Desalination and Water Treatment
www.deswater.com
6 (2009) 281–288

Influence of feed conditions on the rejection of salt and dye in aqueous solution by different characteristics of hollow fiber nanofiltration membranes

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Received 6 January 2009; accepted 15 March 2009

ABSTRACT

Three different characteristics of self-made hollow fiber nanofiltration (NF) membranes with different structural and electrical properties were evaluated for their suitability for removing salt and colour as a function of feed conditions. Results showed that NF achieved promising dye retention for the reactive dyes used at different concentrations and varied slightly with pH. The retention of salt was found to be dependent on feed properties and membrane electrical properties where higher retentions were achieved under alkaline condition and by using the most negatively charged NF membrane. The enhancement in the membrane negative surface charge resulting from the addition of sulfonated poly (ether ether ketone) (SPEEK) during membrane preparation had also made the NF less fouling-sensitive to dye absorption and improving the economic viability of the treatment process.

Keywords: Membrane separation; Reactive dyes; Salts; Sulfonated poly (ether ether ketone); Surface charge properties

1. Introduction

Recently, the development of nanofiltration (NF) membranes for various industrial applications has drawn much attention from membrane scientists due to their unique characteristics in the separation of small neutral and charged molecules in aqueous solution. Although a number of studies has been documented in the literature to investigate the separation performance of NF membrane in the treatment of textile discharge, rare work has been done to study the influence of NF properties on solute separation under various feed conditions [1–6]. Membrane properties such as pore radius and effective charge density are preferentially required in NF membrane selection so that one would fully explore how these properties take place in the removal of dyestuffs and dissolved salts from wastewater [7]. Basically, rejection mechanisms of NF involve size sieving (steric hindrance) incorporating Donnan exclusion (electrostatic interaction of charged solutes with charges attached to the membrane matrix). This combination allows NF membranes to be effective for separating mixtures of organic molecules (neutral or charged) and salts.

In the literature, it is found that information on membrane properties given by membrane manufacturers is not really explicit as molecular weight cut off and solute separation efficiency of membrane may be varied depending upon the protocols used by various manufacturers [8]. Furthermore, it is always lack of data about the quantification of the membrane charge. Mostly, only NF membranes with either negatively

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charged or positively charged are disclosed without taking into consideration the value of effective surface charge density, X_d. Bowen and Mohammad [7] reported that a high value of X_d could maximize the Donnan effects of membrane which resulted in effective removal of dye and salt from a dye-salt-water waste stream. Ismail and Hassan [9,10] also found that the appearance of charge density determined the extent of NF performances towards NaCl rejections. Besides the effect of charge density on retention characteristics, membrane surface charge is also important to minimize fouling tendency by reducing the absorption of negatively charged foulants in the feed water [11]. Due to these reasons, NF membrane with known pore size and effective charge density is really required in solute separation applications in order to obtain precise control of separation efficiency and achieve purification goal.

Previously, Ismail and Hassan had succeed to deduce the structural and electrical properties of NF membranes by means of solute retention based on the theoretical models [9,10,12]. By having these properties it becomes possible for potential users to choose NF membrane which is best suited to particular process requirement. Despite membrane properties are often identified as the primary factors affecting the separation efficiency for the solution containing dye-salt-water components, other factors, e.g. the chemical nature of the solution are also found to be the parameters contributing to the membrane separation [8,13–16]. Most of the experiments reported in literature have been performed in a wide range of salt concentrations and with mixtures of salts and dye solutes. In the case of electrolyte solutions, the separation mechanism is remarkably related to the NF membrane surface charge and the external solutions. It is known that pH is able to change the membrane charge with consequence in the performance of NF. Membrane surface would become more negatively charged at higher pH, leading to increased membrane selectivity due to the greater electrostatic repulsion between electrolytes and membrane surface. However, the main drawback in NF process of dye solutions is the fouling problem, which results in an undesirable flux decline. According to Al-Bastaki [17], the extent of dye adsorption on membrane surface and its reversibility are determined by the factors of dye-membrane physicochemical interactions which in turn depend on the feed water chemistry as well as the operating conditions. The fouling phenomena however could be diminished with using a strongly negatively charged NF and/or with an adequate membrane cleaning process [3,6].

