



Effect of MCM-48 nanoparticles on the performance of thin film nanocomposite membranes for reverse osmosis application



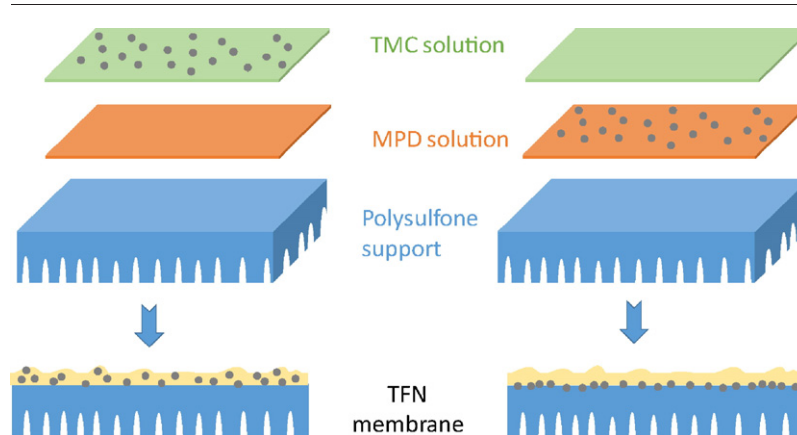
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HIGHLIGHTS

- MCM-48 nanospheres with 3-D pore structure were used to modify the TFC membrane.
- The TFN membranes were prepared by dispersing MCM-48 in the aqueous or organic phase.
- The hydrophilicity of MCM-48 modified TFN membranes was higher than TFC membrane.
- MCM-48 modified TFN membranes exhibited improved desalination performance.
- MCM-48 is stable in TFN membranes by dispersing ether in organic or aqueous phase.

GRAPHICAL ABSTRACT



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ABSTRACT

Monodispersed spherical MCM-48 nanoparticles with three-dimensional cubic mesostructure were synthesized and used as nanofillers dispersed in the aqueous or organic phase to prepare thin film nanocomposite (TFN) membranes through the interfacial polymerization of *m*-phenylenediamine and trimesoyl chloride. Scanning and transmission electron microscope images of the isolated polyamide (PA) active layer confirm that MCM-48 nanoparticles were embedded throughout the PA layer with dispersion in the organic phase as they were clipped between the PA layer and the polysulfone support with dispersion in the aqueous phase. Water flux was measured at 16 bar with 2000 ppm NaCl solution. Furthermore, water flux increased gradually from 24 L/m²·h to 40 L/m²·h with the increase in MCM-48 content in the organic phase, without significantly affecting salt rejection (>95%). Water flux increased from 24 L/m²·h to 68 L/m²·h, whereas salt rejection decreased from 97% to 80% with the increase in MCM-48 content in the aqueous phase. Less MCM-48 can be used in the aqueous phase than in the organic phase with similar performances. The two kinds of TFN membranes exhibit enhanced long-term durability, which confirms that MCM-48 can stably reside in the TFN membranes by dispersing whether in the organic or aqueous phase.

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1. Introduction

The shortage of fresh water is a global, long-term challenge because of the exponential population growth and the negligent wastewater

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management. Owing to enormous resources of seawater, desalination of seawater is considered the most sustainable and adequate solution to alleviate water scarcity [1]. Current desalination processes are divided into two major categories, namely, thermal distillation and membrane separation [2]. Compared with the membrane desalination technology, the conventional thermal desalination process is more robust, but is inefficient in its use of energy and particularly suffers from corrosion [3]. Therefore, membrane desalination currently dominates the global market because of its relatively lower energy consumption and operational simplicity [2].

Polyamide (PA) thin film composite (TFC) reverse osmosis (RO) membranes, which consist of a microporous polymer membrane on polyester nonwoven fabric as support and an ultrathin PA barrier layer, are applied worldwide because of their high water flux and excellent rejection abilities [4]. Despite the significant improvements in TFC membranes, shortcomings, such as fouling and permeability, still hinder their applications. Increased permeability leads to reduced membrane area, and consequently, a reduction in membrane replacement costs and cleaning chemical usage [5]. Moreover, the increase in permeability can lessen the pressure necessary to drive permeation, thereby diminishing the energy demand of RO desalination [1]. Fouling resistance leads to less frequent rinse and membrane replacement. Therefore, the development of fouling-resistant membranes with high water flux will improve energy usage and decrease water cost [1].

A new concept of interfacial polymerization of thin film nanocomposite (TFN) RO membrane via embedding zeolite nanoparticles in the PA thin film layer has been reported [6]. The water permeability of RO membranes was dramatically improved without any significant change in salt rejection. Other nanomaterials, such as nanoparticles of zeolite [7–9], silica [10], MCM-41 [11], mesoporous carbon [12], reduced graphene oxide/TiO₂ [13], and zeolitic imidazolate framework-8 [14], and nanotubes of aluminosilica [15], titanium [16], halloysite [17], and carbon [18], were used as nanofillers to prepare the TFN membranes afterwards. All resultant TFN membranes exhibited improved performance, particularly increased water flux. Water flux was enhanced because of the superhydrophilicity and the low crosslinking density of the PA layer. In addition, the internal porosity of the nanoparticles/nanotubes provides flow paths for water molecules through the nanocomposite thin film layer. Several researchers observed that TFN membranes possess antifouling properties because of its high hydrophilicity [8,19,20]. Therefore, TFN membranes with high water flux and fouling resistance have become the new generation of membrane utilized to decrease water costs. The nanofillers have a critical role in improving the performance of the membrane. Therefore, ascertaining the position and the action of nanofillers in the TFN membrane is necessary.

