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PbS-NiO nanocomposite material with enhanced magnetic, photocatalytic and antifungal properties



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ABSTRACT

PbS, NiO and PbS-NiO nanocomposites were synthesized by a cost effective chemical route and characterized by techniques like TG-DTA, XRD, SEM, TEM, EDX, FTIR and PL. XRD studies reveal the presence of diffraction peaks related to PbS and NiO in the composite. The visible light photocatalytic tests showed that the PbS-NiO nanocomposite had better photocatalytic activities for the photodegradation of methyl orange (MO) compared to that of pure PbS and NiO nanopowders. Within 210 min of reaction time, nearly 89% decolorization efficiency of MO was achieved by the PbS-NiO photocatalyst, which is higher than that of pure PbS and NiO. The high photocatalytic efficiency of the composite was due to the extended photoresponse range and efficient separation of the electron-hole pairs in the PbS-NiO heterojunction. In addition, the composite also possess excellent antifungal efficiency against *A. niger* fungus strain. Enhanced magnetic properties were observed for the composite.

1. Introduction

Nano-sized semiconductor materials have received considerable attention in recent years due to their novel electronic and optical properties [1]. The composition, structure, morphology, phase, shape, size, distribution and spatial arrangement determine the physical and chemical properties of nanomaterials [2]. Nanosized materials have attracted extensive interest in recent years due to their numerous potential applications in the fabrication of optoelectronic and magnetic devices [3]. Lead sulfide (PbS) is a IV-VI group semiconductor which exhibit strong size quantization effects. It has a direct band gap which varies between 0.41 eV and 2.3 eV. It possesses excellent solar control characteristics and good photoconductive properties [4]. PbS nanosized materials also show excellent near infrared (NIR) emitting optical properties in the wavelength range 700-1600 nm which make them suitable as effective biological labels and light harvesters [2]. The multiple exciton generation (MEG) property possessed, make PbS a suitable candidate for the next generation solar cells [5]. PbS nanoparticles in the size range of 5-8 nm exhibit nonlinear optical property due to strong confinement and the presence of discrete energy levels [6]. Compared to their bulk counterparts, PbS nanoparticles are photoelectrochemically active which makes them suitable as absorber in extremely thin absorber (ETA) solar cells [7]. However, regarding magnetic properties, PbS is a less studied material due to the paramagnetic nature of lead. In recent years, the development of visiblelight driven photocatalysts with high energy transfer efficiency, nontoxicity and low cost has become one of the most challenging tasks, and foremost efforts are dedicated to identify new photocatalytic materials or to modify the existing photocatalysts with enhanced photocatalytic performance. To make PbS suitable as a photocatalyst and to improve its magnetic properties it is essential to dope it with metal ions or to make composite along with some magnetic oxide/sulfide semiconductors. Nickel oxide (NiO) is an important p-type transition metal oxide semiconductor with cubic lattice structure. NiO posses excellent electronic, optical and catalytic properties which make it a raw material for p-type transparent films, gas sensing units, alkaline battery cathodes, dye-sensitized solar cells, photocatalysts, etc. [8]. NiO nanoparticles exhibit different magnetic behaviours such as superparamagnetic, superantiferromagnetic and ferromagnetic order depending on the particle size, shape and synthesis route [9]. Also, presently microbial infection is a serious concern in medical devices, drug delivery systems, water purification systems, textiles and food packaging and storage. Currently, some noble metal oxide/sulfide nanoparticles have been extensively investigated to circumvent the microbial infections [10,11]. The most commonly used metals are gold and silver. However, due to heavy cost other alternative materials should be identified and Cu, Al, Ni are the most promising alternative materials. Recently, NiO based nanomaterials have been widely investigated for their antimicrobial activities [12]. Improved antibacterial activity has been reported for NiO nanoparticles against Escherichia Coli

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by Wang et al. [13]. So in this work, pure PbS, NiO and PbS-NiO nanocomposites were synthesized using a cost effective chemical route. This route has several advantages like simplicity, low cost, low processing temperature, capable of achieving doping, etc. The synthesized samples were characterized by techniques like TG-DTA, XRD, SEM, TEM, FTIR, PL and VSM. To identify the suitability of PbS-NiO nanocomposite as a photocatalyst, photocatalytic activity was performed against methyl orange (MO) under visible light irradiation. Also, antifungal activity was performed against *A. niger* fungus strain by agar well diffusion method and the results obtained are presented in detail.

2. Experimental details

PbS, NiO and PbS-NiO nanocomposites were synthesized by a cost effective chemical route using lead nitrate ($Pb(NO_3)_2$), thiourea ($SC(NH_2)_2$) and nickel chloride ($NiCl_2 \cdot 6H_2O$) as precursor salts.

