1, 6.1F2, 6.2F1, 6.2F2, 6.3F1, 6.3F2, 6.4F1, 6.4F2, 6.5F1 and 6.5F2 of SrS:Eu Ce (Ga,Tb,Dy and La) phosphors respectively. From all the micrographs it is found irregular shape particles with different lengths and breadths. Different clusters are also found, implying that different crystallite particles are agglomerated.

The FTIR spectrums of SrS base and SrS:EuCe (Ga,Tb,Dy and La) are presented in the Chapter-VI. From the FTIR studies we observed that most of the bands from ligands and are Sr-S stretching and Eu-S, Ga-S, Tb-S, Dy-S, La-S stretchings. The variation of different absorption intensities are nothing but, the presence of ligands in different concentrations.
Allowed transitions of Eu ion are as follows
467 transition $^5D_2 \rightarrow ^7F_0$ corresponding energy value is 2.655eV

- The electronic configuration of Eu$^{2+}$ is 4f$^7$. The lowest excited state of 4f levels is located at $28 \times 10^3$ cm$^{-1}$ and is higher than the 4f$^6$ 5d$^1$ level in most of the crystals, so that Eu$^{2+}$ usually gives broad band emission due to f-d transitions.
- The emission wavelength positions of the bands depend very much on hosts, changing from the near-UV to the red. This dependence is interpreted as due to the crystal field splitting of the 5d level.
- As it is reported when increase in crystal field strength, the emission bands shift to longer wavelength. The luminescence peak energy of the 5d-4f transitions of Eu$^{2+}$ and Ce$^{3+}$ are affected most by crystal parameters denoting electron-electron repulsion. On this basis, a good fit of the energies can be obtained. Red luminescence is observed in Eu$^{2+}$-activated SrS, the crystal field is stronger in sulfides than in fluorides and oxides.
- Among the lanthanide ions, the 4f $\rightarrow$ 5d transition energy is the lowest in Ce$^{3+}$, but the energy gap from the 5d$_1$ state to the nearest level (2F$^{7/2}$) below is so large that the 5d level serves as an efficient light-emitting state.
- The luminescence photon energy depends strongly on the structure of the host crystal through the crystal-field splitting of the 5d state

Allowed transitions of Ce$^{3+}$ ion
515 transitions 5d $\rightarrow$ 2F$^{7/2}$ corresponding energy value is 2.408eV
530 transitions 5d $\rightarrow$ 2F$^{5/2}$ corresponding energy value is 2.255eV, these emissions are not influenced the 600nm peak shape.

From the above discussions the emissions observed in SrS:EuCe, SrS:EuCe (La,Tb,Dy and Ga) are the allowed transitions of both the dopants.

To obtain even smaller particles of SrS: EuCe, SrS:EuCe (La,Tb,Dy and Ga), the influence of the reaction temperature, the duration of the reaction and the amount of starting materials could be investigated. However, lowering the reaction temperature and the reaction time might reduce the yield of the final reaction product and PL intensity.
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