

Nano-Ag particles doped TiO₂ for efficient photodegradation of Direct azo dyes

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Abstract

Silver nanoparticles doped TiO₂ has been prepared and characterised by surface analytical methods such as BET surface area, scanning electron micrographs (SEM), X-ray diffraction (XRD), energy dispersive X-ray micro analysis (EDX), electron spin resonance (ESR) and diffuse reflectance spectroscopy (DRS). We have investigated the photocatalytic degradation of two Direct diazo dyes, Direct red 23 (DR 23) and Direct blue 53 (DB 53) in the aqueous suspensions of TiO₂ and Ag deposited TiO₂ nanoparticles under UV-A light irradiation in order to evaluate the various effects of silver deposition on the photocatalytic activity of TiO₂. The presence of silver in TiO₂ was found to enhance the photodegradation of DR 23 and DB 53. The higher activity of silver doped TiO₂ is due to the enhancement of electron–hole separation by the electron trapping of silver particles.

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1. Introduction

Heterogeneous photocatalysis is an alternative economical and harmless technology of advanced oxidation processes (AOP) for removal of organic impurities. During the process, illuminated semiconductor absorbs light and generates active species which leads to complete oxidation of organic components in waste water. A distinct advantage of the photocatalysis lies in its ability to utilise solar energy in the production of active species OH•. Photocatalysis on semiconductors has been studied in many fields. For example: (i) fuel production: hydrogen from water photolysis [1–3], (ii) removal/recovery of heavy metal ions [4,5], (iii) water detoxification: removal of toxic, harmful or hazardous pollutants [6–11].

A great many photocatalysts have been examined for the degradation of organic pollutants in waste water. Among various semiconductor metal oxides, TiO₂ has been the focus of photocatalysts under UV irradiation because of its physical and chemical stability, low cost, ease of availability, non-toxicity and

electronic and optical properties. Despite the positive attributes of TiO₂, there are a few drawbacks associated with its use; it has a high bandgap ($E_g > 3.2$ eV) and it is excited only by UV light ($\lambda < 388$ nm) to inject electrons into the conduction band and to leave holes into the valence band [12]. Thus, this practically limits the use of sunlight or visible light as an irradiation source in photocatalytic reactions on TiO₂ [13]. In addition, the high rate of electron–hole recombination on TiO₂ particles results in a low efficiency of photocatalysis [14]. For the purpose of overcoming these limitations of TiO₂ as a photocatalyst, numerous studies have been recently performed to enhance electron–hole separation and to extend the absorption range of TiO₂ into the visible range. These studies include doping metal ions into the TiO₂ lattice [15,16], dye photosensitisation on the TiO₂ surface [17–21], addition of inert support [22] and deposition of noble metals [23–34].

In particular, noble metal-modified semiconductor nanoparticles become of current importance for maximising the efficiency of photocatalytic reactions. The noble metals such as Pt [23,24] and Au [25,26] deposited or doped on TiO₂ have the high Schottky barriers among the metals and thus act as electron traps, facilitating electron–hole separation and promotes the interfacial electron transfer process [5,35–37]. Most studies of noble metal-modified TiO₂ photocatalysts have focused

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on the details of the photoinduced electron transfer from the conduction band of UV-irradiated TiO₂ to noble metals for improving the photocatalytic activity of TiO₂ under UV irradiation.

Noble metals doped or deposited on TiO₂ are expected to show various effects on the photocatalytic activity of TiO₂ by different mechanisms. These noble metals act separately or simultaneously depending on the photoreaction conditions. They may (i) enhance the electron–hole separation by acting as electron traps, (ii) extend the light absorption into the visible range and enhance surface electron excitation by plasmon resonances excited by visible light and (iii) modify the surface properties of photocatalysts.

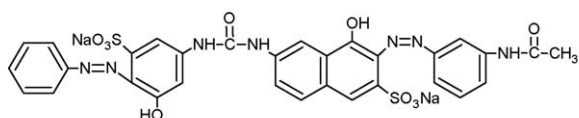
In the recent years, silver ions have attracted the interests of several researchers [38–40], because of both their novel effects on the improvement of photoactivity of semiconductor photocatalysis nanocrystallites [39,40] and their effects on antibacterial activity [38]. These properties can be applied to a tremendous range of applications, for instance, environment, textiles, engineering materials and so on. However, the studies on silver doped photocatalyst nanocrystallites are still limited in the literature [41–49].

