



Short communication

Effect of toluene, an immiscible pollutant, on the photocatalytic degradation of azo dye



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ABSTRACT

An adverse effect of a water-immiscible pollutant, toluene, on the photocatalytic degradation of Direct Red 80 (DR80) with TiO₂ in an aqueous solution was observed when toluene was added as a continuous free layer. However, a more efficient photocatalytic system was obtained when toluene was added in the Pickering emulsion (PE) form with TiO₂. With only UV and two different types of TiO₂ (more hydrophilic and large-sized type A TiO₂ and less hydrophilic and small-sized type B TiO₂), 44% and 59% color removal was observed, respectively. Almost 20% decrease in color removal with toluene in the continuous free layer form was observed, however, the higher color removal was obtained when toluene was added as PE stabilized with type B TiO₂ than with type A TiO₂. The higher color removal was owing to the good stability of PE stabilized with type B TiO₂.

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Introduction

The wastewater generated during the textile dyeing process contains a combination of various pollutants, including dyes. Azo dyes in particular, with the general formula R₁–N=N–R₂, are one of such toxic pollutants and they should be degraded as part of an efficient wastewater treatment process [1–4]. The use of azo dyes is, however, not limited to the textile industry only; azo dyes find application in the paper, foodstuff and cosmetics industries as well [2]. The dye-containing effluent from these industries may not necessarily be toxic but is highly colored. Along with azo dyes, water-immiscible pollutants such as toluene can also be found in the wastewater originating from various industries and academic research laboratories [5,6]. These immiscible pollutants float on the surface of the wastewater and may hinder the degradation of azo dyes present therein. Previous studies have shown that conventional biological and physiochemical treatments of dye-containing effluents, from both industry and academia, were ineffective because of the high stability of modern dyes [7].

Although anodic and photocatalytic oxidation processes for an efficient degradation of Direct Red 80 (DR80) have been studied previously [8,9], no studies were found in the literature regarding the impact of water immiscible pollutants on dye degradation.

In view of the reported failure of biological and physiochemical treatment methods for dye degradation, photocatalytic degradation of DR80 using nano-TiO₂ and UV radiation ($\lambda = 365$ nm) was attempted in this study. In photocatalysis, transition-metal oxide or semiconductor particles are irradiated with UV radiation and electrons move from the valence band (VB) to the conduction band (CB), thereby generating positive holes in the VB of the catalyst. The photo-generated holes then oxidize the dye either directly or *via in situ* generated hydroxyl radicals (OH^{*}) [10,11]. To investigate the effect of water-immiscible pollutants on this process, toluene was added to DR80 aqueous solution, either as a single continuous layer or as a Pickering emulsion (PE) by stabilizing with two different types of TiO₂. Two different types of TiO₂ were selected mainly due to the different properties such as size and wettability of the particles. These properties of the particles have enormous impact on the stability of PE [12]. The main focus of the current study is to highlight the fact that water-immiscible pollutants can affect dye degradation and to make such pollutants an important parameter for wastewater treatment studies.

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Materials and methods

DR80 was selected as a representative water-soluble azo dye, which is used for dyeing silk, wool, cellulose, and cotton. The chemical structure of DR80, which contains four azo groups, is shown in Fig. 1.

DR80 was purchased from Sigma-Aldrich (South Korea) and used as supplied. It was diluted to 50 mg/L aqueous solutions. The two different types of TiO₂ (TiO₂ 232033 and 637254, which were termed as “type A TiO₂” and “type B TiO₂”, respectively) were also purchased from Sigma-Aldrich (South Korea), and 0.05 g/L of each type of TiO₂ (anatase) were used in almost all experiments. The average particle size (Fig. 2b) of TiO₂ (type A: 72 nm; type B: 23 nm) was measured using the N5 Submicron Particle Size Analyzer (Beckman Coulter, USA). BET surface areas of 7.88 m²/g and 44.80 m²/g (obtained by the adsorption-desorption method) and pore sizes of 0.04 cm³/g and 0.33 cm³/g (obtained with the BJH desorption method) for type A TiO₂ and type B TiO₂, respectively, were measured with Autosorb-iQ & Quadrasorb SI (Quantachrome, USA). GR grade Toluene; purity 99.7%, was purchased from Daejung Chemicals (South Korea), used as supplied, and 5 vol% with respect to the aqueous DR80 solution was added either in a continuous layer form or as a PE with TiO₂.

