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Effect of firing temperature on the particles size of Gd_2O_3: Eu doped nanophosphors

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Abstract
The paper reports that synthesis and characterization of Eu^{2+} doped Gd_2O_3 nanophosphors prepared by combustion synthesis. It has been reported that the effect of synthesis temperature on particle size dependence phenomenon of rare earth doped nanophosphors. The sample was characterized by XRD pattern and FTIR study. Synthesized sample shows cubic structure verified by the XRD results. Size of the particle was caculated by scherer equation the avarage size obtained 15, 23 and 55 at 400^0C, 500^0C and 600^0C respectively. In this synthesis urea used as a fuel for created reducing atmosphere during sample preparation.

Keywords: Gd_2O_3: Eu^{2+}, combustion methods, FTIR, XRD.

INTRODUCTION

At present, lanthanide-doped oxide nanoparticles are of special interest as potential materials for an important new class of nanophosphor. For instance, Gd_2O_3 nanoparticle is a promising host matrix for multiphoton and up-conversion excitation [1]. Gd_2O_3 doped with Eu^{2+} (Gd_2O_3:Eu) exhibits paramagnetic behavior as well as strong ultraviolet and cathode-ray excited luminescences, which are useful in biological fluorescent labeling and display applications, respectively [2, 3]. Gd_2O_3 has been frequently used as host material due to abundant resource in nature. It has an additional advantage as Gd is a known contrast agent for magnetic resonance imaging (MRI), and thus the rare-earth ion-doped Gd_2O_3 can be used for dual, MRI and fluorescence imaging applications [4-6] Gd_2O_3 is an excellent host matrix due to its lower phonon energy, better chemical durability and thermal stability [7]

Gd_2O_3:Eu^{2+} has been a promising candidate material owing to improved luminescent properties and well energy transfer between Gd^{3+} and Eu^{2+} ions [8], many methods, including sol–gel method [9,10], emulsion method [11], and precipitation method [12] were used to prepare the nanosized phosphors. Apart from these methods, combustion method has been extensively studied and used in the preparation of nanoparticles, in which glycine was usually used as a fuel for the combustion reaction [13–15]. However, the nanoparticles with monoclinic phase structure and network structure were usually obtained due to higher combustion temperature [16].

Experimental

In this study Gadolinium Nitrate (99.99% Sigma Aldrich), Europium nitrate (99.99% Sigma Aldrich), urea were used as starting raw material. To prepare Gd_2O_3: Eu $^{3+}$, These $Gd(NO_3)_3$ and $Eu(NO_3)_3$ were mixed according to the stoichiometry equation in a beaker and then a suitable amount of urea was added to prepare the precursor solution and kept stirring for 20 min. An amount of $Gd(NO_3)_3$ solution, $Eu(NO_3)_3$ solution (Eu^{2+} doping concentration is 1%, 1.5%, 2% 2.5 %,3%, 3.5%, 4% 4.5% and 5%). Finally this sample was transferred to crucible and fired in a furnace at different temperature (i. e. 400^0C, 500^0C and 600^0C) then water was evaporated quickly and soon a vigorous redox reaction occurred, the whole process went on for a few seconds at different temperature. Finally Gd_2O_3:Eu^{2+} nanoparticles with different concentration were obtained. In order to investigate the effect of firing temperature on structure and luminescence properties of nanoparticles. The Synthesis reaction is

$$(2\text{-}2x)\ Gd(NO_3)_3 + 2xEu(NO_3)_3 + 5NH_2CONH_2 \rightarrow (Gd_{1-x}Eu_x)_2O_3 + 5CO^{2+} 8N^{2+} 10H_2O$$

According the reaction above, the stoichiometry for the preparation of Gd_2O_3:Eu^{2+} from $Gd(NO_3)_3$ and Eu $(NO_3)_3$: Urea was 1:5 was optimized. The structural properties and sizes of the Eu doped Gd_2O_3 were determined by X-ray diffraction studies with Cu Kα radiation (λ=1.5418 A^o).XRD data were collected over the range 20^o-60^o at room temperature. The X-ray diffraction patterns have been obtained data from X-ray Powder diffractometer .The particle size was determined using the scherer's formula.

