Research Article

Facile Synthesis of Indium Sulfide/Flexible Electrospun Carbon Nanofiber for Enhanced Photocatalytic Efficiency and Its Application

Liu Han,1,2 Haohao Dong,1 Dong Mao,1,2 Baolv Hua,1 Qinyu Li,1 and Dong Fang1,2

1School of Chemistry and Environmental Engineering, Yancheng Teachers University, Yancheng 224002, China
2College of Chemical Engineering, Nanjing Tech University, Nanjing 210009, China

Correspondence should be addressed to Dong Fang; fangd@yctu.edu.cn

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Heterojunction system has been proved as one of the best architectures for photocatalyst owing to extending specific surface area, expanding spectral response range, and increasing photoinduced charges generation, separation, and transmission, which can provide better light absorption range and higher reaction site. In this paper, Indium Sulfide/Flexible Electrospun Carbon Nanofiber (In$_2$S$_3$/CNF) heterogeneous systems were synthesized by a facile one-pot hydrothermal method. The results from characterizations of SEM, TEM, XRD, Raman, and UV-visible diffuse reflectance spectroscopy displayed that flower-like In$_2$S$_3$ was deposited on the hair-like CNF template, forming a one-dimensional nanofibrous network heterojunction photocatalyst. And the newly prepared In$_2$S$_3$/CNF photocatalysts exhibit greatly enhanced photocatalytic activity compared to pure In$_2$S$_3$. In addition, the formation mechanism of the one-dimensional heterojunction In$_2$S$_3$/CNF photocatalyst is discussed and a promising approach to degrade Rhodamine B (RB) in the photocatalytic process is processed.

1. Introduction

Nowadays, it is a huge challenge for people to deal with the organic pollutant in the energy crisis environment [1–3]. Certainly, photocatalytic as a novel solution has aroused great interest for people. It has been considered as one of the most effective ways for the solar energy conversion and the destruction of organic pollutant [4, 5]. Up to now, numerous experiments of the degradation of organic pollutants by using photocatalysts have been researched. However, the photocatalytic activity of pure photocatalyst is limited by its low efficiency of light absorption, difficult migration, and high recombination probability of photogenerated electron-hole pairs, and the development of photocatalytic technology is still limited for the photocatalyst [6–8]. Therefore, it is urgent and indispensable to find a novel photocatalyst to improve both the photochemical activity and the stability.

In$_2$S$_3$, as a typical III–VI group sulfide, is an n-type semiconductor with a band gap of 2.0–2.3 eV corresponding to visible light region which attracted intense interest for optical, photoconductive, and optoelectronic applications. Furthermore, In$_2$S$_3$ shows property of high photosensitivity and photoconductivity, stable chemical and physical characteristics, and low toxicity; it has great potential for visible-light-driven photodegradation of pollutants [9, 10]. Realistically, the narrow band gap and the rapid recombination of photogenerated electron-hole pairs causing poor quantum yield are similar to other visible light photocatalysts [11–14]. To meet the practical application requirements, it is urgent and important to enhance the photocatalytic efficiency of In$_2$S$_3$. Up to now, many attempts have been explored to improve the photocatalytic performance of In$_2$S$_3$, such as metal ions doping, coupling with other semiconductors, and carbon materials-based assemblies [15, 16].

As a viable alternative route to boost the efficiency of photocatalysts, CNF-based assemblies have aroused attention [17–19]. CNF is easily synthesized by electrospinning with a large theoretical specific surface area and high intrinsic electron mobility; it possesses physicochemical, superior electronic, mechanical character, and high absorption properties.
In particular, compared with traditional carbon nanofibers obtained by other physical and chemical methods, the carbon nanofibers synthesized by electrospinning (CNF) have stronger electronic transport properties [20, 21]. Therefore, it is an ideal method to enhance photocatalytic activity by coupling In\(_2\)S\(_3\) with CNF to construct In\(_2\)S\(_3\)/CNF.

