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Article

Synthesis and characterization of sulfur-titanium dioxide nanocomposites for photocatalytic oxidation of cyanide using visible light irradiation

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ABSTRACT

A sol-gel method was used to prepare TiO_2 and sulfur- TiO_2 (S- TiO_2) nanocomposites, which were characterized by N_2 adsorption-desorption, X-ray diffraction, X-ray photoelectron spectroscopy, photoluminescence, ultraviolet visible and transmission electron microscopy measurements. The photocatalytic performance of TiO_2 and S- TiO_2 nanocomposites, with respect to the photocatalytic oxidation of cyanide under visible light irradiation, was determined. The results reveal that S is well dispersed on the surface of TiO_2 nanoparticles. Additionally, the surface area of the S- TiO_2 nanocomposites was observed to be smaller than that of the TiO_2 nanoparticles because of blocked pores caused by doping with S. The S- TiO_2 nanocomposite (0.3 wt% S) exhibited the lowest band gap and the highest photocatalytic activity in the oxidation of cyanide. The photocatalytic performance of S- TiO_2 (0.3 wt% S) nanocomposites was stable, even after the fifth reuse of the nanoparticles for the oxidation of cyanide.

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1. Introduction

Cyanide (CN) is an inorganic pollutant that is released in the aquatic environment from different anthropogenic sources. It is produced from different industries including photography, pharmaceuticals, plastics, ore leaching, metal mining, finishing, cleaning, plating, processing, electroplating, automobile parts manufacture, steel mills, steel tempering, and coal coking [1–4]. Cyanide usually exists in the form of a simple salt (KCN or NaCN and HCN) or as part of a complex [2–8]. In soils, cyanides bond weakly to form complexes with metals found in industrial effluents, such as copper, iron, zinc and nickel. Cyanides such as the cyanide anion and hydrogen cyanide are considered the most toxic. Cyanide can also form a weak-acid dissociable complex with copper, iron, zinc and nickel [6,7] and strong-acid dissociable complexes with gold, cobalt, silver and iron [6,7].

Organic cyanides such as propionitrile and acrylonitrile also exist [9]. The concentration of cyanide in environment depends mainly on human activity. Normal cyanide levels in unpolluted stream and lake water is between 0.001–0.05 ppm [10]. The cyanide concentration range for industrial effluents is between 0.01–10.00 ppm [10]; however, levels from effluents of electroplating plants can be as high as 100,000 ppm [10]. It is therefore very important that effluents from different industries are treated to reduce the cyanide concentration level before release into the environment and a maximum concentration for levels in aquatic-biota and drinking water, respectively, were imposed by the US Environmental Protection Agency. In Germany and Switzerland, limits were set at 0.01 ppm for surface water cyanide and 0.5 ppm for sewers [11]. Accordingly, removal, degradation and/or recycling are crucial for reducing the cyanide concentration in different aquatic environments to

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meet regulatory limits. Different methods are known for treating cyanide such as physical, adsorption, complexation, and oxidation [12–14]. Alkaline chlorination is the most common technique for removal of cyanide and increases the total solids dissolved in water [15]. Another method is the photocatalytic degradation of pollutants using solar light, which is an abundant resource, making the process more economic. [16]. The most well-known photocatalyst is TiO_2 [17–21] – as a non-toxic catalyst, it can be used many times without reduction of the photocatalytic activity. The application of TiO_2 particles for the photocatalytic degradation of dyes under ultraviolet (UV) or visible light has attracted much attention, compared with other less efficient conventional treatment methods [19,20]. Nevertheless, the application of TiO_2 as a photocatalyst for wastewater treatment is relatively low because of its poor photodegradation efficiency [22]. Researchers have attempted to enhance this by the deposition and doping of metals [23–27], with mixed results [28]. The addition of adsorbent materials such as zeolites, clay, silica, alumina fiber and glass also was found to improve TiO_2 photocatalytic activity [29–34]. Many recent studies showed that activated carbons could be used as a co-adsorbent and enhance TiO_2 photocatalytic activity because of their high adsorption capacity and very porous structure [30–35].

It has been proposed that TiO_2 photocatalytic activity initiates free radicals such as $\text{HO}\cdot$ to degrade organic compounds [36]. Another suggested mechanism is that organic compounds are first adsorbed on the surface of the photocatalyst and then react with excited superficial e^-/h^+ pairs or $\text{HO}\cdot$ radicals [37]. There are few reports on the adsorption kinetics of organic compounds on photocatalysts [38–40].

