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Activated carbon nanoparticles entrapped mixed matrix polyethersulfone based nanofiltration membrane for sulfate and copper removal from water

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ABSTRACT

Mixed matrix activated carbon nanoparticles (ACNPs) embedded polyethersulfone based nanofiltration membranes were prepared by solution casting technique. SEM images show that utilization of nanoparticles in the membrane matrix causes a decrease of the size of the channels in both top and sub layers. SOM images show a uniform nanoparticle distribution for the prepared membranes. SOM images also show agglomeration of nanoparticles at high additive concentrations. The surface images show a reduction of roughness for membranes filled with ACNPs. The flux decreases at 0.05 wt% nanoparticles loading rate and then increases again by an increase of the nanoparticles dosage from 0.05 to 0.1 wt%. The flux then decreases again at a nanoparticles ratio from 0.1 to 1 wt%. According to the performance test, the membrane with 0.5 wt% nanoparticles indicated the highest sulfate (95%) and Cu (97%) ions removal. The water contact angle was found to decrease from 54° to 43° by increasing the ACNPs concentration. This is assigned to a decrease of the membrane surface roughness due to migration of nanoparticles to the membrane surface during the fabrication process. The water content in the membrane and the porosity were also improved by increasing the nanoparticles ratio up to 0.1 wt%, but decreased for higher additive concentrations. The tensile strength of the membranes was enhanced by utilizing a nanoparticles ratio up to 0.05 wt%, but showed a decreasing trend for higher nanoparticles concentrations. The decrease of the flux ratio (J/J_o) was measured to be 5% for the modified membrane containing 0.5 wt% ACNPs and 63% for the pristine membrane.

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1. Introduction

Nanofiltration (NF) is attractive and effective for the treatment of effluents, especially the removal of heavy metal ions from water, compared to treatment techniques such as adsorption, electrochemical oxidation, ion-exchange and coagulation-flocculation. NF is also useful for concentration/purification in the pharmaceutical and chemical products industries [1–3]. This technique offers several advantages compared with other separation methods such as a relatively low investment cost, low energy consumption, high permeation flux and removal efficiency and singular separation capability for ions of different valences [4–6]. Sulfate and copper ions, harmful contaminants of potable water resources, should be removed because of their risks for humans, plants growth and scaling problems in industrial applications [1,4,5].

Nowadays, various methods have been used for modification of NF membranes with special characteristics in chemical and waste treatment applications such as polymers blending, polymers photo-grafting, plasma treatment, chemical modification, use of different filler additives and many more [7–14].

Elimination of sulfate and copper ions by nanofiltration membrane has been reported. Adsorptive membranes prepared by blending adsorptive polymers such as chitosan and incorporating nanoparticles such as metal oxides and magnetic nanoparticles in the membrane matrix can improve the membrane performance in sulfate and copper ions removal from water [1,4,15–18].

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Utilizing inorganic particles or fillers, especially nanomaterials, into polymeric NF matrices has been examined in many applications to enhance their stability in demanding conditions and the separation properties of membranes, based on a synergism between the organic and inorganic components. An increase of thermal, chemical and mechanical stabilities and an improvement of hydrophilicity, water permeability and rejection and antifouling properties of NF nanocomposite membranes has been observed [2,7,8].

Currently only few studies considered incorporating activated carbon nanoparticles (ACNPs) into NF membranes and the literature is silent on the characteristics of mixed matrix PES based nanofiltration membranes prepared by using ACNPs for sulfate and copper ions removal from water. In this study, activated carbon nanoparticles embedded mixed matrix asymmetric polyethersulfone based nanofiltration membranes were prepared by solution casting to increase the removal efficiency of sulfate and copper ions from water.

Activated carbon nanoparticles are a new class of advanced materials with good chemical stability, high surface area, and good activation/adsorption characteristics due to its unique structure and functionality. These make it to an attractive additive in water treatment [19].

Polyethersulfone (PES) is one of the most used polymers in the preparation of commercial and laboratory nanofiltration membranes. Its thermal stability and outstanding mechanical strength have made this polymer an interesting material in membrane fabrication [20,21].

The use of hybrid systems making use of a membrane with nanoparticles can create additional properties to increase the efficiency of the system based on the synergism between the properties of the different components.

The influence of the concentration of activated carbon nanoparticles in the casting solution on physico/chemical properties, separation characteristics and antifouling ability of PES based nanofiltration membranes was investigated.

