Research Article

The High Photocatalytic Activity of SnO₂ Nanoparticles Synthesized by Hydrothermal Method

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Tin oxide nanoparticles (SnO₂ NPs) were prepared at low temperature by hydrothermal method. Synthesized SnO₂ NPs were confirmed *via* characterization techniques such as UV-visible spectroscopy (UV-vis), X-ray diffraction (XRD), and Transmission Electron Microscope (TEM). The synthesized nanoparticles were in the size of 3 nm and they have high photocatalytic activity. The result showed that SnO₂ NPs degraded 88.88% MB solution after 30 minutes of UV illumination and reached 90.0% for 120 minutes (2 hours) of UV illumination. Moreover, they degraded 79.26% MB solution after 90 minutes (1.5 hours) under assisted sunlight illumination.

1. Introduction

In recent years, nanomaterials attracted much attention due to their high surface-to-volume ratio, enhanced characteristics of quantum size effects, and the high fraction of chemically similar surface sites [1]. Among metal oxide semiconductor materials, tin dioxide (SnO₂) has been attracting a great deal of research interest owing to its outstanding physical and chemical properties [2–4]. SnO₂ is an n-type semiconductor with a wide band gap of 3.6 eV at room temperature, having excellent optical and electrical properties such as peculiar optical transparency, low resistivity, and high theoretical specific capacity [5, 6]. Moreover, SnO₂ possesses a high electron mobility ($\sim 100\text{--}200\text{ cm}^2\text{ V}^{-1}\text{ s}^{-1}$), indicating a faster transport of photoexcited electrons [7–9]. In the last decade, SnO₂ has been studied as promising material with many unique surface properties including luminescence and photocatalytic activity [2]. In particular, as the diameter of SnO₂ is smaller than excitons and carriers are confined in all three dimensions to a nanometer size region, the high band gap energy and high stability of SnO₂ nanoparticles (SnO₂ NPs), unique properties such as the blue shift of the band edge transition energy, and unusual structural and optical

properties take place [10, 11]. Moreover, the high degradation rate of the organic dye on the as-synthesized SnO₂ NPs with size below 10 nm can be attributed to the small size of SnO₂ NPs because the large surface area helps to increase the photocatalytic reaction sites and promotes the efficiency of the electron-hole separation [12–14].

There are many methods to synthesize SnO₂ NPs, such as sol-gel [2, 15], hydrolysis [16], electrochemical oxidation of tin metal sheet in a nonaqueous electrolyte containing NH₄F [17], the chemical coprecipitation route [18, 19], dropping ammonium hydroxide into tin salt solution under sonication [20], and a microwave irradiation method [21, 22]. However, the hydrothermal method with an aqueous solvent as a reaction medium is environmentally friendly because the reactions are carried out in a closed system. Hydrothermal synthesis involves use of water as a solvent at elevated temperatures and pressures in a closed system and certain properties of the solvents such as density, viscosity, and diffusion coefficient change [23, 24]. Furthermore, hydrothermal synthesis is often used due to its simple processes and equipment, without high temperature sintering, allowing the control of the grain size, morphology, and degree of crystallinity by easy changes in the experimental procedure [25].

In the present work, we synthesize SnO_2 NPs powder via a simple hydrothermal method from tin chloride, sodium hydroxide, and hydrazine hydrate. The trick in the hydrothermal method presented here is the application of hydrazine hydrate ($\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$), which served as both the alkali and the ligand to coordinate with Sn ions to form a complex cluster. Furthermore, the hydrothermal process is desirable for the large-scale fabrication of other ultrafine oxide nanoparticles [1]. Besides, the photodegradation of dye organics by solar irradiation is a comparatively greener approach than by UV-light [26]. Previous publications on the photocatalytic activity of SnO_2 NPs often use UV-light as excited source for photoreactions. Herein, we synthesized SnO_2 particles with diameters <10 nm, which presents excellent photocatalytic degradation using MB as a model pollutant in the presence of UV-light and direct sunlight.

