



## Preparation of nanosize $\text{Sr}_2\text{SiO}_4:\text{Ce}^{3+}$ by oxalate precipitation method

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### Abstract

In present work  $\text{Sr}_2\text{SiO}_4$  sample activated by cerium ions were prepared first time by modified oxalate precipitation method after heating at 800°C for 2 hr. Sample are characterized by XRD, photoluminescence excitation, and photoluminescence emission. XRD pattern shows the formation of pure phase and nanosize powder. The emission spectra shows a broad peaks in the blue region (between 420 and 580 nm), due to splitting of the 4f energy level into the  $2F_{5/2}$  and  $2F_{7/2}$  energy levels of 4f1 electron in  $\text{Ce}^{3+}$  and influence by crystal field surrounding it in host material.

Keywords: nanomaterial,  $\text{Sr}_2\text{SiO}_4:\text{Ce}^{3+}$ , photoluminescence, precipitation method, Dip-UV display application.

### 1. Introduction

Phosphors prepared by conventional methods are generally in micron size, but for solid state lighting purpose it should be small nanosized (<400 nm) and of uniform particle size, to reduce light scattering in the device to improve the extraction efficiency of the phosphors. Secondly it is not possible to grow crystals for LEDs that could give an emission at the desired wavelength. Hence it becomes significant to prepare phosphors that could easily be synthesized by novel methods and could produce emission at desired wavelengths. The synthesis of nanosize uniform powder by soft chemical methods is of particular interest as they are simpler and do not need cumbersome equipment and machinery. These methods include precipitation under controlled conditions, thus combustion method, Pechini sol-gel method, hydrothermal method and coprecipitation method are generally used for synthesis of oxide phosphors. In recent years, much attention has been focused on nanosize oxide based luminescent (especially for superior PL and CL properties) materials due to their commercial applications in color television, fluorescent tube, tri color LEDs etc [1,2]. The search for blue phosphors is of particular importance because of limited number of stable blue luminescent materials available. The past twenty years have witnessed the development of AlGaIn-based

UV LEDs with their emitting wavelength shorter than 365 nm [3], (band gap of GaN). In 1998, Han et al. [4] manufactured the first UV LED with an emitting wavelength 353.6 nm. Subsequently, UV LED with emitting wavelength 280, 269 and 265 nm were achieved one by one [5-7] these UV LED can not be commercially utilized because of their lower quantum and luminous efficiency.

Recently developed (222-282 nm) AlGaIn and InAlGaIn based deep-LEDs on high-quality AlN template which can be used as (long lifetime, high color-rendering light) for white light phosphor, and scientist Hirayama and his colleagues introduced an electron-blocking multi quantum barrier (MQB) into an AlGaIn DUV-LED and achieved a dramatic increase in electron injection efficiency to more than 80% succeeding in generating light at a wavelength of 250 nm with a light output of 15 mW. This investigation will completely remove the use of mercury in lamp industries and fulfill the demand of second generation solid state lighting [8-9]. Thus after a decade it will be possible that phosphorus with their exciting bands located in UV region combined with UV LEDs to accomplish a desired white light.

From the literature review it is found that Jong et al. [10], and QIAN Yanmin et al. [11] prepared  $\text{Sr}_2\text{SiO}_4$  with rare earth ion doping by solid state reaction at high temperature, in present work we proposed a new method to

prepare  $\text{Sr}_2\text{SiO}_4$  phosphor activated by cerium ions first time by precipitation method by sintering at  $800^\circ\text{C}$  for 2 hr.

## 2. Experiment

$\text{Sr}_2\text{SiO}_4$  phosphor activated with  $\text{Ce}^{3+}$  ions were synthesized by precipitation method shown in figure (1) in the form of flow chart. Using  $\text{Sr}(\text{NO}_3)_2$  (99.99%),  $\text{Ce}(\text{NO}_3)_3$  (99.99%),  $\text{SiO}_2$  (99.99%) and Oxalic acid were used as starting material. First  $\text{SiO}_2$  were dissolved in dilute  $\text{HNO}_3$  (AR) to make nitrate solution and then  $\text{Sr}(\text{NO}_3)_2$  and  $\text{Ce}(\text{NO}_3)_3$  is added in

silicate nitrate mixture. All sample were weighted with stoichiometric ratio. Then these starting material are heated till transparent solution is obtain at  $80^\circ\text{C}$  near by half hour with continuous stirring. Solution of oxalic acid (dehydrated) was made in double distilled water and warming at 15-20 minute, with continuous stir till transparent solution is obtained. Then solution of oxalic acid was added drop by drop in above mixture of nitrates .

