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Synthesis and Characterization of ZnO/NiO and Its Photocatalytic Activity

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ABSTRACT. Bimetallic ZnO/NiO was synthesized by a simple one-pot solvothermal method with zinc nitrate hexahydrate and nickel nitrate hexahydrate in ethanol medium. Material was characterized by X-ray diffraction, Field emission scanning electron microscope, UV-Vis and FTIR. The proposed mixed oxide was investigated as a suitable photocatalytic material for the degradation of organic dyes under visible light irradiation. The formation of ZnO/NiO hetero-junction improved the separation rate of photogenerated electrons and holes and therefore yields enhanced photocatalytic efficiency.

Introduction. Nanostructured metal oxide materials fascinated the lot of attention due to their multifunctional activities including optical, magnetic, electrical and catalytic properties. These interesting features increases the wide utility of the materials to many promising applications. Zinc oxide (ZnO) as n-type semiconductor with a direct wide band gap of (Eg = 3.2 eV), has high photosensitivity, photocatalytic activity, quantum efficiency, non-toxicity and low cost [1]. Its extraordinary structural and microstructural benefits further extends their applications to gas sensors, solar cells, light-emitting diodes, field effect transistors, varistors and piezoelectric devices. To date several morphologies such as nanotubes, nanoflowers, nanowires and nanorods of ZnO have developed and studied as the photocatalysts for degradation of organic pollutants. Recent years it has been well established that the coupling of two different semiconductors with different energy levels of photogenerated electron–hole pairs enhanced their functional properties due to their interfacial activity. In particular, the combination of p and n type binary semiconductor oxides could form the p-n junction at the interface and lead to the effective separation of electron-hole pairs. Among various p-type oxides nickel oxide (NiO) is a highly active material with wide band gap (3.6eV to 4.0 eV) and extensively studied for various applications such as catalysis, gas sensing, battery cathodes, magnetic materials, electrochromic films, chemical sensors and photovoltaic devices [2]. In this study, the NiO–ZnO binary composite oxide has been synthesized by one-pot solvothermal synthesis and investigated as the visible light catalyst for photodegradation. The photogenerated electron–hole pairs were effectively separated, which facilitates to enhance their photocatalytic activity.

Experimental

Materials

Zinc nitrate, nickel nitrate and sodium lauryl sulphate was purchased from SRL. Absolute ethanol was used as a solvent without purification. 2mmol Zn(NO₃)₂, 6H₂O and 2mmol Ni(NO₃)₂.6H₂O were dissolved in 40 mL of ethanol. The mixture was stirred for 15 min and then 0.1g of SDS was added as a surfactant. 1M NaOH was used to adjust pH before the mixture was sealed in a Teflon-
lined stainless-steel autoclave. The autoclave was heated to 140°C for 12 h, then cooled to room temperature. The final product was collected by centrifugation and washed with ethanol and deionized water for five times. Then the product was dried and annealed at 450°C for 3 h.

**Instruments.** The powder X-ray diffraction (XRD) of the sample were performed on Rich Siefert 3000 diffractometer Cu-Kα1 radiation (λ=1.5406Å). Ultraviolet visible spectral analysis was performed on Perkin Elmer lambda 650. The morphology of the samples was analyzed by FE-SEM using a HITACHI SU6600 filed emission - scanning electron microscopy.

**Results and discussion**

Fig. 1 (a) shows the XRD pattern of ZnO/NiO binary composite. The diffraction peaks at 31.2°, 33.7°, 35.6°, 46.7°, 55.8°, 67.3°, 68.6° of ZnO were well matched with standard (JCPDS No. 76-0704). In the meantime the ZnO is in good agreement with the hexagonal phase. Moreover, diffraction peaks at 42.5°, 62.2°, are related to the NiO and directly indexed to (JCPDS No. 65-2901). The observed peaks indicates the NiO phase is the face centered cubic structure. And there is no other impurity phase or secondary phase in the XRD pattern. Typical UV- Vis spectrum of ZnO/NiO composite is shown in Fig.1 (b). It exhibit absorption band at 200-400nm indicating the extended UV-vis absorption property of the mixed oxide.

![Fig. 1. (a) XRD pattern and (b) UV Vis spectrum of ZnO/NiO.](image)

Fig. 2 shows the FT-IR spectrum of ZnO/NiO composite. It shows the absorption the region of 1128 cm⁻¹, 1392 cm⁻¹, 1613 cm⁻¹ and 3442 cm⁻¹. The absorption band at 400 cm⁻¹ to 600 cm⁻¹ corresponds to M-O bond [4]. The band observed at 1128 cm⁻¹ corresponds to C-O stretching vibration. The band appearing at 1392cm⁻¹ attributed to C-H bending vibration. The band at 1613 cm⁻¹ corresponds to the symmetrical of C-O. The band at 3442 cm⁻¹ corresponds to stretching vibration of the –OH group.

The FESEM image and EDS spectrum of ZnO/NiO composite are given in Fig. 3. The FESEM image of ZnO/NiO composite shows undefined coarse morphology. The non-uniform particles are agglomerated and forms irregular shaped particles. Fig. 3 shows the EDS spectrum which confirms the presence of Zn, Ni, O and no other impurity present in the sample.
**Photocatalytic activity.** The photocatalytic degradation of methylene blue was studied by taking 100ml double distilled water containing 0.001M of methylene blue and 0.05g of ZnO/NiO. The reaction mixture was irradiated under visible light. For irradiation purpose, 500W Halogen lamp was used in photo reactor chamber. Fig.4 shows the absorption spectrum of methylene blue subsequently the visible light irradiation for different time interval. It can be seen that the intensity of the absorption peaks decreased as the reaction progressed with ZnO/NiO nanocomposite as a catalyst. After 60 min of irradiation, the intensity of the absorption peaks decreased to 30% of the initial methylene blue solution. When the irradiation time was increased to 180 min, the degradation of MB was nearly ~85%. This result shows that the ZnO/NiO exhibit good photocatalytic activity. The surface interface of the combined systems ZnO/NiO significantly influenced on the structure relation properties. Thereby, the photocatalytic activities has been increased. This p-n hetero-junction forms the negative and positive charges in p-NiO and n-ZnO regions respectively. In the ZnO/NiO composite the photogenerated e/h^+ pairs form at the interface. The photogenerated electrons can easily migrate from NiO to ZnO because conduction band-NiO is more negative than the conduction band-ZnO. At the same time, the hole transfer take place in opposite direction from valence band-ZnO to valence band-NiO. So the e/h^+ recombination processes decrease significantly and increase the photocatalytic activity [3, 5].
Summary. ZnO/NiO composite was synthesized by one-pot solvothermal method. The synthesized ZnO/NiO was confirmed by XRD analysis and the material was characterized by various techniques. The morphologies of the sample found to be aggregated nanoparticles. The photocatalytic results show the ZnO/NiO composite has better degradation efficiency under visible light irradiation towards MB. Hence it can be suggested that the formation of p-n hetero junction enhances the separation rate of photogenerated electrons and holes and therefore yields enhanced photocatalytic efficiency.

References


