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Facile synthesis and immobilization of Ag–TiO₂ nanoparticles on electrospun PU nanofibers by polyol technique and simple immersion

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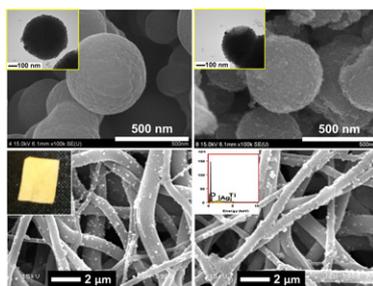
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HIGHLIGHTS

- ▶ One-pot polyol method led to synthesis of Ag NPs deposited on TiO₂ NP substrate.
- ▶ The synthesized Ag–TiO₂ NPs were decorated on electrospun PU mat by simple immersion.
- ▶ Well-dispersed Ag–TiO₂ NPs were found to be strongly adhered on the PU surface.
- ▶ The Ag–TiO₂ NP-decorated PU composite mat showed superior photocatalytic efficiency.
- ▶ The present composite mat could have potential use in water treatment application.

GRAPHICAL ABSTRACT



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ABSTRACT

A simple polyol process is presented here to synthesize silver (Ag) nanoparticles (NPs) on titanium nanosphere substrate (Ag–TiO₂), which are subsequently decorated on the surface of electrospun polyurethane (PU) nanofibers by immersion in hot colloidal solution. Various spectroscopic and physical characterization techniques are utilized to study the physico-chemical properties of the prepared samples. The polyol-synthesized Ag–TiO₂ decorated PU nanofibers show superior antibacterial properties under UV light, and are potentially useful for water filtration application.

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1. Introduction

Electrospinning is a popular technique in the effective and easy production of nanofibers that has evoked significant interests in

different fields [1,2]. In recent years, research focusing on polymer-based composite materials, especially by incorporation or deposition of metal particles in/on various inorganic host matrices is being widely investigated in materials sciences due to their enhanced properties and potential application in optics, energy storage, catalysis, sensor and environmental remediation [3–6]. Several nanoparticles (NPs) like Au, Ag, ZnO, TiO₂, etc. have been incorporated in various polymeric matrices. A few studies have tried the

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combination of different NPs in a polymeric matrix to enable synergistic effects and further enhance their functionality. For instance, it has been known that the presence of Ag^+ ion and metallic silver (Ag^0) nanoparticles (NPs) in/on titanium dioxide (TiO_2) matrix not only can lead to enhancement of the electron–hole separation and interfacial charge transfer which increases the photocatalytic activity, but it can also extend the working area to the visible light region [7,8]. Therefore, embedding or surface functionalization of organic nanofibers by silver (Ag) and TiO_2 NPs or its composite structures could present new or additional functionalities such as improved mechanical, optical, photocatalytic, and antimicrobial properties than their single-polymer material counterpart.

A number of processing techniques have been carried out to decorate or incorporate TiO_2 NPs in/on polymeric matrices such as sol–gel and sputter coating [9], layer-by-layer assembly [10], and electrospray deposition [11]. For the deposition of Ag NPs, techniques such as in-situ decoration [12], chemical reduction of AgNO_3 precursor [13], and controlled assembly [14] have been widely studied. However, most of the studies in literature used two separate steps in the decoration of TiO_2 NPs and Ag NPs on polymeric nanofiber surfaces. This is because the TiO_2 and Ag NPs were also separately synthesized and prepared prior to their subsequent incorporation in the composite nanofiber. For example, Jeun et al. [15] recently prepared titania/polyacrylonitrile (TiO_2 /PAN) nanofibrous mats containing silver nanoparticles by electrospraying method and then by e-beam irradiation process. Also, Pant et al. [16] obtained silver-impregnated TiO_2 /nylon-6 nanocomposite mats using similar method and they suggested that the enhanced hydrophilicity (antifouling capacity) and superior photocatalytic and antimicrobial properties of the spider-wave-like structure of the nylon-6 composite mat were due to the presence of TiO_2 and Ag NPs on the surface of the nanofibers.

Based on our review of the literature, the immobilization of composite nanoparticles, which were synthesized by one-pot method, on/in electrosprayed nanofibers is relatively less investigated. In the present study, we synthesized two different nanoparticles (i.e., Ag NPs in/on TiO_2 NPs) by a one-pot polyol method. Subsequently, we functionalized the surface of electrosprayed polyurethane (PU) nanofibers using the synthesized Ag NPs on/in TiO_2 sphere by immersion in Ag– TiO_2 solution at different duration, which needs no other binding agent or surfactant except ethylene glycol solution.

