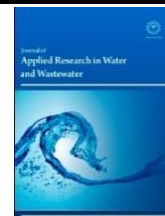




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Original paper

## Preparation and characterization of a high antibiofouling ultrafiltration PES membrane using OCMCS-Fe<sub>3</sub>O<sub>4</sub> for application in MBR treating wastewater

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### ARTICLE INFO

#### Article history:

Received 13 October 2013

Received in revised form 13 December 2014

Accepted 18 March 2014

Available online 25 March 2014

#### Keywords:

Membrane bioreactor

Antifouling

Ultrafiltration

Carboxymethyl chitosan-Fe<sub>3</sub>O<sub>4</sub>

### ABSTRACT

An innovative method based on the membrane bioreactor (MBR) technology was developed as a potential remedy for the water shortage. MBRs attracted much attention in the field of wastewater treatment and reuse. It is reported from many researchers that membrane bioreactor technology is feasible and an efficient method for the treatment of wastewater. However, MBRs are faced to membrane fouling which lead to short membrane lifetime and increase operating costs. Here we was modified polyethersulfone (PES) ultrafiltration membrane by blending of O-carboxymethyl chitosan/ Fe<sub>3</sub>O<sub>4</sub> nanoparticles in a PES solution (14% polymer weight) and casted by a phase inversion process. Membranes with four different weight percentage of O-Carboxymethyl chitosan bound Fe<sub>3</sub>O<sub>4</sub> magnetic nanoparticles (OCMCS-Fe<sub>3</sub>O<sub>4</sub>) to PES of 0.05, 0.10, and 1 wt. % were tested. The OCMCS-Fe<sub>3</sub>O<sub>4</sub> nanoparticles were prepared by the binding of carboxymethyl chitosan (CC) onto the surface of Fe<sub>3</sub>O<sub>4</sub> magnetic nanoparticles, which were prepared by co-precipitating method. The synthesized nanoparticles were characterized by the Fourier transform infrared (FTIR) technique. Moreover, OCMCS-Fe<sub>3</sub>O<sub>4</sub> nanoparticales blend membranes were also characterized using scanning electron microscopy (SEM), and permeation tests. Antifouling performance was studied using activated sludge as a biological suspension and measuring the pure water flux recovery ratio (FRR). The 0.1 wt. % OCMCS-Fe<sub>3</sub>O<sub>4</sub>-PES membrane revealed the highest FRR value (89%). The results exhibited that addition of OCMCS-Fe<sub>3</sub>O<sub>4</sub> nanoparticales lead to membranes with high pure water flux compared to the unmodified PES membrane.

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### 1. Introduction

Due to increasing the water source shortage concerns, recently technology of membrane bioreactors (MBRs) is become an attractive option for the treatment and reuse of municipal and industrial wastewaters because of many favorable features that it offers: high quality of processed water, reduction in excess sludge, controllability of solids and hydraulic retention time, and minimization possible in required footprint (Le-Clech et al. 2010; Kraume et al. 2010; Judd. 2008). However, a major issue associated with MBR is membrane fouling that the bioreactor suffers from it. Complicated interactions between membrane material and various components of activated sludge mixed liquor result in biofouling of the membrane. Unlike physical or chemical fouling, biofouling can irreversibly damage membrane surfaces and often causes permanent permeability loss, which makes the MBRs for wastewater treatment costly (Lee et al., 2013; Flemming et al. 1991; Liu et al. 2010). Therefore, it is highly desirable to have a membrane with antifouling capability, or anti-biofouling membrane.

