



Mechanically enhanced PES electrospun nanofiber membranes (ENMs) for microfiltration: The effects of ENM properties on membrane performance

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ABSTRACT

The application of electrospun nanofiber membranes (ENMs) as microfilters for the process of water purification requires the substrate to possess suitable strength, permeability, and a smooth surface. Therefore, the fiber homogeneity, inter-fiber adhesion, and surface roughness of the ENMs must be carefully controlled. Concurrently, an understanding of the ENMs' rejection mechanism for contaminants is necessary for the effective application of ENMs. In this study, we demonstrate the fabrication of polyethersulfone (PES) ENMs, which are useful for water purification as water treatment membranes. An optimum fabrication condition that can significantly improve the mechanical property and surface roughness of the PES membrane is also illustrated. This technique induces the solvent remaining on the fiber's surface after the electrospinning process, and the mechanical properties and surface roughness of the membrane are improved by the solvent-induced fusion of the fiber. The fabricated PES ENMs also show higher clean water productivity. Additionally, we show that a particulate contaminant in water is mainly rejected on the ENM surface by using a water filtration test. Based on our conclusions, we suggest the appropriate ENM regeneration method and confirm that the fabricated ENMs show excellent regeneration ability.

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1. Introduction

Water scarcity is becoming one of the most serious problems that the world faces; this problem is due to industrial advancement, population growth, and global warming. However, treating water to become drinkable will require people to become conscious of the issue. New technologies, innovative techniques, and novel engineering designs will be needed to tackle water scarcity. Among the various existing water treatment techniques, the most advanced membrane technologies are expected to solve the world's water crisis (Ulbricht, 2006). Although membrane technology currently requires high capital and operating expenditures, with improvements in technologies, water membrane treatment costs are anticipated to continue to decline, which will drive demand (Shannon et al., 2008). However, for liquid filtration, conventional porous polymeric membranes have their intrinsic limitations (for

example, low-flux and high-fouling performances). These drawbacks are due to the geometrical structure of the pores, the corresponding pore size distribution, and the undesirable macro-void formation across the entire membrane thickness. It appears that electrospun nanofiber membranes (ENMs) can overcome some of these limitations.

Among the various fiber synthesis methods, electrospinning is one method that produces nanofibers. Unlike mechanical fiber spinning techniques, electrospinning is a widely used technology for electrostatic fiber formation. It utilizes electrical forces to produce polymer fibers with diameters ranging from 2 nm to several micrometers by using polymer solutions of both natural and synthetic polymers. Electrospinning has seen a tremendous increase in research and commercial attention over the past decade, not only due to its versatility in spinning a variety of polymeric fibers but also due to its ability to consistently produce fibers in the submicron range. Producing tiny fibers is difficult using the standard mechanical fiber-spinning technology (Reneker et al., 2000; Schreuder-Gibson et al., 2002). Electrospinning technique offers unique capabilities for producing novel natural nanofibers and

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fabrics with controllable pore structures (Zussman et al., 2002; He et al., 2005). With smaller pores and higher surface areas than regular fibers, electrospun fibers have been successfully applied to various fields; these fields include nanocatalysis, tissue engineering scaffolds, protective clothing, filtration, biomedical, pharmaceutical, optical electronics, healthcare, biotechnology, defense and security, and environmental engineering (Luu et al., 2003; Subbiah et al., 2005; Ramakrishna et al., 2006; Modgill et al., 2015; Llorens et al., 2015; Turaga et al., 2012; Welle et al., 2007).

In the environmental field, electrospun nanofibers have been widely used in air filtration processes, such as microparticle removal (Wang et al., 2016). However, they have hardly been applied to water filtration because ENMs do not have enough mechanical strength to be used as water treatment filters (Huang et al., 2014). To overcome the mechanical limitations of ENMs for water treatment, a polymer fabric, such as Polyethylene phthalate (PET), has been used as a support layer (Homaieghor et al., 2010).

In this study, we demonstrate practical approaches to enhancing the mechanical properties of PES ENMs and estimate the membrane performance of support-less PES ENMs. PES was selected as the membrane material due to its high thermal and chemical resistance. PES can also be considered to be a model membrane material since it is widely used for commercial microfiltration (MF) and ultrafiltration (UF) membranes. In addition, N-methyl-pyrrolidinone (NMP) was used as a solvent due to its high solubility in PES and high viscosity compared to other solvents; however, it has rarely been used in the electrospinning technique due to its low volatility. The drawbacks of the solvent's low volatility were overcome by an additional process: the ENM wetted process. In the wetted process, the remaining solvent removal and fiber solidification were induced.

The goal of the present study was to further the understanding of the relationships between the structural parameters of PES ENMs (i.e., the average fiber diameter, ENM porosity, and mechanical strength) and ENMs' microfiltration performance in water purification.

