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Microwave-assisted electrospun PVB/CdS composite fibers and their photocatalytic activity under visible light



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ABSTRACT

Degradation of dyes is a standard method for checking the photocatalytic activity of any type of photocatalyst. In this paper, one-dimensional (1-D) poly-vinyl butyral (PVB)/cadmium sulfide (CdS) nanofibers have been first prepared by using cadmium nitrate and thiourea dissolved in a solvent of ethanol and the polymer used poly-vinyl butyral via electrospinning approach. The as-spun 1-D PVB/CdS nanofibers were employed as precursors to fabricate cotton- and fiber-shaped PVB/CdS composite materials via annealing treatment and microwave irradiation. The structure, morphology, composition, and optical properties of the samples were characterized using X-ray diffraction (XRD), transmission electron microscopy (TEM), energy dispersive X-ray spectroscopy (EDS), and scanning electron microscopy (SEM). The photocatalytic activity of two kinds of resultant PVB/CdS composite materials under visible light irradiation was evaluated by degrading methylene orange (MO).

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1. Introduction

Photocatalysis of organic and biological molecules is a promising technique for solving the environmental problem of organic pollutants and toxic pollutants in water [1–6]. Semiconductor photocatalysts have attracted attention for application in the degradation of organic pollutants and the production of H₂ by water splitting [7,8] because of their environmental friendliness, low cost, and reusability.

Cadmium sulfide (CdS), which belongs to the II–VI group, has a direct band gap of 2.1–2.4 eV and a suitable band position with visible light response. It is widely applied in photocatalysts, photoelectric conversion devices, solar cells, light-emitting diodes, optical devices, and environmental sensors [9–13].

Unfortunately, the S²⁻ ions in CdS rather than H₂O are the holes acceptor in CdS particles, which results in photocorrosion in aqueous solution under illumination [14]. To improve the photocatalytic activity of CdS, the photocorrosion problem must be solved. Polymeric materials are a candidate additive. Some specific polymers are dispersible in water and can form a stable suspension. Poly-vinyl butyral (PVB) is a non-toxic, odorless, and environmentally benign polymer. Such the polymer shells allow the photocatalyst nanoparticles to be suspended

in water. Furthermore, photocorrosion can be minimized by dispersing the nano-size crystallites over the surface of a polymeric support [15–18]. It is an early report that the cubic CdS nanoparticles hybrid carbon spheres (CdS@C) displayed structurally enhanced photocatalytic activity [19].

Techniques such as spray pyrolysis [20], the two-stage process [21], thermal evaporation [22], chemical vapor deposition [23], electrodeposition [24], electrospinning, and the solvothermal methods [25] have been employed for the preparation of CdS materials. Among them, electrospinning is an easy method for preparing one-dimensional structures on a large scale. The process can be used to synthesize various organic/inorganic hybrid composite nanofibers. A microwave-assisted reaction can be conducted at normal atmospheric pressure with short reaction times.

In this work, PVB/CdS composite materials are synthesized via a microwave-assisted electrospinning process and their photocatalytic performance is evaluated in terms of dye degradation. The crystal structure, morphology, and optical properties of CdS nanocrystals were investigated in detail by using X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). Furthermore, the photocatalytic activities in the degradation (de-coloration) of methylene orange (MO) over the CdS photocatalysts were also investigated under visible light irradiation ($\lambda \geq 420$ nm) in air at room temperature for various catalyst compositions.

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2. Material and methods

2.1. Materials

Cadmium nitrate ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) and thiourea ($\text{CH}_4\text{N}_2\text{S}$) were purchased from Alfa Aesar (Ward Hill, MA, USA), and PVB (average $M_v \sim 40$ kDa) was obtained from Chang Chun Group Company (Kaohsiung, Taiwan). Ethanol (99%) was purchased from PA Panreac (Barcelona, Spain). These substances were used as received without further purification. The deionized water (DI water) used in this work was obtained from the EMD Millipore Corporation-Direct-Q 3 system (Billerica, MA, USA).

2.2. Preparation of electrospinning solution and annealing treatment

In a typical synthesis, 1 mmol of cadmium nitrate, 2 mmol of thiourea, and 10 wt.% PVB (PVB/ethanol content ratio = 8:92) were dissolved in 10 mL of ethanol under magnetic stirring at room temperature for 2 h to obtain a homogenous precursor of PVB/CdS composites. The electrospinning setup consisted of a syringe and needle (with an internal diameter of 0.21 mm), a syringe pump (KD Scientific Inc., Model 200, Holliston, MA, USA), a high-positive-voltage power supply (You-Shang Technical Corp., Kaohsiung, Taiwan), and a collection board. A stainless steel electrode was connected to a high-voltage power supply, which could provide a DC voltage of up to 30 kV. The homogeneous precursor of PVB/CdS composite solution was prepared and used immediately for electrospinning. The electrospun PVB/CdS nanofibers were obtained, followed by transferring to a combustion boat, and calcined at 300–500 °C for 1 h in N_2 atmosphere.

2.3. Microwave-assisted electrospinning route

For the microwave-assisted electrospinning route, the as-spun PVB/CdS nanofibers were put in a high-power microwave oven (700 W) operated using a pulse regime of 100% power for 1 min repeated twice in intervals of 10 min. The precipitates were then centrifuged and washed with DI water and ethanol several times, and then dried in a 60 °C oven for 6 h.

