

Photocatalytic degradation of methylene blue by a combination of TiO₂ and activated carbon fibers

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Abstract

Photocatalytic degradation of methylene blue (MB) in aqueous solution was investigated using TiO₂ immobilized on activated carbon fibers (ACFs). The TiO₂ and ACF combination (TiO₂/ACF) was prepared by using epoxy as the precursor of the link between TiO₂ and ACFs, followed by calcination at 460 °C in a N₂ atmosphere. The TiO₂/ACF composite prepared was easier to handle than the original TiO₂ powder in suspension. More significantly, the TiO₂/ACF composite can be used repeatedly without a decline in photodegradation ability. After six cycles, the amount of MB removal for the TiO₂/ACF composite was still slightly higher than that for fresh P25 TiO₂ in suspension. Through measurement of chemical oxygen demand in the solution and the concentration of ammonium generated during degradation of MB, it was confirmed that MB molecules are mineralized instead of adsorbed by ACFs.

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1. Introduction

Colored wastewater is released in textile effluents and poses a potential environmental hazard. Within the ecosystem, this colored wastewater is a dramatic source of pollution, eutrophication, and perturbations in aquatic life. Moreover, a variety of organic chemicals are produced during the dyeing process, and some have been shown to be carcinogenic [1]. With the growing awareness of decreasing available water resources, many methods, including physical, chemical, and biological methods, are being used in wastewater treatment and recycling. Among them, heterogeneous photocatalysis appears to be an emerging destructive technology leading to the total mineralization of most organic pollutants [2–7]. TiO₂ is the most widely used photocatalyst because of its good activity, chemical stability, commercial availability, and inexpensiveness. However, because the fine

TiO₂ which represents high photoactivity and a significant quantum effect is commonly in nanometer size, the problem of separation and recovery of photocatalyst from the reaction medium exists. An alternative method is immobilization of TiO₂ powder on an inert and suitable supporting matrix [8–14].

Activated carbon (AC) is widely used as a support in gas and water remediation because of its good adsorption, and supported TiO₂ exhibits a synergism that has marked effects on the kinetics of disappearance of pollutants, each pollutant being more rapidly photodegraded [15–18]. However, AC is commonly used in the forms of granules and powder, and there remain some difficulties in filtering and recovery from water. Activated carbon fibers (ACFs), a new formulation of activated carbon, have received increasing attention in recent years as an adsorbent. Compared with AC, ACFs are produced in the form of felt or cloth, with high BET surface area and micropore volume [19]. But, TiO₂ loading on ACFs and photodegradation behavior toward organic pollutants in water have seldom been reported.

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In the current work, ACFs were selected as an adsorptive support for TiO₂ loading. The combination was performed using acetone solution containing TiO₂ and epoxy to impregnate ACFs, followed by heat treatment at suitable temperature in an inert atmosphere. The epoxy was introduced as the precursor of the link between TiO₂ and ACFs. Methylene blue (MB) was chosen as the model pollutant to determine the photocatalytic efficiency of the combination.

2. Experimental

2.1. Materials preparation

The titanium dioxide used in this study is Degussa P25, mainly anatase, with a BET surface area of 50 m²/g and an average particle size of 30 nm. ACFs 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. The fibers were washed with deionized water and dried at 105 °C before use. Diglycidyl ether of bisphenol A (DGEBA) epoxy resins and methylene blue (C₁₆H₁₈ClN₃S) were reagent grade and purchased from Shanghai Reagent Corporation.

The combination of TiO₂ and ACFs, denoted TiO₂/ACF, was prepared as follows. Five grams of TiO₂ was added to 120 mL acetone solution containing 1.1820 g epoxy, and the resulting mixed suspension was stirred vigorously with a high-shear-dispersion machine, allowing the TiO₂ particles to remain suspended in the solution. The mixed suspension was added drop by drop onto a 6.4-g ACF felt; then, it was dried at room temperature and, finally, calcined at 460 °C for 2 h in a N₂ atmosphere.

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 the density functional theory (DFT) method. The morphology of small TiO₂ particles on ACFs was determined by scanning electron microscopy (SEM, Hitachi S-4500) and transmission electron microscopy (TEM, Hitachi-800). After UV irradiation, the variation of the TiO₂ surface was characterized by diffuse reflection infrared Fourier transform (DRIFT) spectroscopic analysis on a Nicolet FTIR spectrometer. Chemical oxygen demand (COD) was measured with a CM-02 COD analyzer (Beijing Shuanghui Corp.) based on the method of acidic oxidation by dichromate. During photoreaction, ammonium was formed and the amount was determined using a UV–Vis spectrophotometer according to the standard method (China, GB7479-87).

2.3. Measurement of photocatalytic efficiency

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 major emission at 365 nm, located in the center of the 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 assess the photocatalytic activity of TiO₂/ACF, pure TiO₂ powder was also tested. The amount of TiO₂ powder chosen was 1 g/L, which was adequate under our conditions without disturbing the UV light entering the reactor. The initial MB concentration was 85 mg/L and the temperature of the reaction solution was maintained at 30 ± 0.5 °C. The TiO₂/ACF sample was used repeatedly, and each cycle lasted 3 h. Before the beginning of the next cycle, the remaining solution was replaced with 85 mg/L fresh MB solution, and 4-ml samples were withdrawn at different intervals. The residual concentration of MB was measured at 664 nm using a spectrophotometer (UV762, Shanghai Analysis Co.).

3. Results and discussion

3.1. Pore size distribution and morphology of TiO₂/ACF

Nitrogen adsorption isotherms and pore size distributions for ACF and TiO₂/ACF are shown in Fig. 1. The surface area of ACF is 748.9 m²/g, mainly in the micropore range. For the TiO₂/ACF sample, surface area decreased to 504.3 m²/g. The presence of a residue of epoxy resin was responsible for the decline in surface area. From pore size distribution curves, it can be seen that there was little change in the micropore size distribution of TiO₂/ACF sample other than introduction of a small portion of macropores from TiO₂ in comparison with ACF. This result seems to indicate that the pore structure of ACF remained the same after loading with TiO₂, and most of the TiO₂ coated the surface of the ACF.

The morphology of TiO₂ deposited on ACFs was observed by SEM (Fig. 2). It is clear that the particles of TiO₂ aggregated into clusters and were fixed on the surface of ACFs by epoxy residue. Furthermore, the TEM images in

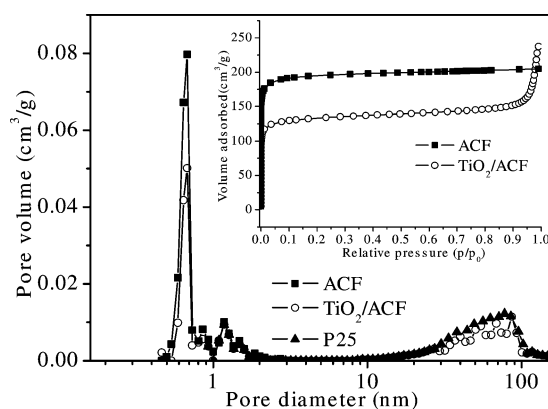


Fig. 1. Pore size distributions of original ACF, TiO₂/ACF, and P25. Inset: N₂ adsorption isotherms of original ACF and TiO₂/ACF.

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