In this work, the CNF was fabricated by electrospinning technique, and In\(_2\)S\(_3\)/CNF composites were fabricated through a one-pot hydrothermal reaction as shown in Scheme 1. The photogenerated electrons on the conduction bands (CB) of In\(_2\)S\(_3\) could easily be transferred to CNF for the positive synergetic effect, in brief, because the formation of interface junction can improve the optical absorption property and simultaneously facilitate the separation of photoinduced electron-hole pairs. In addition, the promising applications of In\(_2\)S\(_3\)/CNF composites have excellent performance for the degradation of organic pollutants. This study shows a reliable method to degrade organic pollutants.

2. Experimental Section

2.1. Materials. All the reagents were of analytical grade and were used as received without further purification. InCl\(_3\)-5H\(_2\)O, thioacetamide (TAA), and other chemicals were of analytical grade and purchased from Sinopharm Chemical Reagents Co., Ltd. Polycrylonitrile (PAN) (\(M_w = 150,000\) g mol\(^{-1}\)) was purchased from Sigma-Aldrich.

2.2. Fabrication of CNF. According to previous reports [22], PAN nanofiber was synthesized from PAN by a modified electrospinning method. Firstly, 1 g PAN was dissolved completely in 9 mL N,N-dimethylformamide (DMF). Then, the mixture was transferred to 5 mL plastic syringe by two times for electrospinning (voltage: 20 kV, injection rate: 0.2 mm min\(^{-1}\)). In order to obtain CNF, the PAN was carbonized at 500°C for 2 h under an inert atmosphere with a heating rate of 2 K min\(^{-1}\).

2.3. Fabrication of In\(_2\)S\(_3\)/CNF. In\(_2\)S\(_3\)/CNF with different In\(_2\)S\(_3\) loadings was then prepared by a facile one-pot hydrothermal method. Briefly, a certain amount of InCl\(_3\)-5H\(_2\)O (351, 702, or 1053 mg) and thioacetamide (120 mg) was dissolved in ethyl alcohol (40 mL) under ultrasound conditions. The CNF (50 mg) was then immersed in the above solution, which was then transferred to a Teflon-lined autoclave and heated in a homogeneous reactor at 180°C for 12 h. According to this method, different weight ratios of the In\(_2\)S\(_3\) to g-CNF samples were synthesized and labeled as In\(_2\)S\(_3\)/CNF-1, In\(_2\)S\(_3\)/CNF-2, and In\(_2\)S\(_3\)/CNF-4, respectively. By controlled trial, the In\(_2\)S\(_3\) was fabricated by the same method.

2.4. Characterization. Scanning electron microscopy (SEM; Hitachi S-4800) coupled with X-ray energy dispersive spectroscopy (SEM-EDS) and transmission electron microscopy (TEM; Hitachi H600) were used to observe the morphology, structure, and size of the In\(_2\)S\(_3\)/CNF and its components. The effect of the In\(_2\)S\(_3\) and CNF contents of In\(_2\)S\(_3\)/CNF on its structural properties were investigated by X-ray photoelectron spectroscopy (XPS; Axis Ultra HAS), Raman (Raman; Axis Ultra HAS), and X-ray diffraction (XRD; X‘ Pert-Pro MPD). The optical properties and the dye concentration were determined by UV-visible diffuse reflectance spectroscopy (UV-vis DRS, Shimadzu UV-3600).

2.5. Photocatalytic Activity Measurements. The photocatalytic activities of samples were evaluated by measuring the photodegradation of Rhodamine B (RB) under visible light. In a typical measurement, 40 mg photocatalysts were suspended in 100 mL of 50 ppm aqueous solution of RB. The solution was stirred in the dark for 30 min to obtain a good dispersion and to reach the adsorption–desorption equilibrium between the organic molecules and the catalysts surface [23]. Then the suspension was illuminated with a 250 W xenon lamp. The concentration change of RB was monitored by measuring the UV-vis absorption of the suspensions at regular intervals (take samples every 10 minutes). The suspension was filtered to remove the photocatalysts before...
hexagonal In heterostructures show all the diffraction peaks assigned to normalized temporal concentration changes of the photocatalysts before irradiation, respectively. The indexed to that of In absorption profile at a given time interval [25].