As shown in literature, membrane properties and feed properties are very important in controlling NF separation performances. Therefore, in this paper the separation performances of three different characteristics of self-made NF membranes were investigated under various properties of aqueous solutions containing dissolved salt and/or dye molecules. The characteristics of NF were altered by varying the content of sulfonated poly (ether ether ketone) (SPEEK) in the dope solution during membrane fabrication process. The study is expected to provide instructive information on the influences of membrane characteristics and feed properties on the NF performances.

2. Experimental

2.1. Materials

Reactive Black 5, RB 5 (MW 991, λ_{max}: 592 nm) and Reactive Orange 16, RO 16 (MW 616, λ_{max}: 493 nm), supplied by Sigma were used as received. Charged solutes, NaCl and Na_2SO_4 used in determination of membrane separation efficiency, were purchased from Merck. NaOH (0.1 N) and HCl (0.1 N) aqueous solution from Merck were used to modify feed pH. Other chemicals used in this study were used as purchased without further purification. The aqueous solutions were prepared using distilled water with pH value nearly 7 unless otherwise specified.

2.2. NF membranes

The self-made NF membranes used in this study were prepared using a simple dry-jet wet spinning technique [18]. Dope solutions were prepared from 20 wt% PES in 70 wt% NMP which containing different concentrations of PVP K15 and SPEEK. The concentration of SPEEK was varied from zero to 4 wt% in the dope solution in order to produce different membrane properties, as shown in Table 1. The structural and electrical properties of these blend membranes were then deduced using Steric-Hindrance Pore (SHP) model and Teorell-Meyer-Sievers (TMS) model, respectively. The use of irreversible thermodynamic model is also needed in order to determine parameters such as solute permeability P_s and reflection coefficient σ which are required in SHP and TMS model. The detailed theoretical studies can be found in our previous work [19]. From Table 1, it is found that the values obtained were in the range of twenty-nine commercial NF membranes assessed by Bowen and Mohammad [7]. Further, the membrane water flux and charge properties were found to increase with increasing SPEEK content in blend membrane, mainly due to the hydrophilic and highly charged characteristics of SPEEK polymer added.

Table 1
The structural and electrical properties of the self-made hollow fiber NF membranes

Membrane[a]	SPEEK Content (%)	J_v[b] ($\times 10^{-7}$ m/s)	r_p (nm)	$A_k/\triangle x$ (m^{-1})	X_d (mol/m^3)
PES	–	3.75	0.77	3196	−11.39
PES/SPEEK 2	2	5.19	0.99	1986	−19.88
PES/SPEEK 4	4	7.84	0.79	5209	−21.02

[a] Membrane properties were deduced using theoretical models as reported in [19].
[b] J_v, r_p, $A_k/\triangle x$ and X_d represent pure water flux, pore radius, ratio of porosity to membrane effective thickness and effective charge density, respectively.

2.3. Experimental system

Filtration experiments were conducted using a laboratory-scale cross-flow permeation system. Both ends of the hollow fiber were plotted with epoxy resin (E-30CL Loctite® Corporation, USA) and then mounted into a stainless steel test housing. A low pressure booster pump (ROP-BP/KF, Kemflo) was used to control the desired operating pressure (5 bar) through the adjustment of the back-pressure regulator. Feed was pressurized onto the outer membrane surface into lumen side and exited through the open fiber ends. The quantity of permeate was then collected for analysis.

