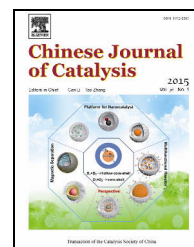


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Article

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 TiO₂ and sulfur-TiO₂ (S-TiO₂) nanocomposites, which were characterized by N₂ adsorption-desorption, X-ray diffraction, X-ray photoelectron spectroscopy, photoluminescence, ultraviolet visible and transmission electron microscopy measurements. The photocatalytic performance of TiO₂ and S-TiO₂ 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 TiO₂ nanoparticles. Additionally, the surface area of the S-TiO₂ nanocomposites was observed to be smaller than that of the TiO₂ nanoparticles because of blocked pores caused by doping with S. The S-TiO₂ 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-TiO₂ (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.

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