2.4. Material characterization

The products were characterized by various instruments. X-ray diffraction (XRD) measurements (Simatsu, XRD6000, Japan) were recorded using a $\text{CuK}\alpha$ ($\lambda = 0.15418$ nm) radiation source operated at 40 kV and 100 mA. The XRD patterns were obtained at a scan rate of 0.03 °/s in the 2θ range of 15° to 85°. The particles were imaged using SEM (Hitachi, 4200A, Japan) at an operation voltage of 10 kV. Detailed microstructure analysis was carried using TEM (JEOL, JEM-2000EX, Japan). The elemental compositions of the particles were obtained using an energy dispersive X-ray spectroscopy (EDS elemental analysis (Horiba, EX220, Japan)) that was attached to the SEM and TEM.

2.5. Photocatalytic performance

The photocatalytic activity was conducted by the photodegradation of organic dyes under visible light irradiation at room temperature. The methylene orange (MO) dyes were chosen to test the photocatalytic activity of as-prepared CdS catalysts. A 300-W Xe arc lamp (Trusttech PLS-SXE 300, China) was used as the light source. It was equipped with a UV cutoff filter to provide visible light ($\lambda \geq 420$ nm). The photocatalysis tests were performed with the photocatalyst (100 mg) suspended in dye aqueous solution (100 mL, 10 mg/L) with constant stirring. The suspension was stirred in the dark for 60 min. The degradation efficiency of MO was monitored by UV–vis spectroscopy at given intervals. According to the Beer–Lambert law, the concentration of MO is proportional

to the absorbance of MO. The degradation efficiency of MO can thus be calculated using [26,27]:

$$R = \frac{C_0 - C}{C_0} \times 100\% = \frac{A_0 - A}{A_0} \times 100\%$$

where A_0 , A and C_0 , C are the absorbance and concentration of MO at reaction times 0 and t , respectively.

3. Results and discussion

3.1. Morphology of electrospun PVB/CdS nanofibers

Fig. 1 shows the SEM images of PVB/CdS nanofibers with various PVB concentrations of (a) 6%, (b) 8%, (c) 10%, and (d) 12% obtained at a working distance of 7 cm. In Fig. 1(a), only a few fibers can be seen due to the low PVB concentration, high conductivity, and low viscosity, which led to the appearance of beaded fibers. With increasing PVB concentration, the amount of beaded fibers decreased. Fig. 1(c) shows PVB/CdS nanofibers with smooth surfaces for a PVB concentration of 10%. The results indicate that increasing the PVB concentration can increase the amount of smooth fibers. The high conductivity of the solvent facilitates ion diffusion and reduces resistance. However, the high viscosity of the solvent may make it more difficult for the ions to diffuse, and thus reduce conductivity [28]. When the concentration of PVB was increased to 12% (Fig. 1(d)), the diameter of the fibers increased and the thickness was uneven [29–31]. Fig. 1(e) and (f) shows the influence of the different operating voltages and flow rates on the diameter of the fibers, respectively. The operating voltage was changed by the high-voltage power supply (provided a DC voltage of up to 30 kV) at any time. As the operating voltage gradually increases, the curve for the diameter of the fibers changes. As the applied voltage was increased from 14 to 16 kV, the PVB/CdS nanofiber diameter increased and the thickness became uneven. At an applied voltage of lower than 11 kV, no fibers formed. In other words, on operating voltage that is too high or too low caused the fiber diameter to increase or increased the number of beaded fibers that are produced [30–32]. When the coulombic, viscoelastic, and surface tension forces were well-balanced, the fiber diameters had a narrow distribution. An increase in the applied voltage increased the coulombic repulsive force of the fluid jet, leading to slim fibers. With a further increase in the operating voltage, the coulombic force became much greater than the viscoelastic force. This likely increased the possibility of the breakage of an over-stretched charged jet during its flight to the target [32]. In this experiment, the optimal operating voltage for preparing PVB/CdS smooth fibers was 14 kV. Fig. 1(f) shows the influence of different flow rates on fiber diameter. The flow rates were changed by the syringe pump. As the flow rate of electrospun PVB/CdS composite solution was increased, the diameter of the PVB/CdS nanofibers increased [33–35]. For a flow rate of 0.7 mL/h, the nanofiber diameters had an uneven distribution. Increasing the flow rate made the distribution of diameters much more even. When the flow rate was increased to 1.5 mL/h, the nanofiber diameter and thickness were uneven, and the fibers aggregated and adhered to each other. The most suitable flow rate for preparing PVB/CdS nanofibers was 1.2 mL/h.

3.2. Characterization of sphalerite PVB/CdS composite material formation

The phase and crystallographic structures of resultant PVB/CdS composite materials were investigated by X-ray diffraction patterns; the detail experimental parameters were summarized in Table 1. Fig. 2 shows the XRD patterns of the PVB/CdS composite materials fabricated by electrospinning at 14 kV with a flow rate of 1.2 mL/h, and working distance of 7 cm followed via the annealing process and the microwave-assisted electrospinning process. Fig. 2(a) shows the XRD patterns of pure PVB fibers and PVB/CdS composite materials obtained

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