2.4. Analytical methods

The removal efficiencies for reactive dye solutes were determined using spectrophotometer (Model DR/4000, Hach) at wavelength specified by manufacturer. The concentrations of salt in the feed and in the permeate were analyzed by conductivity using a portable conductivity meter (EC300, YSI Inc). pH meter (HI 8424, HANNA instruments) was used to measure the pH value of aqueous solution prepared. For the determination of polyvinyl alcohol (PVA) rejection, total organic carbon (TOC) analysis was performed using TOC-$V_{CSH/CSN}$ analyzer (Shimadzu, Japan). The chemical oxygen demand (COD) analysis was carried out following the procedures handbook of DR/4000 spectrophotometer.

2.5. FESEM studies

The fiber samples were prepared by cryogenically fractured in liquid nitrogen and then coated with platinum using auto fine coater (JEOL JFC-1600). To observe the surface of fibers, Field Emission Scanning Electron Microscope (FESEM, JEOL JSM-6701F) was employed.

3. Results and discussion

3.1. Salt rejection

3.1.1. Effect of salt concentration

NaCl and Na_2SO_4 are the most common inorganic salts that have been widely used in dyeing process for the purpose of enhancing the degree of dye fixation onto the fibers. Therefore, the dissolved salt in waste stream must be treated properly before being discharged into environment. Fig. 1 shows the salt rejection of different characteristics of NF at salt concentration in the range of 250–5000 ppm. The membranes showed higher rejection to divalent anions with higher co-ion charge than monovalent anions. Comparing the three membranes studied, it revealed that PES/SPEEK 4 membrane had the highest values followed by PES/SPEEK 2 and PES membrane in the separation of NaCl and Na_2SO_4 solute. With the addition of small amount of SPEEK into PES membrane, it showed that both PES/SPEEK blend membrane displayed higher salt separation than that of PES membrane. It is because of the enhancement in membrane surface charge properties resulting from the sulfonic acid groups. Increase in surface charge properties, however resulted in larger variation in NaCl

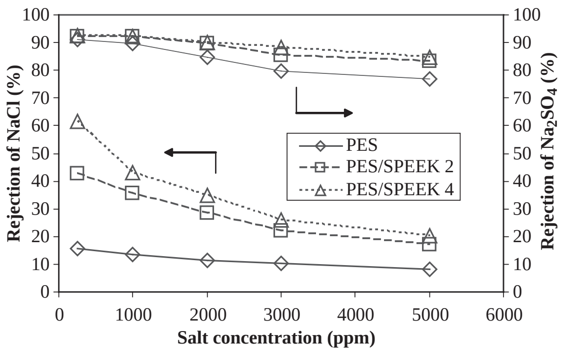

Fig. 1. The effect of salt concentration on the salt rejection of NF.

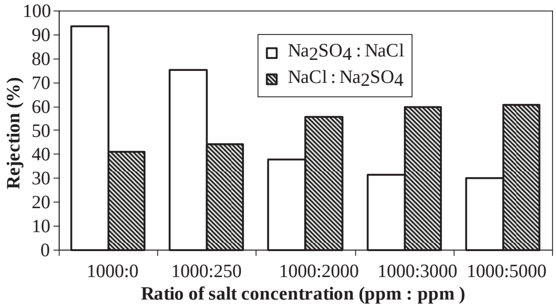

Fig. 2. The effect of mixture ratio of NaCl and Na_2SO_4 on salt rejection of PES/SPEEK 4 membrane.

separation in PES/SPEEK membrane compared to PES membrane. This observation was similar to the work carried out by Van der Bruggen et al. [4], where salt concentration plays an important role in damping out electrostatic repulsion, causing electrolyte retention of charged membrane decreases significantly with increasing concentration.

