



Short communication

Continuous thermal-rolling of electrospun nanofiber for polyamide layer deposition and its detection by engineered osmosis

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ABSTRACT

In this study, electrospun nanofiber was continuously sandwiched between two rubber rollers at high temperature (150 °C). The prepared nanofiber support layer possessed a pore size of 0.4 μm (with narrow distribution) and a fiber diameter of 292 ± 94 nm. Afterward, an interfacial polymerization was employed to deposit the selective polyamide layer on the nanofiber support layer. For the first time, engineered osmosis (EO) was used as a platform for verifying the presence of a polyamide selective layer on a nanofiber support layer. We found that the membrane prepared exhibited an excellent combination of permeability (30 L/m²h) and selectivity (17 g/m²h and 0.57 g/L) under a net pressure of 49 bar at the EO platform. The membrane developed can be applied to generate renewable energy using the EO process. In addition, the EO method can be used to detect selective polymer having a nano-scale thickness.

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1. Introduction

Phase separation for two or more complex fluids is one of the core processes in both chemical and environmental engineering [1]. Nanofiber has been investigated extensively in the fields of energy-storage devices, batteries, tissue engineering, biotechnology, cosmetics, super capacitors, and environmental filters [2,3]. In addition, nanofiber possesses inherently superior structural properties for mixed-media filtration, including a large surface area, high porosity, low tortuosity, and narrow pore-size distribution [4,5]. Although continuous and massive fabrication of polymer or nanomaterial composite polymer have been actively investigated, research on continuous fabrication of nanofiber/polyamide composite membrane still remains a great challenge [6–9].

As a process for generating nanofibers, electrospinning has been used more frequently and extensively than the mechanical fiber-spinning technique. Unlike mechanical spinning, electrospinning enables the production of nanofibers from various materials in

various configurations and assembles them at a lower cost (Fig. S1) [10–12]. However, some critical issues result from electrospinning, including weak mechanical strength and excessive surface roughness, which need to be addressed before electrospun nanofibers can be used in filtration processes [13]. One major concern, weak mechanical strength, has been addressed by reinforcing the fusion of the fibers induced, which was accomplished by tuning the solvent and the phase-inversion process (Table S1–S2, Figs. S2–S3) [14]. However, other concerns, including excessive surface roughness, remain major issues. Excessive surface roughness, which is caused by the inherent semi-woven structure of nanofibers (Table S3), carries a high potential for fouling. In addition, it hinders the deposition of selective polyamide layers, which is important for advanced separation processes, including nanofiltration, reverse osmosis, and forward osmosis [14–19]. Depositing a selective polyamide layer on a rough nanofiber surface is difficult. Nanofibers formed by electrospinning often have a rough surface because they are randomly ordered on a substrate that has a fiber valley [19]. Successfully depositing polyamide layers by interfacial polymerization requires a smooth surface with a pore size of less than 1 μm of substrate, as this prevents delamination of the polyamide layer [20,21]. Furthermore, evaluating the deposition on nanofiber of polyamide that is less than 400 nm in thickness requires super-sensitive characterization [22–24].

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Engineered osmosis, also known as “manipulated osmosis,” can be used as a platform for determining whether the chemical bonding energy between a selective polyamide and nanofibers is strong enough to act as a selective barrier during the filtration process (Figs. S4–S6, see the supplementary data for details about engineered osmosis). The osmotic pressure, which is driven by the differences in concentration of the monovalent ions, continuously pushes the selective polyamide layer to detach from the support layer while applying hydraulic pressure to keep the polyamide on the support layer. To determine whether the selective polyamide layer has peeled off or is still attached under certain pressure, the water molecules and ions transported through the membrane can be measured as a function of the net pressure (osmotic pressure minus applied pressure) (Fig. 2(a)).

The present study made two major remarkable findings. First, it used a continuous thermal-rolling pretreatment to flatten the surface morphology of the electrospun nanofiber to better deposit the polyamide on top of it. Second, it used engineered osmosis using monovalent ions to verify whether a successfully deposited polyamide layer had attached to the nanofiber. Although there are several methods for identifying the binding force between two materials, this method is simple and similar to the actual filtration process.

2. Experimental

The continuous thermal-rolling pretreatment was conducted prior to the interfacial polymerization for polyamide deposition, when deep steps still existed between fibers, even though the junction points of the nanofiber were fused by the controlling phase inversion process (Fig. 1) [14]. Our previous study showed that heat-pressing improves mechanical strength and decreases the porosity, and a smaller pore size creates a denser structure and network among the nanofibers [25], which helps to create a smooth, densely packed surface amenable to accepting deposition of another layer atop it. Unlike thermal pressing for thin-film nanofiber composite (TNC) membranes, a process found frequently in the literature, the continuous thermal-rolling process used in the present study improves the commercial viability of the product [26,27]. Therefore, in the present study, the highly fused nanofiber created using this process was used as a pristine support layer. To deposit the selective polyamide layer on the nanofiber

support, a general interfacial polymerization method was used. For more detailed experimental conditions, including those regarding the membrane synthesis, please refer to the supplementary data.

