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Preparation, characterization and performance of Al_2O_3/PES membrane for wastewater filtration

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ABSTRACT

Membrane bioreactors (MBRs) have been widely used as advanced wastewater treatment processes in recent years. However, membrane fouling and its consequences in terms of plant maintenance and operating costs limit the widespread application of MBRs. Thus great efforts have focused on fouling mitigation. In this study, Al_2O_3 entrapped polyethersulfone (PES) ultrafiltration membranes were prepared and applied to activated sludge filtration in order to evaluate their fouling characteristics. The impact of solvent evaporation time and polymer concentrations on ultrafiltration (UF) membrane characteristics and performance was studied. PES was employed as a base polymer, while *N*-methyl pyrrolidone (NMP) was used as a solvent. The flat sheet membranes, prepared via phase inversion, were characterized using scanning electron microscope (SEM). Membrane performance was changed by the addition of Al_2O_3 nanoparticles to the casting solution. Al_2O_3 entrapped membrane showed lower flux decline compared to that of neat polymeric membrane. Fouling mitigation effect increased with nanoparticle content. No significant effect of the nanoparticles distribution pattern inside the membrane matrix was found on the membrane performance. This study highlights the potential of Al_2O_3 immobilized membranes in MBR application.

1. Introduction

The MBR process has now become an attractive option for the treatment and reuse of industrial and municipal wastewaters. However, the MBR filtration performance inevitably decreases with filtration time due to membrane fouling. More severe fouling is expected when hydrophobic membranes are used in the MBR. Polyethersulfone (PES) has become an important separation membrane material, as it possesses many good physical and chemical characteristics such as good heat-aging resistance and environmental endurance as well as easy processing. However, the inherent hydrophobicity of PES due to its structure leads to a low membrane flux and poor anti-fouling properties, which have a great impact on its application and useful life [1]. Therefore, efforts have focused on increasing PES hydrophilicity either by chemical or physical modifications such as ultraviolet irradiation [2], blending with hydrophilic materials [3], graft polymerization [4], plasma graft [5], and so on. Of the aforementioned methods, blending with inorganic materials, especially nanoparticles, has attracted much interest due to their convenient operation and mild conditions [6]. Moreover, by the way of blending, the modified membrane can combine basic properties of organic and inorganic materials and offer specific advantages for the preparation of artificial membranes with excellent separation performances, good thermal and chemical resistance and adaptability to the harsh wastewater environments [7–10]. Several types of inorganic materials have been blended with polyvinylidene fluoride (PVDF) such as silica [11], zirconium dioxide (ZrO_2) [12], Al_2O_3 [13] and some low molecular weight inorganic salts, such as lithium salts [14]. TiO_2 nanoparticles have also been used in water treatment membrane technology in recent years [8–10]. Molinari et al. [8–10] who tried to report on the promise of photocatalytic membrane reactor for toxic organic removal [8–10], immobilize TiO_2 nanoparticles on flat polymeric ultrafiltration (UF) membranes in two different ways. TiO_2/polymer thin film composite (TFC) reverse osmosis membrane has been investigated to mitigate biofouling by photobactericidal effect under ultraviolet (UV) radiation [15,16]. Bae and Tak [17] investigated the fouling mitigation effect of immobilized TiO_2 UF membranes during the activated sludge filtration. However, studies of blending membranes with nanoparticles focused primarily on gas separation [18–20] and pervaporation membranes [21–23] and have recently been extended to porous membranes for ultrafiltration (especially PVDF membranes) [11–14] and potential nanofiltration applications [24].

Since, no studies have been conducted on Al_2O_3 immobilized membranes for activated sludge filtration despite previous applications in water treatment [13], the novelty in this research lies in introducing Al_2O_3 nanoparticles to PES in order to improve the

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performance of PES membrane for wastewater filtration. Therefore, this research aimed to prepare pure PES membrane and Al_2O_3–PES composite membranes using the phase inversion method by including a small proportion of Al_2O_3 particles to the casting solution. The effects of polymer preparation conditions such as polymer concentration, solvent evaporation time and Al_2O_3-particle concentration in the casting solution on the membrane permeation flux were studied. The membranes morphology was characterized by SEM. Investigation of the fouling mitigation effect of Al_2O_3 immobilized UF membranes during the activated sludge filtration was also included in this study.

2. Experimental

2.1. Membrane preparation

Pure PES flat membranes were prepared by phase inversion [25]. PES Radel A-100 (Solvay Advanced Polymers, Alpharetta, GA, USA) was used as membrane material. NMP solvent was selected in the current study as it is widely accepted as a good solvent for many polymers [11,12,17]. The effect of polymer concentration was tested by preparing casting solutions consisting of 5, 10, 15, 18 and 20 wt%, PES polymer and NMP (Sigma–Aldrich Canada Ltd.). The membranes were casted with a 100 μm casting knife onto a glass plate at room temperature; the nascent membrane was evaporated at $25 \pm 1\,^{\circ}$C for 30 s then immersed in a deionized water coagulation bath maintained at $18 \pm 1\,^{\circ}$C for 2 min. In order to study the effect of solvent evaporation time on membrane preparation, the 18 wt% nascent membranes were casted by the same method described above and evaporated at $25 \pm 1\,^{\circ}$C for 15, 30, 60 and 120 s.