2. Materials and methods

2.1. Materials

Polyethersulfone (PES) provided by (BASF) (Ultrason E6020P, MW = 58,000 g/mol), New Jersey, USA, polyvinylpyrrolidone (PVP, MW = 25,000 g/mol) and N, N Dimethyl acetamide (DMAc, Mw = 87.12 g/mol) provided by Merck Inc., Darmstadt, Germany and deionized water were used as membrane base polymer, pore former, solvent and non-solvent, respectively. Activated carbon nanoparticles (ACNPs), (Bamboo, black powder, spherical, specific surface area (SSA) >1000 m^2/g, iodine adsorption >1000 mg/g, methylene blue number >240 mg/g, negative-ions concentration: 7150/cm^3, decoloration rate >99%, electrical conductivity: 0.4 Ω cm, high activation/adsorption capability, average particle size <100 nm) were obtained from US Research Nanomaterials, Inc., Houston, USA., and used as inorganic filler additive. All other chemicals were supplied by Merck Inc., Darmstadt, Germany.

2.2. Preparation of ACNPs entrapped mixed matrix PES based NF membrane

Asymmetric flat sheet PES base membranes were fabricated by phase inversion induced by the immersion precipitation technique. To this aim, certain amounts of PES (18 wt%) and PVP (1 wt%) were dissolved in DMAc by mechanical stirring for 4 h with a mechanical stirrer (Velp Scientifica Multi 6 stirrer). Afterwards, activated carbon nanoparticles were dispersed in various concentrations into the polymeric solutions. Dispersing was followed by sonication for 1 h in an ultrasonic cleaner bath (Parsonic 11S model, S/N PN-88159, Iran) for breaking up any aggregates between the nanoparticles. The prepared solutions were kept for about 4 h at room temperature without stirring in order to completely remove the air bubbles and then they were cast onto clean glass plates with a film applicator with constant thickness of 150 µm. Subsequently, the glass plates were immersed into distilled water parallel to horizontal plane at ambient temperature. After primary phase separation and membrane solidification, the membranes were kept in fresh distilled water for 24 h to ensure the complete solvent extraction. Then, the membranes were placed between two filter paper sheets for 24 h at room temperature for drying. The composition of the polymeric solutions is shown in Table 1.

Table 1
Composition of casting solutions used in fabrication of membranes.

Samples (No)	PES (wt%)	PVP (wt%)	DMAc (wt%)	ACNPs (wt%)
1	18	1	81	0
2	18	1	80.95	0.05
3	18	1	80.9	0.1
4	18	1	80.5	0.5
5	18	1	80	1

2.3. Characterization of membranes

2.3.1. Scanning electron microscopy (SEM)

The cross-sectional morphology of membranes was observed using a SEM (Seron Technology Inc. Korea) instrument. Before taking SEM images, the membranes were dipped in liquid nitrogen for 5 min and then all the samples were gold sputtered and carefully handled to avoid contaminations. In this way, the membranes were prepared for SEM images and images were taken at 15 kV in high vacuum conditions.

2.3.2. Scanning optical microscopy (SOM)

Scanning optical image analyzing was used for surface characterization and investigation of the distribution of nanoparticles in the prepared membranes. The instrument used for this aim was Olympus; model IX 70, transmission mode with light going through the sample. For taking SOM images, small squares of prepared membranes (1 cm × 1 cm) were cut and placed between two glassy blades.

2.3.3. Three dimensional (3D) surface images

In order to study the surface morphology of the fabricated membranes and the effect of roughness on the separation efficiency, 3D surface images were prepared using optical microscopy and SPIP software (version 6.4) in the area of 8 µm × 10 µm.

2.3.4. Water contact angle

In order to observe the membrane surface hydrophilicity, water contact angles were measured statically by using a contact angle measuring instrument. De-ionized water was the probe liquid in this test. Measurements were done shortly after drop formation on the membrane surface. To minimize the experimental errors, the contact angle was measured in three different locations for all membranes; the average was reported.

2.3.5. Water content

The water content was measured as the weight difference between dried and wet membranes. For the purpose, prepared membrane were cut in square pieces (4 cm × 4 cm) and immersed in distilled water for 72 h. After discharging the samples, their surface were wiped by filter paper and weighed (OHAUS, Pioneer™, readability: 10^{-4} g, OHAUS Corp., USA). The wet membranes were

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