Dependence of defect concentration, optical and photocatalytic properties of β -Ni(OH)₂/ZnO and NiO/ZnO composite powders on phase transformation

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β -Ni(OH)₂/ZnO composite powders were successfully synthesized by hydrothermal method at 180 °C for 15 h whereas NiO/ZnO composite powders formed after the as-prepared powders were calcined at 800 °C for 1 h in air. The X-ray diffractometer (XRD), scanning electron microscope (SEM), UV-Vis spectrophotometer were used to characterize the phase, particle shape as well as size and optical properties, respectively. In this system, it was found that ZnO is a major phase while β -Ni(OH)₂ and NiO are a minor phases. The altered particle shape of ZnO was influenced by addition of Ni(CH₃COO)₂ · 6H₂O whereas the particle shape of the minor phase was changed due to the calcination process. The optical band gap decreased when the amount of minor phase increased. For photocatalytic study, it was found that 6 mol% β -Ni(OH)₂/ZnO composite powders exhibited the best decolorization of methylene blue aqueous solution.

Keywords: ZnO; composites; chemical synthesis; defects; optical properties

1. Introduction

Recently, the textile industry has been one of the largest polluters in the world because they manufacture many products each year. In a typical processing, several manufacturing processes such as dyeing and finishing release many chemicals including aromatic amines, heavy metals, ammonia solutions, alkali salts and pigments, etc. to the surface water sources. Untreated dyes or these chemicals cause the water pollution crisis and this phenomenon significantly affects the trees, animals and humans. Therefore, the treatment of these dangerous dyes or chemicals before being released to the surface water sources is very important to corporate social responsibility that the textile industries are always conscious. As far as we know, there

are many methods [1–4] for textile wastewater treatment, such as biological treatment, electrocoagulation, electrochemical oxidation, ozone, membrane filtration and photocatalytic route. Among these methods, heterogeneous photocatalysis using semiconducting powder such as zinc oxide as a catalyst is of higher interest than the conventional method for removing organic species in the wastewater as it offers several advantages; for example, the reaction is made at room temperature and ambient pressure, no additional reagent except catalyst is used and the final products show little or no toxicity [5]. Variety of catalysts including TiO₂ [6], Bi₂MoO₆ [7], ZnO [8], CuO [9] and m-BiVO₄ [10], etc. have been used during this time; however, many research groups paid attention to ZnO powders. Not only the morphology, but also the synthetic method was taken into account so that the photocatalytic efficiency could be

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enhanced significantly. Focusing on the synthetic methods, ZnO powders can be synthesized easily by various methods such as ultrasonic method [11], combustion [12], hydrothermal [13], sol-gel [14] and precipitation [15]. Under different points of view, hydrothermal method is still popular because of its numerous advantages such as low cost, ease of mass production, simple process, high-yielding, controllable process and unsophisticated apparatus [16]. Additionally, a fabrication of metal oxide heterojunction or metal oxide composite is an alternative way to improve the photocatalytic efficiency. It can retard the recombination of the photogenerated electrons and holes during the photocatalytic processes. In this study, we investigate the phase transformation, optical properties and photocatalytic activities of β -Ni(OH)₂/ZnO and NiO/ZnO composites with different loading contents prepared by a hydrothermal method.

2. Experimental

2.1. Synthesis procedure

Zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$), nickel acetate hexahydrate ($\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 6\text{H}_2\text{O}$) have been obtained from Sigma-Aldrich. Potassium hydroxide has been obtained from Emsure® and polyethylene-block-polypropylene (PEO₁₉-b-PPO₃, M.W. 1000) has been purchased from Jeffamine®. The reagents were used as received without further purification.