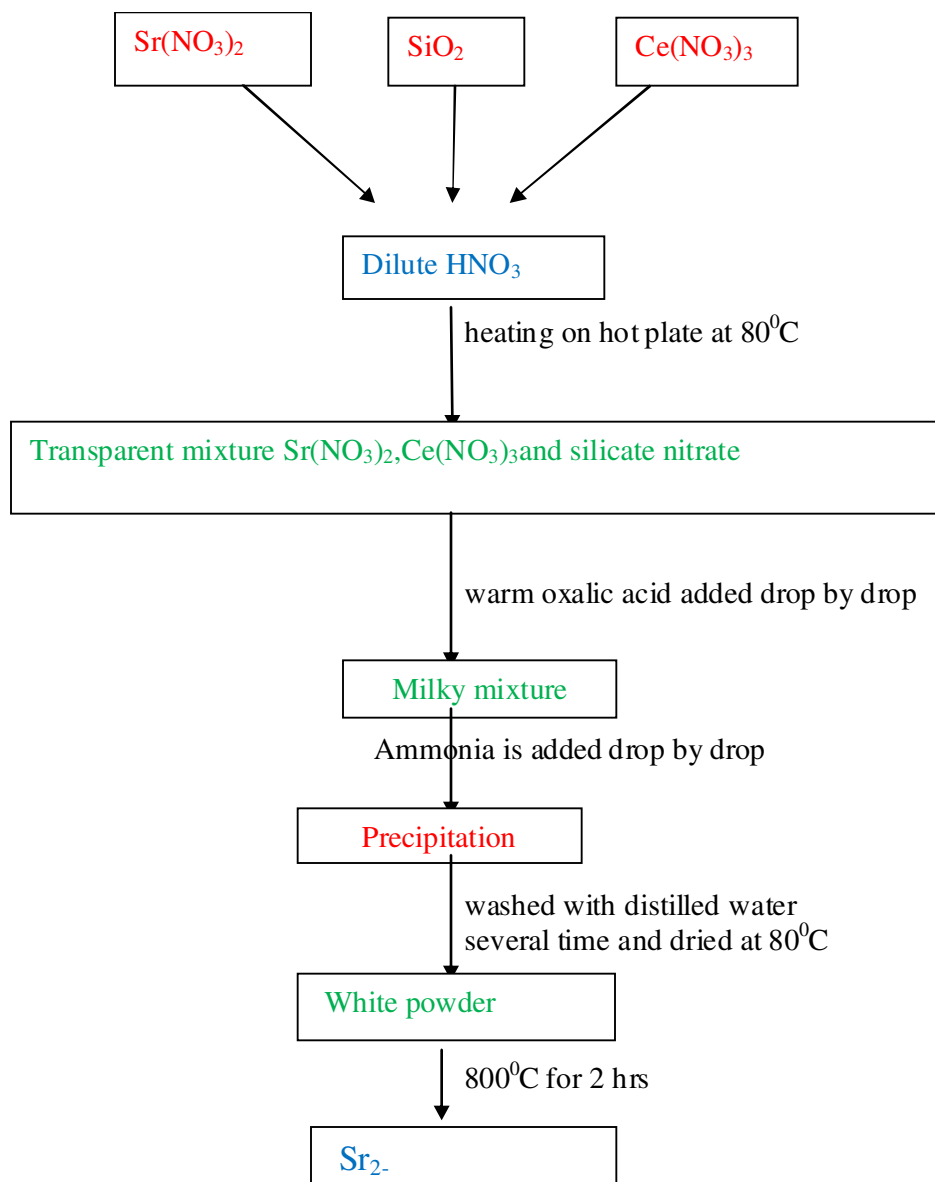


Fig.1. The flow chart for synthesis of  $\text{Sr}_{2-x}\text{SiO}_4:\text{Ce}_x$  by precipitation method.

Then mixture keep well stir with adding ammonia till the precipitation is obtained. Then obtain precipitated was filtered out after cooling using double filter paper with washing with distilled water several times. Then it kept at 90°C on hot plate for half hour for drying. The dry white powder was kept for heating at 800°C for 2h, to remove organic impurities.

### 3. Results & Discussion:

The XRD pattern of Sr<sub>2</sub>SiO<sub>4</sub>:Ce<sup>3+</sup> phosphor with Ce<sup>3+</sup> concentration 1 mol% is presented in fig.2. using Cu K $\alpha$  radiation ( $\lambda = 1.541 \text{ \AA}$ ) with a nickel filter. All patterns agree well with JCPDS file 39-1256, indicating that the doped Ce<sup>3+</sup> ions have not caused any significant change in the host structure. Sr<sub>2</sub>SiO<sub>4</sub> has an orthorhombic crystal structure, and their lattice parameters values are  $a = 0.7079 \text{ nm}$ ,  $b = 0.5672 \text{ nm}$ ,  $c = 0.9743 \text{ nm}$ .

