



Highly reusable TiO₂ nanoparticle photocatalyst by direct immobilization on steel mesh via PVDF coating, electrospinning, and thermal fixation

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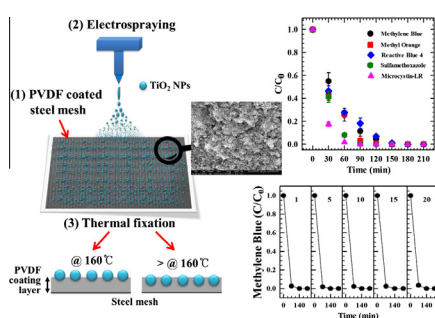
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HIGHLIGHTS

- TiO₂ nanoparticles electrospayed and thermally fixed on PVDF-coated steel mesh.
- Thermal fixation at 160 °C was optimal for photocatalytic activity.
- Immobilized photocatalyst effective for degrading organic pollutants in UV light.
- High photocatalytic efficacy was retained for 20 consecutive runs.

GRAPHICAL ABSTRACT



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ABSTRACT

We have developed a highly reusable photocatalyst based on TiO₂ nanoparticles for degrading organic pollutants in water. The particles were immobilized on steel mesh (SM) by a three-step procedure: (1) formation of poly(vinylidene fluoride) (PVDF) binder interface by dip-coating SM (2.5 cm × 5.0 cm), (2) electrospinning of TiO₂ nanoparticles dispersed in methanol (Degussa P25, 1 mg/mL), and (3) final thermal fixation with a pressure of 100 MPa for improved mechanical stability. When the electrospayed volumes were 10, 20, 30, 40, 50, and 60 mL, the TiO₂ loading on both sides of the PVDF-coated SM increased from 0.20, 0.43, 0.73, 0.97, 1.10, to 1.60 mg respectively. The SM sample loaded with 1.10 mg TiO₂ (SM-TiO₂) was found to be optimal for the photocatalytic oxidation of methylene blue (MB) under UV irradiation, with stable performance for 20 consecutive photocatalytic runs. The SM-TiO₂ thermally fixed at 160 °C exhibited higher photocatalytic efficacy than those fixed at 180 and 200 °C, because at higher temperatures the melted PVDF resin layer (melting point: 165–172 °C) entrapped the TiO₂ nanoparticles and rendered them photocatalytically inactive. The optimized SM-TiO₂ demonstrated good performance on diverse organic pollutants, namely MB, methyl orange, reactive blue 4, sulfamethoxazole, and microcystin-LR, with rate constants of 0.0251, 0.0368, 0.0164, 0.0568, and 0.0725 min⁻¹, respectively.

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1. Introduction

Heterogeneous photocatalytic processes using inorganic semiconductors are very effective for oxidizing a wide range of organic pollutants, including synthetic dyes, pharmaceuticals, and pesticides [1,2]. Titanium dioxide (TiO_2) is the most popular heterogeneous photocatalyst because of its high activity, chemical stability, and commercial availability [3–7]. Nevertheless, the difficulty in recovering and separating TiO_2 slurry from treated water and the growing concerns of particulate toxicity still limit its industrial feasibility [8–10]. As a result, the concept of immobilizing TiO_2 catalysts onto inert supports such as glass, metal, polymer films, and porous materials is being extensively explored for practical applications [11,12].

A number of techniques are available to immobilize TiO_2 nanoparticles (NPs) on various substrates, using proper coating methods that are essential for catalyst immobilization. The most common approach is a sol-gel process, which includes complicated steps such as drying, binder mixing, coating, and calcination. The final calcination at high temperatures (usually above $600\text{ }^\circ\text{C}$) often results in rapid increase of particle size due to sintering, accompanied by the loss of the particles' nanoscale properties, thereby causing reductions of the active surface area and pore volume. Moreover, the high-temperature calcination is not applicable to less heat-resistant support materials [11,13].

As a simpler and more versatile alternative, electrohydrodynamic atomization, also referred to as electrospaying, is a promising technique as it allows uniform deposition in an ambient atmosphere. This technique offers the advantages of low cost, high deposition efficiency under mild conditions, the capability to generate near-monodispersed distribution, and easy control of film thickness [7]. Electrospaying has recently been employed to fabricate nanostructured films from pre-synthesized NP-containing sols [14]. Since the electrospaying of nanosuspensions directly deposits NPs onto the substrate, it thus avoids additional steps such as calcination. Some researchers have prepared TiO_2 and ZnO films via electrospaying the corresponding NP dispersions [15,16]. In general, the efficacy and reusability of the immobilized catalysts are directly related to the bonding strength between the substrate material and catalyst. For metal substrates, the application of a thermoplastic binder together with heat treatment is necessary to obtain highly stable surface-immobilized catalysts [17,18].