2. Experimental

2.1. Materials. Tin chloride ($\text{SnCl}_4\cdot 5\text{H}_2\text{O}$, Merck, 99.99% pure), hydrazine hydrate ($\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$, 99% pure), sodium hydroxide (NaOH , 99%), methanol (CH_3OH , 99% pure), and methylene blue ($\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCL}$, JHD Fine Chemicals, China, 99%) were used. Deionized (DI) water was obtained from Thermal Scientific.

2.2. Synthesis of SnO_2 NPs by the Hydrothermal Method. SnO_2 NPs were prepared using the hydrothermal method. Firstly, 0.9028 g $\text{SnCl}_4\cdot 5\text{H}_2\text{O}$ was introduced into a mixed solution of 25.75 mL DI water and 1.14 mL hydrazine hydrate ($\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$). After that, solutions were adjusted to $\text{pH} = 12$ by the addition of the NaOH solution and transferred to a stainless autoclave. Finally, it is heated at 135°C for 24 hours and cooled naturally to room temperature. The product was centrifuged, filtered out, rinsed with methanol and DI water several times, and then dried at 120°C for an hour in the air.

2.3. Characterization of SnO_2 NPs. The size and morphology of SnO_2 NPs were characterized by TEM (JEM 1400 Instrument, JEOL). XRD analysis was carried out (Bruker D8-Advance 5005) at a voltage of 45 kV with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) to examine the crystalline phase of synthesized nanoparticles. The Fourier transform infrared spectroscopy (FT-IR) spectrum was recorded on a FT-IR spectrometer (Vertex 80, Bruker, Germany) over the range of 400 to 4000 cm^{-1} in transmission mode at room temperature to identify the functional group present on SnO_2 NPs and responsible for the stability of nanoparticles.

2.4. Photocatalytic Activity of SnO_2 NPs for Degradation of the MB Solution. In order to determine the photocatalytic activity of the SnO_2 NPs, we use the MB dye solution as a model of contamination to characterize their photocatalytic activity. In the experiment, 30 mL of the MB solution (10 mg/L) and 0.03 grams of catalyst were used. This solution was stirred for 10 minutes in dark for the equilibrium of the adsorption and desorption process of MB with NPs. After stirring, the solution was irradiated by UV lamp (Osram, Germany) and direct sunlight conditions. The UV lamp has radiation of 350 nm

FIGURE 1: XRD patterns of the (a) as-synthesized and (b) thermally treated SnO_2 .

with 7 W of lamp power. Concentrations of the MB solution initially and after every 15 minutes of irradiation of UV-light were taken by using UV-visible spectrophotometer (U2910, Hitachi, Japan). In case these solutions irradiated by sunlight, the experiments were carried out on a sunny day at Ho Chi Minh City between 10 a.m.–1 p.m. with outside temperature from 35°C to 40°C and these experiments were tested 3 times.

3. Results and Discussion

3.1. X-Ray Diffraction (XRD) Pattern Analysis. The XRD pattern of the product is shown in Figure 1. The peaks at 2θ values of 26.6° , 33.8° , 51.8° , and 65.9° can be associated with (110), (101), (211), and (301), respectively. A matching of the observed and standard planes confirmed that the product is of SnO_2 having a tetragonal structure, which are in good agreement with the literature values (JCPDS card number 41-1445). The average particle size (D) was estimated using the following Scherrer equation [30, 31]:

$$D = \frac{0.9\lambda}{\beta \cos \theta}, \quad (1)$$

where D is the crystallite size, λ is the X-ray wavelength, β is the full width at half maximum of the diffraction peak, and θ is the Bragg diffraction angle of the diffraction peaks. The average particle size of SnO_2 calculated using Scherrer equation of SnO_2 is 3 ± 0.1 nm.

Comparison of the XRD pattern of the as-synthesized samples is as follows: the samples calcined at 300°C in flowing air for 2 hours (Figure 1(b)) do not change characterization peaks of SnO_2 peaks and the intensity of peaks do not increase significantly. Therefore, we only use the as-synthesized samples for survey continuously.