For the entrapped membrane 0.01, 0.03, 0.05 Al_2O_3/PES mass ratios were prepared. Al_2O_3 nanoparticles with average particle size of 48 nm and a surface area of 34 m^2/g (Sigma–Aldrich Canada Ltd.) were dissolved into the NMP solution and was sonicated at $60\,^{\circ}$C for 72 h to obtain a uniform and homogeneous casting suspension. Subsequently, 18 wt% polymer was added and the mixture was sonicated again for a week, the membranes were cast with a 100 μm casting knife onto a glass plate at room temperature. The nascent membrane was evaporated at $25 \pm 1\,^{\circ}$C for 15 s and then immersed in a deionized water coagulation bath maintained at $18 \pm 1\,^{\circ}$C for 2 min. For all prepared membranes, after complete coagulation, the membrane was transferred to a water bath for 15–17 days at room temperature to remove the remaining solvent from the membrane structure before it testing.

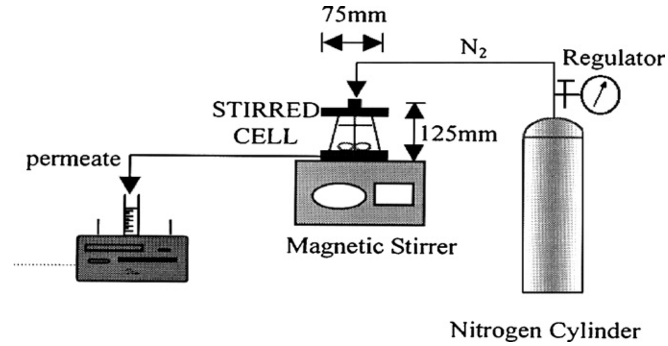

Fig. 1. Schematic diagram of stirred batch cell system.

2.2. Membrane characterization

In order to operate under constant trans-membrane pressure (TMP), membrane filtration was carried out using a stirred batch cell (Model No. 8050, Amicon) as shown in (Fig. 1). The mode of constant TMP is suitable for the study of membrane fouling and there are still many reports about the application of constant TMP for long-term wastewater treatment [26–29]. The deionized water (DIW) flux was determined for the PES control membranes as well as the Al_2O_3 entrapped PES at different TMPs of 0.345, 0.69, 1.034, 1.38 and 1.724 bar). The cross-sectional morphologies of the membranes were characterized using SEM (Leo 1530, LEO Electron Microscopy Ltd) at 1 kV with no conductive coating. To expose the cross-section for SEM characterization, the membranes were cryogenically fractured in liquid nitrogen. The distribution of the Al_2O_3 nanoparticles and the dimension of the membranes were measured using the Java-based image processing program, Image J (National Institutes of Health).

2.3. Activated sludge

Activated sludge used in this study was cultivated in a submerged laboratory scale MBR (Fig. 2) treating synthetic wastewater for more than 5 months. Starch and casein, $(NH_4)_2SO_4$, and KH_2PO_4 were used as carbon, nitrogen and phosphorus sources, respec-

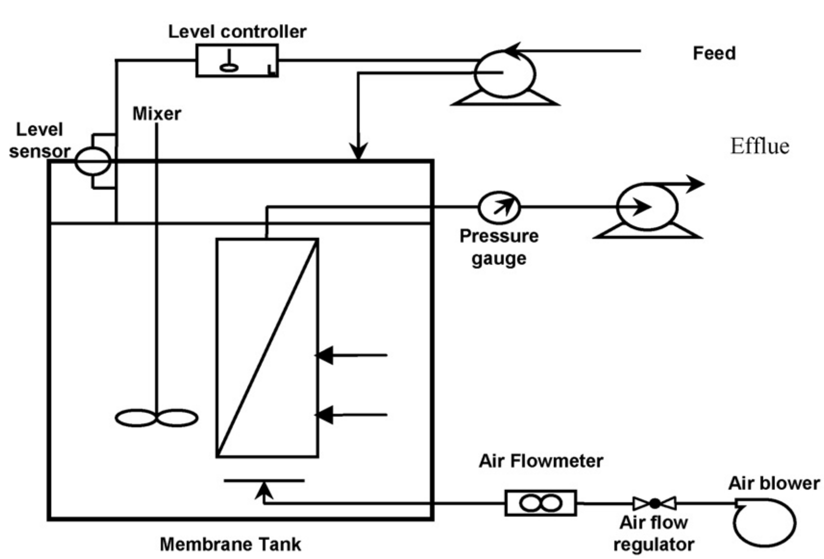

Fig. 2. Schematic diagram of MBR experimental setup.