Moreover, the intensities of the corresponding diffraction In peak of In indexed to the (111), (311), (400), (511), (533), and (444) planes of InS3, respectively. The XRD patterns of In2S3/CNF-2 and In2S3 correspond to the (130) plane of orthorhombic CNF, indicating the existence of In2S3 and CNF in the In2S3/CNF-2 heterostructures. Moreover, the intensities of the corresponding diffraction peaks of In2S3 strengthened gradually along with the addition of the CNF in the In2S3/CNF-2 composites; the formation of heterostructures can be demonstrated.

The morphology of the In2S3 and In2S3/CNF-2 was analyzed by SEM and TEM. The flower-like In2S3 with an average diameter of 5 μm possesses porous structures due to the aggregation of a certain amount of nanosheets (Figure 2(a)). The TEM image of Figure 2(b) further confirms the result. The SEM image of electrospun CNF is shown in Figure 2(c), which shows that the average diameter is about 300 nm and there is no defect in a smooth surface. As shown in TEM images (Figure 2(d)), it is clear that the surface of In2S3/CNF-2 is uniformly covered by the ultrathin In2S3 nanosheets after hydrothermal treatment. Further, there is no aggregation found in the surface of In2S3/CNF-2 composites.

The EDX spectrum shown in Figure 2(e) reveals the presence of In and S elements in a mass fraction ratio of 4.47%: 1.61%, which is close to the expected stoichiometry for In2S3 (Au signal is from FTO substrate).

Figure 3 shows that the different concentration of In2S3 deposited on the surface of CNF nanofibers. A small amount of nanoplate-like In2S3 was found on the smooth surface of CNF nanofibers, which correspond to low concentration. As the concentration increases (Figures 3(c) and 3(d)), In2S3 nanosheets with curled shapes grow vertically on the nanofiber surface and with a uniform distribution. In addition, the surface of nanofiber also turns from smooth to rough. As shown in Figures 3(e) and 3(f), serious aggregation occurred and thick layer In2S3 nanosheets were observed after further increasing the In2S3 concentration. The rapid nucleation of In2S3 at high concentration can be demonstrated.

XPS measurements were carried out to testify the chemical composition and chemical states of elements in In2S3/CNF-2 heterostructure photocatalyst [26]. The full-scale XPS spectrum for In2S3/CNF-2 sample is shown in Figure 4(a), in which the In, S, and C elements could be detected and no other impurities were observed. Figures 4(b), 4(c), and 4(d) show the high-resolution XPS spectra for In2S3/CNF sample. The XPS peaks (Figure 4(b)) at 444.1 and 452.7 eV correspond to the In3s5/2 and In3d3/2 states [27], respectively. The peak at 161.9 eV in Figure 4(c) corresponds to the S2p3/2 state of S2− moieties. The peak at 284.8 eV in Figure 4(d) corresponds to the C1s state. The above XPS

3. Results and Discussion

The X-ray diffraction (XRD) patterns of pure CNF, In2S3, and In2S3 are shown in Figure 1. All of the diffraction peaks can be indexed to that of In2S3 with a cubic phase structure (JCPDS, number 32-0456). Peaks at 2θ of 14, 27, 33, 44, 48, 56, and 60° in the XRD patterns of In2S3/CNF-2 and In2S3 correspond to the (111), (311), (400), (511), (533), and (444) planes of In2S3, respectively. The XRD patterns of the In2S3/CNF-2 heterostructures show all the diffraction peaks assigned to hexagonal In2S3 except the peak at 25° which corresponds to (130) plane of orthorhombic CNF, indicating the existence of In2S3 and CNF in the In2S3/CNF-2 heterostructures. Moreover, the intensities of the corresponding diffraction peaks of In2S3 strengthened gradually along with the addition of the CNF in the In2S3/CNF-2 composites; the formation of heterostructures can be demonstrated.

As shown in Figure 4(c), which shows that the average diameter is about 300 nm and there is no defect in a smooth surface. As shown in TEM images (Figure 2(d)), it is clear that the surface of In2S3/CNF-2 is uniformly covered by the ultrathin In2S3 nanosheets after hydrothermal treatment. Further, there is no aggregation found in the surface of In2S3/CNF-2 composites.