3.2. Transmittance Electron Microscopic Analysis (TEM). The morphology and the particles size of the samples were obtained by TEM images (Figure 2(a)). The image shows that spherical shaped nanoparticles have been formed and they were uniformly distributed except for a few aggregated

FIGURE 2: (a) TEM image of SnO₂ NPs synthesized by hydrothermal method and (b) histogram showing crystallite size distribution.

particles. The diameter of SnO₂ Nps measured from TEM image is in the range of 0.5–5.5 nm with an average of 2.2 nm which is almost the same as that obtained from XRD patterns (Figure 2(b)).

3.3. Fourier Transform Infrared Spectroscopy. The FT-IR spectroscopy was recorded at room temperature for determination of the functional group of materials available in the nanopowders. The FT-IR spectrometer was used for the analysis in a range between 4000 and 380 cm⁻¹, as shown in Figure 3. The band around 3454.29 to 3539.64 cm⁻¹ and 1612.43 cm⁻¹ indicates the presence of O-H group due to stretching vibrations of water molecules or hydroxyl groups adsorbed at the surface of SnO₂ and also to stretching vibrations of Sn-OH groups [26, 32]. The band around 3196.74 cm⁻¹ and 2928.99 cm⁻¹ indicates the presence of C-H group. The peak around 608.14 cm⁻¹ is assigned to Sn-O-Sn stretching mode of surface bridging oxide formed by the condensation of

FIGURE 3: FT-IR spectra of SnO₂ NPs.

adjacent surface hydroxyl groups [25, 26, 32]. Therefore, the presence of these bands confirmed the formation of SnO₂.

3.4. Photocatalytic Activity of SnO₂ NPs. The photocatalytic activity of SnO₂ NPs was examined by the changes in the absorption spectra of the MB dye solution during their photodegradation process. Figure 4(a) shows that commercial SnO₂ degraded 20.49% MB solutions after 30 minutes of UV lamp illumination and it mostly does not degrade when increasing illumination time. Figures 4(b) and 4(c) show the change of the absorption of MB solution when it obtained SnO₂ NPs under different UVA irradiation times. Figure 4(b) shows that the degradation activity of SnO₂ NPs is better than the commercial SnO₂. Figure 4(c) is a magnified image of Figure 4(b). It shows that the typical absorption peak of MB at 664 nm wavelength is significantly decreased. However, when the irradiation time increases from 15 minutes to 180 minutes, the decline of the absorption peak of MB is insignificant. Besides, Figures 4(b) and 4(c) indicate that SnO₂ NPs degraded 88.88% MB solution after 30 minutes of UV illumination and reached 90.0% for 120 minutes (2 hours) of UV illumination. These results show that the more the illumination time increases, the more the concentration of MB dye decreases, which is shown by the decrease in UV-absorbance spectra. It can be seen that the intensity of the absorption peak gradually decreases with increasing UV illumination time.

Figure 5 shows the photocatalytic activity of SnO₂ NPs according to UV illuminated times condition. After 15 minutes of UV illumination, SnO₂ NPs degraded strongly MB solutions and nearly saturated during the time from 30 minutes to 180 minutes. Whereas, the degradation of commercial SnO₂ is weak and unstable.

The mechanism for photodegradation of MB dye can be explained as follows [33, 34].

Because SnO₂ Nps are synthesized by hydrothermal method, there are the generated oxygen vacancies in SnO₂ crystal, and these can induce the formation of new energy levels in the band gap. After the heat treatment of Sn(OH)₄, the hydroxyls remaining in the structure will generate molecular water, while releasing the lattice oxygen and producing a neutral oxygen vacancy (V_O) in its position. Also the high pressure produced in the hydrothermal process is in favor of

FIGURE 4: UV-visible absorbance spectra for photodegradation of MB dye according to illumination time under UVA irradiation: using commercial SnO_2 (a) and using SnO_2 NPs (b) and a magnified image (c).

the removal of hydroxyls and production oxygen vacancies. or
The process can be expressed as follows:

FIGURE 5: Photocatalytic degradation of MB under UVA irradiation.

