

Surface characteristics and photocatalytic activity of TiO₂ loaded on activated carbon fibers

Rusheng Yuan^{a,b}, Jingtang Zheng^c, Rongbo Guan^{c,*}, Yucui Zhao^c

^a Institute of Coal Chemistry, Chinese Academy of Sciences, Taiyuan 030001, PR China

^b Graduate School of the Chinese Academy of Sciences, Beijing 100039, PR China

^c The State Key Laboratory of Heavy Oil Processing, University of Petroleum, Dongying, Shandong 257061, PR China

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Abstract

TiO₂ loaded on activated carbon fibers (ACF) was performed by using epoxy resin as the precursor of the link between them, followed by calcination at different temperatures in N₂ atmosphere, and detailed surface characteristics and photocatalytic activity for methylene blue have been carried out. The pore structure of ACF was preserved well after loading with TiO₂ particles. From SEM and TEM images, it was observed that TiO₂ loaded on ACF was in the form of small clusters, and the primary particles still kept in nanometer size. Upon calcination at different temperatures, the residue of epoxy resin on the surface of TiO₂ resulted in the changes in UV–vis and IR spectra, which reflected in the differences of photocatalytic activity. The sample calcined at 460 °C exhibited the best photocatalytic activity, and still maintained high activity without any decrease when used repeatedly. Even at the sixth cycle, the amount of MB removal for TiO₂/ACF composite was still slightly higher than that for fresh P25 in suspension.

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1. Introduction

The heterogeneous photocatalysis has been proposed as an advanced oxidation process for the mineralization of various environmental organic pollutants in gas and liquid phases [1–5]. TiO₂ is the most widely used photocatalyst due to its good activity, chemical stability, commercial availability and inexpensiveness. However, in field applications, there are at least two obvious problems arising from using fine TiO₂ powders: (1) separation of photocatalyst from the reaction media is difficult and (2) particulate suspensions are not easily applicable to continuous process. An alternative method is immobilizing TiO₂ powders onto an inert and porous supporting matrix such as silica [6], alumina [7,8], zeolites [9],

and activated carbon (AC) [10–17]. Among them, AC is most commonly used support for TiO₂ in gas and water treatment due to its porous structure and adsorption properties, and the supported catalyst has made remarkable effects in the kinetics of disappearance of the pollutants, each pollutant being more rapidly photodegraded. This effect has been explained that by the adsorption of organics on AC followed by a transfer to the TiO₂ surface where it is immediately degraded [10,14]. But, AC is usually in the form of granules and powder, and the problem of filtering and recovery still exists.

Activated carbon fiber (ACF), a new formulation of activated carbon, is in the form of felt or cloth, with high BET surface area and uniform pore structure [18]. It is preferable in recovery catalyst from water than granular supports. But there are only a few reports about the TiO₂ loaded on ACF and its photodegradation behavior for organic pollutants in

* Corresponding author. Tel.: +86 546 8395024; fax: +86 546 8395190.
E-mail address: guanrbo@yahoo.com.cn (R. Guan).

water. Therefore, in the current work, ACF was selected as adsorptive support for TiO₂ loading.

The TiO₂ loaded ACF (TiO₂/ACF) was performed by using epoxy resin as the precursor of the link between TiO₂ and ACF, followed by calcination at different temperatures in inert atmosphere. The photocatalytic activity of TiO₂ powder strongly depends on its microstructural and physical properties [19]. In this sense, we studied the surface characteristics and photocatalytic activities of TiO₂/ACF samples calcined at different temperatures, and methylene blue (MB) was chosen as the model pollutant.

2. Experimental

2.1. Materials preparation

Titanium dioxide used in this study was Degussa P25, mainly anatase, with a BET surface of 50 m²/g and an average particle size of 30 nm. Activated carbon fibers were produced from rayon precursor, in the form of felt, having a BET surface area of 749 m²/g and a total pore volume of 0.25 cm³/g. They were washed with deionized water and dried at 105 °C before use. Diglycidyl ether of bisphenol A (DGEBA) epoxy resin and methylene blue (C₁₆H₁₈ClN₃S) were reagent grade and purchased from Shanghai Reagent Corporation.