The β -Ni(OH)₂/ZnO and NiO/ZnO composite powders were synthesized in the same way by hydrothermal method. In this study, a stoichiometric amount of $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ and $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 6\text{H}_2\text{O}$ was weighed to obtain a series of $\times$ mol% loading contents ($x = 0, 2, 4, 6, 8$ and 10). To synthesize pure ZnO powders, $0.015 \text{ mol } \text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ was dissolved in 100 mL of distilled water and stirred by a magnetic bar stirrer to obtain a clear solution, then $11.25 \text{ mmol PEO}_{19}\text{-b-PPO}_3$ was added into the zinc precursor solution under a continuous stirring for 10 min . Finally, 0.075 mol KOH that dissolved in 100 mL of distilled water was slowly dropped into the PEO₁₉-b-PPO₃-modified zinc precursor

solution. The white precipitates were formed during the addition of the aqueous KOH solution and the precipitates were stirred continuously for 15 min . After that, they were transferred into a Teflon-lined reactor and heated at 180°C for 15 h . When the reactors were cooled to room temperature, the precipitates were filtered and washed with distilled water several times, rinsed with absolute ethanol and dried at room temperature.

To synthesize the β -Ni(OH)₂/ZnO composite powder, $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ and $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 6\text{H}_2\text{O}$ were first dissolved in 100 mL of distilled water and the other processes were followed as for synthesizing pure ZnO powders. On the other hand, the NiO/ZnO composite powders formed when the as-synthesized β -Ni(OH)₂/ZnO composite powders were calcined at 800°C in air for 1 h .

2.2. Characterizations

X-ray diffraction (XRD) analysis was performed on powdered samples using a X'Pert PD, Philips with a $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). Scanning electron microscope (SEM) images were taken by using Quanta 400, FEI. UV-Vis diffuse reflectance spectra (DRS) were measured in a wavelength of 200 nm to 800 nm by Shimadzu 2450 spectrophotometer equipped with an integrating sphere.

2.3. Photocatalytic degradation test

Methylene blue (MB) was used as a dye model to evaluate a photocatalytic degradation of β -Ni(OH)₂/ZnO and NiO/ZnO nanocomposite powders in this study. The photocatalytic activities were studied under the irradiation of UV light. 150 mg of photocatalyst was suspended in a 250 mL aqueous solution of $1 \times 10^{-5} \text{ M}$ in a glass beaker. The suspension was kept and stirred in the dark for 30 min to ensure an establishment of adsorption-desorption equilibrium on the surface of photocatalyst. Subsequently, the suspension was irradiated under three parallel black light fluorescent tubes (SYLVANIA, Blacklight Blue Lamp, wavelength of 316 nm to 400 nm , 18 W) positioned about 15 cm away from the glass beaker. 3 mL of

working suspension was sampled at given time intervals (30 min) and centrifuged at 4000 rpm for 3 min to separate the photocatalyst. The absorbance of the degraded solution was measured using UV-Vis spectrophotometer (UV-Vis Lambda 25, PerkinElmer) and the absorption peak at 664 nm was monitored. The decolorization of MB was calculated by the following equation:

$$D = \frac{C}{C_0} = \frac{A}{A_0} \quad (1)$$

where D is the decolorization, C_0 is the initial concentration, C is the concentration at any time, A_0 and A are the initial and instantaneous absorbance of MB solution at 664 nm, respectively.

3. Results and discussion

3.1. Structural characteristics

Based on the X-ray analysis as shown in Fig. 1, it was observed that the as-prepared samples exhibited a major phase of ZnO (JCPDS Card No. 36-1451) and a minor phase of β -Ni(OH)₂ or theophrasite (JCPDS Card No. 14-0117). On the other hand, the samples calcined at 800 °C for 1 h showed that ZnO was a major phase as in the case of no calcination process, whereas NiO was a minor phase (JCPDS Card No. 04-0835). The amount of both β -Ni(OH)₂ and NiO increased as a function of Ni(CH₃COO)₂ · 6H₂O concentration. In this study, it was clearly seen that the calcination temperature significantly affected the transformation of nickel oxide as shown in equation 2 to equation 4 [17–19]:

It has been well known that different structural characteristics influence various properties of the

samples. Therefore, the different phases of nickel oxides resulted in the optical and photocatalytic properties in a different way. The results were reported in other sections.

Fig. 1. XRD patterns of (a) β -Ni(OH)₂/ZnO and (b) NiO/ZnO composite powders at different loading contents.