Carbon nanotubes (CNTs) have attracted the attention of scientists worldwide because of the special structure of CNTs, and their electronic and mechanical properties, which has allowed them to be used for different applications. Many advanced composites are made of CNTs because of their high mechanical strength [41]. CNTs can be metallic, semi-metallic or semi-conducting, depending on their tube diameter and helicity [42], which has allowed them to be used in nanoscale electro-device applications. Their hollow and layered structures, as well as large specific surface area, have meant they have been applied in the storage of hydrogen [43] and as an adsorbent for different pollutants [44–51]. Consequently, CNTs were considered as promising materials for environmental cleaning. Progress in CNT preparation technology has seen their price decrease significantly and it is now possible to use CNTs in a large-scale operation. CNTs have two distinguishing properties that could be employed in the photodegradation of different pollutants: high adsorption capacity and excellent conductivity. The anatase form of TiO_2 has superior photocatalytic activity in comparison with other photocatalysts. Therefore, the application of CNTs to the photocatalytic degradation of a dye with TiO_2 as a photocatalyst is a promising strategy. Jo et al. [52] prepared aluminum sheet-based, S-doped TiO_2 for the degradation of different toxic organic vapors under visible light. Mohamed et al. [53] prepared P- TiO_2 thin films for photocatalytic degradation of butyl benzyl phthalate under visi-

ble-light irradiation. Lü et al. [54] prepared N and S co-doped- TiO_2 /fly ash bead composite materials for photocatalytic degradation of methylene blue dye under visible light irradiation. To the best of our knowledge, there are no reports on the oxidation of cyanide in aqueous solution by S- TiO_2 . The present study reports the synthesis and characterization of S- TiO_2 nanocomposites and the evaluation of its photocatalytic activity for the oxidation of cyanide in the aqueous phase.

2. Experimental

All chemicals used in this study were used as received, without any further purification.

2.1. Preparation of the photocatalyst

The photocatalyst, TiO_2 , was prepared by the following method: 0.04 g of 36 % HCl was dissolved in 10 mL of water and then added dropwise to dodecylamine solution (3 g in 30 mL water). The resulting solution A was stirred at room temperature for 1 h; 0.8 g of titanium isopropoxide dissolve in 18 mL of isopropyl alcohol and the resulting solution B was stirred at room temperature for 1 h. Solution A was added to solution B and stirred at room temperature for 24 h, followed by filtration and calcination for 5 h at 550 °C. S- TiO_2 samples (containing 0.1 wt%, 0.2 wt%, 0.3 wt% and 0.4 wt% of S) were synthesized using the same method of preparation of TiO_2 ; however, titanium isopropoxide was mixed with thiourea as the source of sulfur.

2.2. Characterization techniques

X-ray diffraction (XRD) analysis was performed at room temperature with a Bruker axis D8 using $\text{Cu-K}\alpha$ radiation ($\lambda = 0.154$ nm). The specific surface area was calculated from N_2 -adsorption measurements, which were obtained using a Nova 2000 series Chromatech apparatus at -196 °C. Prior to the measurements, the samples were treated under vacuum at 100 °C for 2 h. The band gap of the samples was identified by UV-visible diffuse reflectance spectroscopy (UV-Vis-DRS), which was performed in air, at room temperature over the wavelength range of 200–800 nm using a UV/Vis/NIR spectrophotometer (V-570, JASCO, Japan). Transmission electron microscope (TEM) analysis was conducted with a JEOL-JEM-1230 microscope, and samples were prepared by suspension in ethanol, followed by ultrasonication for 30 min. Subsequently, a small amount of this solution was placed onto a carbon-coated copper grid and dried before loading the sample into the TEM. X-ray photoelectron spectroscopy (XPS) studies were performed using a Thermo Scientific K-ALPHA, XPS, England. Photoluminescence (PL) emission spectra were recorded using a Shimadzu RF-5301 fluorescence spectrophotometer. The actual amount of sulfur in prepared samples was ascertained by inductively coupled plasma-mass spectrometry (ICP-MS). It was found that 0.1 wt%, 0.2 wt%, 0.3 wt% and 0.4 wt% S- TiO_2 comprised of 0.95 wt%, 0.20 wt%, 0.30 wt% and 0.39 wt% S, respectively.