A membrane material among synthetic polymers that has widely been used in membrane processes is polyethersulfone (PES), because of having many good physico-chemical characteristics such as desirable thermal and mechanical properties as well as chemical

stability and easy processing (Marchese et al. 2003). However, the natural hydrophobicity of PES due to its structure is caused it is not immune from the biofouling problem and provides a low membrane flux and poor anti-fouling properties (Akar et al. 2013). Therefore, various approaches have been taken to increase the hydrophilic properties of PES either by chemical or physical modifications including blending [Yi et al. 2010; Teli et al. 2012], coating (He et al., 2008) and grafting (Yune et al. 2011; Deng et al. 2009). Akar and coworker (2013), prepared polyethersulfone ultrafiltration membranes with selenium and copper nanoparticles and investigated the morphology, performance and anti-fouling properties of membranes. It was shown that the blending membranes with nanoparticles are considered to be suitable for the prevention of biofouling. Yu et al. (2013) were prepared SiO<sub>2</sub>@N-Halamine/polyethersulfone (PES) ultrafiltration membranes by phase inversion method. It was reported that hybrid membranes showed good antifouling and antibacterial properties, which might expand the usage of PES in water treatment and also could make some potential contributions to membrane antifouling.

In the another study by Huang et al. (2012), mesoporous silica (MS) particles was synthesized as inorganic fillers, and blended with polyethersulfone (PES) to achieve nanocomposite membranes with antifouling properties.

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The results indicated that the nanocomposite membrane with 2% MS exhibited excellent hydrophilicity, water permeability and good antifouling performance.

Chitosan (CS) widely researched for several applications such as commercial, industrial, environmental and biomedical applications due to its non-toxic, biodegradable and biocompatible biopolymer and also due to economic reasons, since it derives from chitin, which is very abundant in nature (Kumar, 2000). Chitosan has also have been a large capacity to fix molecules such as proteins and a high permselectivity for water (Huang et al. 2001). However, unmodified chitosan has few bottlenecks such as acidic solubility, low thermal and mechanical stability (Mansourpanah et al. 2013). To overcome these problems especially its limited solubility in aqueous media, many derivatives of CS have been synthetic. One of the hydrophilic derivatives which has a backbone structure like CS but hydroxyl group (-OH) is substituted by carboxyl group (COOH) is O-carboxymethyl chitosan (OCMCS).

Recently, intensive attention has been paid to Fe<sub>3</sub>O<sub>4</sub> nanoparticles due to unique properties of them such as high specific surface area, magnetic, catalytic activities once used with powerful oxidants and/or UV/VIS light, easy to be synthesized, ecofriendly (Chen et al. 2009; lida et al. 2007). In addition to, magnetic nanoparticles and polymer composites not only can be tailored to reveal some new properties such as good film forming and processing properties, besides electrical, magnetic and optical properties, also do not destroy the polymer intrinsic properties (Guo et al. 2009). Despite those advantages, the magnetite (Fe<sub>3</sub>O<sub>4</sub>) nanoparticles have to be encapsulated to inhibit the agglomeration or to make them mono disperse in suspension. Afterward, it is important to modify the Fe<sub>3</sub>O<sub>4</sub> nanoparticles to improve the stability.

In this work, a novel ultrafiltration membrane was prepared by blending of O-carboxymethyl chitosan coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles in PES matrix using the phase-inversion method. SEM and FTIR analyses together with permeation measurements were carried out to characterize the membranes containing different amounts of the Fe<sub>3</sub>O<sub>4</sub>/OCMCS nanoparticles. Investigation of fouling alleviation properties of Fe<sub>3</sub>O<sub>4</sub>/OCMCS entrapped PES membranes during the activated sludge filtration were studied.

## 2. Materials and methods

### 2.1. Materials

Polyethersulfone (Ultrason E 6020P, M<sub>w</sub>=58,000 g/mol) and dimethylacetamide (DMAc) as solvent were supplied from BASF Co., Germany. Polyvinylpyrrolidone (PVP) with a molecular weight of 25,000 g/mol, sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) (98 wt. %), FeCl<sub>3</sub>·6H<sub>2</sub>O, FeCl<sub>2</sub>·4H<sub>2</sub>O, and ammonium hydroxide (28 %) were purchased from Merck. O-carboxymethyl chitosan was purchased from Chung-mu Industrial Corp (Korea). All chemicals used in the experiments were of reagent grade and used as supplied.