RESULTS AND DISCUSSION

Infrared analysis

In order to determine the behavior of the sample at different synthesis temperature, infrared spectra of Er^{2+} doped gadolinium nanophosphor represent in (Fig. 1). The obtained spectra exhibited characteristic bands corresponding to gadolinium around 1605–1560 cm−1 and 1550– 1500 cm−1 were assigned to C–O and C–C stretching vibrations. At 400 °C and 500 °C also presented these bands and at 600 °C, the observed bands were typical of the cubic phase of Gd_2O_3 (545 cm−1) which demonstrated that crystallization process takes place, since at 600 °C, there was significant elimination of residual organic compounds. It is important to note that

for Er^{2+} doped Gd_2O_3 powders, two low intensity peaks in the region of 1600– 1300 cm−1, due to N–O and CO_3 and other bands observed at ~3450 cm^{-1}, assigned to –OH groups of H_2O, were still visible.

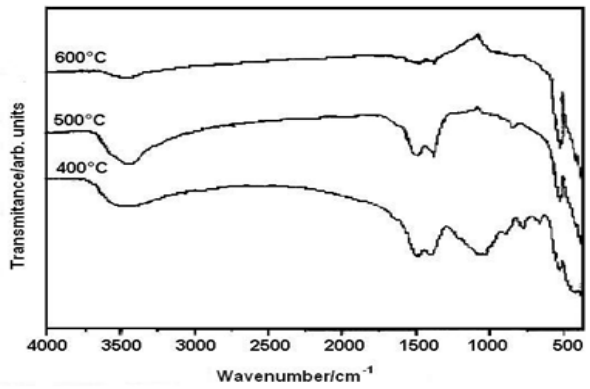

Fig 1. Infrared spectra Gd_2O_3:5%Eu^{2+} nanoparticles prepared under different temperatures

XRD (X Ray Diffraction) ANALYSIS

The prepared phosphor materials were analyzed by XRD to reveal phase compositions and the particle size at different firing temperatures. The crystallite size was calculated from the XRD pattern following the Scherer equation

$d = 0.9\lambda / \beta \cos\theta.$

Here, d is the crystallite size for the *(hkl)* plane, λ is the wavelength of the incident X-ray radiation [CuKα(0.154056 nm)], β is the full width at half maximum (FWHM) in radians, and θ is the diffraction angle for the *(hkl)* plane. The XRD patterns shown in Fig. 2

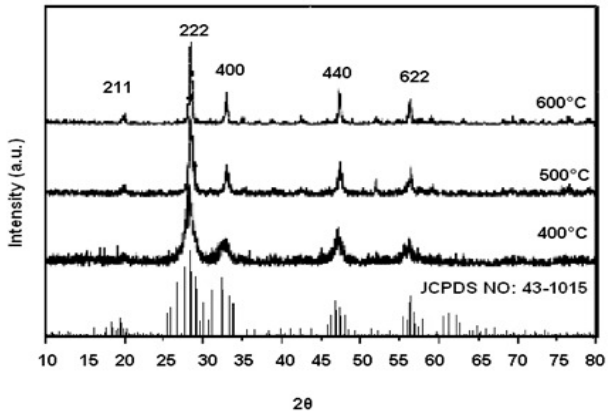

Fig 2. XRD patterns of the precursor and Gd_2O_3:5%Eu^{2+} nanoparticles prepared under different synthesis temperatures

CONCLUSION

Cubic-phased Gd_2O_3:Eu^{2+} nanoparticles with different sizes and different doping Eu^{2+} concentration were prepared by combustion method. The results of XRD show pure phase can be obtained, the average crystallite size could be calculated as 15, 23 and 55 nm for 400^0C, 500^0C and 600^0C respectively.

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