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Incorporation of cadmium sulfide nanoparticles on the cadmium titanate nanofibers for enhanced organic dye degradation and hydrogen release

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Abstract

In this study, the effective cadmium sulfide NPs were successfully encapsulated on cadmium titanate electrospun nanofibers. Calcination of electrospun nanofiber mats composed of titanium isopropoxide, poly (vinyl pyrrolidone) (PVP) and cadmium acetate dihydrate with few drops of ammonium sulfide in air at 600 °C led to the production of good morphology of CdS decorated cadmium titanate nanofibers. The morphology and structure of as-synthesized nanocomposites were characterized by field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD). As-synthesized nanocomposites exhibited a strong photocatalytic activity for decomposition of methylene blue (MB) under visible light and showed good hydrogen release from hydrolysis of the ammonia–borane complex. The perfect recovery of catalyst after reaction and its unchanged efficiency for cyclic use showed that it will be an economically and environmentally friendly photocatalyst. The results indicated that the $CdS/CdTiO_3$ photocatalysts possess improved photocatalytic activity than that of $CdTiO_3$ alone.

Keywords: A. Electrospinning; B. Nanocomposite; E. Photocatalyst; E. H_2 release

1. Introduction

Energy crisis and environmental pollution are the biggest challenge in the 21st century at a global level. Many photocatalysts have been reported for the splitting of water in the presence or absence of sacrificial agents, producing Hydrogen as a clean energy source, and for the degradation of organic pollutants [1–6]. Semiconductor photocatalysts have attracted much attention in recent years because of their potential in solving global energy and environmental problems. Among them, composite systems made of more than two semiconductor components are considered a promising method to develop a high efficiency photocatalyst working under visible light because they can compensate for the disadvantages of the individual component, and induce a synergistic effect, such as an efficient charge separation and improvement of photostability [7,8].

Since the discovery of the photocatalytic activity of TiO_2 for hydrogen production and organic dye degradation, photocatalysis has been considered to be a promising technique for solving energy and environmental issues using abundant sunlight [9]. Titanium based oxides are of great interest due to their high refractive index, direct wide energy band gap and low absorption properties [10,11]. Although TiO_2 has been extensively studied and widely used for water splitting and environment treatment, it has practical limitations in its use due to the requirement of near ultraviolet (UV) irradiation for effective photocatalysis. Therefore, the development of efficient visible-light-responsive photocatalysts is a highly

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challenging but desirable goal for renewable energy resource and environment remediation which can effectively use a maximum of the solar spectrum.

Due to the excellent activity under visible-light and suitable negative conduction band (CB) edge compared to the reduction potential of hydrogen, CdS (2.4 eV) is most extensively used as a photocatalyst [12,13]. However, the low surface area and high recombination rate of photo induced charge carriers owing to its band energies lower the photocatalytic activity of CdS [14,15]. The photocatalytic efficiency of CdS can be enhanced by reducing the recombination rate of photo induced charge carriers. For this strategy, coupling with wide band gap semiconductors such as TiO_2, ZnO, NiO has been carried out [16–18]. One of these oxides, cadmium titanate ($CdTiO_3$), has excellent dielectric, sensing and optical properties [19,20]. By incorporation of CdS nanoparticles to the system the application of cadmium titanate can be broadened as a photocatalyst. Secondary pollution is one of the problems limiting the widespread applications of nanostructured photocatalysts. In other words, most of the reported nanostructured photocatalysts can effectively eliminate the organic pollutants; however, they create a serious issue due to the difficulty of separating the utilized nanostructured photocatalysts from treated water, especially in the case of large-scale processes [21]. Therefore, here, prior formed CdS NPs are incorporated into $CdTiO_3$ nanofibers to achieve better stability. By doping the CdS nanoparticles inside the $CdTiO_3$ nanofibers, we were simultaneously able to solve the problem of photo corrosion and toxicity of CdS nanoparticles. The introduced materials overcame the toxicity problem of CdS as it is well incorporated on the nanofibers.