3. Results and discussion

Under the same experimental conditions, the pretreated membranes not subjected to the thermal-rolling process demonstrated no water flux, because the polyamide layer peeled off and washed away when net pressure was applied. In contrast, when the continuous thermal-rolling pretreatment was used for the TNC membrane (labeled TNC-1 in Fig. 2(b)), water flux was observed, meaning that the polyamide layer was firmly attached to the nanofiber's surface. This was enabled by preparing the nanofiber so that it had a narrow pore size, around $0.4\ \mu\text{m}$ (Fig. 2(c)), which made the nanofiber's surface smoother.

Under every net pressure tested, the water flux through the TNC membrane was two-fold higher than the water flux through the pristine thin-film composite (TFC) membrane (labeled TFC-3 in Fig. 2(b)). For the TNC membrane, the selective polyamide layer was firmly deposited and did not peel off, even at the high net pressure of 24 bar. Therefore, the high porosity (Fig. 2(c) and Table S4) and low tortuosity (Fig. S4) of the nanofiber appear to be advantageous to the phase separation process.

Scanning electron microscopy (SEM) images of the TNC-1 membrane (Fig. 3(a)–(b)) showed that the diameter of the nanofiber tended to be about $292 \pm 94\ \text{nm}$ (Fig. 2(d)). In addition, the nanofiber exhibited excellent uniformity, without any bead or shape structures. Although the support layer of the TNC membrane was composed of nanofibers having a diameter of several hundred nanometers, the fusion of the junction points (Fig. S2–S3) enabled the membrane to endure when filtering various mixtures under high net pressure [14,25]. Therefore, the nanofibers could be used as the sole support layer without an extra substrate to improve their mechanical strength, a process that generally is required during the phase separation process. This finding could be highly beneficial to membrane processes, because it would enable a membrane to maintain its flexibility, even with decreased total thickness. Therefore, this type of single-layer TNC membrane could be more effective than membranes possessing additional outer layers to increase their mechanical strength.

This type of TNC membrane could be used to generate

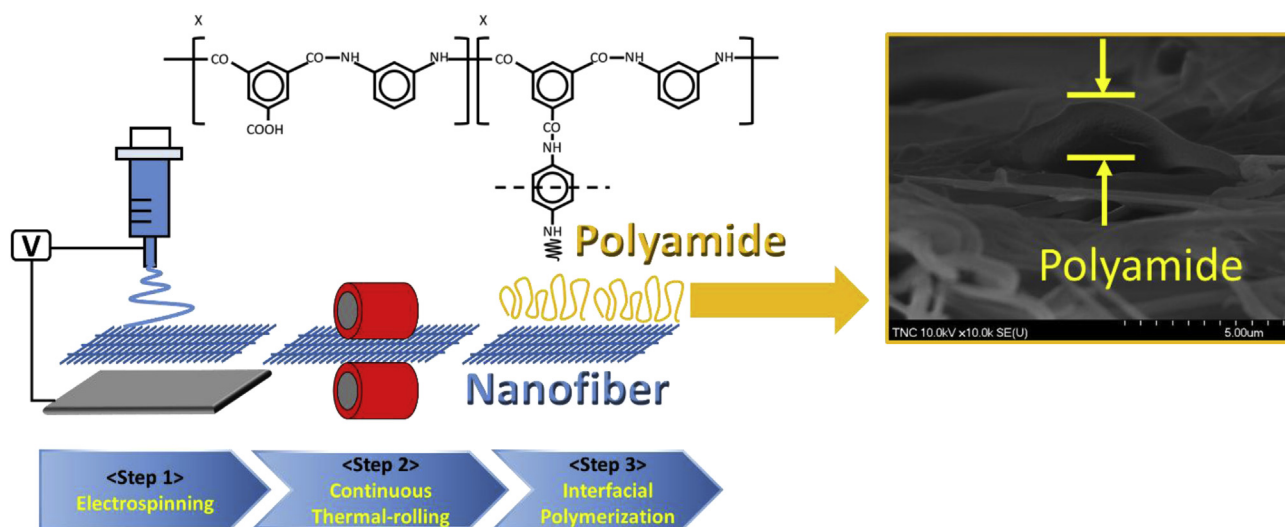


Fig. 1. Schematic of three steps used to synthesis thin-film nanofiber composite (TNC) membrane: electrospinning, thermal-rolling, and interfacial polymerization. Cross-section scanning electron microscopy (SEM) image of single polyamide layer attached on nanofiber support layer.

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