PREPARATION AND CHARACTERIZATION OF PVDF HOLLOW FIBER MEMBRANE

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Abstract

In this study, dual-layer spinneret was used to fabricate poly (vinylidene fluoride) (PVDF) hollow fiber membrane by using PVDF/NMP dope solution and ethanol solution as the inner-layer solution and outer-layer solution, respectively. The effects of air gap distance (corresponding to the stand time of nascent hollow fiber membrane in the ethanol bath) and PVDF polymer dissolved temperature on the morphology and mechanical property were investigated. The result shows that the PVDF hollow fiber membrane with porous surface can be prepared. Moreover, the tensile strength at break as well as elongation at break of the PVDF hollow fiber membrane decreased as increasing the dissolved temperature of PVDF polymer.

Key words：PVDF，spinneret，hollow fiber membrane

Introduction

Membrane processes, with the advantages of easy operation and energy saving, have many applications in separation of mixtures. The applicability of a membrane technique to a separation process strongly depends on if the membrane possessing suitable separation performance can be successfully prepared. Therefore, many researches have concentrated on developing techniques to prepare membranes and to tailor their separation performance. Among the methods for preparation of polymeric membranes, the most widely used one is the phase inversion method [1]. In this method, the phase separation and subsequent solidification (gelation) of the cast polymer solution determine the final membrane morphology and the associated separation performance. There are several methods to induce phase separation during membrane forming: change in the solution temperature, the so-called thermally induced phase separation (TIPS), exchange of solvent with nonsolvent (coagulant), the so-called immersion precipitation or nonsolvent induced phase separation (NIPS), and intake of nonsolvent vapor, the so-called vapor induced phase separation (VIPS) [2,3]. In this investigation, a dual-layer spinneret was used to fabricate the PVDF hollow fiber membrane to investigate the spinning condition effect on the morphology and mechanical property of PVDF hollow fiber membrane.

Experimental

Materials

PVDF polymer (PVDF Kynar® 760) was used as the matrix of hollow fiber membrane. *N*-methyl pyrrolidinone (NMP) of reagent grade, without further purification, was used as the solvent for PVDF. During the spinning of PVDF hollow fiber membrane, ethanol and aqueous ethanol solution were used as the outer layer solution and bore liquid, respectively. Water was used as the external coagulant.

Fabrication of PVDF hollow fiber

PVDF hollow fiber membranes were fabricated via dry/wet spinning process. The PVDF polymer was dissolved in NMP to form a 20 wt.% dope solution. The outer-layer solution, inner layer dope and bore fluid are delivered to the orifice by passing through three independent channels. The hollow fiber was washed with water to remove the residual solvent for at least three days. Table 1 reveals the detailed process parameters and spinning conditions.

Table 1 The spinning condition

Condition	Value
Inner-layer dope solution	PVDF/NMP 20 wt %
Bore liquid	EtOH/H_2O 70/30
Outer-layer dope solution	EtOH
Outer-layer dope flow rate	0.1 ml/min
External coagulant	H_2O
Bore liquid flow rate	0.3 ml/min
Dope extrusion pressure	3 atm
Coagulant temperature	25 ^{O}C
Air gap	0, 3, 10 cm

SEM observation

The morphology of the PVDF hollow fiber membrane was observed with the scanning electron microscope (SEM, Hitachi, Model: S-3000N). The samples were immersed in liquid nitrogen to fracture and then were sputtered with Au.

Mechanical properties measurement

Tensile stress and elongation at break of the fiber were measured by using tensile test machine (Instron 5544 model) at a crosshead speed of 50 mm/min, with a clamp distance of 3 in. The initial Young's modulus was calculated in the range of 0.5–1.0% tensile strain.

Results and Discussion

Effect of polymer dissolved process on the morphology of PVDF hollow fiber membrane

PVDF polymer chips were dissolved in the solvent, NMP, to form 20 wt% spinning dope at 30 and 80 ^{O}C, respectively. Fig. 1 shows the cross-section morphology of PVDF hollow fiber membranes which were spun via wet spinning process by using 30 and 80 ^{O}C dissolved PVDF/NMP polymer solution. From Fig. 3, it can be found that there existed a uniform pore distribution near the part of inner skin layer for the 30 ^{O}C dissolved system, while a