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Original Article

Surface modification of polyamide thin film composite membrane by coating of titanium dioxide nanoparticles

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ABSTRACT

In this paper, the coating of TiO₂ nanoparticles onto the surface of a polyamide thin film composite nanofiltration membrane has been studied. Changes in the properties and separation performance of the modified membranes were systematically characterized. The experimental results indicated that the membrane surface hydrophilicity was significantly improved by the presence of the coated TiO₂ nanoparticles with subsequent UV irradiation. The separation performance of the UV-irradiated TiO₂-coated membranes was improved with a great enhancement of flux and a very high retention for removal of residual dye in an aqueous feed solution. The antifouling property of the UV-irradiated TiO₂-coated membranes was enhanced with higher maintained flux ratios and lower irreversible fouling factors compared with an uncoated membrane.

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1. Introduction

During the last decades, interest in the use of membrane technology has emerged for wastewater treatments as well as for the production of drinking water [1]. Particularly, fouling is one of the main problems in any membrane separation process. Surface modification of membranes has been considered to be the most sustainable solution to reduce the fouling. Among various approaches, hydrophilization of membranes is a potential fouling mitigation method [2,3]. The idea is to introduce hydrophilic groups into a polymeric membrane surface, so that the overall membrane material becomes more hydrophilic and thus less prone to organic fouling. The polyamide thin film composite (TFC-PA) membranes have been widely used for water treatments due to their superior water flux, good resistance to pressure compaction, wide operating pH range, and good stability to biological attack; however, it has also significant drawbacks due to the membrane fouling [1,4].

Titanium dioxide (TiO₂) nano-sized particles are a popular photocatalysts. They attract much attention from both fundamental research and practical applications for the removal of contaminants

from water because of the high photoactivity and chemical stability [5–9]. It is well known that TiO₂ would generate electrons and empty holes under ultra-violet (UV) irradiation [10]. There have been numerous studies about this material in recent years due to its innocuity, resistivity, photo catalytic and superhydrophilicity properties [3,5]. Two different schemes [11] can explain the self-assembly (Fig. 1a, b) behavior of TiO₂ on the surface of polymer containing COOH and the COOH groups. One way is to link TiO₂ with oxygen atoms via coordination to Ti⁴⁺ cations (Fig. 1a). The other way is to form a hydrogen bond between COOH groups and the hydroxyl group of TiO₂ (Fig. 1b).

Many experiments have been carried out for modifying the ultrafiltration (UF) and microfiltration (MF) membranes using TiO₂ nanoparticles [2,5–8,12–14]. Rahimpour et al. [7] successfully prepared two types of the modified polyethersulfone (PES) membranes via entrapping or coating TiO₂ nanoparticles along with UV irradiation. However, the separation performance and antifouling properties of the UV-irradiated TiO₂-coated membranes were higher than those of the UV-irradiated TiO₂-entrapped membranes. The optimum conditions for the preparation of TiO₂-coated membranes were determined when using 0.03 wt.% of a TiO₂ colloidal suspension, followed by 15 min UV irradiation at 160 W. Li et al. [12] successfully coated TiO₂ nanoparticles onto an ultrahigh molecular weight poly(styrene-*alt*-maleic anhydride)/poly(vinylidene fluoride) (SMA/PVDF) membrane surface. It was demonstrated that

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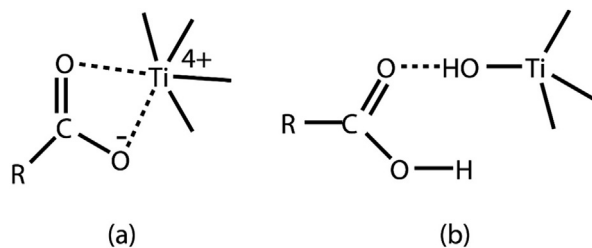


Fig. 1. Mechanism of self-assembly of TiO₂ nanoparticles [11].

TiO₂ particles were tightly absorbed on the surface of SMA/PVDF membranes and the amount of TiO₂ increased with the increase of –COOH groups hydrolyzed from SMA in membranes. The hybrid membranes exhibited extraordinary hydrophilicity, superior permeability and excellent fouling resistance in contrast with an original SMA/PVDF membrane. Madaeni et al. [10] coated TiO₂ nanoparticles and subsequently irradiated UV light onto the cellulose ultrafiltration membrane surface. The results indicated that the stable whey flux of the coated TiO₂ nanoparticle membrane was higher than that of the uncoated one. After an exposure of the membrane surface under the UV light, two phenomenon can be occurred: photo catalytic and ultrahydrophilicity, which lead to the decomposition and removal of the foulant and increase the membrane flux.