To investigate the influence of nickel concentration on particle size and shape of the samples, the SEM was used and the results are presented in Fig. 2 and Fig. 3. Considering Fig. 2, pure ZnO powders exhibit platelet structures. As the Ni(CH₃COO)₂ · 6H₂O was added into the precursor solution, the morphology of the samples altered to another shape. A bundle of hexagonal ZnO prism and β -Ni(OH)₂ platelet formed in the as-prepared samples. The β -Ni(OH)₂ platelet adhered to the hexagonal ZnO prism. In this study, it has been found that the particle size of ZnO increased when Ni(CH₃COO)₂ · 6H₂O was added into the precursor solution because of the increase in nucleation of particles which enhanced

the grain growth of ZnO [20]. The morphological change was not caused by the hydrothermal conditions because they were kept constant throughout the experiment. As we know, ZnO crystal is polar in nature. The crystal contains Zn terminated (0 0 0 1) positive plane and oxygen terminated (0 0 0 $\bar{1}$) negative plane. Generally, the positive (0 0 0 1) plane is more reactive than the negative (0 0 0 $\bar{1}$) plane and this gives rise to an improvement of anisotropic growth along the *c*-axis. Additionally, the higher symmetry (C_{6v}) of positive (0 0 0 1) plane compared to the other planes causes the highest growth rate along the *c*-axis as well [21]. In this study, pure ZnO powder exhibited a platelet structure since the positive (0 0 0 1) planes usually expose terminal OH^- ligands to the solution. This prevented the further extensive deposition of Zn species on (0 0 0 1) planes, giving rise to a suppression of crystal growth and its sideways growth along the (2 $\bar{1}$ $\bar{1}$ 0) direction resulting in the forming of platelet shape. Interestingly, when $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 6\text{H}_2\text{O}$ was added into the precursor solution, the Ni^{2+} ions could react with OH^- and form a $\beta\text{-Ni}(\text{OH})_2$ phase. Therefore, the OH^- could react decreasingly at (0 0 0 1) positive planes. This phenomenon could accelerate the deposition of ZnO species on (0 0 0 1) planes instead, the bundle of hexagonal ZnO prism generated and $\beta\text{-Ni}(\text{OH})_2$ platelets were adhered to the hexagonal ZnO prism [22].

For calcined samples as shown in Fig. 3, the morphology of ZnO structure did not change, but the $\beta\text{-Ni}(\text{OH})_2$ platelets changed to spherical NiO particles. The particle size in the sample slightly decreased when the samples were calcined at 800 °C for 1 h. This might be due to the fact that some Ni^{2+} ions could substitute at the Zn^{2+} sites, when calcining at high temperature, but the ionic radii of Ni^{2+} and Zn^{2+} did mismatch. This gave rise to the suppression of the particle growth.

3.2. Optical properties

Optical properties of the samples were investigated because they are important in many applications. In this study, the effect of $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 6\text{H}_2\text{O}$ concentration on the optical

Fig. 2. SEM images of $\beta\text{-Ni}(\text{OH})_2/\text{ZnO}$ composite powders at different loading contents.

band gap (E_g) was studied. The absorption behavior of the samples was characterized by diffuse reflectance spectroscopy (DRS). It had been observed that the as-prepared composite powders ($\beta\text{-Ni}(\text{OH})_2/\text{ZnO}$) showed the different absorption behavior to the calcined composite powders (NiO/ZnO). In this study, $\beta\text{-Ni}(\text{OH})_2/\text{ZnO}$ composite powders had a lower absorbance in a UV region than pure ZnO powders and the absorbance decreased when the $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 6\text{H}_2\text{O}$ concentration was increased. On the contrary, $\beta\text{-Ni}(\text{OH})_2/\text{ZnO}$ composite powders showed lower absorbance than pure ZnO powders at the wavelength range of about 410 nm to 450 nm and 550 nm to 800 nm. The absorption edge of the $\beta\text{-Ni}(\text{OH})_2/\text{ZnO}$ composite powders shifted to the higher wavelength or lower energy compared to pure ZnO powders. This meant that $\beta\text{-Ni}(\text{OH})_2/\text{ZnO}$ composite powders had narrower E_g value than that of pure ZnO powders.

Fig. 3. SEM images of NiO/ZnO composite powders at different loading contents.