As shown in Fig. 2, salt rejection decreased considerably with NaCl concentration when it was added into electrolyte mixture solution containing 1000 ppm Na_2SO_4. The decrease in retention may be explained by the decrease in membrane repulsion force due to higher ionic strength in solution [20]. Opposite tendency of retention however was observed when Na_2SO_4 was added into solution containing NaCl. The increase in the retention is consistent with quantitative aspects of Donnan exclusion principle where divalent anions, SO_4^{2-} are highly rejected by negatively surface charged membrane than that of monovalent anions, Cl^-. The rejected SO_4^{2-} ions would require an equivalent number of counter ions Na^+ to achieve electroneutrality in the solution and as a result, higher retention can be obtained. Fig. 3 illustrates the responsibility of dissociated sulfonic acid groups on PES/SPEEK membrane in rejection of different ions in electrolyte solution. It becomes clear that SO_4^{2-} ions are largely repelled from passage through the membrane while it is not for Cl^- ions in a mixture of electrolytes.

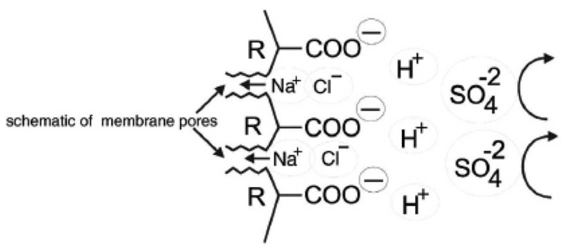

Fig. 3. A schematic diagram of PES/SPEEK NF membrane in the rejection of mixture salt solution.

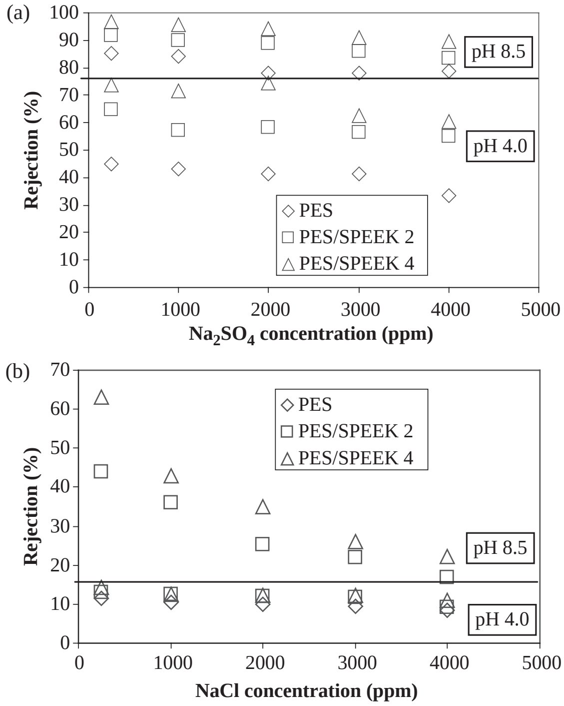

Fig. 4. The effect of pH on the salt rejection of NF membranes at different salt concentrations, (a) Na_2SO_4 and (b) NaCl.

3.1.2. *Effect of pH value*

It has been acknowledged that alkaline environment is always the best condition for enhancing the degree of dye fixation during dyeing process, though acidic condition would also be considered for certain textile operations. Fig. 4 presents the effect of feed pH on the NaCl and Na_2SO_4 rejections. Higher rejection rates were found in the case of PES/SPEEK membranes which were claimed to have SO_3^- ions on the membrane surface resulting from the addition of SPEEK. The PES/SPEEK blend membranes become increasingly more negative in alkaline solution due to the presence of more OH^- ions in solutions [21,22]. This leads to an enhancement in electrostatic repulsion between a negatively charged solute and membrane which results in higher retention. However, decreasing the pH from 8.5 to 4 decreased the salt rejection of all the membranes. This indicates pH has a direct effect on the charge of membrane due to the disassociation of functional groups. The H^+ ions dissociated from HCl can be absorbed onto membrane surface, shielding the negative charges and consequently causes significantly decrease in rejection of electrolytes. Meanwhile, with the use of HCl solution to modify the feed pH, it causes an increase of Cl^- ions in permeate which may also further decrease the rejection rate. Therefore, the salt rejection varied significantly depending on the

