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Optimization of preparation conditions of polyamide thin film composite membrane for organic solvent nanofiltration

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Abstract−Separation performance of polyamide composite membranes is affected by several parameters during formation of thin upper layer via interfacial polymerization. We investigated the effect of various polyamide synthesis conditions on the performance of organic solvent resistant polyamide composite membranes through the model equations designed by 2-level fractional factorial design. The dewaxing solvent recovery was selected as separation process. Five factors were changed in two level includin; TMC concentration (0.05-0.1%), MPD concentration (1-2%), support immersion time in organic solution (2-4 min), support immersion time in aqueous solution (1-2 min), and curing temperature (70-80 °C). The resultant equations showed 93.48% and 94.82% of the variability (R^2_{adj}) in data used to fit oil rejection and permeate flux models, respectively. The analysis of variance revealed that both models were high significant. It was also observed that TMC concentration, MPD concentration and immersion time in TMC have more pronounced effect on the oil rejection and permeate flux than other factors and interactions. Optimal polyamide preparation conditions were obtained using multiple response method for 94% oil rejection as target value. According to the results, the best value of permeate flux (8.86 $l/(m^2{\cdot}h)$) was found at TMC concentration of 0.1%, MPD concentration of 1.94%, immersion time in TMC of 3.88 min, immersion time in MPD of 1.95 min and curing temperature of 71.96 °C with desirability factor of 1.

Keywords: Organic Solvent Nanofiltration, Thin Film Composite Membrane, Factorial Design, Polyamide, Interfacial Polymerization, ANOVA

INTRODUCTION

Thin film composite (TFC) membranes prepared via interfacial polymerization (IP) have been used in membrane processes, such as reverse osmosis (RO) and nanofiltration (NF) [1,2]. Most of the commercial TFC membranes consist of upper ultra thin polyamide layer coated over porous support layer by interfacial polymerization of an aromatic polyamine in an aqueous phase and one or more polyacyl halides in an organic phase [3]. Among polyamide composite membranes, the one fabricated by IP of m-phenylenediamine (MPD) as diamine monomer and trimesoyl chloride (TMC) as acid chloride monomer shows the most successful performance for separation processes, especially in aqueous systems [4-7].

Although polyamide composite membranes are generally used in water and wastewater treatment, they have been found suitable for non-aqueous separation processes. There are a few studies which focus on the performance of polyamide thin film composite membranes in organic solvent separation [8-10].

Both support layer and ultrathin selective layer should be controlled and optimized to tailor superior TFC membrane performance. Among all polymers used for the fabrication of support layer, polysulfone and polyethersulfone have been more considered by researchers. Although PSf support membrane has widely been applied as support film of commercial TFC membranes, it has low chemical stability in harsh environment such as high or low pH medium, high temperature processes and organic solvent separation [4]. Therefore, other candidates such as PAN (polyacrylonitrile), PP (polypropylene), PI(polyimide), PEI (polyetherimide) introduced in literature to overcome these limitations [8-13]. In addition to chemical stability, surface chemistry (hydrophilicity) and morphology of support layer have an influential effect on permeability and selectivity of membrane [4]. To assess the relation between pore size distribution of PSf support membrane and polyamide layer properties, Singh et al. [14] found that PSf substrate with the pore size about 0.07 μm has higher salt rejection in comparison with one including larger pore size (0.15 μm). Furthermore, Gosh and Hoek [15] prepared PSf support membrane with wide range of hydrophilicity and pore structure, and presented conceptual models to give a comprehensive understanding of the impact of support on polyamide layer structure. In this case, TFC membrane with different permeability and selectivity through water treatment can be fabricated.

Apart from the support layer, separation performance of polyamide composite membranes can also be affected by several parameters during thin film polyamide formation, such as monomer concentration, reaction time, solubility and diffusivity of diamine monomer in organic solvent, and heat curing. Earlier, the dependency of polyamide composite performance on the abovementioned parameters was considered in literature by the one-factor-at-a-time experiment method which focuses on aqueous separations [5,7,16-20]. In this case, only one parameter is changed at a time and large number

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