



Fabrication of Fe-doped TiO₂ nanoparticles and investigation of photocatalytic decolorization of reactive red 198 under visible light irradiation



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ABSTRACT

In this research, Fe-doped TiO₂ nanoparticles with various Fe concentrations (0.01, 1, 5 and 10 wt%) were prepared by a sol-gel method. Then, nanoparticles were characterized by X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), energy-dispersive X-ray analysis (EDX), BET surface area, photoluminescence (PL) spectroscopy and UV-vis diffuse reflectance spectroscopy (DRS). The photocatalytic activity of the nano-particles was evaluated through degradation of reactive red 198 (RR 198) under UV and visible light irradiations. XRD results revealed that all samples contained only anatase phase. DRS showed that the Fe doping in the titania induced a significant red shift of the absorption edge and then the band gap energy decreased from 3 to 2.1 eV. Photocatalytic results indicated that TiO₂ had a highest photocatalytic decolorization of the RR 198 under UV irradiation whereas photocatalytic decolorization of the RR 198 under visible irradiation increased in the presence of Fe-doped TiO₂ nanoparticles. Among the samples, Fe-1 wt% doped TiO₂ nanoparticles showed the highest photocatalytic decolorization of RR198 under visible light irradiation.

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1. Introduction

In recent years, growing worries about increasing water contamination owing to the quick growth of various industries and the great content of wastewater made [1]. Textile industry is the major consumers of water and consequently causing broadly water pollution. The wastewater produced contains organic matter, products derived from fixing agents, detergents, reactive dyes and salts, therefore must be treated before being discharged or reused [1–2]. Large amounts of reactive dyes that are generated in dyeing and finishing processes, are one of the most important environmental problems. It is well known that some of the dyes are not poisonous but after propagation within the aquatic environment their degradation products are frequently carcinogenic. Therefore, they can cause critical damage to aquatic life by increasing chemical oxygen demand, toxicity and as well barrier photosynthetic phenomena through decline of light penetration [3–5].

Several physical, chemical and biological methods such as coagulation-flocculation, chemical degradation, adsorption, precipitation, membrane filtration, electrochemical techniques, ozonation

and biosorption have been employed for the removal of dyes [3,6–7].

These existing technologies for decolorization have some drawbacks including large volume sludge [8]. Moreover, these methods do not result in dye degradation but merely transform dyes in solution into solid forms [9–10]. Advanced oxidation process, such as photocatalytic process, is one of the most important techniques that generally involve the generation of the very powerful oxidizing agent, hydroxyl radical (OH[•]) which completely destroy wide ranges of organic pollutants from industrial wastewater [2,10].

Nano-particles material are used frequently in photocatalytic process. Their properties such as large surface area, high number of reactive atoms and large number of vacant reactive surface sites make them one of the incomparable photocatalysts [11–12]. Among nano-particles material, most investigations have utilized TiO₂ as a model photocatalyst due to its chemical and biological stability, availability, low cost, non-toxicity and high efficiency in environmental treatment [10,13]. However, TiO₂ has a high band gap energy (3–3.2 eV) and can only absorb UV region of the solar light. For efficient photocatalytic activity, it is necessary to extend the photoresponse of the TiO₂ from the UV to the visible region by modification of its optical properties. Extensive studies have been

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conducted to convert the absorption edge of the TiO₂ from the UV to the visible light region by transition metals doping [14].

The most popular transition metals dopants used for modification of the TiO₂ optical properties are Cr, Fe, Ni, V, Mn, and Cu [15–17]. In the literature, many researchers have been demonstrated that Fe doping enhanced the photocatalytic activity of the TiO₂ [18–23]. Based on our knowledge there are not a reports on the photocatalytic decolorization of reactive red 198 under visible light irradiation in the presence of Fe-doped TiO₂. In the present work, TiO₂ nanoparticles doped with different concentrations of Fe were produced by a sol–gel method. Then, the photocatalytic activity of the samples under visible light irradiation was investigated.

2. Experimental

2.1. Preparation of the Fe-doped TiO₂ nano-particles

Ultrasound assisted synthesis of Fe doped TiO₂ was carried out as follows. Firstly, to prepare TiO₂ sol, 5 mL Tetrabutyl titanate [Ti(OC₄H₉)₄, 98%] was mixed with 50 mL absolute ethanol (46.07 M) and 0.33 mL H₂SO₄ (98%), followed by the addition of 250 mL deionized water and continuously stirred. Then, different amounts of Iron (III) Chloride 6-hydrate powder under stirring for 30 min dissolved in the TiO₂ sol and sonicated for 10 min (Ultrasonic processor, SONOREX SUPER RK 106, 35 kHz). After sonicated, the mixture was kept in a water bath at 80 °C for 2 h. Finally, the mixture was cooled to room temperature and washed with deionized water. Samples were dried at 100 °C, and calcinated at 600 °C for 1 h. The Fe concentrations in the samples were 0, 0.1, 1, 5 and 10 wt%. The undoped TiO₂ was synthesized by the same procedure without the addition of Fe.