2. Experimental section

2.1. Synthesis of TiO_2 spheres

The titania precursor spheres were synthesized according to the reported procedures with minor modifications [17]. In a typical procedure, 2 ml titanium tetra-isopropoxide (TIPP, guaranteed reagent, Junsei) was added to 50 ml ethylene glycol (EG, Showa) and was magnetically stirred for 8 h at room temperature; then, the mixture was poured into a solution containing 170 ml acetone (Showa) and 2.7 ml water and was vigorously stirred for 1 h. The white precipitate was separated by vacuum filtration, followed by washing with ethanol and drying at 60 °C. In order to obtain anatase phase of TiO_2 , 0.2 g titanium glycolate precursor was added to 50 ml water and was heated by reflux with stirring. After refluxing for 3 h, the white precipitate was obtained by vacuum filtration, followed by washing with water for five times and was dried at 60 °C for further usage.

2.2. Synthesis of Ag– TiO_2 nanocomposite

In a three-necked round bottom flask fitted in a heating mantle, 0.15 g TiO_2 was added to EG and the mixture was refluxed under vigorous stirring at 150 °C for 30 min. Then, 10 ml EG, which was

dissolved in 0.17 g PVP (Alfa Aesar, MW = 58,000) was added into the hot solution. The PVP here was used to protect the to-be-synthesized silver NPs from agglomeration. Soon afterward, 0.5 M aqueous silver nitrate (AgNO_3 , Showa) was introduced into the mixed solution and was stirred at 150 °C for 30 min. A brownish colloidal solution of Ag– TiO_2 composite NPs was obtained and it was used to functionalize the surface of PU nanofibers.

2.3. Electrospraying of PU nanofibers

Neat polyurethane (10 wt%) solution was prepared by dissolving appropriate amount of PU pellets (Skythane® X595A-11) in DMF/MEK (50/50, wt:wt%, Showa) solvent solution. Electrospraying was carried out at 11 kV, tip-to-collector distance of 150 mm and solution feed rate of 1 ml h⁻¹ [18]. During electrospraying, the nozzle (inner diameter = 0.51 mm) kept on moving laterally (i.e., back and forth) on its axis for a distance of 150 mm and a linear speed of 100 mm min⁻¹ controlled by LabVIEW 9.0 (National Instruments). Four ml of PU solution was electrosprayed onto a grounded flat collector, which was perpendicularly-oriented to the nozzle. After electrospraying, the neat PU nanofibrous mat was dried at 80 °C for 48 h to remove the residual solvents.

2.4. Surface functionalization of electrosprayed PU nanofibers with Ag– TiO_2 NPs

Electrosprayed PU nanofibrous mats (5 cm × 5 cm) were immersed in a previously-prepared colloidal EG solution containing Ag– TiO_2 NPs in an oven at 150 °C. Mat samples were taken out after 4 h and 12 h of immersion, and were then thoroughly rinsed with deionized water and dried at 60 °C for 24 h. For ease of discussion, the composite mat immersed at 4 h and 12 h will be referred herein as CM4 and CM12, respectively.

2.5. Bacterial inactivation test

Bacterial inactivation test was carried out in a batch set-up. Gram-negative *Escherichia coli* (*E. coli*) Top 10 strain was used as the model microorganism, which was inoculated following the method used in our previous study [19]. One colony of *E. coli* was taken out from the original stock in an agar plate and was cultured in lysogeny broth (LB) medium at 500 µl. This bacterial solution was incubated at 35 ± 0.1 °C, 200 rpm for 24 h using a shaking incubator. The working suspensions were prepared by adding 200 µl of inoculated LB medium to a 50 ml sterilized distilled water in a beaker. The electrosprayed nanofibrous mats, i.e. neat PU, CM4 and CM12 cut to a size of 3 cm × 3 cm were each placed at the bottom of a Petri dish, and *E. coli* suspensions of 8 ml (initial concentration = 10⁷ CFU ml⁻¹; CFU: colony forming unit) was placed over the mats. Ultraviolet (UV) light (wavelength = 320–500 nm) was then irradiated directly to the Petri dish at room temperature for a duration of 180 min. The UV light intensity was maintained at 5.9 W cm⁻² using a mercury vapor lamp (OmniCure, EXFO). At given time intervals, 100-µl suspension was collected and diluted appropriately by serial dilution in distilled water. Sampling was done in triplicates and the average was calculated together with the standard deviation. To count the bacterial concentration, ready-to-use petrifilm (3M Petrifilm, USA) and prepared agar plates were used. After incubation for 48 h, the number of bacteria was manually counted using a colony counter.

2.6. Characterization

The surface structure and morphology of the present TiO_2 , Ag– TiO_2 composite spheres and Ag– TiO_2 /PU composite nanofibrous mats were

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