### 2.2. Preparation of O-carboxymethyl chitosan-Fe<sub>3</sub>O<sub>4</sub>

Firstly, Fe<sub>3</sub>O<sub>4</sub> nano particles was synthesized by co-precipitation from Fe<sup>2+</sup> and Fe<sup>3+</sup> ions by ammonia solution in N<sub>2</sub> condition at 70 °C, based on the method published previously (Mai et al. 2012). Briefly, ferric and ferrous chlorides were added into three-neck flask (molar ratio 2:1). Chemical precipitation was obtained under vigorous stirring by adding 2 M NH<sub>4</sub>OH solution. During the reaction process, the temperature and the pH were maintained about 70 °C and 10, respectively. The precipitates were washed several times with distilled water and ethanol and resultant precipitates were dried for 24 h in vacuum condition at 50 °C. To prepare OCMCS modified Fe<sub>3</sub>O<sub>4</sub> nanoparticles according to the method of Mai et al. (2012), 20 mg of Fe<sub>3</sub>O<sub>4</sub> powder was dissolved in 10 ml of 2 mg ml<sup>-1</sup> CC solution (pH= 7). Then, the mixture was stirred for 24 h. Fe<sub>3</sub>O<sub>4</sub>-OCMCS nanocomposite were finally separated from the reaction mixture by magnetic bar and then were washed with water and ethanol.

### 2.3. Preparation of PES/Fe<sub>3</sub>O<sub>4</sub>-OCMCS blended membranes

Phase inversion method has been used for fabrication of membranes. At first step, different precise amount of the Fe<sub>3</sub>O<sub>4</sub>-OCMCS nanoparticles were dissolved into DMAc and sonicated for 30 min for good dispersion. The composition of the casting solutions is detailed in Table 1. After dispersing nanoparticles in the solvent, the first PVP and next PES polymer were added to the dope solution and

dispersed uniformly by using continuous stirring for 24 h at around 25°C. After that, the solution was left overnight to release the dissolved bubbles. The resulted polymer/nanofiller solution was again sonicated 10 min to remove air bubbles. In the next step, the solution was cast in a glass plate by using casting knife with the same thickness of 150 μm and was put into the water immediately. The fabricated membrane was separated from the glass plate. After primary phase separation, the membrane was formed. The prepared membranes were stored in coagulation bath for 24 h for separation of residual solvent and then kept between filter papers at room temperature to dry for another 24 h.

Table 1. Specifications of the prepared membranes.

Membrane code	PES (wt. %)	OCMCS-Fe <sub>3</sub> O <sub>4</sub> (wt. %)	PVP (wt. %)
M <sub>1</sub>	14	-	2
M <sub>2</sub>	14	0.05	2
M <sub>3</sub>	14	0.1	2
M <sub>4</sub>	14	1	2

### 2.4. Characterization of the nanocomposite UF membranes

The Fourier transform infrared (FTIR) spectra of unmodified and OCMCS modified Fe<sub>3</sub>O<sub>4</sub> nanoparticles for functional group determination was recorded on an a Bruker FTIR spectrometer (Model: TENSOR 27). Membranes morphology studies were performed based on Philips-X130 and Cambridge scanning electron microscopes (SEM). Briefly, the membrane pieces were frozen in liquid nitrogen for 60–90 s, then broken and air dried. After sputtering with gold, they were viewed with the microscope at 26 kV.

### 2.5. Permeation experiments

The performance of the prepared ultrafiltration membranes were characterized by measuring pure water flux and activated sludge fouling tests by using a dead-end stirred cell filtration system connected with a nitrogen gas line. The schematic of the dead end system are depicted in Fig. 1 (Vatanpour et al. 2012). Each membrane was primarily immersed in distilled water for 30 min and next pressurized with distilled water at 3 bar for 30 min to compaction. After that, the pressure was reduced to the operating pressure value of 2 bar. The flux (J) via the cake and the membrane may be described as following:

$$j_{w,1} = \frac{M}{A \Delta t} \quad (1)$$

Where M is the weight of the permeate pure water (kg), A is the membrane effective area (m<sup>2</sup>) and Δt is the permeation time (h).

### 2.6. Membrane antifouling property

After pure water flux measurement (J<sub>w,1</sub>), the solution reservoir was rapidly refilled with 1000 ppm activated sludge suspension as the feed and the flux (J<sub>p</sub>) based on the water quantity permeating the membranes at 2 bar for 6 min was attained. The flux was calculated according to the Eq. (1).

