

PHASE INVERSION PREPARATION AND MORPHOLOGICAL STUDY OF POLYVINYLIDENE FLUORIDE ULTRAFILTRATION MEMBRANE MODIFIED BY NANO-SIZED ALUMINA

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Received 13 June 2009; Revised 9 July 2009

In this study, polyvinylidene fluoride (PVDF) membranes with various structures ranging from dense to highly asymmetric morphologies were obtained by changing the effective parameters in the phase inversion process. The effect of some important processing parameters such as solution concentration, harshness of the precipitation bath, exposure time before immersion in coagulation bath and bath temperature was studied and the mentioned parameters were optimized. The membranes were then modified with nano-sized alumina powder in order to improve the hydrophilicity of the PVDF membranes. The surface morphology, surface and cross-sectional structures of the membranes were examined by scanning electron microscope (SEM) and atomic force microscope (AFM). The specific surface area of the membranes was determined using the Brunauer–Emmett–Teller (BET) method. The contact angles between water and the membranes' surfaces were measured in order to study the hydrophilicity changes of the membrane surface. The results indicated that the addition of nano-sized alumina to the casting dope increased the hydrophilicity of the PVDF membrane surface.

Keywords: Polyvinylidene fluoride; membrane; morphology; phase inversion.

Ultrafiltration is widely used in many membrane separation processes, particularly in the wastewater treatment field, oil–water and protein effluent separation.^{1–7} Asymmetric polymer membranes are ideal candidates for these purposes. Polymeric membranes are usually produced by phase inversion techniques. In these techniques, an initially homogeneous polymer solution becomes thermodynamically unstable due to different external effects and phase separates into polymer–lean and polymer–rich phases. The polymer–rich phase forms the matrix of the membrane, while the polymer–lean phase, rich in solvents and nonsolvents, forms the pores.^{8,9} Polyvinylidene fluoride (PVDF) is a fluorinated thermoplastic polymer that can form such asymmetric membranes. This polymer is thermally stable and resistant to most mineral and organic acids and also oxidizing environments. PVDF membranes show outstanding anti-oxidation behavior, strong thermal and hydrolytic stabilities as well as UV stability and good mechanical and filmforming properties.^{10,11} PVDF membranes can thus be used in many ultrafiltration processes and applications especially in aggressive environments. The hydrophilicity and porous structure of the membrane play important roles in membrane separation processes. A suitable porous

membrane must have high permeability, good hydrophilicity, and excellent chemical resistance to the feed streams.¹⁰ In order to obtain high permeability, membranes should have high surface porosity and good pore structure. The asymmetric membranes are characterized by a very thin, but relatively dense skin layer supported by a more open porous sublayer.^{7,12}

In the present study, an effort was made to identify the dominant mechanisms responsible for the morphology development during phase separation of PVDF solution under different processing parameters including solution concentration, harshness of the precipitation bath, exposure time before immersion in coagulation bath and bath temperature. After optimization of the mentioned parameters, the membranes were modified with nano-sized alumina powder in order to improve the hydrophilicity of the PVDF membranes.

The morphology of the membranes was examined using Scanning Electron and Atomic Force Microscopy. The specific surface area of the membranes was determined by Brunauer–Emmett–Teller (BET) method. The contact angles between water and the membrane surface, before and after the modification with nano-sized alumina powder, were measured with a contact angle measurement apparatus.

Polyvinylidene fluoride (PVDF) with an average molecular weight of $534000 \text{ g}\cdot\text{mol}^{-1}$ as the membrane forming

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