2.3. Analysis of hydroxyl radicals

The formed hydroxyl radical ($\cdot\text{OH}$) on the surface was measured by a photoluminescence method [55,56], which uses terephthalic acid as a probe molecule. The intensity of the PL signal of 2-hydroxyterephthalic acid, produced by the reaction of terephthalic acid with $\cdot\text{OH}$, was measured at 425 nm. Therefore, the amount of $\cdot\text{OH}$ produced on the surface of TiO_2 is proportional to the PL intensity of 2-hydroxy terephthalic acid. To measure $\cdot\text{OH}$ radicals, 0.1 g of the prepared sample was dispersed in a terephthalic acid aqueous solution (20 mL, 5×10^{-4} mol/L) with NaOH (2×10^{-3} mol/L) in a dish with a diameter of approximately 9.0 cm. The light source was a 125 W high pressure Hg lamp, held 10 cm above the dishes. The integrated visible light intensity measured with a visible light radiometer (Model: FZ-A) was 2.9 mW/cm², and the wavelength range was 400–1000 nm. Photoluminescence spectra of the generated 2-hydroxyterephthalic acid were measured on a Shimadzu RF-5301 fluorescence spectrophotometer. After irradiation every 10 min, the reaction solution was filtered to measure the increase in the PL intensity at 425 nm.

2.4. Photocatalysis experiment

The application of synthesized nanocomposites for the photodegradation of cyanide was investigated under visible light. The experiments were carried out using a horizontal cylinder annular batch reactor. The photocatalyst was irradiated with a blue fluorescent lamp (150 W) and the reactor doubly covered with a UV cut filter. In a typical experiment, photocatalyst was added to a 300-mL, 100-mg/L potassium cyanide (KCN) solution (pH 10.5, which was adjusted by ammonia solution to avoid the evolution of HCN gas). The reaction was carried out isothermally at 25 °C and samples of the reaction mixture were taken at different intervals for a total reaction time of 1 h. The CN^- (aq) concentration in the samples was estimated by volumetric titration with silver nitrate, using potassium iodide to determine the titration end point. The removal efficiency of CN^- (aq) has been measured by applying the following

Fig. 1. XRD patterns of TiO_2 and S- TiO_2 nanocomposites.

Fig. 2. XPS spectrum of S 2p (0.3 wt% S- TiO_2 sample).

equation.

$$\text{Removal efficiency (\%)} = (C_0 - C)/C_0 \times 100$$

Where C_0 is the initial concentration of uncomplexed CN^- (aq) in solution, C is the concentration of unoxidized CN^- (aq) in solution.

3. Results and discussion

3.1. Structural, morphological and compositional characterizations

Figure 1 shows the XRD patterns of the TiO_2 and S- TiO_2 nanocomposites. The results show that the nanocomposites are mainly composed of TiO_2 anatase and indicate a lack of diffraction peaks from S or SO_2 in the patterns of the S- TiO_2 samples. This is because the wt% of S is below the XRD detection limit, or perhaps because S is well dispersed on surface of the TiO_2 nanoparticles.

Figure 2 shows the XPS spectrum of S 2p for a sample of S- TiO_2 . The peak for S 2p at 169.2 eV confirms the presence of S in the sample.

Figure 3 shows TEM images of S- TiO_2 nanocomposites. The

Fig. 3. TEM images of S- TiO_2 nanocomposites, where the wt% of S is 0.1 (a), 0.2 (b), 0.3 (c), 0.4 (d).

Table 1Textural parameters of TiO₂ and S-TiO₂ nanocomposites.

Sample	S_{BET} (m ² /g)	S_{t} (m ² /g)	S_{micro} (cm ² /g)	S_{ext} (cm ² /g)	V_{p} (cm ³ /g)	V_{micro} (cm ³ /g)	V_{meso} (cm ³ /g)	r (nm)
TiO ₂	56.00	56.00	36.00	23.00	0.220	0.174	0.060	3.5
0.1 wt% S-TiO ₂	53.00	53.00	34.00	19.00	0.165	0.130	0.035	4.5
0.2 wt% S-TiO ₂	51.00	52.00	33.00	18.00	0.142	0.110	0.032	5.5
0.3 wt% S-TiO ₂	49.00	49.00	32.00	18.00	0.135	0.105	0.030	6.0
0.4 wt% S-TiO ₂	44.00	44.00	25.00	16.00	0.120	0.095	0.020	6.5