After filtration of activated sludge suspension, the membranes were washed with distilled water for 10 min and the water flux of cleaned membranes was measured (J<sub>w,2</sub>). In order to evaluate the membrane antifouling property of the membrane, the flux recovery ratio (FRR) was calculated the following equation:

$$FRR (\%) = \left( \frac{J_{w,2}}{J_{w,1}} \right) \times 100 \quad (2)$$

## 3. Results and discussion

### 3.1. Characterization of Fe<sub>3</sub>O<sub>4</sub>-OCMCS nanoparticles

For showing the successful coating of O-carboxymethyl chitosan on the Fe<sub>3</sub>O<sub>4</sub> surface, spectroscopic analysis performed. Fig. 2 shows the FTIR spectra of unmodified and OCMCS surface modified magnetic particles. The band of 581 cm<sup>-1</sup> is the characteristic peak of Fe–O–Fe in the unmodified Fe<sub>3</sub>O<sub>4</sub>. Presence of the peaks at 1633 cm<sup>-1</sup>

and  $1391\text{ cm}^{-1}$  shows the C=O stretches of  $\text{Fe}_3\text{O}_4/\text{OCMCS}$ , indicating the carboxylate anion interacting with the FeO surface resulting in the formation of the iron carboxylate. The signal at  $1057\text{ cm}^{-1}$  could be attributed to the C-O/C-N stretching vibration. Also, a broad band due to the stretching of O-H and N-H bond seemed at  $3429\text{ cm}^{-1}$ . These results prove that O-carboxymethyl chitosan was adsorbed on the magnetic particles (Barroso et al. 2011; Shi et al. 2011; Vatanpour et al. 2012; Xu et al. 2004).

### 3.2. Morphology of the prepared membranes

To evaluate the changes induced in the skin-layer and sub-layer of the membranes, cross-sectional SEM images of the unmodified PES membrane and nanoparticle blended membranes are displayed in Fig. 3. A typical asymmetry structure composed of a thin skin-layer and a porous bulk with a finger-like structure can be clearly seen from the SEM images. According to the SEM images (Fig. 3), addition of the  $\text{Fe}_3\text{O}_4/\text{OCMCS}$  nanoparticles to the casting solutions causes the increased number of finger-like channels, which led to increase of the porosity. However, the addition of more than 0.1 wt. % of the  $\text{Fe}_3\text{O}_4/\text{OCMCS}$  results in denser skin-layers with increased thickness and sub-layers with lower porosities. This was may be due to the increased viscosity of the  $\text{Fe}_3\text{O}_4/\text{OCMCS}$  /PES blend solution and agglomeration of the nanoparticle on the surface of the membranes.

Increase of the viscosity usually delays the exchange of solvent and non-solvent. This suppresses the formation of large pore radius and reduces the porosity of membrane.

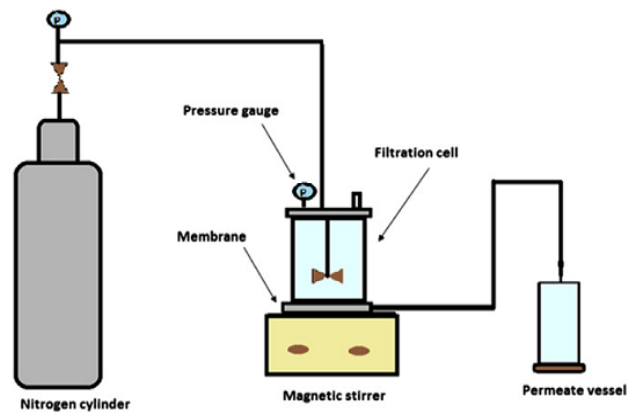


Fig. 1. Schematic of dead end system.