The TiO₂/ACF samples were prepared as follows. A 5 g TiO₂ was added to 120 ml acetone solution containing 1.1820 g epoxy resin, and the resulting mixed suspension was stirred vigorously with high-shear-dispersion machine allowing the TiO₂ particles to remain suspended in the solution. The mixed suspension was added drop by drop on a 6.4 g ACF felt, then, it was dried at room temperature. Finally, it was calcined at 400, 460, 500 and 580 °C for 2 h in N₂ atmosphere, respectively. The sample dried at room temperature was denoted as T₀, and the others were named as T₄₀₀, T₄₆₀, T₅₀₀ and T₅₈₀, according to the heat treatment temperature, respectively.

2.2. Characterization

The BET surface area and pore volume of composite TiO₂/ACF were obtained by N₂ adsorption at 77 K using a micromeritics ASAP 2000. The pore size distribution was calculated by density functional theory (DFT) method. TG and DTA were carried out in a Netzsch STA409PC thermoanalyzer. The samples were heated in N₂ flow from 25 to 700 °C at a rate of 10 °C/min. X-ray diffraction patterns (XRD) were performed on Rigaku Dmax-2000 diffractometer with Cu Kα irradiation of wavelength 0.154 nm in the range 10–70° (2θ) with a scan rate of 2 °/min. The morphology of small TiO₂ particles on ACF was examined by scanning electron microscope (SEM, Hitachi S-4500 model) and transmission electron microscope (TEM, Hitachi-800). UV–vis spectra were recorded on Shimadzu UV-2101PC instrument in the range 200–600 nm at room temperature in air, using BaSO₄ as ref-

erence substance. When TiO₂/ACF samples were calcined at different temperatures, the change of TiO₂ surface groups was characterized by diffuse reflection infrared Fourier transformed (DRIFT) spectroscopic analysis on a Nicolet FTIR Spectrometer Magna-760.

2.3. Measurement of photocatalytic activity

The photoreaction was conducted in a 350 mL cylindrical vessel with a water-cooled quartz jacket. Irradiation was provided by a 500 W high-pressure mercury lamp with a major emission at 365 nm, located in the centre of quartz jacket. A magnetic stirrer was equipped at the bottom of the reactor to achieve effective dispersion. Air was bubbled through the reaction solution from the bottom to ensure a constant dissolved O₂ concentration. To compare the photocatalytic activity of TiO₂/ACF, the pure TiO₂ powders were also tested. The amount of TiO₂ powders was chosen as 1 g/L, which was adequate in our conditions without disturbing the UV light entering the reactor. The initial MB concentration was about 85 mg/L and the temperature of reaction solution maintained at 30 ± 0.5 °C. The TiO₂/ACF sample was used repeatedly, and each cycle lasted 3 h. Before the beginning of next cycle, the remaining solution was replaced by fresh MB solution with 85 mg/L. The 4 mL samples were withdrawn at different intervals of time to determine the residual concentration of MB by using a spectrophotometer (UV762, Shanghai Analysis Company) at 664 nm.

3. Results and discussion

3.1. Pore size distributions

The pore size distributions for original ACF and TiO₂/ACF samples that were calcined at different temperatures are shown in Fig. 1. All TiO₂/ACF samples presented a sharp pore distribution in the range of 0.6–0.7 nm, and a small amount of macropores appeared due to the introduction

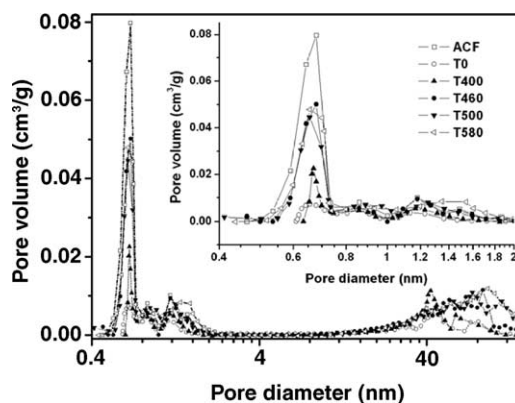


Fig. 1. Pore size distributions of original ACF and TiO₂/ACF samples. The inset shows the enlarged pattern from 0.4 to 2 nm for all samples.

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