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Innovative high flux/low pressure blend thin film composite membranes for water softening

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ABSTRACT

Nanofiltration (NF) thin film composite membranes were prepared via interfacial polymerization using piperazine with isophthaloyl chloride on porous PES/PAN blend membrane. The prepared membranes were characterized through chemical structure, morphology, surface charge studies and separation performance. All the variables are taken into consideration including reactive monomers amount. The monomers ratio were 1: 0.3: 0.3: 0.3 that corresponds to piperazine: sodium hydroxide: dodecyl sodium sulfate: isophthaloyl dichloride respectively. Soaking time for amine solution was 30 min, while it was 10 min in organic chloride solution. After interfacial polymerization the membranes have been annealed at temperature 80 °C for curing time 10 min. These were the optimum conditions which greatly enhanced the flux and the salt rejection of NF thin film composite membranes. The membranes have rejection percent in the order of 99.9, 99, 82, and 72% for Na_2SO_4, $MgSO_4$, $CaCl_2$, NaCl respectively without reducing water productivity. This study offers separation of 7000 ppm salt solution with low operating pressure (5 bars) compared with previous studies. The fabricated membranes were applied for 30 days with steady-state flux keeping their polyamide layers. As well; the fabricated membrane with the optimum operating conditions has a better fouling resistance for humic acid and molecular weight cut off 600 Da.

1. Introduction

For water softening, there are several techniques such as ion- exchange resin, zeolite, and lime-Soda ash. Meanwhile, the first technique has been restricted due to the high sodium chloride content in the produced water and the required resin recovery, while the last one produced a lot of waste sludge that required huge land utilization [1–4].

Recently, nanofiltration (NF) highly produced with low-vitality utilization pressure-driven membrane technique that has characterized by safety operating conditions for the environment and applied in numerous applications. For example, partial water softening, saline water desalination, dye removal, lowering chemical oxygen Demand (COD), and thus on [5–8].

There are numerous techniques for NF membranes fabrication in the form of thin film composite membranes (TFCMs) or typical asymmetric membranes such as the phase inversion [9], photo-initiated polymerization [10], chemical cross-linking [11], interfacial polymerization (IP) [12], electrostatic layer-by-layer (LbL), and self-assembly [13].

TFCMs have been improved both selectivity and efficiency with less vitality utilization. So, most commercialized NF membranes uses thin film composite (TFC) flat sheet membranes that had been made by the development of a polyamide (PA) selective layer on the surface of a permeable substrate by IP of aqueous di-amine solution with acyl chloride in the organic solvent such as NF series by Dow, the UTC series from Toray, and the Desal series by GE-Osmonics [14,15]. Meanwhile, many researchers explored different strategies to improve NF membranes fabrication including adjusted IP [5,16], surface grafting [10], and incorporation of nanomaterials [17].

The thin polyamide (PA) selective layer that covers the permeable substrate is the fundamental element of the TFC membrane with a variable thickness between 0.2 μm – 0.3 μm [18,19].

Several NF TFCMs with a variable thickness structure were prepared by IP of piperazine (PIP), *m*-phenylene diamine (MPD) alone or in the blend as diamines, and acyl chlorides similar to trimesoyl chloride (TMC) and isophthloyal chloride (IPC) individual or mixed [20].

Improving NF membranes with high flux rate and great dismissal capacity would extraordinarily build the effectiveness of membrane innovation. Large studies have been centered on enhancing the thin selective layer, for example, usage of new monomers [20–22] and surfactants [23] through IP technique.

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Table 1
The ratios of PIP: NaOH: SDS.

Membrane	PIP	NaOH	SDS
TFCM	0.5	0.2	0.2
TFCM 1	0.75	0.3	0.3
TFCM 2	1.5	0.3	0.3
TFCM 3	1	0.4	0.4
TFCM 4	1	0.3	0.3

Recently, the effect of the substrate properties on the fabrication of TFC membranes and high permeability has been taken into account. Few reviews were directed to explore the relation between the features of the substrate and the preparation of the PA selective layer [24,25]. In the previous studies, the interfacial polymerization was carried out using PSF, PES, PEI, and PAN as membrane substrates [26].

Abdallah et al., [27] manifested that the PES/PAN blend membrane had permeate flux and rejection percent for humic acid 57 LMH and 82% respectively at 2 bar.

In this study, modification and development of the previously fabricated UF blend membrane is presented. Where, PA selective layer is introduced on the porous PES/PAN through IP process of PIP and IPC. The physicochemical properties of TFCMs were fully presented by SEM, the PA layer thickness, FTIR, and the NF performance. Accordingly, the current study would give a hand in the enhancement of better TFCMs including NF, membranes.

