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The potential of membrane distillation in recovering water from hot dyeing solution

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ABSTRACT

The possibility of recovering water from hot dyeing solution has been investigated in this work by using membrane distillation (MD) made of polyvinylidene fluoride (PVDF). A series of PVDF hollow fibre membranes with various polymer concentrations (12–18 wt%) were prepared by dry-jet wet inversion method and were designed for direct contact membrane distillation (DCMD) application. Prior to the treatment of hot dyeing solution of 80 °C, the PVDF membranes were characterized in terms of scanning electron microscopy, atomic force microscopy, gas permeation, liquid entry pressure, contact angle and membrane porosity. It is reported that the surface roughness, contact angle and liquid entry pressure of PVDF membrane were increased while mean pore size and porosity decreased with increasing PVDF concentration in the dope from 12 to 18 wt%. Based on the DCMD results, it is found that 12 wt% PVDF membrane showed the highest permeate flux due to its smooth surface, high membrane porosity, large mean pore size as well as big macrovoids formed at the middle cross-section of the hollow fibre membrane. Although MD flux tended to decrease with increasing polymer concentration, all the resulting membranes were still able to achieve excellent dye rejection (at least 99.78%), indicating that MD is very potential to be employed in treating dyeing solution which normally discharged in hot condition from textile industry.

1. Introduction

Current concerns about environmental issues and water shortage have driven scientists and researchers to find effective ways to reduce the amount of contaminants released to the river as well as potential treatment process to reuse process water in industry. The reuse of industrial wastewater is practically applied in many developed countries such as United States, Australia, South Africa and Japan as most of the industrial projects in these countries used the treated wastewater for irrigation process [1]. From the various types of industrial wastewaters, the effluents produced by textile industry is of big concern to many countries due to the huge amount of water discharged daily [2–4]. Statistics revealed that textile factories consume average 0.06–0.40 m^3 of fresh water for each kg of finished product [1,5,6]. Furthermore, textile industries are considered as one of the largest generators of toxic chemical wastewater in the world as they contained more than 2000 types of chemicals and over 7000 types of dyes [7].

Various techniques are used for the conventional treatment of textile wastewater such as coagulation flocculation, biotechnology, electrochemical oxidation and adsorption [1,8,9]. Treatment of textile wastewater with biotechnology is most preferred over other technologies because the process is more environmentally friendly and cost-effective [10,11]. However, most of the conventional treatment processes are associated with significant drawbacks, i.e. relatively low rejection of salt and insufficient to remove completely colour from the textile effluents. Since 1990s, vast research of pressure-driven membrane processes (i.e. ultrafiltration (UF), microfiltration (MF), nanofiltration (NF) and reverse osmosis (RO)) in textile wastewater treatment was widely reported in literatures [8,9,12,13]. It is shown that membrane separation technologies are an attractive alternative to the conventional treatment processes due to their potential to either produce purified water or allow reuse of the auxiliary chemicals used for dyeing process. However, the major problem in these pressure-driven membrane processes is the rapid decline of the permeation flux that arises from membrane fouling during operation [8,13].

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Membrane distillation (MD) is seen as a potential candidate in treating textile effluents as this membrane process has unique advantages over other membrane processes. Unlike NF and RO membrane which strongly rely on external osmotic pressure to operate, the existence of vapour pressure difference between the hot solution (wastewater) and cold water across the membrane could act as driving force which potentially minimizes fouling tendency in long run. Furthermore, MD in principle has the ability to completely eliminate ions, macromolecules, colloids, cells, and other non-volatile organic compounds from the wastewater [14]. It must be pointed out that the hot effluent discharged (80–90 °C) from textile industry has particularly made MD process as a cost effective and energy efficient separation process [15]. A literature search revealed that not many research works have been conducted to employ MD process for the treatment of textile wastewater [15–17]. Besides, the previous published works on the treatment of textile effluents via MD processes were only based on the commercial membranes made of polypropylene (PP) (either Enka Microdyn, USA or Membrana GmbH, Germany).

In order for the MD process to be practically applied in industrial sectors, the study on the laboratory-made MD membranes for different range of wastewaters is very necessary. Currently, polyvinylidene fluoride (PVDF) has been highlighted as the most commonly used polymer material for MD especially in seawater desalination process [18–20]. This is due to its advantage of dissolving in variety of solvents at room temperature. The main objective of this work is to study the potential of PVDF membrane as a new polymer type for textile wastewater treatment in MD process. The hollow fibre membranes were fabricated via dry-jet wet spinning process and were characterized with respect to surface morphology, cross-sectional structure, porosity, hydrophobicity and liquid entry pressure. This paper will study the effect of polymer concentration on the properties of PVDF hollow fibre membrane as well as the MD performances of the membranes in treating hot dyeing solution.