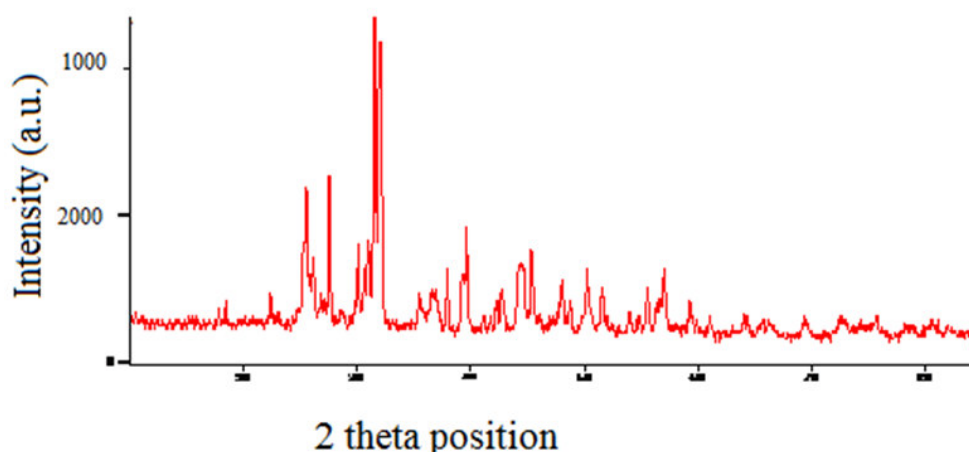


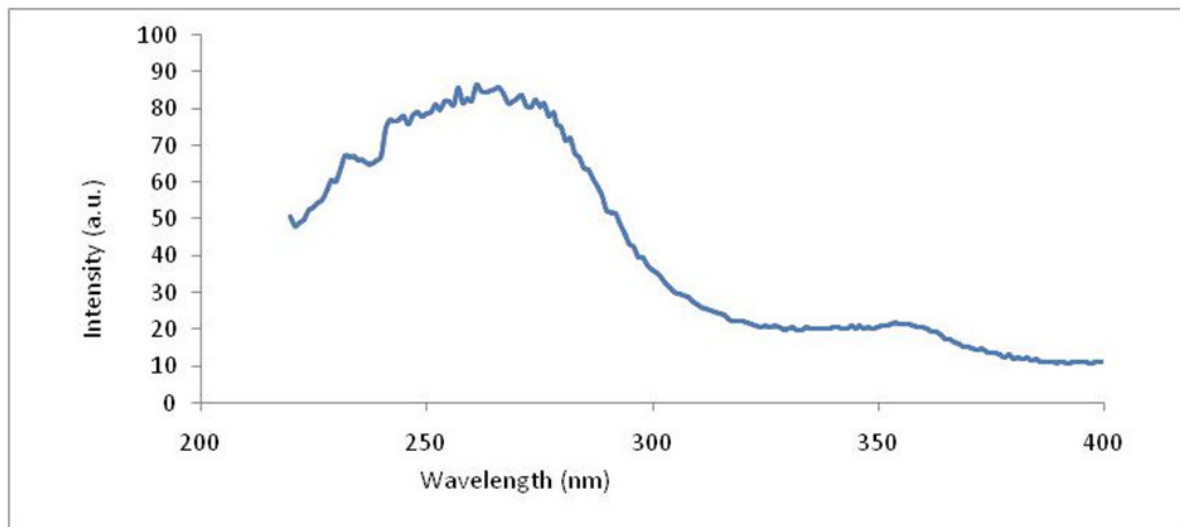
Fig.(2). XRD pattern of Sr<sub>2</sub>SiO<sub>4</sub>:Ce<sup>3+</sup>

The crystalline size was calculated from the broad XRD peaks using the Scherrer equation (1).

$$d = \frac{0.9\lambda}{\beta \cos \theta} \quad (2)$$

Where  $d$  is average grain size and  $\beta$  is the diffracted full width at half maximum and  $\lambda$  is incident wavelength,  $h, k, l$  are Miller indices and  $\theta$  is Bragg's angle. The average crystalline size is found to be around 60 nm.