Hydrogen energy is one of the most important kinds of energy; it is pollution-free compared with petroleum products. Storage of hydrogen in large quantities for on-board applications remains a major constraint for its widespread usage. The low density of H_2 makes it difficult to store it in compressed or liquefied form. In recent years, hydrogen was stored in solid materials; they were considered a reservoir for hydrogen. Among these materials, the ammonia–borane complex (AB, H_6BN) has been identified as one of the leading candidates as a hydrogen reservoir owing to its high hydrogen content (19.6 wt%) which exceeds that of gasoline [22], solubility/stability in aqueous solution and stability toward hydrolysis in aqueous solutions for at least 4 days [23,24]. It can release hydrogen by two main routes; pyrolysis or a hydrolysis route. Recently, the hydrolysis of AB was accomplished by the use of noble and non-noble metallic, bimetallic, and tri metallic nanoparticles (NPs) due to their good catalytic activity and large surface area to volume ratios. The development of efficient, low cost and stable catalysts is very important for practical application to overcome the high cost of the reported precious metals [25].

Electrospinning has attracted much attention as a simple and versatile technique capable of generating continuous nanofibers not only from polymers [26,27], but also from inorganic [28] and hybrid (organic–inorganic) compounds [29–31] which have different applications in a wide range of areas, e.g., filtration, protective clothing, catalysis, sensors, energy storage, biomaterials [31–33]. Here, we report the synthesis of ilmenite rhombohedral phase $CdTiO_3$ nanofibers with simultaneous formation of CdS NPs throughout the surface of nanofibers via the electrospinning technique followed by calcination in air. This is a simple and efficient approach to synthesize the composite of cadmium titanate decorated with CdS nanoparticles. Imran et al. reported a humidity sensor based on electrospun $CdTiO_3$ nanofibers [34,35]. Similarly, bulk $CdTiO_3$ and its thin films have been investigated widely [36–38]. However, the synthesis of $CdTiO_3$ nanofibers containing CdS NPs has not been reported yet. To explore a new application, the CdS/$CdTiO_3$ nanocomposite is primarily employed as a photocatalyst for release of hydrogen by the hydrolysis of ammonia–borane and the degradation of methylene blue under visible light irradiation to our knowledge.

2. Experimental procedure

2.1. Materials

Polyvinylpyrrolidone (PVP), titanium tetraisopropoxide (Ti (Iso), 97%), cadmium acetate dihydrate 98%, ammonium sulfide (40–48 wt%), and the ammonia–borane complex (AB, 97.0%) were obtained from Sigma-Aldrich. Acetic acid was obtained from Showa Chemicals Co. Ltd., Japan. All the chemicals were analytic grade and used without further purification.

2.2. Preparation of CdS/$CdTiO_3$ nanocomposite

At first, 1.5 g of titanium tetraisopropoxide was mixed with 3 g acetic acid. After 10 min of stirring, 0.45 g of PVP and 4 g of ethanol were added to the solution followed by the addition of 1 g of cadmium acetate and 0.5 g of ammonium sulfide (drop-wise). After stirring for 12 h at room temperature, the resulted mixture solution was subjected to the electrospinning process in a plastic syringe fitted with a pointed nozzle, which was clamped at about a 20° angle relative to the horizontal axis. The positive pole copper electrode was secured inside the solution. An electric voltage was applied at 20 kV. A rotating steel drum covered with alumina foil used as a collector of the electrospun nanofibers and was positioned 15 cm from the tip of the syringe. The collected nanofibers were dried at room temperature for 12 h and vacuum dried for further 12 h. After calcination in air at 600 °C for 3 h, the sample was used for further analyses.

2.3. Characterization

The surface morphology of nanofibers was studied by using a JEOL JSM-5900 scanning electron microscope, JEOL Ltd., Japan, and a field-emission scanning electron microscope equipped with EDX (FE-SEM, Hitachi S-7400, Japan). The phase and crystallinity were characterized by using a Rigaku X-ray diffractometer (Rigaku Co., Japan) with Cu Kα (λ=1.54056 Å) radiation over 2θ range of angles, from 10°

to 80°. High-resolution images and selected area electron diffraction patterns were observed by a JEOL JEM 2010 transmission electron microscope (TEM) operating at 200 kV (JEOL Ltd., Japan). The concentration of the dyes during the photodegradation study was investigated by spectroscopic analysis using an HP 8453 UV–visible spectroscopy system (Germany). The spectra obtained were analyzed by the HP ChemiStation software 5890 Series.

