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Treatment of dyeing solution by NF membrane for decolorization and salt reduction

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ABSTRACT

A synthesized dyeing solution consisted of reactive dye, salts, and polyvinyl alcohol was prepared and treated by two different types of commercial nanofiltration (NF) membranes, i.e. NF90 and NF270 under various process conditions. These NF membranes were evaluated for permeate flux, salt, and color rejection as well as fouling propensity for a certain period of operation. Results revealed that NF90 suffered significant flux decline compared to NF270 after four operation cycles of treatment process, indicating its high sensitivity to foulant attachment. With respect to color removal, it is found that NF270 demonstrated greater stability in maintaining its separation efficiency (between 94 and 98% rejection) regardless of the feed properties and number of operation cycles. Though the salt rejection of NF270 was not as high as NF90, its consistent separation performance throughout the study period proved its reliability in long run. Recommended chemical cleaning process was also conducted in an effort to recover the membrane water flux due to the fouling. The results revealed that 90% of the initial water flux of NF270 was retrieved after a chemical cleaning. The water flux of NF90, however, was not able to be recovered. A water flux coefficient of 1.74 l/m^2 h bar was reported after a chemical cleaning process in comparison to its virgin state of 5.43 l/m^2 h bar. The irreversible fouling on NF90 was strongly linked to the pore blockage resulted from the presence of dye molecules in the feed solution. Based on these findings, it is showed that NF270 is more reliable and sustainable to be used in treating industrial dyeing wastewater. The high water productivity of NF270 coupled with high resistance against dye absorption may outweigh its relatively low rejection rate of salt in industrial applications.

Keywords: Nanofiltration; Textile industry; Salt rejection; Color removal; Fouling; Chemical cleaning

1. Introduction

Nanofiltration (NF) membrane has been developed for more than 40 years [1]. Before the term of NF was coined by membrane scientists in the second half of 1980s, this type of membrane used to be categorized as either tight ultrafiltration (UF) or loose reverse osmosis (RO) membranes [1]. The unique characteristics of NF which lies between UF and RO have made it very attractive to many industrial applications, e.g. industrial effluent treatment [2–4], molecular separation involving organic solvent [5,6], micropollutants removal [4,7], water softening [8,9], whey demineralization [1], etc.

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In the particular case of textile wastewater treatment, NF membrane appears as an excellent candidate over other pressure-driven membrane processes owing to its well-balanced performance between water flux and solute rejection as well as relatively low operation and maintenance costs [10–13]. In addition to this, NF has also been proven to be more suitable and reliable compared with the conventional treatment methods such as ozonation, coagulation and flocculation, and chemical oxidation [1,12]. In general, the wastewater generated from the textile industry is known to contain many types of dyes, salts, detergents, surfactants, and in some cases heavy metals, depending on the processing regime [12]. The wastewater thus can be considered to pose serious threat to the environment if the effluent is discharged into receiving stream without going through proper treatment process.

Numerous research works on textile effluent treatment using thin film composite nanofiltration (TFC-NF) membrane have been documented in the literature. It is, however, found that most of the performances of NF were assessed within a very short period of operation time [14–16] and/or tested using simple dye–salt mixture solution [17–19] which are hard to convince the readers of its reliability. Furthermore, many have common perception on the complete decolorization of NF in textile wastewater treatment without seriously taking into consideration the role of membrane pore structure and surface charge properties. It must be pointed out that different properties of NF might have different degrees of fouling resistance against dye adsorption, leading to difference in performance stability. In addition to this, dye molecules present in the effluent can also be varied in terms of molecular weight (M_W) and charge number (depending on the kind of product being processed in the textile industry), and results in different characteristics of effluent produced. This as a consequence may have significant impact on NF performance in particular water permeation rate in long process run. In this regard, selecting an ideal property of NF is particularly important to ensure maximum separation performance with minimal fouling.

The main objective of this study is to investigate the performances of two commonly used commercial TFC-NF membranes for the dyeing wastewater treatment process. These membranes with different properties will be evaluated in terms of water flux, salt rejection, and color removal under prolonged operation time so as membrane fouling tendency can be assessed. Chemical cleaning process will also be performed to determine the sustainability of NF membrane in treating dyeing effluent. It is expected that the outcomes of this study can provide instructive information on the ideal properties of NF membranes used in textile industry.