The coating of TiO₂ particles followed by UV radiation could improve membrane flux and the self-cleaning property increases with the longer UV irradiation time [8,9,15–19]. It is important to mention that TiO₂ nanoparticles have the ability to temporarily keep their photo-induced superhydrophilicity after switching off the UV light.

In this work, the surface of a TFC-PA NF membrane was modified by coating TiO₂ nanoparticles with a subsequent UV irradiation. Changes in the membrane surface characteristics were determined through the scanning electron microscope (SEM) images, time of flight secondary ion mass spectroscopy (ToF-SIMS) analysis, Fourier transform infrared spectroscopy – attenuated total reflectance (FTIR-ATR) spectra, and water contact angle (WCA) measurements. The changes in the membrane separation performance were evaluated through water permeability, flux, and retention for removal of reactive red dye in an aqueous feed solution. The antifouling property of the membranes was determined through a maintained flux ratio and an irreversible fouling factor for filtration of the dye and protein feed solutions.

2. Experimental

2.1. Materials

A commercial TFC-PA membrane (Filmtec BW30) was used as the substrate material for the surface coating of TiO₂ nanoparticles. It consists of a topmost ultrathin polyamide active layer on a reinforced polysulfone (PSf) porous substrate and demonstrates up to 99.1% NaCl rejection with flux as high as 42.5 L/m²h at a pressure of 5.5 MPa [20]. The membrane samples were cut to have a diameter of 47 mm and soaked in a 25 v/v % aqueous solution of isopropanol (99.9%, Sigma-Aldrich) for 60 min; next, they were carefully rinsed with deionized water, and then kept wet until they were used for surface coating. The commercial TiO₂ nanoparticles in aggregated form with primary particle size of 14 nm and anatase phase of 89.38% were used for the surface coating. Reactive red dye RR261 (China) and pure-grade bovine serum albumin (BSA) (Wako, Japan) were used for the preparation of aqueous feed solutions for membrane filtration tests.

2.2. Coating of TiO₂ nanoparticles onto membrane surface

The solutions of TiO₂ nanoparticles in suspension were prepared by ultrasonic method. The TFC-PA membrane substrate was dipped in the TiO₂ colloidal solution containing 10–80 ppm of TiO₂ nanoparticles. The membrane was then washed with deionized water and exposed to UV light (UV-B lamp, 300 nm, 60 W) for different time periods, from 15 s to 90 s. The coated TiO₂ membranes were kept wet in deionized water until they were used for characterization.

2.3. Membrane characterization

2.3.1. Morphology

The membrane surface morphology was observed through the scanning electron microscopy (SEM), using a field-emission scanning electron microscope (FE-SEM, Hitachi S-4800). The micrographs were taken in high vacuum conditions at 5 kV. The membrane samples were sputter coated with a 3 nm thick platinum layer prior to imaging.

2.3.2. ToF-SIMS analysis

The existence of TiO₂ nanoparticles on the surface of a TiO₂-coated TFC-PA membrane was also determined through time of flight secondary ion mass spectroscopy (ToF-SIMS), using MiniSIMS (SAI Scientific analysis instruments Ltd.). Gallium ions (Ga⁺) with energy of about 6 keV were used as the primary ion beam for a nominal incident angle of 90° to the surface.

2.3.3. Functionality

The surface chemical functionality of the membranes was characterized by the attenuated total reflectance Fourier transform infrared spectroscopy (FTIR-ATR, Spectro100 Perkin Elmer) for a nominal incident angle of 45°, with 100 scans at a resolution of 4 cm⁻¹. All membrane samples were dried at 25 °C under vacuum before characterization.

2.3.4. Wettability

The wettability of the membrane surface was examined through the water contact angle measurements, using a goniometer (DMS012) equipped with a camera, which captured images of deionized water drops on the dried surfaces of the membranes at 25 °C. The contact angles were then calculated from the captured images. For each sample, three drops (3 μL) were placed at different positions on the membrane surface, and the average value of the contact angles was obtained.

2.3.5. Evaluation of the membrane filtration properties

The membrane filtration experiments were performed in a dead-end membrane filtration system, consisting of a stainless steel cylindrical cell with a volume of 300 cm³ supplied by Osmonics (USA) and a stirrer connected to a nitrogen gas cylinder, which provided a working pressure through a membrane area of 13.2 cm². Filtration experiments were carried out at room temperature. The membrane was compacted by deionized water at 15 bar for 15 min before carrying out the filtration measurements. In all experiments, the membrane cell was carefully rinsed with deionized water before and after using. The water flux was determined by

$$J_w = [V_w / (A \times t)] (L / m^2 \cdot h)$$

where V_w is the deionized water volume obtained through a membrane area of A within a filtration time of t.

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