For calcined samples, pure ZnO powders and NiO/ZnO composite powders exhibited similar absorbance in UV region, but the NiO/ZnO composite powders showed higher absorbance in visible region. The absorbance in visible region of NiO/ZnO composite powders is quite different from β -Ni(OH)₂/ZnO composite powders, namely the NiO/ZnO composite powders showed the absorbance consistently in the whole range of visible region.

From the DRS spectra, the E_g value can be estimated by using the Tauc formula [2]. Typically, Tauc plot shows the quantity E on x-axis and the quantity $(\alpha E)^{1/n}$ on the y-axis, where α is the absorption coefficient of the material and E is the photon energy. The value of the exponent n denotes the nature of the transition: $n = 1/2$ for direct allowed transitions, $n = 3/2$ for indirect allowed transitions, $n = 2$ for indirect allowed transition and $n = 3$ for indirect forbidden transitions. In this study, the

ZnO, β -Ni(OH)₂/ZnO and NiO/ZnO powders exhibited a direct transition, and the Tauc plots for evaluating the E_g value of the samples are shown in Fig. 4 and Fig. 5. The extrapolation of the linear region to the x-axis gives the E_g value and the obtained E_g values are given in Table 1.

Fig. 4. Plots of $(\alpha E)^2$ versus E or photon energy for evaluating the E_g value of β -Ni(OH)₂/ZnO composite powders at different loading contents.

As shown in Table 1, it was found that the E_g values of both β -Ni(OH)₂/ZnO and NiO/ZnO composite powders decreased with the increase in $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 6\text{H}_2\text{O}$ concentration. In this study, the particle size and shape of all samples obtained in all studied synthetic conditions were quite equivalent as observed in SEM images, therefore, the dependence of E_g values on the particle size and particle shape was not considered. As far as we are concerned, ZnO semiconductor powder contains numerous defects in its lattice, such as zinc interstitials, oxygen vacancies, zinc vacancies and oxygen antisites [2]. These defects normally generate the energy bands between the conduction band and valence band. This phenomenon could alter

Fig. 5. Plots of $(\alpha E)^2$ versus E or photon energy for evaluating the E_g value of NiO/ZnO composite powders at different loading contents.

the density of states with the degenerated band tail stretched into the band gap, resulting in a reduction of band gap of ZnO powders. Therefore, in ZnO having higher defects concentration, the E_g values are narrower [23].

In this study, the Urbach law was applied to determine the defect concentration and disordered structures. The relationship is presented as follows [24]:

$$\alpha = \alpha_0 \exp[(E - E_g)/E_u] \quad (5)$$

where α is an absorption coefficient, α_0 is a constant, E is the photon energy, E_g is a band gap and E_u is the Urbach energy.

As shown in equation 5, the E_u value can be evaluated by plotting $\ln(\alpha)$ versus E and the reciprocal of a slope of straight line as shown in Fig. 6 and Fig. 7 represents the E_u value of ZnO powders. The estimated E_u values are given in Table 1.

Fig. 6. Plots of $\ln(\alpha)$ versus E or photon energy for evaluating the E_u value of β -Ni(OH)₂/ZnO composite powders at different loading contents.

3.3. Photocatalytic activities

In this study, the photocatalytic activity was investigated by decolorization of aqueous MB solution under UV irradiation for 180 min. The photocatalytic activity was investigated by monitoring the absorbance of degraded MB aqueous solution at a wavelength of 664 nm. The relationship between C/C_0 and irradiation time for β -Ni(OH)₂/ZnO and NiO/ZnO composite powders is presented in Fig. 8.

Fig. 8a shows the photocatalytic degradation of MB aqueous solution using pure ZnO and β -Ni(OH)₂/ZnO composite powders as a photocatalyst. In this study, it was found that ZnO loaded with 2 mol%, 4 mol% and 6 mol% exhibited better photocatalytic degradation of MB aqueous solution than pure ZnO powders. In the presence of pure ZnO powders as a photocatalyst, it was only about 85 % of photocatalytic degradation after being irradiated for 60 min, whereas, the photocatalytic degradation of 95 %, 95 % and 97 %

Table 1. Dependence of E_g and E_u values on loading content.