2. Experimental

2.1. Materials

Reactive Black 5, RB5 ($M_W = 991$ g/mol) supplied by Sigma and inorganic salts, i.e. NaCl and Na_2SO_4 purchased from Merck together with polyvinyl alcohol (PVA) ($M_W = 61{,}000$ g/mol and degree of hydrolysis = 98–98.8%) from Fluka were used as received to synthesize dyeing solution. The feed solutions were prepared using distilled water with pH value nearly 7. All the experiments were conducted at room temperature unless otherwise specified.

2.2. NF membranes

Table 1 shows the properties of two NF membranes purchased from DOWFILMTEC™ for the treatment of synthesized low contaminated textile dyeing solutions. Though these membranes are

Table 1
Properties of commercial NF membranes from the literature

Membrane	Selective skin layer	MWCO/pore diameter	Zeta potential (mV at pH 7)	PWF coefficient (l/m^2 h bar)	[a]$MgSO_4$ rejection (%)	Contact angle
NF90	1,3 phenylene diamine and benzenetricarbonyl trichloride [22]	100 Da [22] 0.68 nm [23]	∼−17 [25]	5.2 [22] 6.4 [23] 10.2 [27]	>97.0	80.3° (±3.2)
NF270	Piperazine and benzenetricarbonyl trichloride [22]	155 Da [23] 300 Da [24] 270 Da [28] 0.84 nm [23]	∼−44 [25] ∼−67 [28]	8.5 [22] 13.5 [23] 14.8 [24] 27.5 [27]	>97.0	14.1° (±5.9)

[a]Salt rejection based on the following test conditions: 2,000 ppm $MgSO_4$, 25°C, and 15% recovery at the pressure of 4.8 bar [26].

consisted of a very thin polyamide selective layer, the exact fabrication conditions are remained unknown to public mainly due to the trade secret in manufacturing these membrane products. From the table, it is clear that NF270 possesses relatively larger pore dimension and higher surface charge than that of NF90. The difference in membrane pore structure also leads to different permeation rates in which membrane having larger pore size demonstrates greater water flux and vice versa. In addition to this key parameter—pore structure, Bowen and Mohammad [20]—has reported that the changes in the membrane porosity and polyamide layer thickness might also play a role in changing NF water flux.

2.3. Membrane contact angle measurements

The contact angle measurement was performed using an optical contact angle analyzer (Model: OCA 20, DataPhysics Instruments GmbH, Germany). Ten measurements on different locations of the membrane sample were performed and averaged to yield the contact angles and their standard deviation.

2.4. Filtration experiments

Filtration experiments were conducted in a self-stirred membrane permeation cell (Model: HP4750, STERLITECH™) under nitrogen atmosphere. The cell which containing a circular-shape flat membrane (with 14.6 cm^2 effective area) was then placed on a magnetic stirrer in which a Teflon-coated magnetic bar was fitted into the cell and stirred at constant rotation of 250 rpm, providing agitation to minimize concentration polarization effect during filtration. The cell was operated in batch mode by charging the cell with 300 ml feed solution. During the filtration experiments, 25°C temperature and 3–18 bar operating pressure were used. The water flux of membrane, J ($l/m^2\,h$), at different pressures will be expressed as follows:

$$J = \frac{Q}{At} \tag{1}$$

where Q is the quantity of permeate (l), A is the effective membrane area (m^2), and t is time to obtain the quantity of Q (h).

Prior to NF of dyeing solution treatment, single salt filtration using 1,000 ppm NaCl and 1,000 ppm Na_2SO_4 aqueous salt solution was used to determine the efficiency of NF membranes on monovalent and divalent salt rejection. It is assumed that the concentration of the feed solution was remained almost constant as only a small quantity of the sample volume was taken for conductivity measurement and the permeate (after measurement) was recycled back to the feed solution. To assess the separation efficiency of salt, the rejection rate, R (%), was calculated according to the following formula:

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \tag{2}$$

where C_p and C_f are the solute concentration of the permeate (mg/L) and the feed (mg/L) solution, respectively, when the solute is a single electrolyte. The conductivity was measured using a portable conductivity meter (EC300, YSI Inc) and was converted into concentration according to the calibration curve (conductivity vs. salt concentration). For the artificial dye solution, C_p and C_f are the conductivity of the permeate and the feed solution, respectively, representing the concentration of all electrolytes involved or the concentration of the dye in the permeate and the feed solution, respectively, determined by measuring the absorbance at maximum wavelength (λ_{max}) of 592 nm using spectrophotometer (Model DR5000, Hach).

