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Original research article

Optical and Structural characterization of pure and zinc-doped lead oxide nanostructures synthesized by chemical root method



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ABSTRACT

Our goal in this research was to obtain pure and zinc doped lead oxide nanostructure through chemical root method. 0.04 M aqueous solution of lead nitrate and zinc chloride was mixed with 0.08 M aqueous solution of sodium hydroxide for making reaction solution. 0.001 M aqueous solution of TEA ((Tri Ethanol Amine) was also added to reaction solution. The prepared pure and Zn doped lead oxide nanostructure was characterized by Fourier transformation infrared spectroscopy(FTIR), X-ray diffraction (XRD), energy dispersive X-ray analysis (EDAX), scanning electron microscopy (SEM) and high resolution-transmission electron microscopy (HRTEM) and selected area electron diffraction pattern (SAED) pattern reveals the crystalline nature of the sample. The prepared pure and doped lead oxide consists of the average crystallites about 12 and 5 nm respectively. Optical band gap of pure and doped lead oxide sample are 5.17 and 5.5 eV obtained respectively. The photoluminescence (PL) spectra were investigated for both the sample. The PL spectrum shows peaks in UV region at 356 nm, blue regions around 421 nm and in green region around 544 nm. The spectra show a strong emission band around 421 nm.

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1. Introduction

In recent years, considerable interest has been focused on the synthesis of nanomaterials in the various scientific and industrial fields due to its different applications such as electronics, magnetic and optoelectronic, biomedical, pharmaceutical, cosmetic, energy, environmental, catalytic, sensors, fuel cells, paints, rechargeable batteries, pigments and so on [1–4].

Lead oxide is one of these semiconductors that have important applications in storage batteries, glass industry and pigments [5]. We have lots of oxide forms of lead like PbO (α , β and amorphous), Pb₂O₃, Pb₃O₄ and PbO₂ (α , β and amorphous). Lead oxide is an important industrial material due to its unique electronic, mechanical and optical properties and its potential applications in nano-devices and functionalized materials [6]. PbO itself has two forms: yellow β -PbO, which is stable at high temperature and red α -PbO, which is stable at low temperature. The α -PbO phase transformation to β -PbO takes

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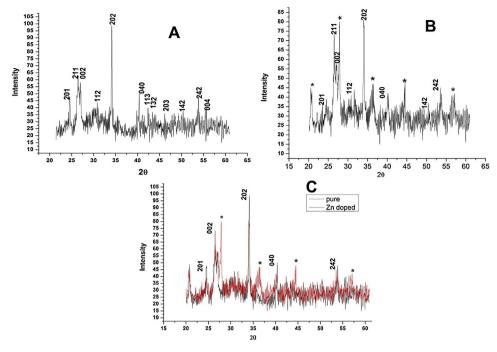


Fig. 1. XRD patterns of [A] pure lead oxide [B] Zn doped lead oxide [C] comparative patterns of pure and Zn Doped lead oxide.

place at about 490 °C [7]. Reliability, easy to design, low production cost, and relative safety are some quality, due to this, research is still interesting. It has to improve different properties of lead oxide and lead dioxide.

Nano range particle have been prepared by different methods, like combustion method, sol gel, oxide-assisted growth, Sold state reaction, solution-liquid growth in organic solvent and chemical bath deposition method [8–13]. Here we used Chemical route method because of its simplicity, convenience and low cost. In this paper, we have reported plate like morphology of pure and Zn doped lead oxide nano powder by using complexing agent (TEA). Our previous results have shown that low concentration of TEA is responsible for synthesis of nanoparticles [14,15]. This was rarely observed ever before. To our knowledge there is no literature available on the optical effects of doping Zn into the lead Oxide host material in this method.

2. Experimental procedure

2.1. Powder preparation

For making first reaction solution $0.04\,\mathrm{M}$ aqueous solution of lead nitrate and second reaction solution $0.04\,\mathrm{M}$ aqueous solution of both lead nitrate and Zinc chloride (both in same quantity, 50-50%) are mixed with $0.08\,\mathrm{M}$ aqueous solution of sodium hydroxide (all AR grade 99.9% pure) respectively. After, we have added $8\,\mathrm{ml}$ of $0.001\,\mathrm{M}$ aqueous solution of TEA in both solutions [14,15]. The reaction solution is allowed to stand for $20\text{-}24\,\mathrm{h}$. The powder is thoroughly washed out (more than $8\,\mathrm{times}$) with distilled water. It is filtered and dried in sunlight. It is then annealed furnace at $290\,^{\circ}\mathrm{C}$. The resultant product was in powder form and appeared yellow in colour (pure) and light yellow in colour (doped).

2.2. Measuring instruments

Energy dispersive X-ray (EDX), field emission scanning electron microscopy (FESEM), XRD and transmission electron microscopy (TEM) are employed to characterize the sample. Energy dispersive X-ray analysis [EDX] is used for elemental analysis of the powder. FESEM is used for morphological characterization of sample. The surface morphology of the yellow

Powder was determined by field emission scanning electron microscope (FESEM) JSM-7600F. The structural parameters of the powder were determined using X-ray diffraction technique. The XRD patterns were recorded with Bruker D8Advanced X-ray diffractometer using a Cu K α radiation source ($\lambda\pm1.54056$ Å). The X-rays were detected using a fast counting detector based on silicon strip technology (Bruker Lynx Eye detector). Particle diameter and surface morphology of yellow powder were determined by transmission electron microscope using Philips CM -200. Absorption spectrum was measured by UV spectrophotometer (Varian) [14,15]. The excitation and emission spectra were recorded by using RF 5301 PC spectroflurophotometer by SHIMADZU, the Xenon lamp (365 nm) as excitation source when measuring throughout the measurement.

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