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Membranes based on electrospun lignin-zeolite composite nanofibers

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ABSTRACT

Lignin-zeolite composite nanofiber membranes were prepared by the electrospinning technique. The membranes were characterized in terms of morphology, mechanical properties, hydrophilicity, permeation, and particulate separation performance. The experimental results indicated that lignin-zeolite composite nanofiber membranes exhibited significant differences in surface properties and mechanical properties because of the addition of inorganic particles. Adding one weight percent of zeolite nanoparticles improved the tensile strength, tensile modulus, hydrophilicity, permeability, and separation factor of the membranes. However, the addition of higher weight percent of zeolite nanoparticles resulted in the decline of mechanical properties, permeability, and particulate retention.

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1. Introduction

Membranes have been implemented in purifying and separating gases, water treatment, food and beverages, metallurgy, pulp and paper, textile, automotive, dairy, pharmaceutical, medicine, bioengineering, biotechnology, chemical, electronic, and nuclear energy sectors and industries. These membranes, used for separating physical, chemical, and biological mixtures in both gases and liquids, can cost considerable amount of energy and money. However, these costs can be reduced by improving the membrane chemical and thermal stability, durability, selectivity, and flux/permeation rate. Conventional membranes strive to overcome such intrinsic limitations, such as poor selectivity, low-flux and high-fouling performance, as a result of relatively low porosity (pore density), pore geometrical structure and pore size distribution [1–8].

Electrospinning is a non-mechanical technique for the formation of nanoscale fibers electro-statically from polymer solutions or melts. It can produce a nonwoven membrane of submicron/-nanofibers with large pore networks, whilst decreasing the fiber diameter from the micrometer range to nanometer range, thereby filtering finer particles. In addition to fiber scale, the technique is capable of producing multifunctional fibrous composites [9,10].

Even though (virgin) polymers, as the main materials in membrane technology, have the advantages of good membrane forming ability, flexibility, and low cost, their applications may be limited because of mechanical failure, adoptability to different environments, and functionality. In order to overcome the limitations of polymers, composite membranes have been developed by combining polymer and inorganic materials. These composite membranes have specific advantages, including excellent separation properties and membrane-forming, good thermal, mechanical, and chemical resistance, and adaptability to severe environments. Therefore, electrospun nanofibrous membranes and their composites not only have a great potential for various applications in the membrane industry, but also offer promising results and provide a good opportunity for overcoming the existing conventional membrane challenges [10–16].

Among polymers, lignin, the second most abundant polymer on earth, exceeded only by cellulose, could play a central role as a new chemical feedstock, particularly in the formation of supramolecular materials and aromatic chemicals. In fact, lignin possesses significant potential for being a component of value-added products. Lignin's antioxidant, antibacterial, and antimicrobial properties [17–23], in addition to its non-cytotoxicity toward human cells [24,25], make it one of the best candidates for replacing imminent membrane materials.

Zeolites, crystalline aluminosilicates containing pores of molecular dimensions, have been implemented in fiber, film, and coating forms in membrane technology. Coupled with the advantages of

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inorganic membranes and perfect shape selectivity, the application of zeolite membranes have been reported in separation membrane and catalytic membrane reactors. For instance: (a) in enhancing a chemical reaction, such as de-hydrogenation, partial oxidation, isomerization, esterification, or acetalization; (b) in separation of the gaseous/liquid mixtures, such as water, hydrogen, carbon dioxide, p-Xylene, or alcohol; (c) in functional films, such as chemical sensors, electrode, opto-electronic device or low dielectric constant material, protection or insulation layer, corrosion-resistant coatings, hydrophilic antimicrobial coatings or sulfonated zeolite for proton exchange membranes; (d) in biotechnology, biomedicine, and medical applications, such as creatinine adsorption in dialysis membranes (artificial kidney). It must be noted that zeolite properties and catalytic performances are affected by their sizes and shapes; as a result, zeolite nanoparticles have demonstrated enhanced catalytic performance in many reactions [26–37].

In the present work, 1–5 weight percent (wt%) of zeolite nanoparticles were incorporated into lignin electrospun nanofibers (LENs) in order to compare them with the virgin (pure) LENs. The aim is to improve the mechanical properties, and the flux and fouling performance of the (filtration) membranes through heat treatment and uniform dispersion of nanoparticles in the lignin nanofibrous membrane. The influence of adding zeolite nanoparticles into the membrane is discussed on the basis of mechanical properties, permeation, and hydrophilicity, as well as the microstructures of the nanofibers in the membranes.