2.2. Characterization of samples

The structure of the nano-particles was determined using a Bruker X-ray diffractometer (XRD, D8ADVANCE, Germany, Ni-filter, Cu K_α radiation λ = 1.5406 Å). The surface morphologies of the nano-particles were measured by field emission scanning electron microscopy (FE-SEM, Hitachi S4160, Cold Field Emission, voltage 20 KV). Energy-dispersive X-ray analysis (EDX, voltage 20 KV, Takeoff Angle 35.0°) was also used for the chemical analysis of the nano-particles. The specific surface area of the nano-composite was evaluated using the BET method. The BET surface area was determined by nitrogen adsorption–desorption isotherm measurement at 77 K. The sample was degassed at 150 °C prior to actual measurement.

A UV–vis spectrophotometer (Shimadzu-UV525) was used to record the diffuse reflectance spectra of the TiO₂ and Fe doped TiO₂ nano-particles. BaSO₄ was used as a reflectance standard in the UV–vis diffuse reflectance experiment. Excitation and emission spectra of the samples were recorded by photoluminescence spectrophotometer (Avantes, Avaspec-2048-TEC) using a Xenon lamp as excitation source operating at 240 nm.

2.3. Photocatalytic activity

The photocatalytic activities of the nano-particles under irradiation were evaluated by the decolorization rate of the reactive red 198 (RR 198, Dystar). In a typical experiment, 1 g/L catalyst (Fe-doped TiO₂ or pure TiO₂) was added to 100 mg/L RR 198 solution under stirring. Then, the solution was irradiated with UV (2 lamp, each power 15 W, wavelength 254 nm) and visible light (250 W mercury lamp) under magnetic stirring, separately. Before lamp turning, the dispersion was stirred in a dark place for 1 h. During photoreaction, solution samples were taken for analysis

at regular intervals. The light absorbance of the RR 198 solution was measured using a UV–vis spectrophotometer at 518 nm, which is the maximum absorption of the RR 198 [24]. Then, the decolorization rate of the RR 198 was used to quantify the photocatalytic activities of the catalyst, and calculated using following equation [24]:

$$\eta = \frac{A_0 - A}{A_0} \times 100 \quad (1)$$

where A_0 is the light absorbance of the RR 198 before the irradiation and A is the light absorbance of the RR 198 after the irradiation.

3. Results and discussion

Fig. 1 shows the XRD patterns of the TiO₂ and Fe-doped TiO₂ nano-particles. The characteristic diffraction peaks of anatase at 25.4, 38, 48, 54.7, 63.1 correspond to the reflections from the (101), (112), (200), (105), (204), (220) and (215) planes were observed and no rutile peaks were detected. Therefore, the XRD measurements showed that both TiO₂ and Fe doped TiO₂ nano-particles contain only anatase phase [24]. No other impurity phase attributed to iron oxide can be detected in the XRD patterns because the radii of Ti⁴⁺ and Fe³⁺ ions are very similar, so all Fe³⁺ ions might substitute Ti⁴⁺ ions and incorporated into the structures of the TiO₂ [20,25]. It is well accepted that the intensity of the diffraction peaks reveals the crystallization of the samples. As shown in Fig. 1, the intensity of the Fe-doped TiO₂ peaks decreased in comparison with the undoped TiO₂ peak. It is indicated that the addition of Fe³⁺ ions decreased the crystallinity of the TiO₂ nanoparticles [18,26].

The crystallite sizes were estimated from the Scherrer equation on the anatase diffraction peaks:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (2)$$

where D is the crystal size, $\lambda = 1.5406$ Å, the wavelength of the Cu K_α source, β is the full width at half maximum (FWHM) of the diffraction peak, K is a constant (0.94) and θ is the diffraction angle at the peak maximum. The lattice parameters were determined using following equations:

$$d_{(hkl)} = \frac{\lambda}{2 \sin \theta} \quad (3)$$

$$\frac{1}{d_{(hkl)}^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \quad (4)$$

where $d_{(hkl)}$ is the distance between crystal planes of (hkl) , λ is the X-ray wavelength, θ is the diffraction angle of crystal plane (hkl) , hkl is the crystal plane index, and a , b , and c are lattice parameters (in anatase phase of TiO₂, $a = b \neq c$) [27].

The crystallite sizes and lattice parameters of the Fe-doped TiO₂ nanoparticles were calculated and indicated in Table 1. According to the Table 1, it can be seen that the crystallite sizes of the Fe-doped TiO₂ decreased with increasing of Fe concentration. Yu et al. [28] indicated that the addition of additives to TiO₂ had a suppressive effect on the crystal growth of the TiO₂, since the additives hindered contact between TiO₂ particles and inhibited crystal growth during the heat treatment.

Although the ionic radius of the Fe³⁺ ion is close to Ti⁴⁺, a lattice distortion in the TiO₂ is generated by the substitution of Fe³⁺ in Ti⁴⁺. As a result of crystal lattice distortion, the crystallite growth of the Fe-doped TiO₂ nano-particle during the heat treatment is restrained [25,28–29].

The a and c lattice constant values estimated for TiO₂ are 1.9 Å and 4.9 Å, respectively, whereas those estimated for Fe-doped TiO₂

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