



High performance thin film composite polyamide reverse osmosis membrane prepared via *m*-phenylenediamine and 2,2'-benzidinedisulfonic acid

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ABSTRACT

Thin film composite (TFC) polyamide membranes for reverse osmosis (RO) were successfully prepared via interfacial polymerization of *m*-phenylenediamine (MPD), 2,2'-benzidinedisulfonic acid (BDSA) and trimesoyl chloride (TMC) on a polysulfone support. The physico-chemical characteristics of the membranes were determined using ATR-FTIR, XPS, zeta potential measurement, SEM and AFM. Membrane performance experiments were investigated using NaCl and MgCl₂ salt solutions. The near region spectra show that the active layer of TFC membrane is an aromatic polyamide, with carboxyl and amino groups, as seen in the ATR-FTIR result. The sulfonyl group addition from the BDSA monomer is evident from the higher sulfur concentration as illustrated in the XPS results and due to more negative zeta potential values. Also, the SEM and AFM pictures show that the surface morphology became smoother as the BDSA content was increased. Due to the modification of the active layer, pure water permeability and rejection of NaCl and MgCl₂ salts were significantly enhanced.

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1. Introduction

Interfacial polymerization (IP), developed by Morgan et al. [1], is used to produce thin films such as polyamides (PA). This process is based on two monomers reacting in a two phase system, where the polymerization takes place in the interface between the aqueous and organic phases. Cadotte et al. [2] introduced the thin film composite (TFC) membranes, composed of a thin polymer film mounted on a porous support layer, via interfacial polymerization. Since the introduction of membrane FT-30 prepared via interfacial reaction between 1,3-benzenediamine and trimesoyl chloride, IP-based polyamide TFC membrane expanded worldwide [3]. Since then, the development of polymeric membrane materials has gone through three main stages: (i) the experimental trial-and-error synthesis and testing of polymers; (ii) opting for appropriate polymerization reactants based on a better understanding of polymer chemistry; and (iii) closely scrutinizing membrane morphology with the aid of advanced characterization tools [4].

Syntheses of polyamide thin film composites based from different chemistries have been performed to fabricate RO membranes with high flux and high rejection. One of the ways to obtain high flux is to incorporate new amines during PA polymerization. Adding sulfonated cardo poly (arylene ethersulfone) into *m*-phenylenediamine (MPD) has shown to significantly improve water permeability of PA membranes, which made use of the hydrophilic poly (arylene ether sulfone); meanwhile, the relatively high salt rejection was observed, and this was

attributed to the copolymer chain stiffness and high degree of cross-linking. Excessive incorporation of poly (arylene ether sulfone), however, led to an abrupt decline in salt rejection [5]. On the other hand, Li et al. synthesized some new acid chlorides to react with MPD instead of TMC, such as 3,4,5-biphenyl triacyl chloride, 3,3,5,5-biphenyl tetraacyl chloride, 2,2,4,4-biphenyl tetraacyl chloride and 2,2,5,5-biphenyl tetraacyl chloride [6,7]. The resultant membranes from 2,2,4,4-biphenyl tetraacyl chloride and 2,2,5,5-biphenyl tetraacyl chloride showed better flux (50.0 LMH and 54.2 LMH, respectively) but slightly lower salt rejection (~97.5%), compared with MPD–TMC membranes (37.5 LMH and 98.4%) [7]. Similarly, 5-isocyanato-isophthaloyl chloride polymerized with MPD produced membranes with higher water flux and salt rejection rate than the traditional commercial MPD–TMC membrane [8].

An effective approach for improving water permeability is attachment of hydrophilic ionic groups (such as sulfonate or carboxylate moieties) onto the polymer [9–14]. Example of such is the synthesis of ternary copolyamides from aromatic diamines with a hydrophilic group such as 3,5-diaminobenzoic acid and 2,4-diaminosulfonic acid, 3,3- or 4,4-diaminodiphenylsulfone and iso- or terephthaloyl chloride. In this study, a mixture of 3,3-diaminodiphenylsulfone and 3,5-diaminobenzoic acid was dissolved in NMP and was cooled below 5 °C [13]. Also, aromatic polyamides containing sulfonic groups were synthesized via dissolution of bis-(4-aminophenyl) ether and 2,4-diamino-benzenesulfonic acid and cooling down to –10 °C under vigorous stirring with isophthaloyl chloride [14]. These studies [13,14], however, were obtained by low temperature solution polymerization and not by interfacial polymerization.

