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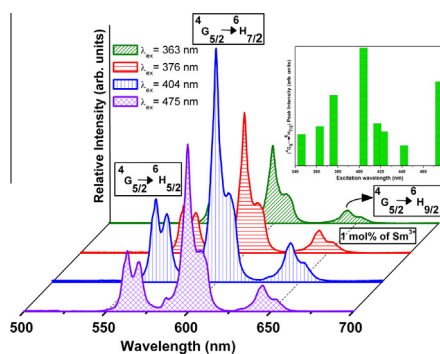
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journal homepage: www.elsevier.com/locate/saaLuminescent properties of orange emissive Sm^{3+} -activated thermally stable phosphate phosphor for optical devicesB.V. Ratnam^a, M. Jayasimhadri^{b,*}, Kiwan Jang^{a,*}^a Department of Physics, Changwon National University, Changwon 641-773, Republic of Korea^b Department of Applied Physics, Delhi Technological University, Delhi 110 042, India

HIGHLIGHTS

- Successfully synthesized Sm^{3+} doped NaCaPO_4 (NCP) phosphors.
- Structural and luminescent properties have been investigated.
- Strongest excitation peak nearly matches with the emission of GaN based LEDs.
- CIE coordinates of Sm^{3+} :NCP phosphor indicate pure orange emission.
- Sm^{3+} doped NCP is suitable for UV based pc-LEDs and other optoelectronic devices.

GRAPHICAL ABSTRACT



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ABSTRACT

Rare earth ion activated orthophosphates have a great deal of interest due to their thermal stability for white light emitting diodes. In this regard, thermally stable Sm^{3+} doped NaCaPO_4 (NCP) phosphor was synthesized by conventional solid state reaction technique. The phase and the structure of the as prepared powders were characterized by X-ray diffraction (XRD), field emission scanning electron microscope (FE-SEM), FT-IR, emission and excitation properties were extensively investigated for NCP phosphors. X-ray diffraction analysis confirmed the formation of NaCaPO_4 with orthorhombic structure. The excitation spectra indicate that this phosphor can be effectively excited by UV light from 350 to 500 nm. All the transitions in the excitation spectrum of Sm^{3+} start from the ground state $^6\text{H}_{5/2}$ to various excited states. The emission spectra indicated that the emitted radiation was dominated by the emission peak wavelength at 599 nm originated from the transition of $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$. The optimum concentration of Sm^{3+} is determined as 1.0 mol% based on the concentration dependent emission spectra. These results suggest that the $\text{NaCaPO}_4:\text{Sm}^{3+}$ phosphor is a promising orange emitting phosphor under 404 nm excitation with CIE coordinates of $x = 0.545$, $y = 0.41$, which might be used in the development of materials for LED's and other optical devices in the visible region.

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Introduction

Considerable attention has been paid recently to the solid state lighting due to its great potential to significantly surpass the energy

efficiencies of traditional incandescent and fluorescent lighting [1–6]. In general, there are several ways to make white LEDs. The present strategy to produce white light utilizes combination of blue LED with yellow luminescence from $\text{Y}_3\text{Al}_5\text{O}_{12}:\text{Ce}^{3+}$ (YAG: Ce^{3+}) phosphor materials [7,8]. However, there is a possibility to obtain white light emission in different crystalline hosts with single phased phosphors by mixing of blue, cyan and orange emitting

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phosphors by exciting with near UV chip, which leads for major improvement in lumen output and also enhancement in CRI by adding a fourth color emitting in the deep red region [9,10]. Thus, the development of near ultraviolet LED based orange emitting optical materials are essential and may be expected to have potential applications in the field of solid state lighting, displays and optoelectronic devices, which inspired us to do research work on different materials to synthesize pure orange emissive phosphor.

Recently, many novel materials have been studied in order to develop phosphors for solid state lighting applications and also for displays [11–14]. It is well-known that the properties such as strong UV absorption, high conversion efficiency, good thermal and chemical stabilities are required for the phosphors to use in the applications of illumination and plasma display panels (PDPs). Particularly, orthophosphates doped with rare earth ions have been considered as an excellent phosphor with strong UV absorption, high thermal and chemical stability [8,15,16]. Nevertheless, only a rather small number of orthophosphates have been investigated as luminescent materials. On the other hand, Sm^{3+} ion is generally considered as an excellent candidate for reddish-orange/orange-red emission, whose energy levels contain several metastable multiplets [17–19]. Generally, samarium can enter in the host matrix either in its divalent form and/or as trivalent form [20–22]. In addition, only few papers have dealt with luminescent properties of Sm^{3+} ions in near ultraviolet (370–410 nm) light excitation. Hence, this work motivated us to design and fabricate pure orange emissive Sm^{3+} -doped NaCaPO_4 phosphor.

In this paper, NaCaPO_4 phosphor samples have been synthesized with different Sm^{3+} concentrations by solid state reaction method. The phase structures, morphologies, emission and excitation properties of the synthesized phosphors have been investigated. The effects of Sm^{3+} concentration on the luminescent properties were discussed in detail. The most important observation was that of the intense pure orange emission in orthophosphates. This study would be useful to examine the suitability of the Sm^{3+} doped NaCaPO_4 for white-LED's and other optical devices in the visible region.