2. Materials and method

2.1. Materials

PES (Solvay Korea Co., Gafone 3000P) with a molecular weight (Mw) of 62,000–64,000 g/mol was used for PES ENM synthesis. NMP was obtained from Sigma-Aldrich and used to prepare the solvent for PES electrospinning. A commercial microfiltration membrane (Model V0.2, Synder Membrane Technology) with a nominal pore size of 0.2 μm , which is the most representative size to define the MF filter and a commercial tight microfiltration membrane (Model LX, Synder Membrane Technology) with 300,000 Da (Da) molecular weight cut-off (MWCO) was also tested for comparison purposes. As a feed system for the rejection tests, kaolin particles, which are a standard material for making the 100 Nephelometric Turbidity Unit (NTU) solution, were obtained from Sigma-Aldrich; they were used to prepare the feed solution.

2.2. PES ENM fabrication by electrospinning

The PES was dissolved in the NMP mixture at room temperature for 1 day until it became a homogeneous solution. Different concentrations (i.e., 25–40 g of PES in 100 mL NMP) of PES/NMP mixtures were prepared. The PES/NMP solution was electrospun directly onto aluminum foil using an automatic electrospinning instrument, consisting of a metallic spinneret, a syringe pump, a high-voltage power supply, and a stainless steel collector. All samples were electrospun using a spinneret with a 19 gauge

diameter, an ambient temperature of 25–30 °C, and a humidity of 50–60%.

2.3. PES ENM characterization

The morphologies of the PES ENMs were observed with a scanning electron microscope (SEM) (S-4700, Hitachi, Japan). The mechanical properties of the electrospun PES membranes were measured with a universal testing machine (UTM) (Shimadzu AG-X, Japan) that had a 50 kN capacity load cell at an ambient temperature. The ENMs were severed in a rectangle measuring 20 mm $\times$ 0.5 mm. The specimen thickness was about 500 μm , and the crosshead speed was 200 mm/min. The mean pore size of the ENMs and their porosity were determined using a capillary flow porometer (CFP-1500AE, Porous Materials, USA) and automatic porosimeter (Autopore III 9420, Micrometrics, USA), respectively. A multimode atomic force microscope (AFM) (MultiMode 8, Bruker, Germany) with a Pt/Ir-coated cantilever was employed to investigate the roughness of the ENMs.

2.4. PES ENM performance test

A dead-end stirred ultrafiltration cell equipped with an air pressure control was used to measure the pure water flux and 1 μm size polystyrene particle rejection and to do a turbidity removal test. The stirred cell (Model 8200, Millipore, USA) with an active filtration area of 28.7 cm^2 under a feed pressure of 1 bar was used to determine the flux, and the amount of permeate was measured using a graduated cylinder. The membrane cleaning effect test was performed by a cross-flow filtration system to identify the ENM roughness effect on membrane fouling. The 1 g/L bovine serum albumin (BSA) solution was tested using a membrane cross-flow system with an effective area of 18.36 cm^2 at room temperature and a 0.25 bar, with a cross-flow rate of 400 mL/min. All chosen PES ENMs possessed a thickness of $200 \pm 10 \mu\text{m}$. The pure water flux was defined as the volume of water passing through the membrane per unit of time, per unit of area and per unit of transmembrane pressure. Two commercial membranes—a 220 μm thickness PES UF scale membrane with 300,000 Da of MWCO (Synder Membrane Technology, China) and a 205 μm thickness PVDF MF scale membrane with a 0.2 μm pore size—were used as the control group. In the membrane performance test, the PES ENMs and commercial membranes were completely wetted first, and then they were tested at ambient temperatures (20–25 °C) and 1 bar pressure. The pure water flux was measured using an analytical balance as a function of time and pressure. The materials that cause turbidity rejection were tested in the same dead-end filtration cell. A suspension of kaolin particles with a diameter of 0.4–3.0 μm was diluted in water to prepare a solution of 100 NTU turbidity to serve as the feed solution. The test was carried out at 1 bar pressure. A turbidimeter (TN-100, Eutech Instruments, USA) and UV spectrometer (Optizen 2120 UV, Mecasys, South Korea) were used to measure the contaminant concentration of the feed solution and the permeation.

3. Results and discussion

3.1. PES ENM morphologies with various synthesis parameters

The morphologies of the PES ENMs prepared with different concentrations were investigated using SEM. In Fig. 1, the beads morphology was seen in the ENM fabricated by the 2.5 g PES in the 10 mL NMP solution. In contrast, the uniform fiber morphology was found in the membranes from 3.0 to 4.0 g PES in the 10 mL NMP solution. The average fiber diameter increased from 550 nm to

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