To the best of our knowledge, no studies have been conducted so far to study the optimization conditions of the PA separating layer on the porous blend membrane. So, the main objective of this study is to optimize the preparation conditions of the thin PA layer formed on the top of the blend UF membrane and to compare the membrane performance of TFC blend substrate with that of TFC neat PSF, PES, and PAN membranes.

2. Materials and methods

2.1. Materials

The polymer materials to fabricate the UF substrate for TFC membrane were Polyethersulfone (PES with MW = 58,000 g/mol-BASF Company -Germany) and Polyacrylonitrile (PAN with MW = 150,000 g/mol-SigmaAldrich). Piperazine hexahydrate (PIP) and isophthaloyl dichloride (IPC) were purchased from Sigma Aldrich and Merk-Germany respectively (purity > 99%) which are used to establish the PA active layer on the substrate. Sodium hydroxide (NaOH) and inorganic salts $MgSO_4$, $CaCl_2$, $NaSO_4$, NaCl for membrane evaluation were purchased from Sigma Aldrich (purity > 98%). N-hexane was obtained from Acros Organics (USA). Sodium dodecylsulphate (SDS) was obtained from Merk-Germany (purity > 99%). Potassium iodide and iodine were supplied by Sigma Aldrich (purity > 98%). Polyethylene glycol (PEG) with nominal MW of 400, 600, 1000, 3000 and 6000 Da were obtained from Sigma Aldrich. Hydrochloric acid (HCl) (purity > 98%) was purchased from Sigma Aldrich.

2.2. Fabrication of TFC nanofiltration membrane

2.2.1. Preparation of UF PES/PAN membranes as substrate

UF PES/PAN membrane with ratio PES: PAN (12.5:1.5 wt%) was prepared via inversion process according to Abdallah et al., [27] and used as a substrate to produce TFC nanofiltration membranes by surface coating via IP process.

2.2.2. Preparation of polyamide active coat over the substrate

The TFC nanofiltration membranes were fabricated via IP process between PIP and IPC through several optimization steps. Initially, the fabricated substrate UF PES/PAN blend membrane was immersed in deionized water for 1 h then the membrane was dried in air for optimization steps.

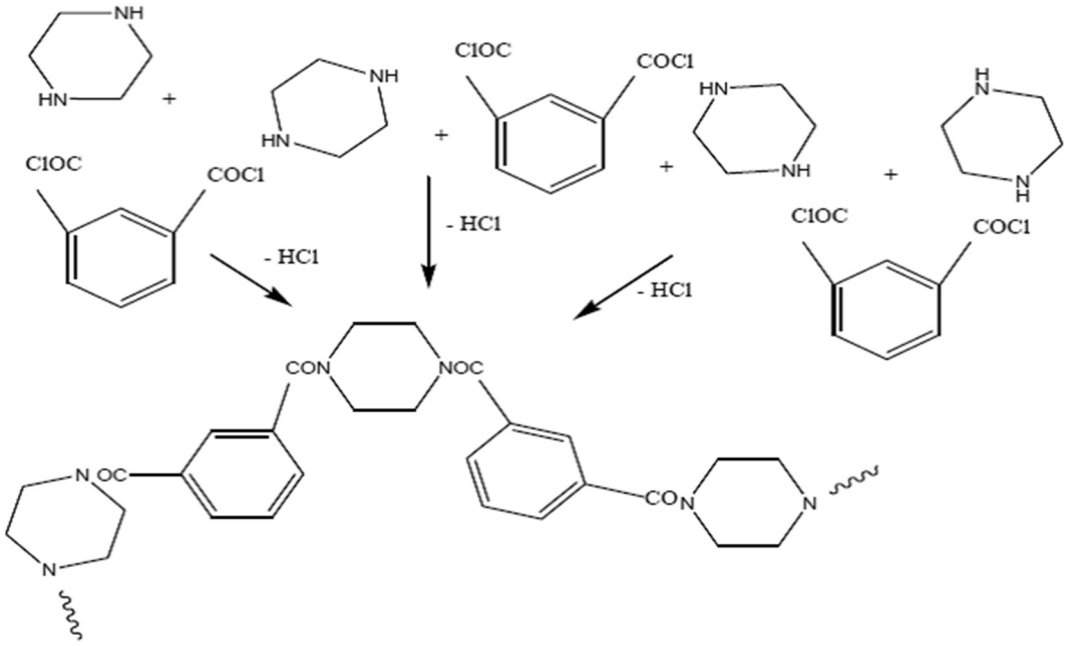

Fig. 1. Scheme for the interfacial polymerization between PIP and IPC.