The aim of the present work is to prepare Ag doped TiO₂ (anatase) by a photodeposition method and to compare the activity of the photocatalyst before and after surface modification with metallic silver for the degradation of two azo dyes DB 53 and DR 23.

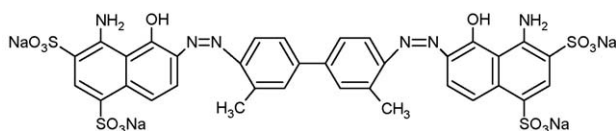
2. Experimental

2.1. Materials

Titanium dioxide (anatase) with a BET surface area of 21.53 m²/g and perchloric acid were supplied by Qualigen. Direct red 23 (DR 23), C.I. 29160 from S.D. fine and Direct blue 53 (DB 53) C.I. 23860 from CDH were used as such. The structure of these dyes are shown in Fig. 1. Silver nitrate (99.5 wt.%) analytical grade from Merck was used as a silver source for the preparation of Ag doped TiO₂ photocatalysts. Double distilled water was used for all the experiments.



Structure of DR 23



Structure of DB53

Fig. 1. Structures of DR23 and DB53.

2.2. Preparation of Ag doped TiO₂ photocatalysts

The Ag doped TiO₂ catalysts were prepared by photoreducing Ag⁺ ions to Ag metal on the TiO₂ surface as per the procedure given below [41].

First pH of the TiO₂ suspension was adjusted to 3 prior to reaction using perchloric acid. Aliquots of various amounts of Ag⁺ ions, prepared by dissolving silver nitrate salt in deionised water, were added into the suspension of TiO₂ such that the Ag⁺ concentration was 0.5, 1.0, 1.5 and 2.0 at.% in relation to TiO₂. The mixtures were then irradiated with UV light by eight mercury lamps (8 W) for 3 h with continuous air supply. The suspensions were then filtered, washed and dried to give Ag deposited TiO₂ catalysts.

2.3. Analytical methods

A Varian Cary 5E UV–VIS–NIR spectrophotometer equipped with an integrated sphere was used to record the diffuse reflectance spectra (DRS) and to measure the absorbance data of the solution samples. The baseline correction was performed using a calibrated reference sample of barium sulfate. The reflectance spectra of the silver doped TiO₂ catalysts were analysed under ambient conditions in the wavelength range of 200–800 nm.

Powder X-ray diffraction patterns of TiO₂ and Ag doped TiO₂ catalysts were obtained using a Philips PANanalytical X'pert PRO diffractometer equipped with a Cu tube for generating a Cu K α radiation (wavelength 1.5406 Å) at 40 kV, 25 mA. The particles were spread on a glass slide specimen holder and the scattered intensity was measured between 20° and 85° at a scanning rate of 2 θ = 1.2° min⁻¹. Peak positions were compared with the standard files to identify the crystalline phases.

The electron paramagnetic resonance (EPR) spectra of the silver doped TiO₂ catalysts were recorded by using a JEOL-JE 100 ESR spectrometer at room temperature. The TiO₂ powder was placed in a thin-walled quartz EPR tube to produce cylindrical samples with identical dimensions. The EPR spectrometer settings were microwave power of 1.00 mW, microwave frequency of 9.39 GHz. Center field of 335 ± 100 mT, modulation amplitude of 0.2 mT and scan time of 0.03 s.

Scanning electron microscopic (SEM) analysis was performed on platinum coated samples using a JEOL apparatus model JSM-5610 LV, equipped with an INCA EDX probe for the energy dispersive X-ray micro analysis (EDX).

The specific surface areas of the samples were determined through nitrogen adsorption at 77 K on the basis of BET equation using a Sorptomatic 1990 instrument.

2.4. Photocatalytic studies

The photocatalytic experiments were carried out in a Heber multilamp photoreactor model HML-MP 88 (Fig. 2) which consists of eight medium pressure mercury vapour lamps (8 W) set in parallel and emitting 365 nm wavelength. It has a reaction chamber with specially designed reflectors made of highly polished aluminium and built in cooling fan at the bottom.

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