In this study, interfacial polymerization of thin film composite polyamide membranes for reverse osmosis has been successfully prepared

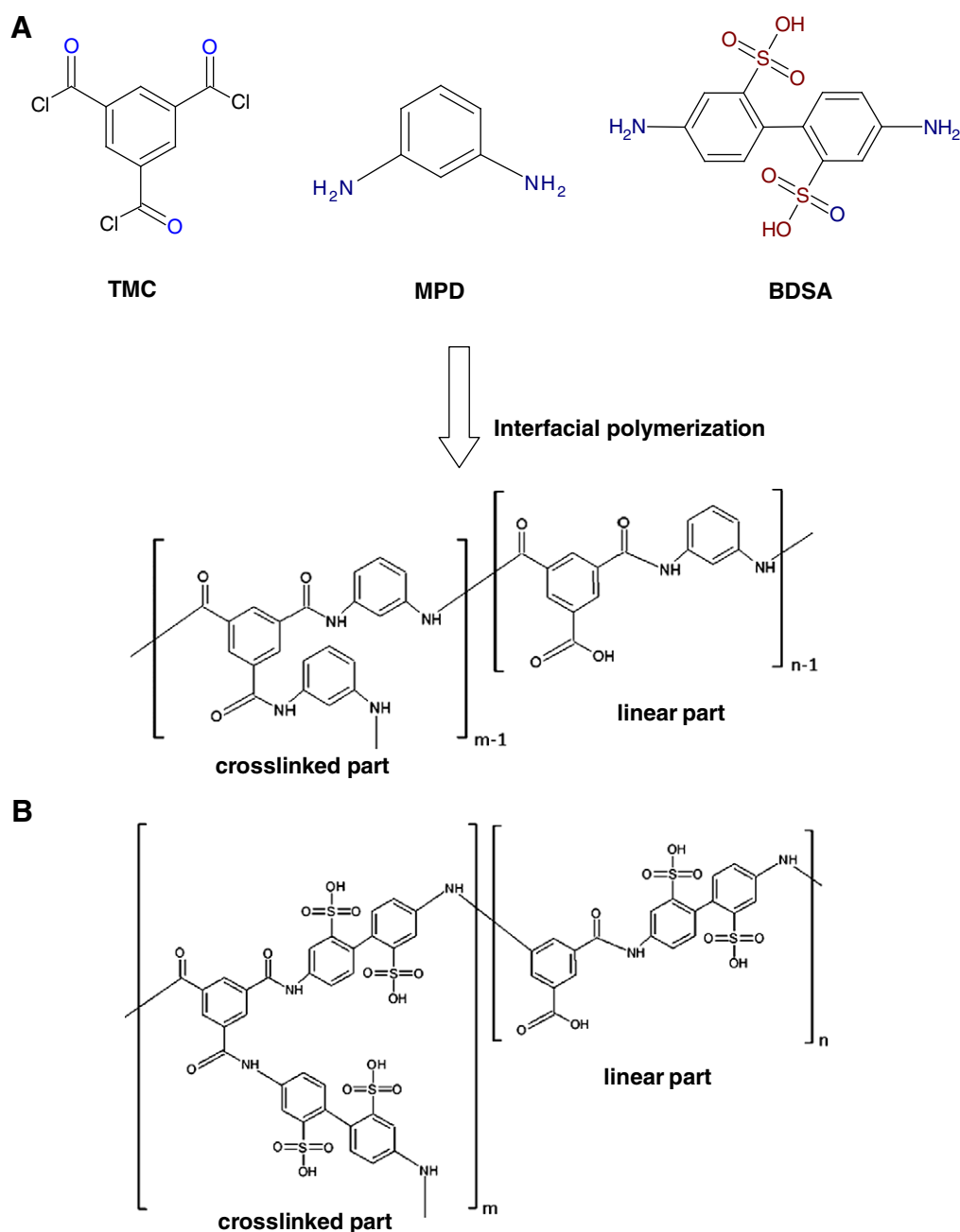
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using *m*-phenylenediamine (MPD), 2,2'-benzidinedisulfonic acid (BDSA) and trimesoyl chloride (TMC). The BDSA monomer is incorporated in the interfacial polymerization of common polyamide monomers, namely MPD and TMC, to be a source of sulfonate groups that will enhance water permeability and increase the overall negative charge of the membrane. The polymerization reaction of the three monomers is shown in Fig. 1. Fig. 1A shows the polymerization product of MPD and TMC monomers while Fig. 1B shows the product of BDSA and TMC. The modified polyamide TFC membranes were characterized using infrared and X-ray photoelectron spectroscopies, SEM and AFM, zeta potential and permeation experiments. The improved RO performance in relation to chemical structure, morphology, and charge effect was investigated.

2. Experimental

2.1. Materials

m-Phenylenediamine, $\geq 99\%$ (MPD) and 1,3,5-benzenetricarbonyl chloride, 98% (trimesoyl chloride) (TMC) were purchased from Sigma Aldrich, Korea. 2,2'-Benzidinedisulfonic acid (contains 30% water at maximum) (BDSA) (Tokyo Chemical Industry Co., Ltd., Japan) was received and used without further purification. The commercial polysulfone support used was from Woongjin Chemical Co., Ltd., Korea. Deionized water purified using MilliQ system (Millipore, USA), with resistivity greater than $18 \text{ M}\Omega \text{ cm}^{-1}$, was used throughout the experiment.



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