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results confirm that the composites are composed of In$_2$S$_3$ and CNF.

Raman analysis was explored to confirm the presence of CNF and In$_2$S$_3$ in In$_2$S$_3$/CNF-2 sample (Figure 5). D-peak (D band) represents the defects of C atomic lattice, and G-peak (G band) represents the expansion vibration of the surface of C atom sp$^2$ hybridization. And the representative Raman spectrum in a range of Raman shift from 100 to 2000 cm$^{-1}$ of the CNF shows mainly two peaks centered around 1369 cm$^{-1}$ (D band) and 1590 cm$^{-1}$ (G band) for CNF. Furthermore, the degree of fibrosis can be measured by the intensity ratio of the G to D band ($I_G/I_D$) [28–30], where $I_G/I_D$ is the intensity ratio of D-peak and G-peak. A slight increase in the $I_G/I_D$ ratio is observed in the spectrum of In$_2$S$_3$/CNF-2 composites, the D/G integral intensity ratio ($I_D/I_G$) for CNF in the In$_2$S$_3$/CNF-2 sample (1.13) is slightly higher than that of CNF (1.12), it is indicated that a certain amount of In$_2$S$_3$ deposited on the surface of CNF during the chemical reduction process,

| Element | Wt%  | At%  |
|---------|------|------|
| C K     | 68.45| 96.31|
| Au M    | 25.46| 2.18 |
| S K     | 1.61 | 0.85 |
| In L    | 4.47 | 0.66 |
| Total   | 100.00| 100.00|

Figure 2: SEM images of In$_2$S$_3$ and CNF ((a) and (c)), TEM images of In$_2$S$_3$ and In$_2$S$_3$/CNF-2 ((b) and (d)), and EDX pattern of In$_2$S$_3$/CNF-2 (e).
and the conjugated CNF network was reestablished [31]. The two peaks for D and G band of the composite no shift appears, indicating that only a small amount of In$_2$S$_3$ deposited on the surface of CNF.

The optical properties of the three samples were detected by UV-vis DRS absorption spectroscopy (Figure 6). Obviously, CNF shows the best performance and its absorption peaks appear in the visible light and UV light regions. It should be noted that In$_2$S$_3$/CNF-2 with the addition of CNF showed an increased photocatalytic performance compared to In$_2$S$_3$ (Figure 6(a)). The band gap energy ($E_g$) of samples was calculated by Tauc’s equation [32, 33] and the result was shown in Figure 6(b); the $E_g$ values of In$_2$S$_3$ and In$_2$S$_3$/CNF-2 in Figure 6(b) are approximately 2.70 and 3.08 eV. The band gap of In$_2$S$_3$/CNF-2 was higher than In$_2$S$_3$, which is close to the value of In$_2$S$_3$ and In$_2$S$_3$/CNF reported in other literatures [10, 34]. Thus, it is indicated that the as-prepared In$_2$S$_3$/CNF-2 heterojunction structures have the appropriate $E_g$ for photodegradation of organic pollutants under visible light irradiation.

In order to detect the ability of photodegradation, different photocatalysts were used to photodegrade organic pollutant under visible light irradiation, then the samples of products were analyzed. The results are shown in Figures 7(a)
Figure 4: XPS spectra of the In$_2$S$_3$/CNF-2: survey spectrum (a), In 3d (b), S 2p (c), and C 1s (d).

Figure 5: Raman spectra of CNF and In$_2$S$_3$/CNF-2.
Langmuir-Hinshelwood apparent first-order kinetics model when the initial degradation reactions followed a Langmuir-Hinshelwood degradation of RB was explored to confirm it. The above characterisation absorption spectra of RB at 570 and 580 nm. Owing to the strong absorption ability of CNF, a certain amount of RB was attached to the CNF before irradiation. Furthermore, the dissociation and adsorption reach equilibrium, the concentration change of Rhodamine B, which is degraded by In$_2$S$_3$ and In$_2$S$_3$/CNF-2, is the same. The concentration of RB does not significantly change after irradiation as shown in Figure 7(a). The change in the concentration of RB in Figure 7(b) is significantly greater than Figure 7(a), which can be further confirmed through the change of solution color before and after degradation with different photocatalysts.