Herein, we have successfully prepared TiO_2 nanoparticles immobilized on steel mesh (SM- TiO_2) via coating the mesh with poly(vinylidene fluoride) (PVDF) as a binder and electrospaying TiO_2 NP dispersion, followed by thermal fixation at a relatively low temperature ($160\text{ }^\circ\text{C}$). The efficacy of the resulting photocatalyst was evaluated by the degradation of methylene blue (MB) as a

model dye under UV irradiation. The synthesis parameters, such as substrate type, catalyst loading, and fixation temperature were optimized for the photocatalytic activity of SM- TiO_2 . Its applicability and reusability for the degradation of different dyes and micropollutants, i.e. sulfamethoxazole (SMX, an antibiotic contaminant), and microcystin-LR (MC-LR, a toxin from cyanobacteria), were also tested.

2. Materials and methods

2.1. Materials

SM (Fibertech, Korea) of $244.4\text{ }\mu\text{m}$ thickness, PVDF (Kynar 761, Arkema, USA), TiO_2 powder (P25, Degussa, Germany), *N,N*-dimethylacetamide (DMAc), methanol (MeOH), MB, methyl orange (MO), reactive blue 4 (RB4), SMX, and MC-LR were used in this study. All chemicals were of either reagent or analytical grade, and used as received.

2.2. Fabrication of SM- TiO_2

Fig. 1 illustrates the synthesis flow of immobilizing TiO_2 NPs on SM. First, the SM ($2.5\text{ cm} \times 5.0\text{ cm}$, total surface area = 25 cm^2) was cleaned by sonicating in a 0.3% solution of Micro-90 detergent (International Products Corp.) for 1 h, followed by washing with hot water and drying at $60\text{ }^\circ\text{C}$ under vacuum for 5 h. The PVDF solution was prepared by adding an appropriate quantity of PVDF powder into DMAc at $70\text{ }^\circ\text{C}$ and stirring for 24 h. To produce PVDF-coated SM (SM-PVDF), the cleaned SM was dipped into the PVDF solution for 5 min, then the excess PVDF solution was drained off for 30 min and the mesh was dried at $145\text{ }^\circ\text{C}$ under vacuum for 2 h. The SM-PVDF samples were coded as SM-PVDF-X/Y, where X = PVDF concentration (wt.%) and Y = number of coatings performed. A dispersion of TiO_2 NPs (1 mg/mL) in MeOH was electrospayed onto both sides of SM-PVDF that was placed on the counter electrode, at a voltage of 20 kV and a flow rate of 6 mL/h. The tip-to-collector distance was kept at 10 cm. The obtained samples were denoted as SM- TiO_2 -X, where X = the total volume (mL) of TiO_2 solution electrospayed. After electrospaying, the mesh was subjected to heat treatment at 140, 160, 180, or $200\text{ }^\circ\text{C}$ under 100 MPa applied pressure. Finally, the fixed SM- TiO_2 -X samples were stirred in deionized (DI) water overnight to remove loosely attached TiO_2 NPs, and dried at $60\text{ }^\circ\text{C}$ under vacuum for 5 h.

2.3. Characterization

The morphology of SM- TiO_2 samples was characterized by field emission scanning electron microscope (Fe-SEM; FEI Inspect F).

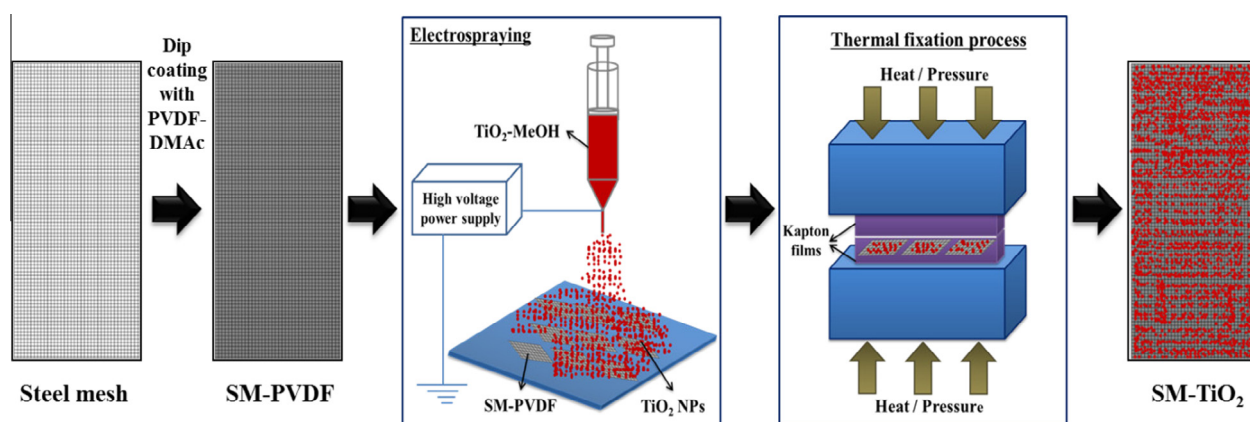


Fig. 1. Schematic of experimental procedure for preparing TiO_2 immobilized on SM.

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