2. Experimental

2.1. Materials

Commercial PVDF polymer (Kynar® 760, MW = 440,000 g/mol) purchased from Arkema Inc., Philadelphia, USA was used as the base polymer. N-methyl-2-pyrrolidone (NMP) with purity more than 99.5% was purchased from Merck and used as solvent without further purification. Polymer concentration in the range of 12–18 wt% was used to fabricate the hollow fibre membrane. Methanol (99.9%) and *n*-hexane (99%) from Merck were then used as membrane post-treatment. Reactive black 5 (RB5, MW = 991 g/mol) from Sigma–Aldrich was used to prepare dyeing solution by dissolving it in DI water.

2.2. Fabrication of PVDF hollow fibre membrane

Prior to dope solution preparation, the polymer pellets were dried in a vacuum oven at 60 ± 2 °C over 24 h to remove moisture content. The spinning dopes containing 12–18 wt% (see Table 1) were prepared through homogeneous stirring of the mixture at temperature 60 °C and stirring rate around 450 rpm for several hours. The viscosity of the dope solutions was measured at the constant temperature of 25 °C by using a basic viscometer (Model: EW-98965-40, Cole Parmer). Then, the dopes were degassed to remove air bubbles prior to spinning process. The spinning conditions of each parameter applied in this work are summarized in Table 2 while the detailed description of the spinning process can be found in our earlier work [21]. After spinning, the spun fibres were immersed in water for few days to completely remove residual solvent. The fibres were then post-treated with methanol followed by *n*-hexane (to minimize fibre shrinkage) before drying at room temperature.

Table 1
Polymer dope composition and viscosity.

Spinning dopes	12PVDF	15PVDF	18PVDF
PVDF (wt%)	12	15	18
NMP (wt%)	88	85	82
Viscosity (cP at 25 °C)	2279.6	3410.8	9544.3

2.3. Liquid entry pressure (LEP) and contact angle measurements

To perform LEP analysis, a test module (0.22 m long) filled with DI water was required in which one unit of hollow fibre membrane was attached at the bottom of the test module while the other end was connected to a diaphragm pump. The water was then pressurized slowly into the lumen side of the fibre at 0.5 bar interval. At each pressure interval, the membrane module was kept at the constant pressure for 10 min to check if any water has permeated through the outer layer of dry membrane. At least three measurements were performed to yield the average LEP. To evaluate the degree of membrane hydrophobicity, a contact angle goniometer (OCA15plus, DataPhysics), equipped with image-processing software was used. A technique called sessile drop was used to measure the contact angle of the outer surface of the fibres. 15 contact angle measurements were performed at various positions on the same sample to yield average result.

2.4. Gas permeation measurement

The membrane with known effective length was first potted into a fitter before putting the membrane sample into module. Nitrogen gas was used during gas permeation test to measure gas permeance, mean pore size and membrane effective porosity. The gas pressure was varied starting from small magnitude until certain pressure. Usually, the upstream pressure is in the range of 0.5–4 bar for a porous membrane. The measurement is based on the volume displacement method by using a burette to measure the collected air bubble volume in predetermined time. Based on the common gas permeation method by Wang et al. [22], gas permeance, J_G for porous membrane can be expressed as

$$J_G = \frac{2r_p\varepsilon}{3RTL_p}\left(\frac{8RT}{\pi m}\right)^{0.5} + \frac{r_p^2\varepsilon}{8\mu RTL_p}\bar{P} \quad \text{or} \quad J_G = K_o + P_o\bar{P} \tag{1}$$

where J_G is the gas permeance (mol/m^2 s Pa), r_p and L_p are the pore radius and effective pore length (m), respectively, ε is the surface porosity, R is the gas constant (8.314 J/mol K), μ is the gas viscosity (kg/m s), M is the gas molecular weight (0.028 kg/mol N_2), T is gas temperature (K), and $\bar{P}$ is the mean pressure (Pa). By plotting J_G with mean pressures according to Eq. (1), mean pore size and effective porosity over pore length, ε/L_p can be calculated from the

Table 2
PVDF hollow fibre spinning conditions.

Spinning conditions	Value
Bore fluid flow rate (mL/min)	2.0
Dope extrusion rate (mL/min)	4.0
Bore fluid composition	100% DI water
Coagulation medium	Tap water
Spinneret OD/ID (mm/mm)	1.15/0.55
Air gap distance (cm)	17.5
Spinning dope temperature (°C)	25 (±1)
External coagulation temperature (°C)	25 (±1)