Table 2
The effect of reactive dye concentration on colour removal of NF membrane

Reactive dye	Conc. (ppm)	Colour removal (%)		
		PES	PES/SPEEK 2	PES/SPEEK 4
RB 5 (991 Da)	50	99.69	99.62	99.54
	100	99.21	99.79	99.96
	250	99.77	99.90	99.82
	500	99.57	99.92	99.78
RO 16 (616 Da)	50	99.47	98.14	98.34
	100	98.60	96.99	96.96
	250	97.23	95.38	95.59
	500	93.71	93.77	94.10

feed properties and the membrane electrical properties. In order to achieve greater electrolyte selectivity under alkaline condition, the use of the most negatively charged NF membrane is hence highly recommended.

3.2. Dye removal

3.2.1. Effect of dye concentration

After filtrating electrolyte compounds, the three membranes were tested for their performance in the presence of reactive dye. Table 2 shows almost complete rejection of RB 5 and 93.7–99.5% rejection of RO 16 were achieved depending on type of NF and dye concentration used. As MW of reactive dyes get larger, sieving effects due to steric hindrance increase and the higher MW of solute is rejected by the membrane more often than the lower MW of solute. Due to this, the membrane pore size may be an important driving factor in the rejection of reactive dye by NF membranes. It is reported that the effective hydrodynamic radius for RB 5 and RO 16 are approximately 2 nm and 1.5 nm, respectively [23]. Thus, it is less possible for dye molecules from passage through the membranes which have relatively smaller pore size (see Table 1). In general, the separation of dye solute is ideally suited for NF separation process since most of the dyes used for the dyeing process have a molecular weight range between 600 and 1000 Da, which is typically bigger than the pore size of NF membrane [24]. In addition to sieving effect, Jiraratananon et al. [25] reported that Donnan exclusion effect should also be taken into consideration during dye separation. The effect however was found little dependence in this study, most probably due to the large size of dye molecules relatively to membrane pore size. Nevertheless, it is believed that Donnan exclusion mechanism may take place in separation when reactive dye used has the effective hydrodynamic radius which is relatively equal or even smaller than the membrane pore radius.

3.2.2. Effect of pH value

Fig. 5 details the reactive dye retention of membranes at different pH of dyeing aqueous solutions. As can be clearly seen, the variation of pH values did not significantly affect the dye retention [26]. No large discrepancies were observed between the values obtained under different pH conditions using either PES or PES/SPEEK membranes. Nevertheless, one can note that the values obtained by PES/SPEEK membranes with solution pH 8.5 were slightly higher than those obtained with solution pH 4. This could be partly due to the enhancement in electrical properties of PES/

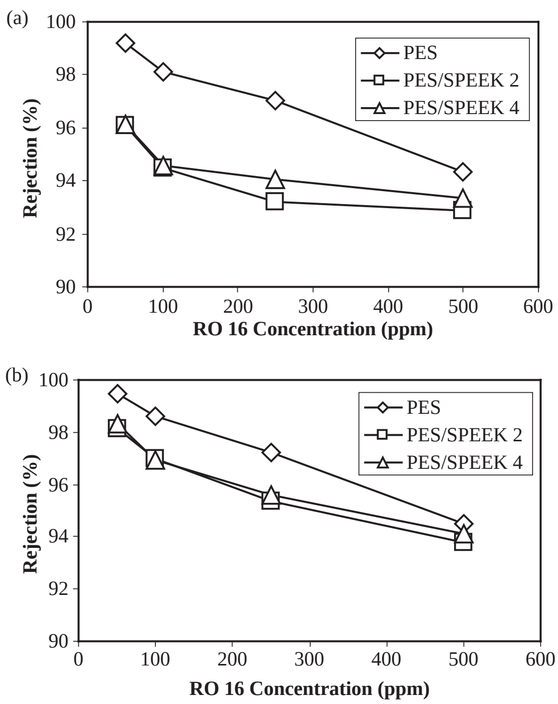

Fig. 5. The effect of pH on dye removal of NF membrane, (a) pH 4 and (b) pH 8.5.