Temperature [°C]	NiO or β -Ni(OH) ₂ loading content [mol%]	E_g [eV]	E_u [eV]
Room temperature	0	3.236	0.055
	2	3.205	0.069
	4	3.195	0.070
	6	3.192	0.072
	8	3.190	0.073
	10	3.187	0.076
800	0	3.219	0.058
	2	3.193	0.098
	4	3.184	0.102
	6	3.184	0.117
	8	3.167	0.130
	10	3.167	0.130

Fig. 7. Plots of $\ln(\alpha)$ versus E or photon energy for evaluating the E_u value of NiO/ZnO composite powders at different loading contents.

was obtained when the β -Ni(OH)₂ content was increased from 2 mol% to 6 mol%, but the photocatalytic degradation decreased when the β -Ni(OH)₂ content exceeded 6 mol%. It can be explained

as follows: when pure ZnO powders were used as a photocatalyst, theoretically the photogenerated electrons were excited to the conduction band (CB) and photogenerated holes were left in the valence band (VB) when the ZnO photocatalyst gained enough energy from light source. Nevertheless, the rapid recombination of photogenerated electrons and holes occurred, influencing the reduction in photocatalytic degradation of MB aqueous solution. Once β -Ni(OH)₂ was introduced to ZnO system to form β -Ni(OH)₂/ZnO composite powders, the electrons photogenerated in the CB of ZnO powders could be transferred to the CB of β -Ni(OH)₂ powders and then the recombination of the photogenerated electrons and holes could be retarded [25, 26]. Therefore, the photocatalytic degradation of MB aqueous solution by β -Ni(OH)₂/ZnO could enhance. However, the photocatalytic degradation decreased significantly when the β -Ni(OH)₂ loading exceeded 6 mol%. This might be due to the fact that many β -Ni(OH)₂ particles formed and covered the ZnO particles, preventing the interaction of light and reactive species on the surface of ZnO, resulting in the reduction of the photocatalytic degradation as mentioned in the previous work [27].

Considering the photocatalytic degradation of MB solution as shown in Fig. 8b, it was found that the photocatalytic degradation of MB

Fig. 8. Photocatalytic decolorization of (a) β -Ni(OH) $_2$ /ZnO and (b) NiO/ZnO composite powders at different loading contents under UV irradiation.

aqueous solution also increased as a function of the NiO loading content in a range of 0 mol% to 6 mol% similar to the case of β -Ni(OH) $_2$ loaded ZnO composite powders. It is clearly observed from Fig. 8a and Fig. 8b that β -Ni(OH) $_2$ /ZnO composite powders can more degrade the MB aqueous solution compared with NiO/ZnO composite. This phenomenon might be due to the narrower E_g value of NiO (3.5 eV) [28] compared with β -Ni(OH) $_2$ (5.83 eV) [29], so the recombination of photogenerated electrons and holes in NiO/ZnO composite is faster than that of β -Ni(OH) $_2$ /ZnO composite powders and lower photocatalytic degradation is obtained for NiO/ZnO composite powders.

4. Conclusions

The composite powders formed with a major phase of ZnO and minor phase of β -Ni(OH) $_2$ or

NiO, depending on calcination process. The ZnO platelet altered to hexagonal prism structure after Ni(CH $_3$ COO) $_2$ ·6H $_2$ O was added into the zinc precursor solution, but its structure did not change after calcination process. On the other hand, the platelet β -Ni(OH) $_2$ shape changed to spherical NiO after being calcined at 800 °C in air. The optical characteristics of β -Ni(OH) $_2$ /ZnO and NiO/ZnO slightly differed due to the differences in minor phases. The E_g value estimated by Tauc formula showed that the E_g value decreased as a function of the loaded minor phase content because of the generated sub-energy level within the forbidden zone of the samples. The β -Ni(OH) $_2$ /ZnO composite powders exhibited better photocatalytic activity compared to NiO/ZnO composite powder because the recombination of photogenerated electrons and holes was more obstructed and then the photocatalytic process could progress significantly. The most appropriate sample for decolorizing the MB aqueous solution is 6 mol% β -Ni(OH) $_2$ /ZnO composite powder, where the decolorization percentage reached to 97 % within 60 min of UV irradiation.

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