These neutral vacancies may be singly ionized vacancies as $V_{\text{O}}^{\bullet}$ or $V_{\text{O}}^{\bullet\bullet}$. Semiconductor photocatalytic activity has attracted great interest due to its potential applications in the degradation of environmental pollutants and organic pollutant transformation.

So, electron (e^-) is excited from the valence band into the conduction band, generating hole (h^+) when a photon with energy of $h\nu$ matches or exceeds bandgap energy, E_g , of semiconductor SnO_2 :

In the photocatalytic reaction, $\bullet\text{O}_2^-$ and $\bullet\text{OH}$ radicals are the extremely strong reduction and oxidation agent, respectively.

FIGURE 6: UV-visible absorbance spectra for photodegradation of MB dye using SnO_2 under direct sunlight irradiation.

Figure 6 shows the UV-visible absorbance spectra for the photodegradation of MB dye of commercial SnO_2 and SnO_2 NPs under direct sunlight. Sunlight assisted photodegradation property of SnO_2 NPs was investigated and it shows the photodegradation efficiency of 79.26% MB solution after 90 minutes (1.5 hours) under direct sunlight illumination. Otherwise, the commercial SnO_2 powder only degrades 36.23% of MB solution. Besides, we compared with previous studies of other researchers for the photocatalytic degradation of MB using SnO_2 NPs. These results show that the degradation ability of SnO_2 NPs with MB solution is very high under both UV illumination and sunlight. It is indicated that only a very small amount of SnO_2 NPs (0.03 grams) was degradable more over 90% MB solution under UV illumination condition (power of lamp is 7 W) for 2 hours.

A comparison between the present study and studies conducted by other researchers is shown in Table 1. The comparison also showed that the synthesized SnO_2 NPs in this research have extremely high photocatalytic activity.

4. Conclusion

SnO_2 NPs of average size 3 nm synthesized by hydrothermal method from tin chloride, sodium hydroxide, and hydrazine hydrate. The photodegradation efficiency of SnO_2 NPs with MB solution is very high under both UV illumination and sunlight. SnO_2 NPs degraded strongly MB solutions (>80.0%) after 15 minutes of UV illumination and degraded 79.26% MB solution after 90 minutes under sunlight irradiation.

TABLE 1: The comparison of the photocatalytic degradation of dyes using SnO₂ NPs.

Catalysts (shape and particles size)	Light source	Dye (initial concn)	Volume (mL)	% degradation	Irradiation time (mins)	Catalyst concn (g)	Ref
SnO ₂ NPs (~3 nm)	7 W UV lamp, visible light $\lambda = 350$ nm Direct sunlight	MB, 10 mg/L	60	90.0	120	0.03	This work
SnO ₂ NPs (15–40 nm)	Low pressure 125 W UV lamp, visible light $\lambda = 254$ nm	MB, 20 mg/L	100	93.3	120	0.2	[3]
SnO ₂ NPs (~4 nm)	UV lamp, $\lambda = 365$ nm (power of lamp: nondetermined)	Phenol red, 1 mL, 10^{-4} M	1.0	100	120	0.25	[27]
Microspherical (0.4–1.8 μ m diameter)	Four 8 W UV lamps (Philips UV-A, $\lambda = 350$ nm)	Aniline, 20 mg/L 4-Nitroaniline (20 mg/L) 2,4-Dinitroaniline (20 mg/L)	10	80 70 50	120	0.1	[28]
Spherical SnO ₂ quantum dots (~2.5–4.5 nm)	Direct sunlight	Rhodamine B (10^{-4} M) MB (10^{-4} M)	200	83.9 56.8	420 360	0.01	[26, 29]

Competing Interests

The authors declare that they have no competing interests.

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