2.5. Artificial dyeing solution

In this study, the mixture dyeing solution composed of 1,000 ppm RB5, 5,000 ppm Na_2SO_4, 3,000 ppm NaCl, and 500 ppm PVA was prepared for NF performance evaluation. The concentration of each solute used in synthesizing dyeing solution is mimicking effluent discharged from textile factory as reported in the work of Mo et al. [10]. The reason for including PVA into synthesized dyeing solution is because PVA is used during sizing process to help smooth and strengthen fibers. It is believed that the presence of small quantity of PVA in the dyeing solution would not affect significantly membrane water flux and selectivity and thus was not considered as a main factor influencing membrane performance. It has been reported previously that PVA could be potentially retained by microporous UF membranes owing to its relatively high M_w [21].

To conduct this experiment, the NF membranes used for dyeing solution treatment process were first assessed by increasing the operating pressure from 10 to 18 bar. For each specified operating pressure, the membrane water flux and solute rejection rate for both salt and color were recorded. This was followed by NF fouling evaluation under prolonged operation time.

2.6. Cleaning process

Membrane cleaning process was carried out in accordance to the recommendations of DOWFILMTEC™. Both NF90 and NF270 membranes were immersed into a NaOH aqueous solution (0.1%) of 30˚C for 8 h to remove any possible foulants. The pure water flux of the cleaned membranes was then determined and compared with the virgin membranes in order to determine the flux decline due to irreversible fouling.

3. Results and discussion

3.1. Pure water permeability

The pure water permeability of the two commercial NF membranes is shown in Fig. 1. The filtration experiments were performed at pressure range of 3–11 bar and room temperature. As can be clearly seen, both membranes demonstrated linear line of high coefficient ($R^2 > 0.98$) in which NF270 showed greater pure water flux coefficient, L_p (20.51 l/m^2 h bar) than that of NF 90 membrane (5.43 l/m^2 h bar). The higher value of L_p for NF270 can be well-explained by its larger molecular weight cut-off (MWCO) as given in Table 1. The larger the MWCO of the membrane, the faster the water molecule is transported through membrane due to its relatively low transport resistance. It must be pointed out that the NF270 tested in this work demonstrated much higher L_p than that of reported previously [22–24], except the one reported by Hilal et al. [27]. The significance difference between these water fluxes of NF270 might be caused by the pretreatment procedure and/or operation at various pressure ranges. The main reason for the flux discrepancy, however, still remained unknown.

3.2. Salt rejection

Due to the insufficient data provided by the membrane manufacturer, filtration of single salt solution using NF membranes was carried out and the results of rejection and water flux are presented in Fig. 2. Studies on the salt removal rate are very important because NaCl and Na_2SO_4 are the most common salts being used in textile industry during dyeing process to enhance the dye fixation degree onto the fabric. From Fig. 2, it is found that NF90 showed higher salt rejection for both NaCl and Na_2SO_4 compared to NF270, despite its lower water permeation rate. The higher separation efficiency of NF90 could be mainly due to its relatively smaller pore structure which restricted the passage of dissolved salt. The salt rejection of Na_2SO_4 for both tested membranes was apparently much higher than NaCl rejection and this is consistent with the Donnan exclusion mechanism where divalent anions are highly rejected by NF membrane of negatively surface charge than that of monovalent anions. In addition to Donnan exclusion effect, Vrijenhoek and Waypa [29] on the other hand attributed the high rejection rate of Na_2SO_4 to the effect of size exclusion where the observed order of ion rejection was $SO_4^{2-} > Na^+ > Cl^-$.

Compared to the NaCl rejections of NF270 and NF90 reported in the literature, it is found that the rejections shown in this study were in good agreement with the one reported by Dalwani et al. [28] for NF270 and Al-Zoubi and Omar [30] for NF90 at the same operating pressure. Nevertheless, the rejection of NF270 against Na_2SO_4 was found to be lower than the literature data [31,32] and this might be partly due to different testing conditions (e.g. salt concentration, feed pH, applied pressure, etc.) used during characterization. It must be also pointed out that the presence of various types of salt in feed solution could also

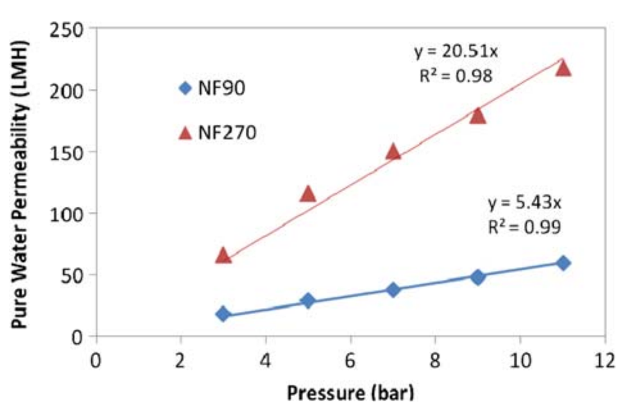

Fig. 1. Pure water permeability of NF membrane at different operating pressures.