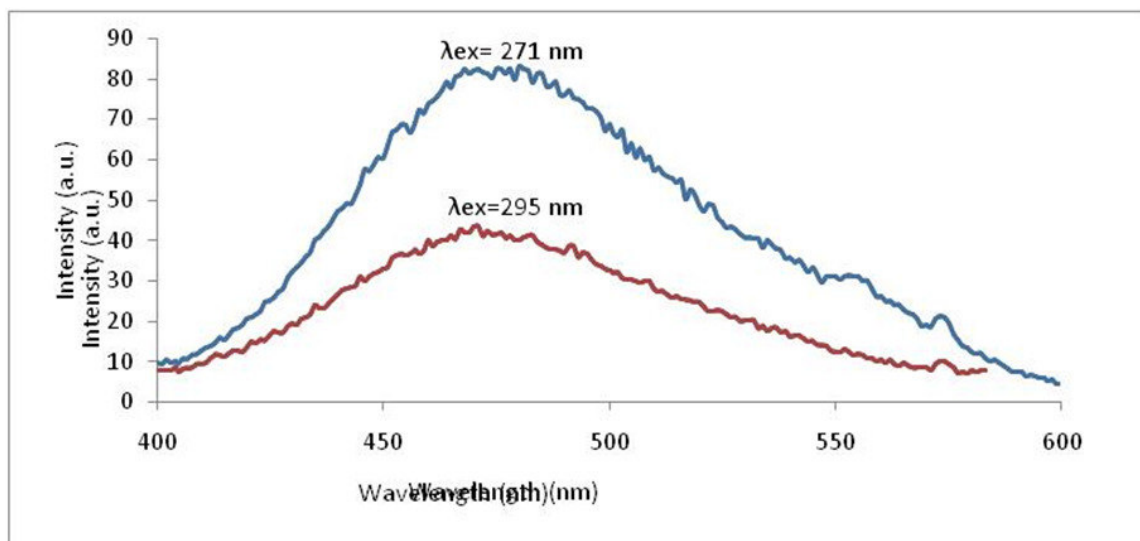
In the present work Sr<sub>2</sub>SiO<sub>4</sub>:Ce<sup>3+</sup> have been studied in order to understand the The average crystalline size and luminescent behavior for application in the lighting industry and display applications. Figure (3) demonstrate the PLE spectra of Sr<sub>2</sub>SiO<sub>4</sub>:Ce<sup>3+</sup>. Photoluminescence spectrum ( $\lambda_{em} = 470 \text{ nm}$ ) shows a broad band from 222 nm to 300 nm that is at deep-UV region. Figure (4) shows the photoluminescence spectrum, which shows a broad band in blue region of electromagnetic spectra.



**Fig.3. Excitation spectra of Sr<sub>2</sub>SiO<sub>4</sub>:Ce<sup>3+</sup> =1m% at emission wavelength 470 nm .**

This luminescence in Sr<sub>2</sub>SiO<sub>4</sub>:Ce<sup>3+</sup> occurs due to characteristic transition (in the Ce<sup>3+</sup> ion itself). When Sr<sub>2</sub>SiO<sub>4</sub> doped with an activator such as Ce<sup>3+</sup> creates an energy level structure inside the band gap of Sr<sub>2</sub>SiO<sub>4</sub>, where the 5d to 4f transition takes place. Splitting of the 4f energy level into the <sup>2</sup>F<sub>5/2</sub> and <sup>2</sup>F<sub>7/2</sub> energy

levels is due to the 4f<sup>1</sup> electron in Ce<sup>3+</sup> having the ability to exhibit a +1/2 and -1/2 spin [12,13]. This creates the expectation of a luminescent spectrum with two main peaks in the blue region. However; reports on broad band single spectra (between



**Fig.4. Emission spectra of Sr<sub>2</sub>SiO<sub>4</sub>:Ce<sup>3+</sup> =1m% .**

420 and 580 nm) have been found, as shown in figure (3), this broad band in blue region indicates presence of strong crystal field on  $Ce^{3+}$  site in the proposed host material. Since  $Sr_2SiO_4$  exhibit excellent physical and chemical stability. Besides, it absorbed ultraviolet radiations and emit the blue light when activated by cerium ions thus it may play positive in display applications.

#### 4. Conclusion:

An simple and effective precipitation method is was used to prepare nanosize and single phased  $Sr_2SiO_4$  activated by cerium ions. Average crystalline size is found to be around 60nm. Phosphors showed a intense broad band emission in the blue region when it was excited with deep ultraviolet radiations. Presence of broad band indicates, existence of strong crystal field on  $Ce^{3+}$  site in  $Sr_2SiO_4$ : $Ce^{3+}$ . Thus proposed phosphors can be used for display applications under deep-UV LED excitations.

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