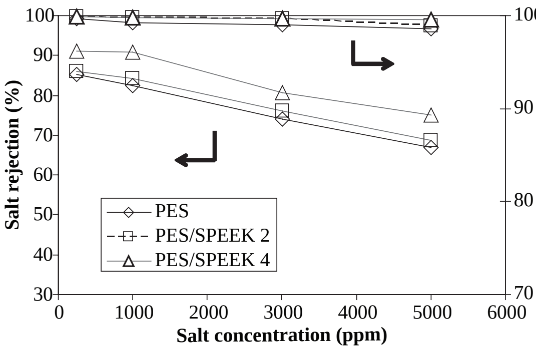

Fig. 6. The effect of salt concentration on dye removal of NF membranes.

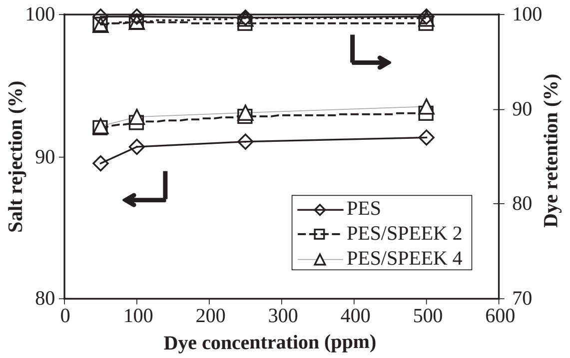

Fig. 7. The effect of dye concentration on salt removal of NF membranes.

SPEEK membranes as discussed in Section 3.1.2. Overall, it was found that dye retention was strongly dependent on membrane structural properties rather than electrostatic properties.

3.3. The salt and dye removal in mixture solution

3.3.1. Effect of salt concentration on NF separation

Fig. 6 shows the experimental results on solutions which containing both salt and dye solutes. The concentration of RB 5 was fixed at 250 ppm while the concentration of Na_2SO_4 was varied in the range between 250 and 5000 ppm. With the presence of Na_2SO_4 in solution, both the salt and dye retention decreased as salt concentration increased. The dye retention value however could be maintained as high as 98% even at high salt loadings of 5000 ppm, giving a decolourized permeate which is suitable for possible discharge or reuse. Compared to Fig. 1, it was experienced that membranes suffered greater decrease in salt rejection with the presence of dye solutes in feed water. It is because dye molecules may deposit on certain portions of membrane surface and form an additional barrier which in turn decreasing Donnan exclusion effect.

3.3.2. Effect of dye concentration on NF separation

Fig. 7 displays the rejections of RB 5 and Na_2SO_4 when different concentrations of RB 5 were added to the Na_2SO_4 aqueous solution. Dye retention remained unchanged while salt rejection increased slightly with increasing dye concentration from 100 to 500 ppm. High removal efficiency of RB 5 could be easily achieved despite the variation of dye concentration in feed. These results revealed that the dye retention is mainly controlled by the sieving effect (membrane pore size). At high solute concentration, the dye molecules would result in the formation of additional barrier together with salt concentration polarization layer, resisting the passage of ions through the membrane. As a consequence, slightly increase in salt rejection was experienced. Considering the presence of –SO_3H groups on membrane surface, PES/SPEEK 4 membrane is able to achieve excellent separation performance, especially in the rejection of ions.