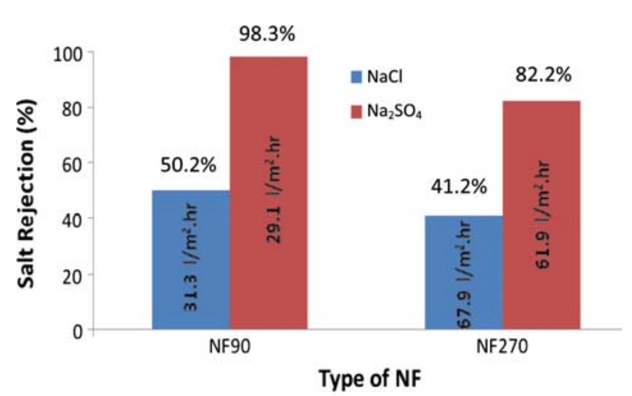

Fig. 2. Single salt rejection and water flux of NF membranes operated at 9 bar.

play a role in altering NF selectivity owing to different charge densities and hydrated sizes of ions [33].

Fig. 2 also shows the water flux of membranes operated at 9 bars under various types of salt solution. The results revealed that at the same concentration of salt solution, the NF membranes tested using divalent salt solution demonstrated lower water flux in comparison to when they were used for filtering monovalent salt solution. This phenomenon can be explained by the higher osmotic pressure resistance created by Na_2SO_4 in the aqueous solution [1].

3.3. NF of dyeing wastewater

Fig. 3 shows the performances of NF membranes of dyeing wastewater at different operating pressures. Compared to the pure water permeability shown in Fig. 1, the water flux of the membrane was significantly reduced in the treatment of dyeing wastewater. It is mainly because of the presence of various components in the feed solution, leading to considerable build up of solution's osmotic pressure. Significant increase in osmotic pressure of dyeing solution is unavoidable, but it can be offset with higher operating pressure during treatment process. It must be pointed out that NF90 showed unexpected flux behavior with increasing operating pressure. Only minor flux increase was experienced with an increase in pressure compared to the remarkable flux increment in NF270. The insignificant flux increase of NF90 can be explained by the fact that the membrane is more susceptible to compaction when it is operated at high pressure.

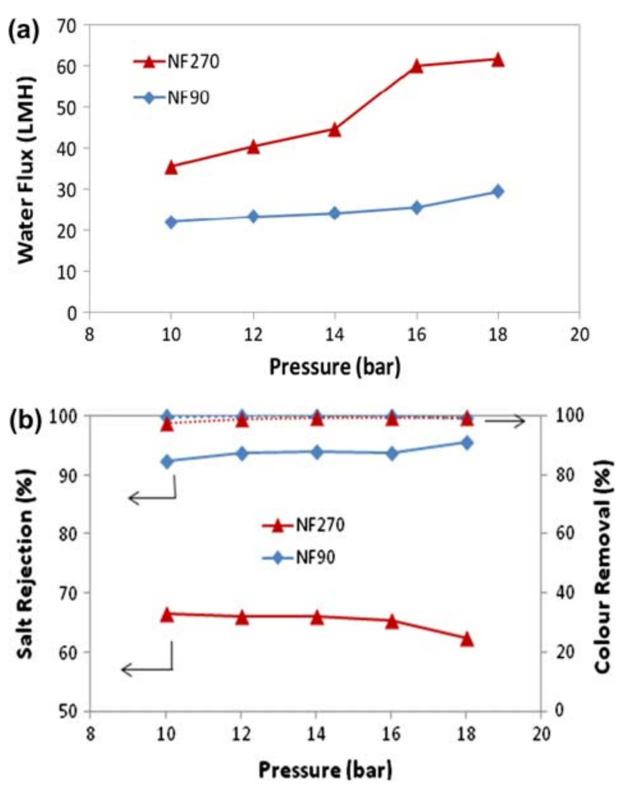

Fig. 3. NF of textile wastewater at different operating pressures (a) water flux and (b) solute rejection rate.