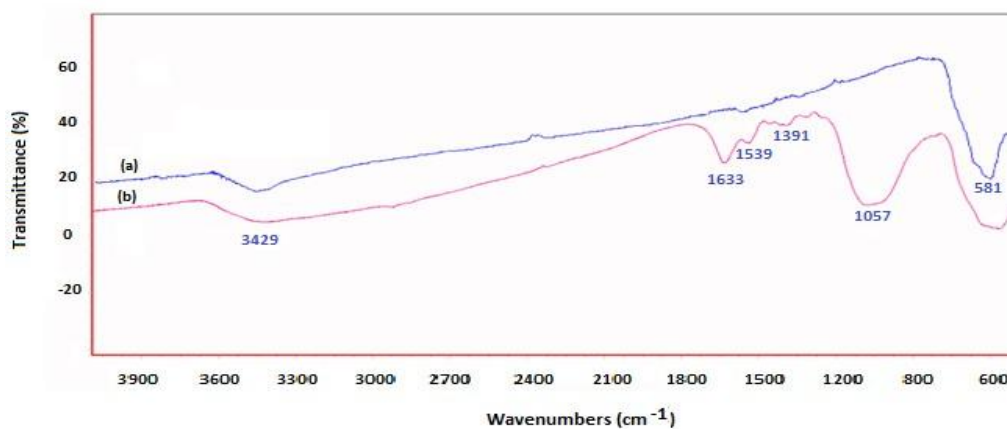


Fig. 2. FTIR spectra of unmodified (a) and CC modified  $\text{Fe}_3\text{O}_4$  particles (b).

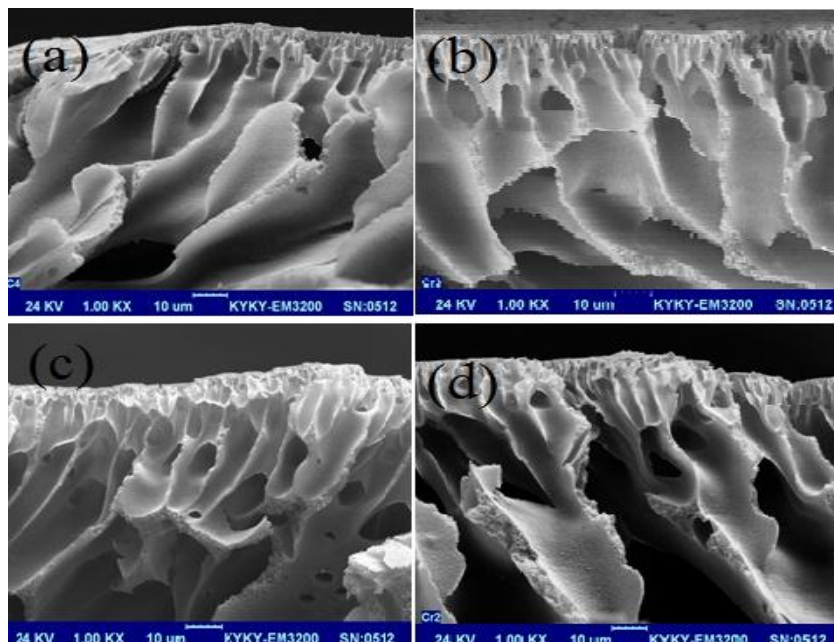


Fig. 3. Cross-sectional SEM images of the prepared membranes, (a)  $M_1$ : Unfilled PES, (b)  $M_2$ :  $\text{Fe}_3\text{O}_4/\text{OCMCS}$  0.05 wt. %, (c)  $M_3$ :  $\text{Fe}_3\text{O}_4/\text{OCMCS}$  0.1 wt. % and (d)  $M_4$ :  $\text{Fe}_3\text{O}_4/\text{OCMCS}$  1 wt. %.

### 3.3. Pure water flux

The pure water flux (PWF) of different types of  $\text{Fe}_3\text{O}_4/\text{OCMCS}$  embedded PES membranes as a function of the nanoparticle

concentration shown in Fig. 4. As shown, the pure water flux of the membranes increased from 77.5 to 277  $\text{kg/m}^2\text{ h}$  as the additive quantity increased from 0 to 0.1 wt. %. Although, the 1 wt. %  $\text{Fe}_3\text{O}_4/\text{OCMCS}$  content membranes have the highest percent of

nanoparticles, but its pure water flux was the lowest. This contrary behavior can be ascribed to agglomeration of nanoparticles which cause to clogging of membranes pores (Vatanpour et al. 2012) as well as dense skin-layer of this membrane as shown in the SEM images (Fig. 3d).

