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An innovative solar photoactive system N-WO₃@polyester fabric for degradation of amaranth in a thin-film fixed-bed reactor



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ABSTRACT

A new, highly photoactive system consisting of N-WO₃ coated on polyester fabric is prepared. The catalyst coating is done through a simple heat attachment method. The photocatalytic activity of the photoactive system (N-WO₃@PF) is evaluated for degradation of a recalcitrant dye, amaranth, in a recirculation thin-film fixed-bed reactor (TFFBR) under solar light, as well as under ultraviolet A and visible light for comparison. Analyses by X-ray diffraction, Fourier transform infrared spectroscopy, and scanning electron microscopy confirm the presence of N-WO₃ evenly coated on the fabric surface. Based on diffuse reflectance spectroscopy analysis, N-WO₃@PF shows remarkable absorbance in the visible and near-infrared region. The photocatalytic degradation of amaranth under solar light increases with increasing catalyst loading and decreases with increasing flow rate, reactor inclination, and salinity. The inhibition effect of inorganic ions is in the order of Ca²⁺ > CO₃²⁻ > SO₄²⁻ > NO₃⁻. At chosen experimental conditions (irradiation time of 4 h, dye concentration of 10 mg/L, catalyst loading of 16.4 g/m², pH of 7, flow rate of 80 mL/min, and inclination angle of 20°), dye degradation efficiencies under solar, ultraviolet A, and visible light are 98.2, 63.5, and 56.7%, respectively. After 12 runs under identical experimental conditions, dye degradation efficiencies are reduced to 86.9 (solar), 56.2 (ultraviolet A), and 41.3% (visible). On the basis of figure-of-merit recommended by International Union of Pure and Applied Chemistry (A_{CO} of 2.9 m²/(m³ order)), N-WO₃@PF shows a better performance compared to some existing photoactive systems. Overall, results show that the photoactive system N-WO₃@PF can be successfully used for the degradation of amaranth in a TFFBR under solar light and could be a suitable option for environmental photocatalysis.

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1. Introduction

Dyes are extensively used in textile industries. During the dyeing process, a large quantity of dyes with intensive color and toxicity can be released into the water bodies due to incomplete dye fixation [1] and improper treatment of effluents. This leads to serious environmental problems. Coagulation, filtration, and adsorption with activated carbon are effective methods for dye removal [2], but they merely transfer the dyes from the liquid to the solid phases and thus generate secondary pollutants. Biological treatment is a cost-effective method, but the majority of dyes are resistant to biological processes [3]. Therefore, finding destructive yet cost-effective methods for the treatment of dyes is of important concern.

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Heterogeneous photocatalysis with inorganic semiconductors such as ZnO and TiO₂ offers clean, solar driven process for degradation of recalcitrant compounds including dyes. Several studies have been reported on photocatalytic degradation of dyes in slurry systems involving suspensions of catalyst particles [4,5]. However, from a practical viewpoint, recovering the used catalysts, especially those in the nanoform, is a tricky task as it requires considerably long settling times for sedimentation and can be expensive for filtration. Light scattering by catalyst particles is also another challenge.

For overcoming such issues, immobilization of the catalyst on a stationary, solid support can be a suitable option. A variety of supporting materials such as alumina and glass beads [6], glass plate [3], pebbles [7], Cuddapah stones [8], and activated carbon fiber [9] have been investigated. Textile fabrics made from cotton [10], flax [11], nylon [12], and polyester [13], have also been explored as catalyst support due to their flexibility, relative stability, and low-cost. Among different types of fabrics, polyester

fabrics appear suitable due to their strength, combined with their resistant to UV, chemicals, abrasion, stretching, shrinking, and wrinkling [14]. However, due to the intrinsic hydrophobic and inert nature of constituent polymer in polyester fabrics and lack of functional groups on the fabric surface, the stability of the coated catalyst particles is relatively low [15].

To increase hydrophilicity of fabric surface, pretreatment with alkali has been proposed [14,16,17]. The alkali treatment can alter the chemical, physical, and morphological properties of the fabric by increasing the amount of surface functional groups, improving the accessibility of functional groups on the newly exposed surfaces, and increasing roughness and porosity of the surface, respectively [14]. Due to increased hydrophilicity of the surface, more catalyst can be coated through bonding with the functional groups on the fabric surface. Fabric treatment with UVC light has also been reported to induce the formation of chelating agents to bind the catalyst particles on the surface [18,19]. As a result, high stability of the catalyst particles on the fabric surface can be obtained.

In this study, a photoactive system, employing highly active N-doped WO_3 photocatalyst coated on a polyester fabric ($\text{N-WO}_3\text{@PF}$), is prepared. The fabric is pretreated with NaOH solution followed by irradiation with UVC light to improve its hydrophilicity. The photocatalytic activity of $\text{N-WO}_3\text{@PF}$ is evaluated for the degradation of amaranth (AM) under solar light, as well as under visible and UVA light in some experiments, with a thin-film fixed-bed reactor (TFFBR) in recirculation mode. A TFFBR is used due to its high optical efficiency, simple construction, and low investment costs [20]. The effects of operating parameters on the process efficiency are investigated. For the purpose of comparison, the IUPAC figures-of-merit of the photoactive system $\text{N-WO}_3\text{@PF}$ are estimated and compared with those of some existing photoactive systems.