2.4. *Photocatalytic activity measurements*

The photocatalytic activity of the prepared nanocomposite photocatalysts was evaluated by degradation of methylene blue aqueous solution under visible light irradiations. In the durability test of catalyst in the photodegradation of MB, three consecutive cycles were tested. At the beginning 25 mg of CdS/$CdTiO_3$ nanocomposite was put in 50 mL of a 10 ppm MB aqueous solution. Under magnetic stirring, the mixed solution was irradiated under sunlight. Then samples were taken out at regular intervals of time. After centrifugal separation, the concentration of MB in supernatant was analyzed by a spectrophotometer. Direct sunlight was used in the present study and all the experiments were conducted in September between 10:00 a.m. and 3:00 p.m., when the solar intensity fluctuations were minimal. The mixture underwent three consecutive cycles. After each cycle, the catalyst was filtered and washed thoroughly with water and fresh MB solution of the same concentration was used.

2.5. *Catalytic hydrolysis of ammonia–borane*

The catalytic activity of CdS/$CdTiO_3$ nanocomposites toward AB hydrolysis under sunlight was evaluated according to our previous experiment [25]. Briefly, before starting the catalytic activity test, a flask containing a Teflon-coated stir bar was placed on a magnetic stirrer and irradiated to sunlight. Then, a graduated cylinder filled with water was connected to the reaction flask to measure the volume of the hydrogen gas to be evolved from the reaction. Next, the 70 mL aqueous dispersion of the catalyst was transferred into the reaction flask and 20 mg AB was added into the catalyst solution under 600 rpm stirring rate under sunlight. The volume of the hydrogen gas evolved was measured by recording the displacement of the water level every minute. The reaction was ceased when no hydrogen gas generation was observed. For the control experiment, the same experiment was repeated without any catalytic material; it was observed that no appreciable hydrogen gas evolved.

3. Results and discussion

Fig. 1A shows the FE-SEM image of CdS-TiO_2/PVP composite nanofibers fabricated by electrospinning. Each individual nanofiber was uniform in cross section. The diameter of nanofiber was found to be approximately 280 nm. Since $Ti(OiPr)_4$ can be rapidly hydrolyzed by moisture in the air, continuous networks (gels) of TiO_2 sols were able to form in the nanofibers once they had been ejected from the orifice [1]. Fig. 1B shows the FE-SEM image of CdS/$CdTiO_3$ nanocomposites calcined at 600 °C. As the PVP was selectively removed by burning the sample in air at 600 °C, the nanofibers remained as continuous structures, and their average diameter was decreased to 175 ± 15 nm. Moreover, CdS nanoparticles (approximately 8 nm) were formed during heat treatment and attached on the cadmium titanate nanofibers (Fig. 2A). The formation of CdS nanoparticles and their growth on the cadmium titanate nanofiber might take place simultaneously during the calcination. FE-SEM EDX confirms the presence of Cd, Ti, O, and S. Furthermore, the TEM image in Fig. 3A confirms that the anchored CdS nanoparticles were obviously sintered on the cadmium titanate nanofiber during calcination. Well-attached nanoparticles can be seen in the HR-TEM image in Fig. 3B. The size of the nanoparticles was very uniform.

Fig. 4 represents the typical XRD patterns for the different samples obtained from the electrospinning process, and after the calcination. Before calcination (Fig. 4a), the peaks at 2θ values of 26.2°, 43.50°, and 52.6° oriented along the (111), (220), and (311) directions stand for CdS, which is in agreement with the value reported by JCPDS 10-454 [39]. After the calcination process (Fig. 4b), along with these peaks a strong peak appeared at 2θ values of 24.80°, which can be assigned to the (100) phase of the hexagonal structure of CdS [40]. The polycrystalline structure of the ilmenite

