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ScienceDirect

Procedia Engineering

Procedia Engineering 148 (2016) 588 - 593

www.elsevier.com/locate/procedia

4th International Conference on Process Engineering and Advanced Materials

Synthesis and Characterization of Polyethersulfone/Carbon Molecular Sieve Based Mixed Matrix Membranes for Water Treatment Applications

Danial Qadir*, Hilmi Mukhtar, Lau Kok Keong

Department of Chemical Engineering, Universiti Teknologi PETRONAS, 32610 Seri Iskandar, Perak Darul Ridzuan, Malaysia

Abstract

Novel mixed matrix membrane was prepared by incorporating the carbon molecular sieves (CMS) into polyethersulfone (PES) matrix. Flat sheet membranes of different filler concentrations were synthesized through phase inversion technique. Scanning electron microscope and Thermogravimetric analyzer were used to investigate the morphology and thermal stability of synthesized membranes respectively. Finally membranes were tested for their pure water flux and sodium chloride (NaCl) rejection (100ppm aqueous solution). Investigation has shown that all synthesized membranes had asymmetric structure with thin dense top and well-defined macropores in sublayer. It is noticed that inclusion of inorganic filler has improved the thermal stability as well as pure water flux of mixed matrix membranes (upto 33.8LMH at 3 bar). Moreover, synthesized mixed matrix membranes also showed better NaCl rejection (upto 26.13% at 3 bar) than pure polymeric membranes.

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Peer-review under responsibility of the organizing committee of ICPEAM 2016

Keywords: Mixed matrix membranes, carbon molecular sieves, polymeric membranes, performance of mixed matrix membrane

1. Introduction

For the past several decades, synthetic polymeric membranes are being used for a wide variety of liquid separations such as microfiltration, ultrafiltration, reverse osmosis, nanofiltration. A common method for the preparation of polymeric membranes is the phase separation process [1]. Different techniques have been employed over the years to improve the different properties membranes to make it viable for range of applications [2]. Inclusion of inorganic material to enhance the properties such as flux, antifouling property, thermal and mechanical properties etc. Inclusion of any filler in form of disperse form in continuous matrix is defined as mixed matrix membrane [2, 3]. Polyethersulfone (PES) is widely used as a membrane material because of its commercial availability, processing ease, favorable selectivity, permeability characteristics and good mechanical and thermal properties. PES is an amorphous glassy and hydrophilic polymer containing sulfone groups [4]. Carbon molecular sieve (CMS) is added to this matrix a an inorganic filler to achieve the higher flux and higher retention since carbon based sieves are reported to be good adsorbent for heavy metal ions and also provide excessive filtration area for permeation [5, 6]. In this study, polyetehrsulfone is used as continuous matrix whereas carbon molecular sieve is added to polymer matrix as an inorganic filler to achieve a better overall performance in terms of flux and salt retention at low pressures.

* Corresponding author. Tel.: +601129155624 *E-mail address*: danial2715@gmail.com

2. Methodology

2.1. Materials

Polyethersulfone (PES) was used as polymer to synthesize the membrane and it was purchased from BASF Co. (Ultrason® E6020P). Since, PES is hydrophilic in nature and absorbs moisture rapidly; it was dried for 5 hrs in a dry oven at 100 °C before its use in casting solution preparation. N-methyl-2-pyrrolidone (NMP) from Merck Co. was used as solvent. Deionized water at room temperature was used as a non-solvent in coagulation bath. Sodium chloride (NaCl-pure) was purchased from R & M chemicals Ltd. to study the membranes rejection for sodium salt. Carbon molecular sieve was purchased in granular form from Japan Enviro Chemicals, Ltd. and then it was grinded to achieve the desired size for mixed matrix membrane preparation.

2.2. Flat Sheet Membrane Preparation

Casting solution for flat sheet membrane was prepared by dissolving 15wt% PES into 85wt% of NMP. This solution was then stirred overnight at 200 rpm for complete dissolution of polymer into solvent. Then prepared solution was kept for 45 mins to remove any bubbles from solution. Flat sheet membranes were prepared by pouring the casting solution on immaculate glass plates and membranes were cast using casting knife at thickness of 200 microns. Glass plates were then immersed in water bath till membranes came off naturally after solvent exchange. Afterwards, synthesized membranes were put into separate water (deionized) bath for complete removal of solvent.

2.3. Carbon Molecular Sieve (CMS) Powder Preparation

Carbon molecular sieve was grinded by "Mortar Grinder" (RockLab) for 90 mins. Particle size analysis and distribution was determined by using "Mastersizer" particle size analyzer. After achieving the distribution of CMS particles, mechanical sieving was done using (63 microns) sieve.

2.4. Mixed Matrix Membrane Synthesis

For synthesis of mixed matrix membrane, known amount (1 wt%) of inorganic filler was dissolved into solvent (NMP) for 45 mins. at 200 rpm. 10% of polymer was then added to the casting solution and solution was stirred at 200 rpm for 1 hr. Later, remaining polymer was added to the solution and it was left for overnight stirring. Casting solution was then kept at room temperature for air bubbles removal for 45 mins. Finally, solution was sonicated for 45 min before membrane casting. Membrane casting was done on glass plates at 200 microns thickness with Elcometer casting knife. Table 1 presents the casting solution composition of synthesized membranes.

Membrane Code	PES	CMS	Casting Method	Casting Thickness (µm)
	(wt%)	(wt%)		
E5	15%	0%	wet phase inversion	200
E5C1	15%	1%	wet phase inversion	200
E5C5	15%	5%	wet phase inversion	200

Table 1. Composition of Casting Solution Used for Membrane Synthesis

2.5. Characterization

2.5.1. Scanning Electron Microscope (SEM)

Surface morphology and cross section of prepared membranes was carried out using Variable Pressure Scanning Electron Microscope (VPFESEM, Zeiss Supra55 VP). All membrane samples were dipped in liquid nitrogen before imaging to achieve clean samples. Samples were attached to plates with two sided adhesive tapes in a lateral position. Membrane thickness and structural information was evaluated through SEM analysis.

2.5.2. Thermogravimetric Analyzer (TGA)

Thermogravimetric analysis was accomplished to determine the thermal stability of the modified and unmodified membranes with PERKEN ELMER simultaneous Analyzer STA6000 under nitrogen atmosphere 100ml/min at a heating rate of 10°C/min.

2.5.3. Filtration Experiment

Performance of flat sheet polymeric and mixed matrix membrane was tested using a stirred dead end filtration cell (UHP-90) from ADVANTEC. A 90 mm diameter circular membrane cut was used for testing while active surface area of membrane was

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