2. Experimental

2.1. Materials and chemicals

An undyed 100% polyester fabric, obtained from a local market, was used. All chemicals were of analytical grade. Tungstic acid (H_2WO_4 , Aldrich), hydrogen peroxide solution (H_2O_2 , 35 wt%, Aldrich), urea ($\text{CO}(\text{NH}_2)_2$, Aldrich), amaranth ($\text{C}_{20}\text{H}_{11}\text{N}_2\text{Na}_3\text{O}_{10}\text{S}_3$, Aldrich), sodium chloride (NaCl, Chameleon reagent), sodium nitrate (NaNO_3 , Aldrich), sodium carbonate (Na_2CO_3 , Chameleon reagent), sodium sulfate (Na_2SO_4 , Chameleon reagent), and calcium sulfate (CaSO_4 , Chameleon reagent) were used as received without further purification. DI water was used in all of the processes.

2.2. Synthesis of N-WO_3

N-WO_3 was synthesized by thermal decomposition of peroxotungstic acid–urea complex [21]. Peroxotungstic acid was obtained through the reaction of H_2WO_4 with H_2O_2 . About 3.75 g of H_2WO_4 was dispersed in 75 mL of water with continuous stirring, followed by addition of 25 mL of aqueous H_2O_2 . The reaction mixture was further stirred for 24 h at 40 °C. After drying, the obtained powder was dissolved in 75 mL of water with continuous stirring for 1 h at 75 °C. The solid product was subsequently ground with 1.8 g of urea in an agate mortar for 30 min. The ground mixture was calcined at 500 °C for 3 h in a muffle furnace to form crystalline N-WO_3 with orange-red color. The N-WO_3 was washed with water followed by ethanol, prior to drying at 100 °C for 24 h.

2.3. Preparation of $\text{N-WO}_3\text{@PF}$

To remove impurities, the fabric with a size of 20 cm × 20 cm was first scoured with detergent at 80 °C for 30 min and rinsed with a large amount of water. After being cleaned with acetone at room temperature for 30 min, it was immersed in 1 M of NaOH solution at 80 °C for 2 h, repeatedly rinsed with water, and dried at 80 °C for 24 h. Subsequently, the alkali-treated fabric was irradiated with UVC light at room temperature for 5 h, immersed in 16 g/L of N-WO_3 suspension at room temperature for 24 h under agitation on a shaking bath at 200 rpm, and dried at 80 °C for 24 h. Before immersion, the N-WO_3 suspension was sonicated in an ultrasonic bath for 2 h to de-agglomerate the N-WO_3 particles. Immersion-drying process was undertaken five times in succession to increase the total thickness. The coated fabric was further subjected to heating at 200 °C for 30 min. To remove weakly attached particles, it was rinsed with water followed by drying at 80 °C for 24 h. The catalyst loading on the PF surface is found to be 16.4 g/m². For comparison, photoactive $\text{N-WO}_3\text{@PF}$ systems with lower catalyst loadings were also prepared by the same method but using different concentrations of N-WO_3 suspension. The catalyst loading of 12.8 and 5.5 g/m² were obtained with 8 and 4 g/L of N-WO_3 suspension, respectively.

2.4. Characterization of $\text{N-WO}_3\text{@PF}$

The infrared spectra were recorded by a Fourier transform infrared (FTIR) spectrometer (Nicolet 6700) in attenuated total reflection (ATR) mode. The X-ray diffraction (XRD) patterns were collected on a Bruker D8 diffractometer (Cu K α radiation, $\lambda=1.5406$ Å). The morphological properties were observed by scanning electron microscopy (SEM) (S-3400N, Hitachi). The diffuse reflectance spectra (DRS) were obtained from a UV–vis–NIR spectrophotometer (Cary 5000, Agilent). The specific surface areas were determined based on the Brunauer–Emmett–Teller (BET) isotherm measured at liquid nitrogen temperature of 77 K by an adsorption instrument (ASAP 2020, Micromeritics).

2.5. Photocatalytic activity evaluation

A thin-film fixed-bed reactor, consisting of a covered reservoir, a peristaltic pump, and a sloping plate for collecting light (Fig. 1), was used. The operation regime during all experiments is continuous flow with recirculation. The $\text{N-WO}_3\text{@PF}$ was attached to the sloping plate having a workable area of 20 cm × 20 cm. The dye solution with a volume of 0.5 L was transferred into the reservoir. The pH of the dye solution was adjusted to a desired level using dilute NaOH and HCl. The dye solution falls as a thin film from the top of the plate, allowing effective oxygen transfers from the air into the water film. The system was further irradiated with solar light to initiate a photocatalytic reaction. The solar irradiance was measured regularly during the experiments at the same angle as that of the sloping plate and the average solar irradiance was found to be 577.2 W/m². For comparison, visible and UVA light were used in some experiments. A metal halide lamp 250 W (Philips) with an irradiance of 210.5 W/m² and a blacklight lamp 11 W (General Electric) with an irradiance of 53.1 W/m² were used as visible and UVA light sources, respectively. When visible or UVA light irradiation was applied, the reactor was put in a box chamber, in which the inner wall is covered with aluminum foil to reflect light. After a given irradiation time, the solution was withdrawn and centrifuged to measure its absorbance at λ_{max} of 521 nm using a UV–vis spectrophotometer (Genesys 10S, Thermo Fisher Scientific). To determine the dye concentration, calibration plots based on Beer–Lambert's law were established by relating the absorbance to the

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