MCM-48 nanoparticles with three-dimensional (3-D) cubic *1a3d* channel network will serve as a highly opened porous host for the easy and direct access of guest species [21]. Compared with two-dimensional (2-D) hexagonal *p6mm* MCM-41, guest species can more easily diffuse throughout the pore channel of MCM-48 without pore blockage [21]. MCM-41 has been adopted as nanofiller to prepare TFN membranes with enhanced performance. To the best of our knowledge, MCM-48 has not been used as nanofiller for TFN before.

In this study, MCM-48 with controlled particle size was prepared and used to modify TFC RO membranes. Scanning electron microscope (SEM), X-ray diffraction (XRD), and nitrogen adsorption-desorption were used to characterize the morphology, pore structure, and pore size of MCM-48 nanoparticles, respectively. MCM-48 nanoparticles were dispersed in the organic or aqueous phase to prepare the TFN membrane. Surface morphology, roughness, and hydrophilicity of the TFN membranes were characterized by SEM, atomic force microscope (AFM), and contact angle analyzer. The PA thin layer was separated from the polysulfone support and was characterized by SEM and transmission electron microscope (TEM) to ascertain the position and distribution of the MCM-48 nanoparticles in the TFN membrane. The effects of different concentrations of MCM-48 nanoparticles in various phases

on the desalination performance of TFN RO membranes were evaluated on a flow cell test kit with 2000 ppm NaCl aqueous solutions at 16 bar and 20 °C.

2. Experimental

2.1. Materials and reagents

Triblock copolymer F127 (EO₁₀₆PO₇₀EO₁₀₆; Sigma-Aldrich, USA), tetraethyl orthosilicate (TEOS; Aladdin), cetyltrimethylammonium bromide (CTAB; Aladdin), ammonium hydroxide (NH₄OH; Sinopharm Chemical Reagent Co. Ltd.), and ethanol (Sinopharm Chemical Reagent Co. Ltd.) were used to synthesize MCM-48 nanocomposite. Trimesoyl chloride (TMC; TCI Co. Ltd.), *m*-phenylenediamine (MPD; Sigma-Aldrich), sodium dodecyl sulfate (SDS, Sigma-Aldrich), and *n*-hexane (Sinopharm Chemical Reagent Co. Ltd.) were used to prepare the PA layer on the polysulfone support. Polysulfone substrate was purchased from the Hangzhou Water Treatment Center (Hangzhou, China). All chemicals were of analytical grade and used without further purification.

2.2. Preparation of MCM-48 nanoparticles

MCM-48 nanoparticles were synthesized by using CTAB as a structure directing agent, TEOS as a silica source, and F127 as a particle dispersion agent under basic conditions [21]. In a typical synthesis procedure, 0.5 g of CTAB and 3.4 g of F127 were dissolved in a mixture of 96.0 mL of deionized water, 34.0 g of EtOH, and 10.0 g of 25–28 wt% ammonium hydroxide solution to provide a clear solution at 30 °C. Afterward, 1.8 g of TEOS was immediately added to the reaction mixture. After 1 min of stirring at 1000 rpm, the mixture was kept at a static condition for 24 h for further silica condensation. The white solid product was recovered through ultrahigh speed centrifuge and was washed with water. Samples were dried at 70 °C in air. Finally, the sample was calcined in air at 550 °C for 5 h to remove surfactants.

2.3. Preparation of MCM-48 incorporated TFN membranes

The MCM-48 incorporated TFN RO membrane was obtained through the interfacial polymerization of MPD with TMC. The MCM-48 nanoparticles were incorporated into the membrane by dispersing MCM-48 (0.004% to 0.14% w/v) in the aqueous phase (2% w/v MPD and 0.15% w/v SDS in deionized water) or organic phase (0.1% w/v TMC in hexane) using ultrasonication prior to the occurrence of the interfacial polymerization reaction. The MPD aqueous solution was poured on the polysulfone support membrane surface and allowed to soak for 2 min. Afterward, the membrane was drained and air dried for 9 min. Then, the TMC hexane solution was poured on the top surface of the membrane for 1 min, which formed a thin film over the surface of the polysulfone support membrane. Afterward, the hexane solution was drained and the membrane was heat cured at 115 °C for 3 min in an oven. Finally, the membrane was stored in deionized water for the performance test.

Notably, the TFC membrane is meant to represent the PA TFC membrane without MCM-48, whereas the TFN-*x*-A membrane denotes the MCM-48/PA TFN membrane with dispersion of *x*% w/v of MCM-48 in the aqueous phase and the TFN-*x*-O membrane denotes the MCM-48/PA TFN membrane with dispersion of *x*% w/v of MCM-48 in the organic phase.

2.4. Characterization methods

The XRD of the MCM-48 sample was recorded on a Bruker D8 ADVANCE instrument (40 kV, 100 mA) equipped with a Cu K α radiation within the range of $2\theta = 1^\circ$ to 7° at the rate of $1^\circ/\text{min}$. The nitrogen

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