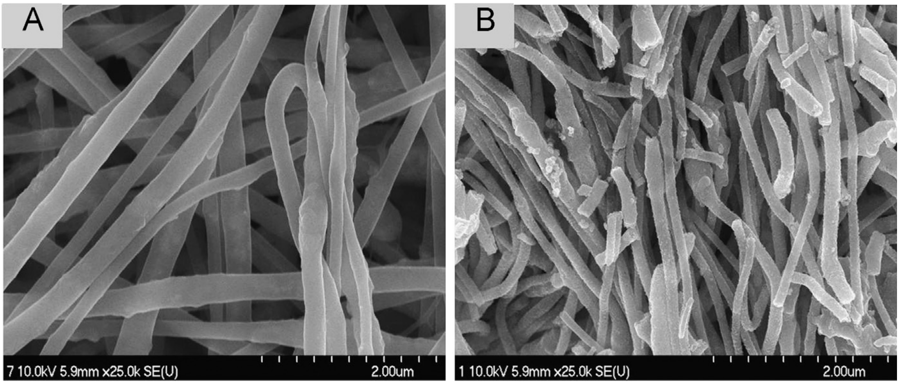
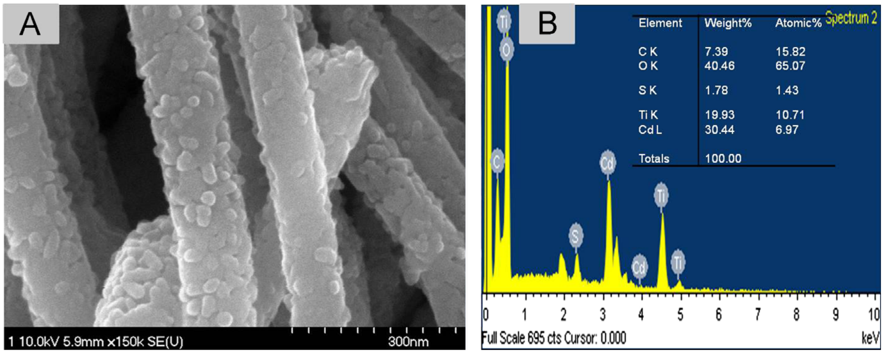


Fig. 1. FE-SEM images before (A) and after (B) calcination.



Element	Weight%	Atomic%
C K	7.39	15.82
O K	40.46	65.07
S K	1.78	1.43
Ti K	19.93	10.71
Cd L	30.44	6.97
Totals	100.00	

Fig. 2. High magnification FE-SEM image (A) and FE-SEM EDS (B) after calcination in air.

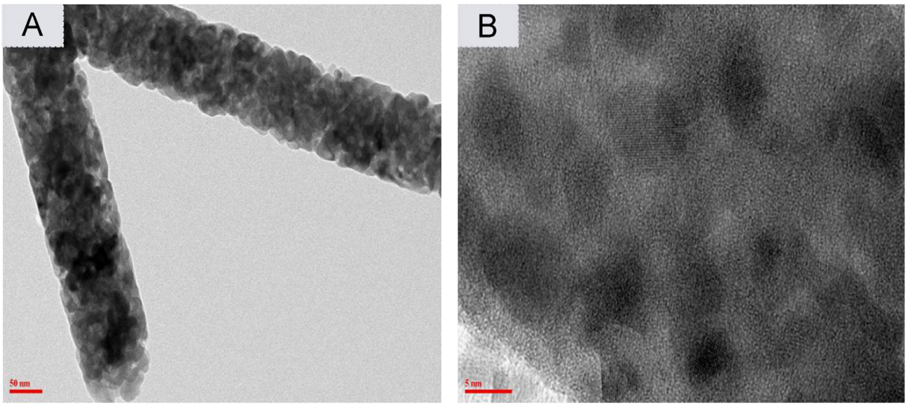


Fig. 3. TEM (A) and HR-TEM images (B) after calcination in air.

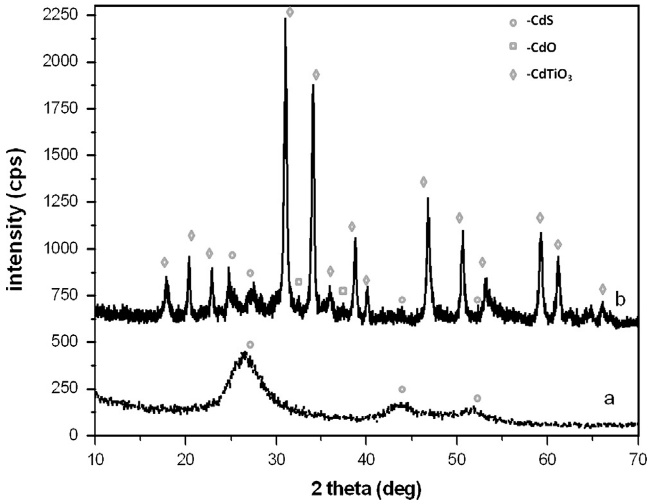


Fig. 4. XRD spectra of as-synthesized nanofiber (a) and nanocomposite after calcination (b).