Note: S_{BET} —BET-surface area, S_{t} —surface area derived from V_{t} plots, S_{micro} —surface area of micropores, S_{ext} —external surface area, V_{p} —total pore volume, V_{micro} —volume of micropores, V_{meso} —volume of mesopores, r —mean pore radius.

results show that an increase in S content increases the dispersion of S on the surface of the TiO₂ nanoparticles. Additionally, an increase of up to 0.3 % S increases the homogeneity of the S particle size on the TiO₂ nanoparticle surface. This homogeneity decreases at higher concentrations i.e., 0.4 wt% S, which suggests that there is an optimum content of S ions that controls the size and homogeneity.

3.2. Surface area analysis

The texture parameters of the TiO₂ and S-TiO₂ nanocomposites are presented in Table 1. The S_{BET} values for TiO₂ and 0.1 wt%, 0.2 wt%, 0.3 wt%, and 0.4 wt% S-TiO₂ were determined to be 56, 53, 51, 49, and 44 m²/g, respectively. The total pore volume of S-TiO₂ samples is smaller than that of the TiO₂ sample because of the blocking of some pores by deposition of sulfur. The presence of mesopores in all samples was confirmed by the similar values of S_{BET} and S_{t} in most samples, as presented in Table 1.

3.3. Optical characterization

Figure 4 shows the UV-Vis diffuse reflectance spectra of TiO₂ and S-TiO₂ nanocomposites. The results demonstrate that the doping of sulfur onto the surface of TiO₂ leads to a shift in the absorption edge of TiO₂ from 394 to 455 nm. The UV-Vis spectra were used to calculate the direct band gap of the TiO₂ and S-TiO₂ nanocomposites based on a method by Mohamed [29]. The band gap energies were calculated using the following equation:

Fig. 4. UV-Vis absorption spectra of TiO₂ and S-TiO₂ nanocomposites.

$$E_g = 1239.8/\lambda$$

Where E_g is the band gap (eV) and λ is the wavelength (nm) of the absorption edges in the spectrum (Table 2). The results reveal that an increase in sulfur from 0.1 wt% to 0.3 wt% decreases the band gap energy from 3.15 to 2.75 eV. However, there is no significant effect on the band gap at a content of S greater than 0.3. Therefore, there is an optimum content of deposited S that controls the band gap.

We investigated the separation and recombination of photogenerated charge carriers and the transfer of the photogenerated electrons and holes by gathering photoluminescence emission spectra. Figure 5 shows (PL) spectra of TiO₂ and S-TiO₂ nanocomposites. The results indicate that an increase in S content from 0.1 wt% to 0.3 wt%, which was doped onto the TiO₂ nanoparticles, leads to a decrease in the PL intensity. However, there is no significant effect on the PL intensity at a content of sulfur above 0.3 wt%. Therefore, there is an optimum content of deposited S that yields the carrier lifetime required for electron-hole recombination, in agreement with the UV-Vis results.

Table 2Band gap energy of TiO₂ and S-TiO₂ nanocomposites.

Sample	Band gap energy (eV)
TiO ₂	3.15
0.1 wt% S-TiO ₂	3.05
0.2 wt% S-TiO ₂	2.91
0.3 wt% S-TiO ₂	2.75
0.4 wt% S-TiO ₂	2.74

Fig. 5. PL spectra of TiO₂ and S-TiO₂ nanocomposites.

Fig. 6. Effect of the content of S on the photocatalytic activity of TiO₂ nanocomposites for the oxidation of cyanide.

3.4. Photocatalytic activity

Figure 6 shows the effect of changing the content of S on the photocatalytic activity of TiO₂ nanoparticles on the oxidation of cyanide under visible light irradiation. The experiment was performed under the following conditions: KCN (100 ppm, 500 mL) and 0.2 g photocatalyst. The results reveal that the photocatalytic activity increased from 10% to 100%, with an increase in sulfur from 0 to 0.3 wt%. However, increasing sulfur above 0.3 wt% led to a decrease in the photocatalytic activity from 100% to 99%. This decrease was observed because a high content of sulfur hinders the penetration of light to the surface of TiO₂, thereby decreasing the photocatalytic performance of TiO₂, with respect to the oxidation of cyanide.