Experimental

The phosphor samples $\text{NaCa}_{1-x}\text{PO}_4:\text{xSm}^{3+}$ ($x = 0.1, 0.5, 1.0, 1.5, 2.0$ and 3.0 mol%) were synthesized by using solid state reaction method. High purity oxide materials, such as $\text{NH}_4\text{H}_2\text{PO}_4$, Na_2CO_3 , CaCO_3 and Sm_2O_3 (Sigma–Aldrich) were used as starting materials. Proper amounts of the starting materials were weighed stoichiometrically and then mixed thoroughly in an agate mortar for 1 h with methyl alcohol as wetting medium. The well-blended mixture was taken in an alumina crucibles and then calcined at different temperatures firstly at 185°C for 2 h, then 714°C for 1 h and finally at 950°C for 3 h in air.

The X-ray diffraction (XRD) investigations were performed with a commercial X'Pert Philips diffractometer over 2θ range from 10° to 75° . The compounds were also characterized by Fourier transform infrared spectroscopy (FT-IR) and Raman spectra, with the help of Jasco, FT/IR-6300 spectrometer (Tokyo, Japan), and Jasco, NRS-3300 laser Raman spectrophotometers (Tokyo, Japan) equipped DPSS laser (532 nm), respectively. The morphology and particles size of the powder phosphors were examined by using field emission scanning electron microscope (FE-SEM) (Tescan, MIRA II LMH, Czech Republic). Luminescent properties were measured with a Shimadzu, RF-5301PC spectrofluorophotometer.

Results and discussion

X-ray diffraction and FE-SEM

To verify the formation of NaCaPO_4 structure, the samples were examined by measuring XRD and compared with the standard JCPDS data of NaCaPO_4 . Fig. 1 shows the XRD patterns of the as-prepared, 1 mol% samarium doped NaCaPO_4 phosphors and the corresponding powder diffraction data (JCPDS NO: 76-1456). It can be seen from Fig. 1 that the XRD patterns of the 1 mol% Sm^{3+} doped NaCaPO_4 was in good agreement with the characteristic peaks of the standard orthorhombic phase of NaCaPO_4 , which belongs to the space group is $Pn2_1a$ symmetry group with lattice constants, $a = 20.39 \text{ \AA}$, $b = 5.412 \text{ \AA}$ and $c = 9.161 \text{ \AA}$. No impurity phases observed indicating that the sample formed with single phase. The peak at $2\theta = 32.578^\circ$ is observed as the strongest peak among the observed peaks, which corresponds to the (6 1 1) plane of NaCaPO_4 . Fig. 2 shows the FE-SEM image for the 1 mol% Sm^{3+} doped NaCaPO_4 phosphor calcined at 950°C for 3 h by solid state reaction method. It can be seen clearly from the image that the product mainly consists of solid micron crystalline structures, which exist some conglomeration among the crystalline grain due to thermal decomposition. The average crystalline sizes of the synthesized phosphor in the micrometer range of about 5–10 μm in dimension.

FT-IR and Raman spectra

The FT-IR and Raman spectra exhibit characteristic bands of the orthophosphates for the as-made 1 mol% Sm^{3+} doped NaCaPO_4 phosphor as shown in Figs. 3 and 4, respectively. The FT-IR spectrum has been measured within the broad range from 500 to 4000 cm^{-1} . Three significant bands observed at frequencies 578, 1038 and 3430 cm^{-1} . The narrow band at 578 cm^{-1} mainly due to the O–P–O asymmetric bending vibrations, where as prominent band at 1038 cm^{-1} due to the asymmetric stretching vibrations in $[\text{PO}_4]^{3-}$ tetrahedron [23]. The broad band around 3430 cm^{-1} is corresponding to the OH^- stretching vibration mode of the H_2O molecule. The Raman spectrum has been recorded from 500 to 1600 cm^{-1} . Raman spectrum shows intense band at around 965 cm^{-1} presenting one shoulder at 898 cm^{-1} , which are attributed to the $(\text{PO}_4)^{3-}$ symmetric stretching vibration [24].

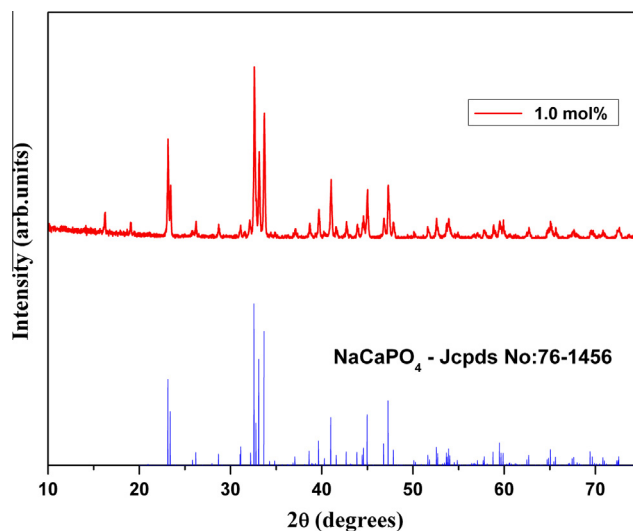


Fig. 1. XRD patterns of 1 mol% Sm^{3+} doped NaCaPO_4 phosphor.

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