3.4. Experiments with synthetic dyeing wastewater

For this experiment, a synthetic dyeing wastewater composed of 1000 ppm RB 5, 500 ppm PVA, 250 ppm NaCl and 750 ppm Na_2SO_4 was prepared and used for evaluation of the membrane separation efficiency. The composition of the synthetic wastewater prepared was very similar to the composition of real dyeing wastewater, making the separation process more practical [6]. The permeate quality was monitored by measuring the removal efficiency of conductivity, colour, TOC and COD retention. Table 3 shows the retention of colour, TOC and COD was not affected by the types of NF used, except the salt retention. The salt retention is found to be dependent on membrane surface charge where retention increases with increasing the surface charge properties. It was also interesting to note that the greater the membrane surface charge, the better the membrane in reducing flux decline, as shown in Fig. 8. The results suggested that cake layer formation of dye molecules on membrane surface, especially the PES membrane was the principal cause of flux decline. In comparison, the use of SPEEK had great influence on

Table 3
Performance of NF membranes for treating synthetic dyeing wastewater

Membrane	Salt (%)	Colour (%)	TOC (%)	COD (%)
PES	60.88	99.62	97.07	98.46
PES/SPEEK 2	63.84	99.85	97.79	98.00
PES/SPEEK 4	72.80	99.89	97.70	97.29

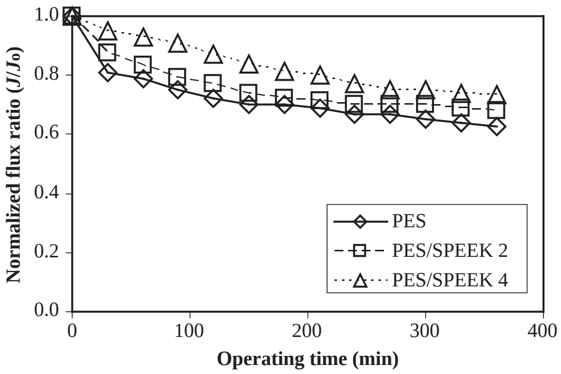

Fig. 8. The normalized flux ratio against operating time of NF membranes for treating synthetic wastewater.

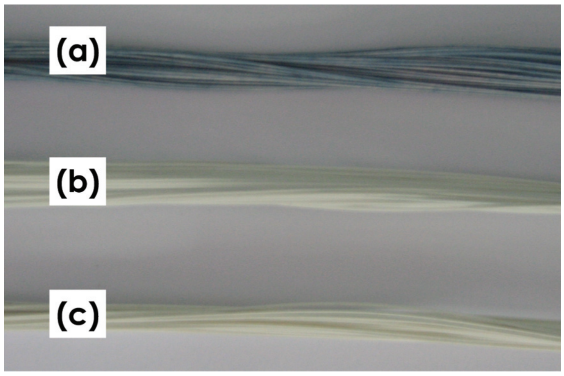

Fig. 9. Direct observation of membrane outer surfaces after chemical cleaning process (a) PES, (b) PES/SPEEK 2 and (c) PES/SPEEK 4.

decreasing flux declines as PES/SPEEK 4 membrane displayed the lowest flux decline among the membrane studied. Fig. 9 shows PES/SPEEK blend membranes were less stained with colour after cleaning process, indicating fouling on these membranes was primarily cake formation (reversible) without permanent particle penetration and pore blockage of the membrane itself. These findings were in agreement with the experimental results where higher flux recovery (86%) could be retrieved using PES/SPEEK membranes compared to 78% by using PES membrane, after a simple chemical cleaning process. Fig. 10 displays PES membrane was still fouled even after the chemical cleaning, indicating foulant due to dye deposition appears to be irreversible. The intact surface of PES/SPEEK 4 membrane on the