The change in PL spectra with increased irradiation time for the 0.3 wt% S-TiO₂ sample in a terephthalic acid solution is shown in Fig. 7(a). The results show that a gradual increase in PL intensity at 425 nm occurs with increasing irradiation time. No PL increase was observed in the absence of visible light or in the 0.3 wt% S-TiO₂ sample not in a terephthalic acid solution. This suggests that the fluorescence is from the chemical reactions between terephthalic acid and •OH formed at the 0.3 wt% S-TiO₂/water interface via photocatalytic reactions. The change

Fig. 8. Effect of loading of the 0.3 wt % S-TiO₂ sample on the photocatalytic oxidation of cyanide.

in PL spectra based on irradiation time for different samples is shown in Fig. 7(b). The results show that •OH is produced from 0.3 wt% S-TiO₂ nanoparticles more quickly than on the undoped TiO₂, 0.1 wt%, 0.2 wt%, and 0.4 wt% S-TiO₂ samples. Thus, the 0.3 wt% S-TiO₂ nanoparticles the highest photocatalytic activity under visible light.

Figure 8 shows the effect of loading the 0.3 wt% S-TiO₂ sample on the photocatalytic oxidation of cyanide solution under visible light irradiation. The experiment was performed under the following conditions: KCN (100 ppm, 1 L) and 0.3 wt% S-TiO₂ nanocomposite. The results reveal that photocatalytic performance after 60 min was increased from 82% to 100%, with an increase in the weight of the photocatalyst from 0.2 to 0.4 g/L. The reaction time required to complete the oxidation of cyanide was 60, 50, and 30 min as the weight of the photocatalyst was increased to 0.4, 0.6, and 0.8 g/L, respectively. However, the reaction time required to complete the oxidation of cyanide was increased to 40 min, with an increase in the weight of the photocatalyst above 0.8 g/L; i.e., the optimum weight of the photocatalyst is 0.8 g/L.

Figure 9 shows the effect on photocatalytic oxidation of cyanide solution under visible light irradiation, which was studied by varying the KCN solution concentration from 25 to 200 ppm

Fig. 7. Fluorescence spectral changes in a NaOH solution of terephthalic acid for 0.3 wt % S-TiO₂ (a) and other catalysts (b).

Fig. 9. Effect of the initial cyanide solution concentration on the photocatalytic oxidation of cyanide solution.

in the presence of 0.3 wt% S-TiO₂ photocatalyst. The results demonstrate that the photocatalytic activity remained nearly unchanged with an increase in the KCN solution concentration after 30 min of reaction time required to complete the oxidation of cyanide. However, for cyanide solution concentrations above 100 ppm, i.e., 150 and 200 ppm, the reaction time required to complete the oxidation of cyanide was increased to 40 and 60 min, respectively. The photocatalytic activity depends on $\cdot\text{OH}$ reaching the surface of the catalyst and then reacting with the cyanide solution; therefore, increasing the concentration increases the probability of a reaction between the free radicals and the cyanide solution, thereby increasing the photocatalytic activity. A further increase in the cyanide solution concentration decreases the photocatalytic activity because the active sites of the photocatalyst are blocked by cyanide solution, preventing visible light from penetrating the surface of active sites.

Testing the photocatalyst for multiple cycles is an important factor for commercial use of the photocatalyst. Figure 10 shows the degradation of cyanide remains at 100% even after the 0.3 wt% S-TiO₂ photocatalyst is reused five times.

4. Conclusions

In summary, a S-TiO₂ nanocomposite photocatalyst was

Fig. 10. Recycling and reuse of the 0.3 wt % S-TiO₂ sample for the photocatalytic degradation of cyanide.

successfully synthesized and proven to be a promising catalyst because of its high efficiency in oxidizing the pollutant cyanide under visible light. The band gap of the TiO₂ photocatalyst can be controlled by adjusting the content of S that is deposited onto its surface. The results of photocatalytic studies reveal that the highest photocatalytic activity and stability were obtained from the 0.3 wt% S-TiO₂ nanocomposite photocatalyst, which can be used to oxidize 100% of a cyanide solution after 30 min exposure.

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Graphical Abstract

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Synthesis and characterization of sulfur-titanium dioxide nanocomposites for photocatalytic oxidation of cyanide using visible light irradiation

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S-TiO₂ nanocomposite photocatalyst can degrade waste materials under visible light. S-TiO₂ nanocomposite has lower band gap and higher photocatalytic activity than TiO₂ nanoparticle. S-TiO₂ nanocomposite can be recycled for five times.

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