



# Effect of flux (NH4Cl)on Y2SiO5:Dy3+ (9 mol %) nanophosphors its Characterization and Structural studies

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# ABSTRACT

Flux (NH<sub>4</sub>Cl) doped on Y<sub>2</sub>SiO<sub>5</sub>:Dy<sup>3+</sup> phosphors were synthesized by auto ignition based low temperature Solution Combustion Synthesis (SCS) using ODH as fuel. Powder X-ray diffraction (PXRD) patterns confirm the nano sized particles corresponding to JCPDS card 36-1476. The crystallite size of the samples estimated from Scherrer's formula. SEM micrographs infer addition of flux gives the enhanced grain growth and it attains smooth surface improves the crystallinity and particle morphology of the sample. FTIR data reveals the presence of M-O bonds and Y-O bonds.

# Keywords—Flux, combustion, FTIR and SEM

# I. INTRODUCTION

White light emitting diodes (LEDS) are given much importance because of their unique properties like low consumption, luminescence efficiency and long life time [1-3]. Addition of flux has several advantages like reducing the processing temperature, increases the luminescence properties as the flux material has low melting point than the solution combustion reaction temperature [4]. This results the material with good crystallinity, particle size distribution. In the present work the effect of various flux namely NH4Cl on the Y<sub>2</sub>SiO<sub>5</sub>:  $Dv^{3+}$ (9mol %) nanophosphor its characterization, morphology are studied. The flux is named as NH<sub>4</sub>Cl -flux 1. The purpose of this work is to investigate the role of flux on the phase formation, surface morphology so that it can be used for further studies.

## **II. EXPERIMENTAL**

## A Synthesis

The phosphors were prepared by combustion method. For the synthesis of phosphors, 0.027g of Dy<sub>2</sub>O<sub>3</sub> and 1.109g of Y<sub>2</sub>O<sub>3</sub> were taken initially as raw materials to which 1:1 HNO<sub>3</sub> is added. This aqueous solution was heated on a sand bath to remove the excess HNO<sub>3</sub>. To this transparent solution 0.3g of SiO<sub>2</sub>, 0.04308g of NH<sub>4</sub>Cl and 1.77g of ODH are added. This mixture is kept in muffle furnace at a temperature of  $\sim$  500°C which undergoes combustion reaction. The obtained product is calcined at 1300 °C for 3h.

0.027g Dy<sub>2</sub>O<sub>3</sub> + 1.109g of Y<sub>2</sub>O<sub>3</sub>  $\rightarrow$  0.3g SiO<sub>2</sub>+ 0.04308g of NH<sub>4</sub>Cl + 1.77g of ODH--(1)

## B Instruments

The important information like lattice parameter, crystallinity of the sample, average crystallite size,

PanalyticalX'Pert pro MPD CuK $\alpha$  (1.541 Å) with nickelfilter. To study the morphology of the samples and to know about the dependence of flux on topography Scanning electron microscopy (SEM) Jeol JSM 7500F Field emission scanning electron microscope is used. In the present work we used Perkin Elmer FTIR spectrometer to study FTIR analysis of the prepared samples.

## **III RESULTS AND DISCUSSION**

A Phase identification of Y<sub>2</sub>SiO<sub>5</sub>:  $Dy^{3+}$  doped with flux1

Phase purity of various flux synthesized samples were investigated by XRD analysis. Fig.1 illustrates the XRD patterns of zero flux and flux1 together with stacked plot of standard pattern of JCPDS card number 36-1476. The diffraction patterns are consistent with the standard data



Fig.1 PXRD patterns of different fluxes used in Y2SiO<sub>5</sub>: Dy<sup>3+</sup> (9 mol %) nanophosphor

which indicates that prepared phosphors are well matched with pure monoclinic phase Y<sub>2</sub>SiO<sub>5</sub>:Dy<sup>3+</sup> phosphors. Adding of flux1 (NH<sub>4</sub>Cl) to the phosphor results in lower phase transformation as flux 1 has decomposition reaction at quite lower temperature and due to this effect plays key role for the cleaning effect of the surface particles. This improves the reaction reactivity and further lowers the synthesis temperature of the samples [5].

#### **B FT-IR Analysis**

FT-IR absorption bands of the zero flux, flux 1 (NH<sub>4</sub>Cl) used in Y<sub>2</sub>SiO<sub>5</sub>:Dy<sup>3+</sup> (9 mol %) nanophosphors recorded are shown in the Fig.3. For zero flux the bands at 593 cm<sup>-1</sup> can be attributed to Y-O stretching vibrations and the bands > 800 cm<sup>-1</sup> can be attributed to the Si-O bands. Similarly for flux 1 the bands at 599 cm<sup>-1</sup> are attributed to Y-O stretching bands, bands at 841 cm<sup>-1</sup> and 881 cm<sup>-1</sup> are the characteristic of M-O bands. The bands above 900 cm<sup>-1</sup> are due to the Si-O vibrations.



Fig.2 Fourier transform infrared spectra of the (a) zero flux, (b) flux 1 (NH<sub>4</sub>Cl) used in Y<sub>2</sub>SiO<sub>5</sub>:Dy<sup>3+</sup> (9 mol %) nanophosphors

### C SEM &EDAX

Surface morphology of the prepared phosphors were analyzed by the SEM shown in the Fig.4. The SEM micrographs in Fig.4 (A) and (B) were recorded with magnification 1µm for zero flux and flux 1 samples respectively. The sample for zero flux has agglomeration, with particle size  $\sim$  159 nm, after introducing flux the number of voids increase for flux 1 shown in Fig.4(B). As the melting point of all the flux i.e., flux 1 (NH<sub>4</sub>Cl -338°C) is less than the solution combustion synthesis reaction temperature (450 °C) this results in melting of flux components there by diffuses into the host reaction region. In the calcining process the melting of flux will eliminate the solidsolid grain boundaries and makes the particles to form a smooth surface [6].



Fig.4 (A) and (B), SEM Micrographs of (magnification  $1\mu$ m) zero flux and 1 (NH<sub>4</sub>Cl) used in Y<sub>2</sub>SiO<sub>5</sub>:Dy<sup>3+</sup> (9 mol %) nanophosphors.

### IV CONCLUSIONS

To improve the luminescence properties of prepared Y<sub>2</sub>SiO<sub>5</sub>:Dy<sub>x</sub><sup>3+</sup> phosphors were doped with flux like NH<sub>4</sub>Cl. It is observed that effect of flux is dependent on XRD, morphology. From XRD data it is observed that effect of flux influence the host by broadening of the peaks and hence it results small crystallite size  $\sim$  20 nm - 22 nm. SEM morphology has agglomeration before adding flux and after adding flux found to have smooth surface as the melting of flux eliminates solid-solid grain boundaries and makes the particles to form a smooth surface [6,7].

#### **ACKNOWLEDGEMENTS**

The author is thankful for co-operation and the support provided by the Dr. A. Jagannatha Reddy, HOD Physics and Management of New Horizon College of Engineering.

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