2. Experimental

2.1. Materials

Lignin was supplied by FPInnovations (Vancouver, BC) and used without further purification. Mesostructured nanosized zeolite particles (with an average diameter of about 20 nm), N,N-Dimethylformamide (DMF, 99.9%), Polyethylene oxide (PEO) (Mw = 1×10^6 g/mol), and Polystyrene (PS) Latex Beads (microparticles with 0.1, 0.5, and 1 μm in diameters) were purchased from Sigma-Aldrich; and used as received.

2.2. Preparation and properties of electrospinning solutions

Zeolite was dissolved in DMF and then lignin/PEO (99/1 w/w) was added to the Zeolite-DMF solution (zeolite/lignin = 1–5 wt%). The Zeolite-DMF-Lignin-PEO solution was placed and stirred in 80 °C oil bath for 2 h, and then cooled to room temperature before electrospinning. Gauge number 18–25 needle was used in combination with proper solution concentration (25–35 wt%). An electrospinning distance of 15 cm was used along with an applied voltage of 12 kV at the 0.02 mL/min pump speed, as shown in Fig. 1. After drying at room temperature for 10 h, the obtained samples were thermo-stabilized (thermally treated) in air atmosphere at a 5 °C/min heating rate to 250 °C for 60 min.

2.3. Characterization of composite membranes

2.3.1. Morphology

The morphology of the electrospun nano-fibrous membranes was observed by a scanning electron microscope (SEM) (Hitachi S-2300) using an acceleration voltage of 5–20 kV. All of the samples were coated with gold before SEM observation. The average diameter of nanofibers was determined by analyzing the SEM images.

2.3.2. Mechanical properties

The mechanical properties of the electrospun membranes were evaluated using a multi-purpose micro-tensile tester (KES-G1, Kato-Tech Co. Ltd) with a 5 kg capacity force transducer. Strip-specimens (0.5 cm W × 5 cm L) were tested with 5 replications for each sample at an extension velocity of 0.1 cm/s. The results from the experiments were calculated in load (gram force) vs. displacement. The strain was calculated by dividing the displacement by the gauge length. The specific stress, expressed in g/tex, was obtained using the following formula [10,38]:

$$\sigma_{specific}\left(\frac{g}{tex}\right) = \frac{Load\ (g)/Width\ (mm)}{Areal\,Density\ (g/m^2)}$$

The areal density is the mass of the sample test strip divided by the area of the test specimen. The result was calculated by the following equation:

$$Areal\,Density\left(\frac{g}{m^2}\right) = \frac{Sample\,Mass\ (g)}{Length\,(m) \times Width\,(m)}$$

The specific stress expressed in g/tex was converted to MPa using the conversion factor shown in the following formula:

$$\sigma_{Eng}\ (MPa) = \sigma_{specific}\left(\frac{g}{tex}\right) \times 9.81 \times \rho_{polymer}$$

2.3.3. Hydrophilicity

The contact angle between water and the external surface of fibrous membrane was measured to assess the membrane hydrophobicity and wetting properties [39]. Each sample was tested five times in order to minimize experimental error. Finally, the average value was obtained according to the result of each sample.

2.3.4. Clean water permeability

Demineralized (clean) water (250 mL) was used to determine the permeability of the membranes (10 cm^2 surface area) placed on the filter support in a flow-through system by the dead-end

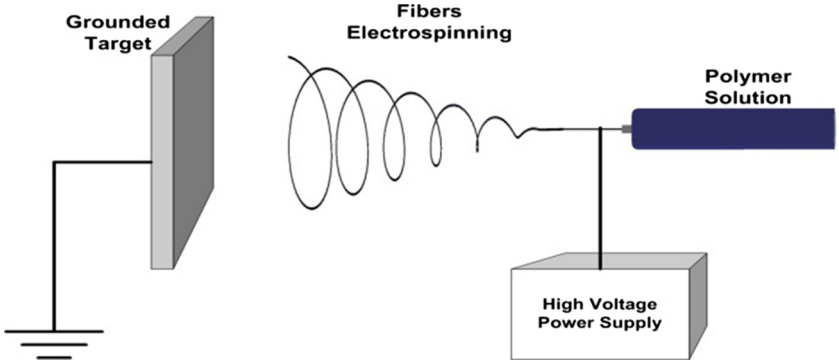

Fig. 1. Schematic of the electrospinning process.

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