The degradation curves of RB on pure In$_2$S$_3$, CNF, and In$_2$S$_3$/CNF-2 composites were shown in Figure 8(a). Obviously, the concentration of CNF almost has no change, indicating that pure CNF has no photocatalytic activity under visible light irradiation. The In$_2$S$_3$/CNF-2 composites have a better photocatalytic efficiency (78.2%) for RB after visible light irradiation for 60 min than that of pure In$_2$S$_3$. It is concluded that In$_2$S$_3$/CNF-2 composites exhibited much higher photocatalytic efficiency compared with the pure In$_2$S$_3$. For a better comparison of the photocatalytic efficiency of In$_2$S$_3$/CNF-2 and pure In$_2$S$_3$, the kinetic analysis of degradation of RB was explored to confirm it. The above degradation reactions followed a Langmuir-Hinshelwood apparent first-order kinetics model [32, 35] when the initial concentrations of the reactants are less than 100 ppm. The Langmuir-Hinshelwood apparent first-order kinetics model is described below:

$$-\ln\left(\frac{C_0}{C}\right) = K_{app}t,$$  \hspace{1cm} (1)

where $K_{app}$ is the apparent first-order rate constant (min$^{-1}$). The determined $K_{app}$ values for degradation of RB with different catalysts are presented in Figure 8(b). It is clear that the as-prepared In$_2$S$_3$/CNF-2 composites show the highest reaction rate among the two catalysts with $K_{app} = 0.0232$ min$^{-1}$, while $K_{app} = 0.0169$ min$^{-1}$ for pure In$_2$S$_3$. The photocatalytic reactivity order is well consistent with the activity studies above.

It is reasonable to presume that the photogenerated electrons ($e^-$) transfer from In$_2$S$_3$ to CNF in the In$_2$S$_3$/CNF system under visible light irradiation. Therefore, the photogenerated electrons first transfer to CNF and then are trapped by O$_2$ and H$_2$O at the surface of photocatalyst or solution to form the active species such as O$_2^-$. These active species could help the degradation of RB dye. At the same time, the photogenerated holes (h$^+$) could react with H$_2$O to form $^{\cdot}$OH, hydrogen ions (H$^+$), and then oxidize RB dye directly [12]. The complete photodegradation process can be summarized by the following reaction steps:

$$\text{In}_2\text{S}_3 + h\nu \rightarrow \text{In}_2\text{S}_3 \left( e^- + h^+ \right)$$
$$e^- (\text{CB, In}_2\text{S}_3) + \text{O}_2 \rightarrow \text{O}_2^-$$
$$h^+ (\text{VB, CNF}) + \text{H}_2\text{O} \rightarrow \text{H}^+ + ^{\cdot}\text{OH}$$  \hspace{1cm} (2)
$$\text{RB} + \left( \text{O}_2^- + h^+ + ^{\cdot}\text{OH} \right) \rightarrow \text{several steps}$$
$$\rightarrow \text{other products} + \text{CO}_2 + \text{H}_2\text{O}$$

4. Conclusion

In summary, an effective method of preparing In$_2$S$_3$/CNF photocatalysts was described in this paper. The incorporation...
of CNF serving as electron collectors realizes a more effective separation of photogenerated electron-hole pairs and greatly boosts the photocatalytic activity of the products compared with the pure In$_2$S$_3$. The In$_2$S$_3$/CNF-2 composites show strong adsorption ability towards the RB, they can degrade 50 ppm of RB in 60 minutes under visible lights, and the excellent degradation RB activities of In$_2$S$_3$/CNF are mainly attributed to the large amount of effectively reactive species like $h^+$ and $O_2^-$. Overall, this study provides a new option to construct the semiconductor/CNF composites with high photocatalytic activity, environmental remediation, and energy conversion.

**Conflicts of Interest**

The authors declare that they have no conflicts of interest.
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