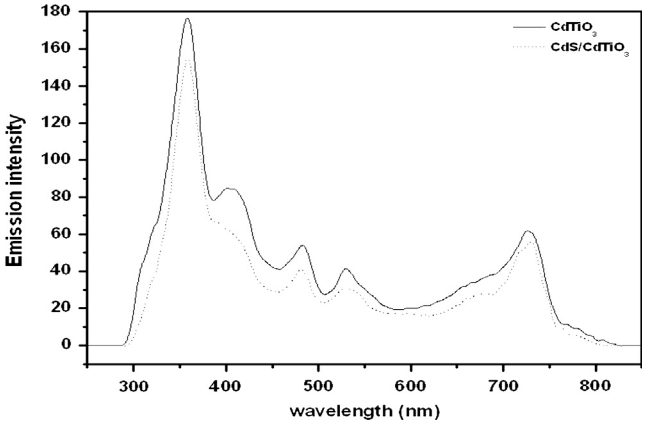


Fig. 5. PL spectra of $CdTiO_3$ and $CdS/CdTiO_3$ nanocomposite.

rhombohedral phase and strong peak observed at 2θ values of 31.22° and 34.11° can be associated with (104) and (110) planes, respectively [34]. All diffraction peaks show good consistency with JCPDS, Card no. 29-0277 of the ilmenite rhombohedral phase of $CdTiO_3$ [41]. Beside these peaks low intensity peaks at 2θ values of 33° and 39° were also found, which can be associated with CdO (JCPDS Card no. 05-0640) [42]. The presence of sharp peaks of CdS along with $CdTiO_3$ in the nanocomposite revealed that CdS is well deposited on the surface of $CdTiO_3$ nanofibers. Furthermore, EDX obtained from FE-SEM images (Fig. 2B) confirms the incorporation of CdS nanoparticles in the cadmium titanate nanofibers and simultaneously supports the XRD analysis.

Photoluminescence (PL) spectra of the as-synthesized photocatalysts were recorded at room condition and shown in Fig. 5. The PL emission spectra were used to reveal the efficiency of trapping, migration, transfer and separation of charge carriers, and to investigate their lifetime in semiconductors since PL emission results from the recombination of electron/hole pairs [43]. As in the figure, the $CdS/CdTiO_3$

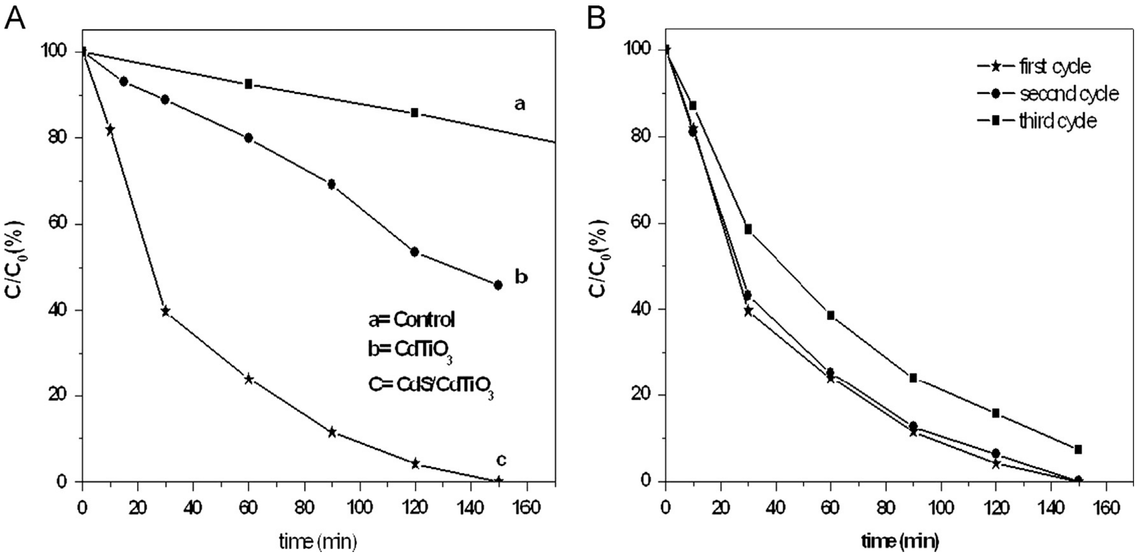


Fig. 6. Degradation of MB by different photocatalysts (A) and up to three cycles by CdS/CdTiO$_3$ nanocomposites (B).

nanocomposite exhibits a lower emission intensity than CdTiO$_3$. A lower PL intensity indicates a lower recombination rate of the excited electrons/holes and low defects in the produced nanofibers [25]. In other words, lower PL intensity indicates low recombination rate, which is preferable in case of utilizing the materials as catalysts in the photoreactions.