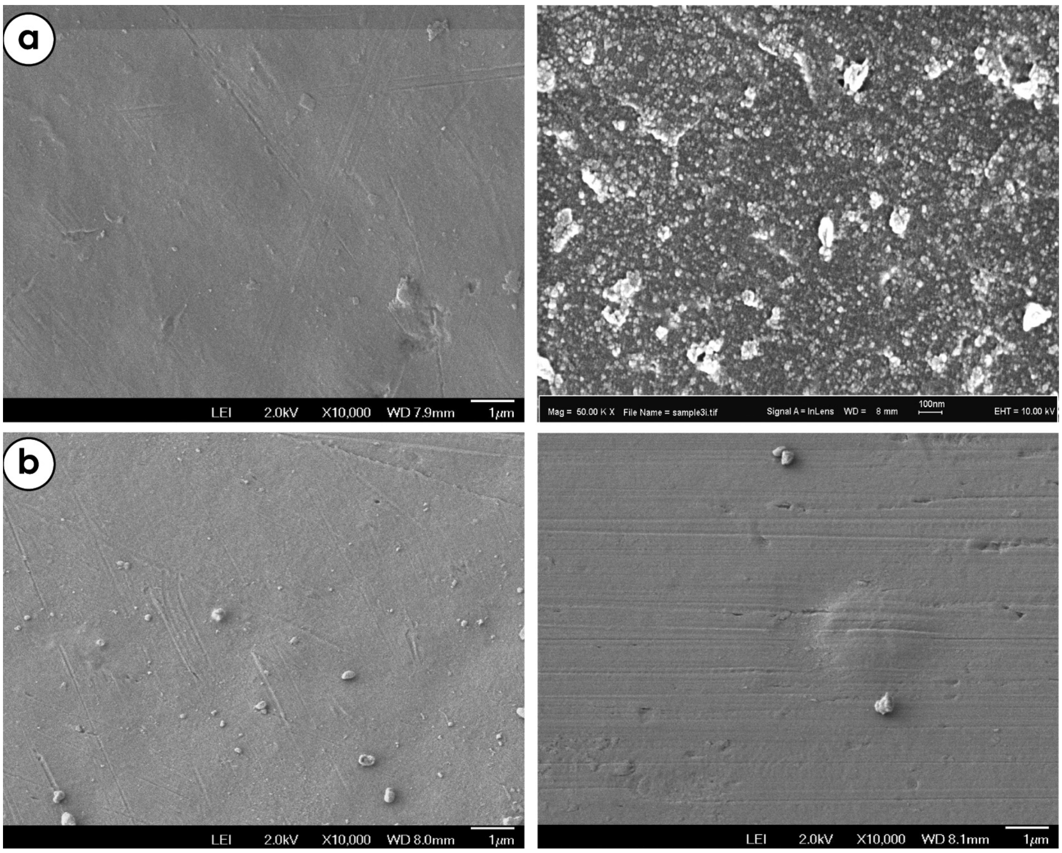

Fig. 10. Scanning electron micrographs of clean membrane surface (left, 10,000×) and used membrane surface after chemical cleaning process (right, 10,000×), (a) PES and (b) PES/SPEEK 4 membrane.

other hand confirmed that dye adsorption could be diminished with the use of strongly negatively charged membranes. The results were supported by Boussu et al. [22] where a hydrophilic and strongly negatively charged membrane seemed to be favorable to reduce the membrane fouling tendency.

4. Conclusions

This study demonstrated the importance of NF characteristics and the feed properties on the NF separation performance. The salt retention was found to be decreased with salt concentration but increased with pH. The surface charge introduced by SPEEK on PES/SPEEK membranes fully explained the better separation performances compared to PES membrane. Since membrane pore size was relatively smaller than the effective hydrodynamic radius of reactive dye used, notably high retention could be easily achieved and the dye retention seemed to be not affected by dye concentration and pH. The results showed that salt retention decreased with salt concentration while increased slightly with dye concentration in the separation of mixture solution containing both dissolved salt and dye molecules. The retention of dye on the other hand remained excellent, irrespective of mixture solution conditions. Membrane fouling can be minimized in dyeing treatment process with the addition of a small amount of SPEEK content into membrane, making the PES/SPEEK membrane less fouling-sensitive to dye absorption. This is in fact helpful in extending membrane lifespan and improving the economics of the process. In summary, a complete understanding of membrane properties and feed properties could lay the basic foundation for achieving high level of water purification during NF membrane applications.

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