2.1. Synthesis of PbS nanopowders

Lead nitrate and thiourea each of 0.1 M were weighed and dissolved in 140 ml de-ionized water and stirred well for 30 min. 10 ml liquid ammonia is added to this solution to raise its pH value to 10. The resultant solution after continuous stirring for 2 h was kept undisturbed for 4 h. The black precipitates thus obtained were washed two times with de-ionized water, dried and then calcined at 200 $^{\circ}\text{C}$ for 1 h in a muffle furnace. The resultant compound was crushed well using an agate mortar to avoid agglomeration and to maintain uniformity in the grain size. The powders were then subjected to heat treatment in the furnace at 200 $^{\circ}\text{C}$ for 1 h to stabilize the microstructure of powders without grain growth.

2.2. Synthesis of NiO nanopowders

To synthesize NiO nanopowders, 0.1 M of NiCl₂·6H₂O was dissolved in 140 ml de-ionized water and the same procedure used to synthesize PbS nanopowders was adopted to get green colored NiO nanopowders.

2.3. Synthesis of PbS-NiO nanocomposite

Lead nitrate and thiourea each of 0.1 M were weighed and dissolved in 140 ml de-ionized water. Nickel chloride of 0.1 M is dissolved in 140 ml de-ionized water. To adjust the pH values of these solutions, 10 ml liquid ammonia was added separately. Both the solutions were stirred well separately for 2 h and kept undisturbed for 4 h. The precipitates obtained was filtered, washed two times with de-ionized water and then mixed together and stirred well for 1 h. The resultant products was calcined at 200 °C for 1 h in a muffle furnace and crushed well with agate mortar to get PbS-NiO nanocomposite. Due the electrostatic adhesion force between the electronegatively charged PbS and electropositively charged NiO their charge gets neutralized to form the composite. The color of the composite was found to be intermediate between the black color of PbS and the green color of NiO.

2.4. Characterization techniques

The thermal behaviour, crystal structure and surface morphologies of the samples were studied using TG-DTA instrument NETZSCH STA 449 F3, X-ray diffractometer (X'Pert PRO analytical-PW 340/60) with CuK α X-ray source ($\lambda=1.5406\,\text{Å}$), scanning electron microscope (HITACHI S-3000H) and 200 KV Tecnai-20 G2 TEM instrument, respectively. Functional groups present in the samples were identified using Perkin Elmer RX-1 FTIR spectrophotometer. Photoluminescence studies were performed using Varian Cary eclipse fluorescence spectrophotometer. Magnetic studies was performed using vibrating sample magnetometer (Lakeshore VSM 7401) Photocatalytic property of the samples was evaluated by the photodegradation of methyl orange (MO)

solution under visible light irradiated from a 100 W incandescent lamp. Antifungal activities were performed against *A. niger* fungus strain by agar well diffusion method.

2.5. Formulae used

The crystallite size (D) values of the PbS, NiO and PbS-NiO nanocomposites were estimated using the Scherrer formula:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \tag{1}$$

where λ is the wavelength of the X-ray used (1.5406 Å), β is the full width at half maximum (FWHM) value of the strongest peak and θ is the Bragg angle.

The photocatalytic efficiency (η) was calculated using the relation:

$$\eta = \left(1 - \frac{C}{C_0}\right) \times 100\tag{2}$$

where C_0 is the concentration of MO before illumination and C is the concentration after illumination.

3. Results and discussion

3.1. Thermal analysis

The TG-DTA curves of the PbS-NiO nanocomposite is shown in Fig. 1. Four steps of weight losses were observed in the TG curve along with three endothermic and two exothermic peaks in the DTA curve. The weight losses of 1.8% and 3.2% observed initially in the TG curve is accompanied with an endothermic peak in the DTA curve at 180 °C and with an exothermic peak at 280 °C which are attributed to the evaporation of water molecules and organic residuals, respectively from the sample. The sharp exothermic peak observed at 380 °C with a weight loss of 2.7% in the TG curve may be attributed to the thermal decomposition of inorganic components present in the precursor material. The exothermic peak above 395 °C in the DTA curve is accompanied with a meagre weight loss of 0.9% in the TG curve which might be due to the release of sulfur ions due to the thermal oxidation from PbS and oxygen ions due to heat treatment from NiO of the composite [14].

3.2. XRD studies

The crystal structure of the PbS, NiO and PbS-NiO nanocomposites were determined from the XRD patterns shown in Fig. 2.

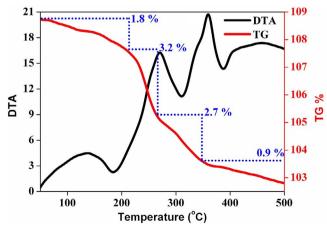


Fig. 1. TG-DTA curves of the PbS-NiO nanocomposite.

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