In order to elucidate the effect of coupling of CdS nanoparticles with CdTiO$_3$ nanofiber, photocatalytic activity was measured by using MB dye. Decolorization of the methylene blue dye was carried out under visible light irradiation. As shown in Fig. 6A, the rate of degradation of methylene blue is highly enhanced by the as-prepared CdS/CdTiO$_3$ photocatalyst. The enhanced degradation of Methylene blue in this experiment could be confirmed by comparing the obtained results with the CdTiO$_3$ nanofiber alone. The result of higher photocatalytic activity of the CdS/CdTiO$_3$ nanocomposite than for other photocatalysts is attributed mainly to the coupling effect of TiO$_2$ and CdS. Furthermore, the porous nature of CdTiO$_3$ nanofiber also acts as a dye adsorbent, resulting in fast degradation of dyes.

In order to verify the reusability of the CdS/CdTiO$_3$ nanocomposite, we carried out three successive cyclic tests with the same nanocomposite. In more detail, the same sample (25 mg) was used to treat 50 mL of the dye solution (10 ppm) successively. Fig. 6B shows the cyclic degradation results of MB under visible light irradiation. As shown in the figure, the efficacy of initially used and reused composite photocatalyst was almost the same for MB degradation for the first and second cycles. However, it was significantly less in the third cycle. The blockage of active sites in the photocatalyst may be the cause of less degradation of MB in the third cycle.

The performance of the as-synthesized nanocomposite on the hydrogen release efficiency by the hydrolysis of the ammonia–borane complex was also measured under visible light irradiation. Fig. 7 depicts the time course of hydrogen evolution by AB hydrolysis under the sunlight. The results showed higher catalytic activity of CdS/CdTiO$_3$ towards hydrogen release under sunlight than that of CdTiO$_3$ alone. This increase in photocatalytic activity may be due to the high degree of crystallinity and the performance of CdS NP [44]. Charge injection takes place from excited CdS to CdTiO$_3$, helping thereby in improved charge separation and catalytic activity in a coupled CdS/CdTiO$_3$ semiconductor system.

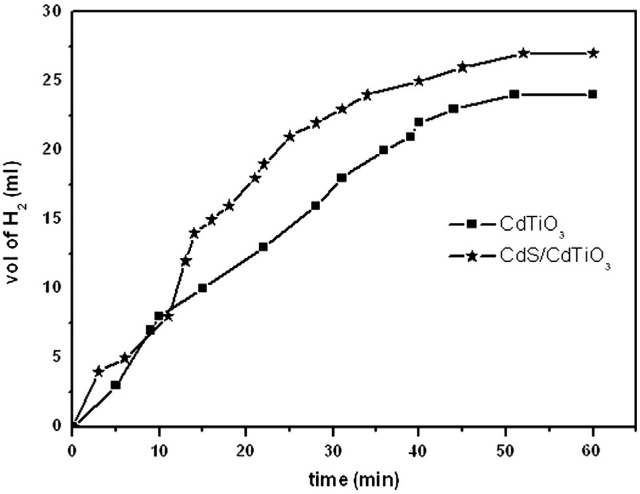


Fig. 7. Hydrogen release from aqueous ammonia–borane in the presence of different catalyst under sunlight irradiation.

4. Conclusions

Successful preparation of CdS distributed cadmium titanate nanofibers were achieved by electrospinning and calcination techniques. The advantage of this strategy lies in its ability; fast degradation of dye and hydrogen release by hydrolysis of the ammonia–borane complex. Incorporation of the CdS nanoparticles enhances the photocatalytic activity of the CdTiO$_3$, so the introduced CdS/CdTiO$_3$ nanocomposite showed high catalytic activity toward releasing of hydrogen from the ammonia–borane complex and photodegradation of methylene blue dye under sunlight irradiation. This proposed strategy might broaden applications of $CdTiO_3$ as an effective photocatalyst.

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