The PE of toluene, stabilized with TiO₂, in the aqueous dye solution was prepared using the VCX-750 High Intensity Ultrasonic Processor (Sonics & Materials, Inc., USA). In the case of the continuous layer form, TiO₂ was only dispersed along with toluene in the aqueous dye solution. The X-ray diffraction (XRD) patterns were obtained by an X'pert APD X-ray Diffractometer (Philips, the Netherlands) using Cu K α radiation to determine the crystallite identity of the TiO₂ powders. Scanning electron microscopy (SEM) images were recorded with Hitachi S-4800 FE-SEM (Hitachi, Japan) and were used to determine the morphology of TiO₂ powders. A Pyrex glass cylindrical photoreactor with 300 ml working volume,

surrounded by a circulating water jacket and equipped with a centrally mounted 125 W medium-pressure mercury lamp and a magnetic stirrer bar, was used in all experiments. The experiments were performed with different combinations of TiO₂ and modes of addition of toluene, and sampling for spectrophotometric analysis was done initially at 15 min intervals and then after every 30 min for a total period of 2 h. After collection, the samples were centrifuged immediately. The chemical oxygen demand (COD) was measured with Hach kits and spectrophotometer. The images of the emulsions were taken using a digital camera (Nikon Coolpix-3700) and an optical microscope (JSB-131, Sam Won Scientific Ind. Co.). UV–vis spectral analysis was conducted with an Agilent-8453 spectrophotometer (Agilent, USA) to investigate dye decolorization.

Results and discussions

The XRD pattern of TiO₂, shown in Fig. 2(a), with principal peaks at $2\theta = 25^\circ, 38^\circ, 48^\circ, 54^\circ, 56^\circ, 62.5^\circ, 68^\circ, 70^\circ,$ and 76° confirmed that both types of TiO₂ were in the pure anatase phase, and no characteristic peak of rutile phase was found. The SEM images (Fig. 3) show the differences in the sizes of type A TiO₂ and type B TiO₂; however, the shape of the particles was found to be almost similar in both types of TiO₂.

To investigate the effect of toluene on the photocatalytic degradation of DR80, the UV–vis data for different combinations of an aqueous solution of DR80 + toluene (as the continuous layer form and the PE form) + TiO₂ (type A and type B) are shown in Figs. 4 and 5. The aqueous mixtures were irradiated with a 125 W medium-pressure mercury lamp ($\lambda = 365$ nm) in a Pyrex glass photocatalytic reactor for 2 h.

Initially, the highest color removal, 75% in 2 h, was observed using only type A TiO₂ without centrifugation. However, a test experiment showed that the hydrophilicity of type A TiO₂ greatly

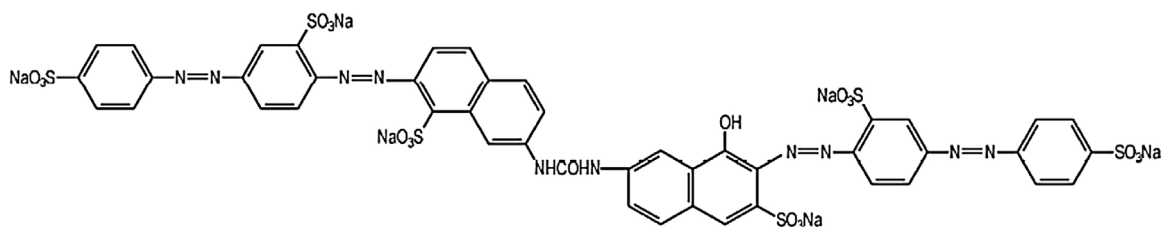


Fig. 1. Molecular structure of DR80 (sulfonic acid groups = 6, azo bonds = 4, aromatic rings = 8, benzene rings = 4, naphthalene rings = 2).

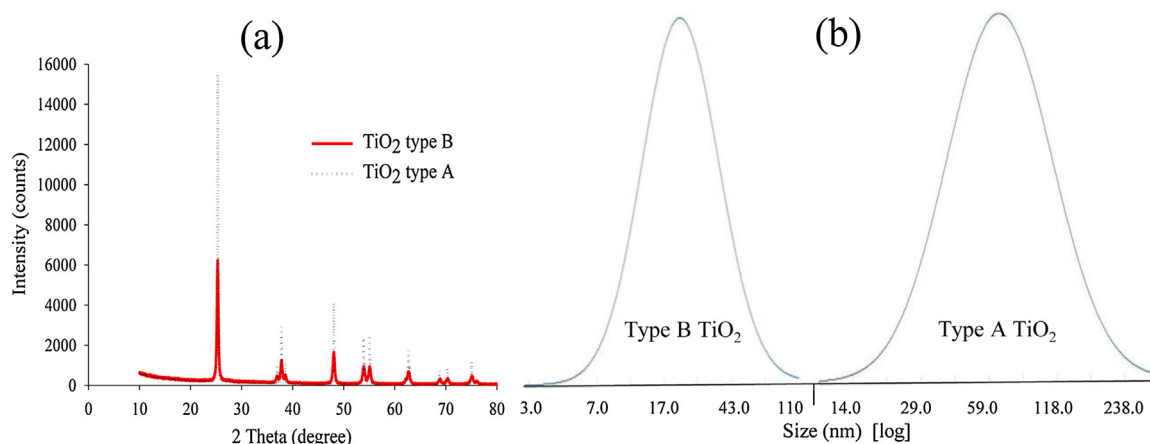


Fig. 2. XRD pattern (a) and average size of TiO₂ (type B and type A) particles (b).

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