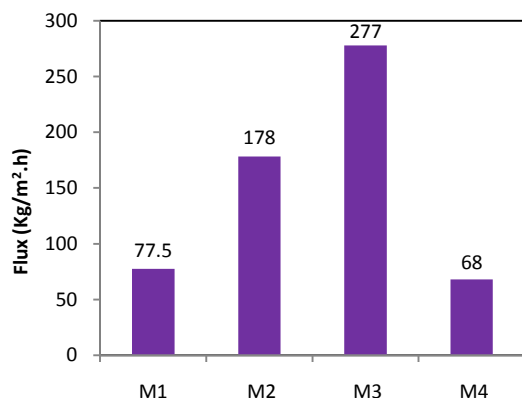


Fig. 4. Pure water flux of the prepared membranes.

### 3.4. Antifouling properties of the membranes

Fouling is the major obstacle of PES ultrafiltration membrane due to the hydrophobic interaction between foulant and membrane surface. The fouling is due to the adsorption and deposition of proteins on membrane surface and introducing of proteins within the membrane pores. Fouling mainly caused flux decline and shortening of membrane life time (Vatanpour et al. 2011). In order to increase membrane permeability and antifouling property, many efforts have been carried out for enhance membrane hydrophilicity (Kumar et al. 2013; Rana et al. 2010), which among these approaches, blending with hydrophilic particles has been considered as an effective and convenient approach for preparation of antifouling membranes (Ng et al. 2013; Zinadini et al. 2014).

The antifouling performance of the unfilled and the filled PES ultrafiltration membranes with nanoparticles was examined by measuring of water flux recovery after fouling by activated sludge suspension, which presented in Fig. 5. The highest FRR indicated that the membranes have more fouling resistant. It could be seen that the FRR values increased when the content of the nanoparticles in the

casting solution increased, and the maximum FRR value reached 89 % for the M<sub>3</sub> blend membrane and then this was dropped to 78 %. This may be attributed to the agglomeration of nanofillers in the pores.

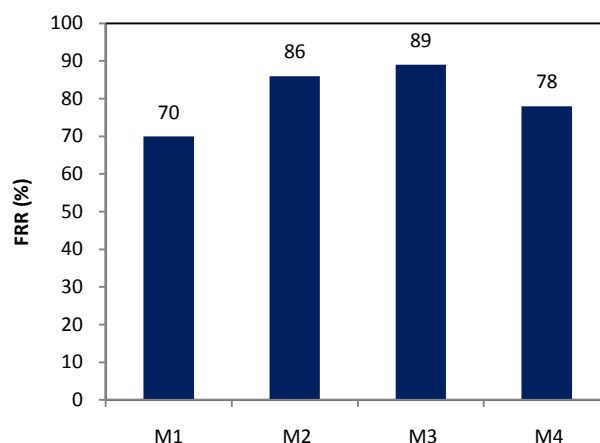


Fig. 5. Water flux recovery of the Fe<sub>3</sub>O<sub>4</sub>/OCMCS nanoparticles blended PES membranes after activated sludge suspension fouling. (Average of three replicates was reported).

### 4. Conclusion

The high performance PES nanocomposite membranes were prepared by addition of synthesized Fe<sub>3</sub>O<sub>4</sub>-OCMCS nanoparticles in the casting solution at different amounts. The water flux of the modified membrane improved by addition of nanoparticles from 0 to 0.1 wt. % and this was followed by a sharp decrease with further addition of Fe<sub>3</sub>O<sub>4</sub>-OCMCS nanoparticles. Similar trend to the pure water flux was observed in this study for antifouling properties and flux recovery ratio of modified membranes. The SEM images of membranes indicated that addition of the Fe<sub>3</sub>O<sub>4</sub>-OCMCS from 0 to 0.1 wt. % led to formation of membranes with the more number of finger-like channels.

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