With respect to color removal, both NF membranes showed almost complete rejection of color regardless of the range of operating pressure studied. The excellent decolorization can be strongly attributed to the relatively smaller MWCO of NF than that of M_w of RB 5. It is generally agreed that sieving effect due to steric hindrance is the dominating factor governing the high rejection of RB 5 in NF membranes [3]. Though both NF membranes were able to achieve almost complete decolorization of dyeing solution, their efficiencies in salt removal, however, were strongly dependent on the membrane type selected, which have been evidenced in Fig. 2. Overall, it can be concluded that the salt rejection which was based on the conductivity of the solution (including the conductivity due to the charged RB 5) was around 93% for NF90 and around 65% for NF270, irrespective of operating pressure.

3.4. Fouling of NF

In order to study the fouling tendency of NF membrane during dyeing wastewater treatment process, filtration experiments were repeated up to four cycles using the same dyeing solution as prepared in Section 3.3. Fig. 4 presents the water flux of NF as a function of time for four continuous operation cycles. For each cycle, no permeate was recycled back to the feed solution and this as a consequence have led to increase in salt and dye concentration in the feed solution, lowering the permeate yield gradually with operation time. In this experiment, new filtration cycle would be conducted using the freshly prepared dyeing solution as soon as the previous cycle was completed. Compared to NF270, NF90 suffered remarkable flux decline every time when a new cycle was started. Initial water flux of NF90 was decreased from 33 l/m^2h at first cycle to around 21 l/m^2h at fourth cycle, recording around 36% flux decline after four continuous operation cycles. The remarkable flux deterioration of NF90 also revealed its low resistance against dye absorption. This finding can be supported by the evidence where the top surface of NF90 was stained with light-blue color due to RB 5 absorption. Comparing with the flux behavior of NF90, it is found that NF270 exhibited quite consistent water flux throughout the four operation cycles. Only around

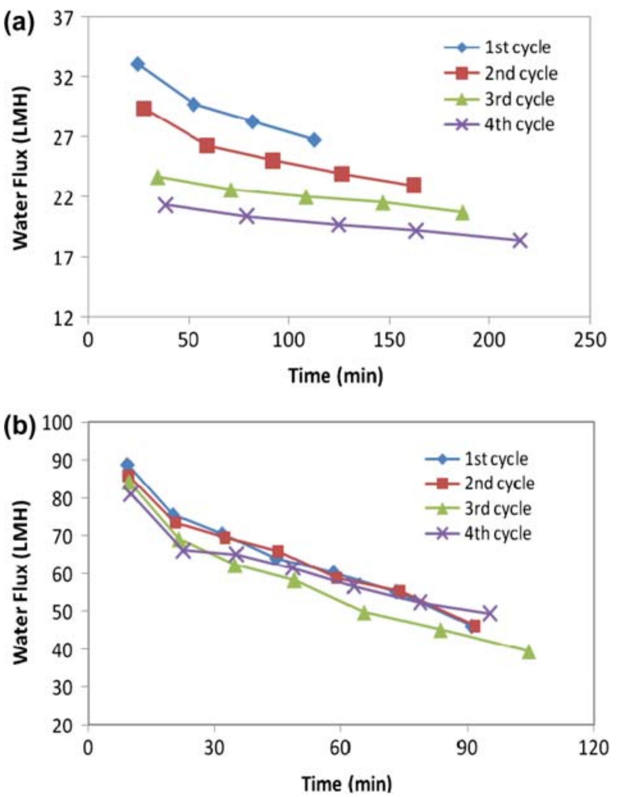

Fig. 4. Flux stability of NF membranes in dyeing wastewater treatment as a function of time, (a) NF90 operated at 12 bar and (b) NF270 operated at 15 bar.

8.9% flux decline was recorded after four cycles of operation. It must be pointed out that the flux decline in NF270 may be even lesser when it is used for industrial applications as NF270, which is sold in spiral wound format in the market comes with turbulent promoter that could provide synergistic effect to minimize fouling effect.

Fig. 5 presents the membrane stability as a function of time at different operation cycles. As the filtration experiment was conducted in batch mode operation, increasing salt concentration in the feed solution against the time was expected and this was confirmed by the significant increase in the feed solution conductivity measured at the end of filtration process of each cycle. In general, the increase in salt concentration would damp out electrostatic repulsion of NF, causing salt rejection rate to decrease. This explanation however seemed does not apply to NF90 where it showed high separation rate of salt (between 89 and 94%) throughout the experiments, irrespective of salt concentration in the feed. In view of this, the salt rejection of NF90 can be generally said to be dominated by sieving effect rather than Donnan exclusion effect. Unlike NF90, the tendency of salt rejection decrease was very obvious in NF270 and this is largely due to the remarkable decreasing Donnan exclusion effect. Since NF270 exhibited relatively high surface charge property compared with NF90, the

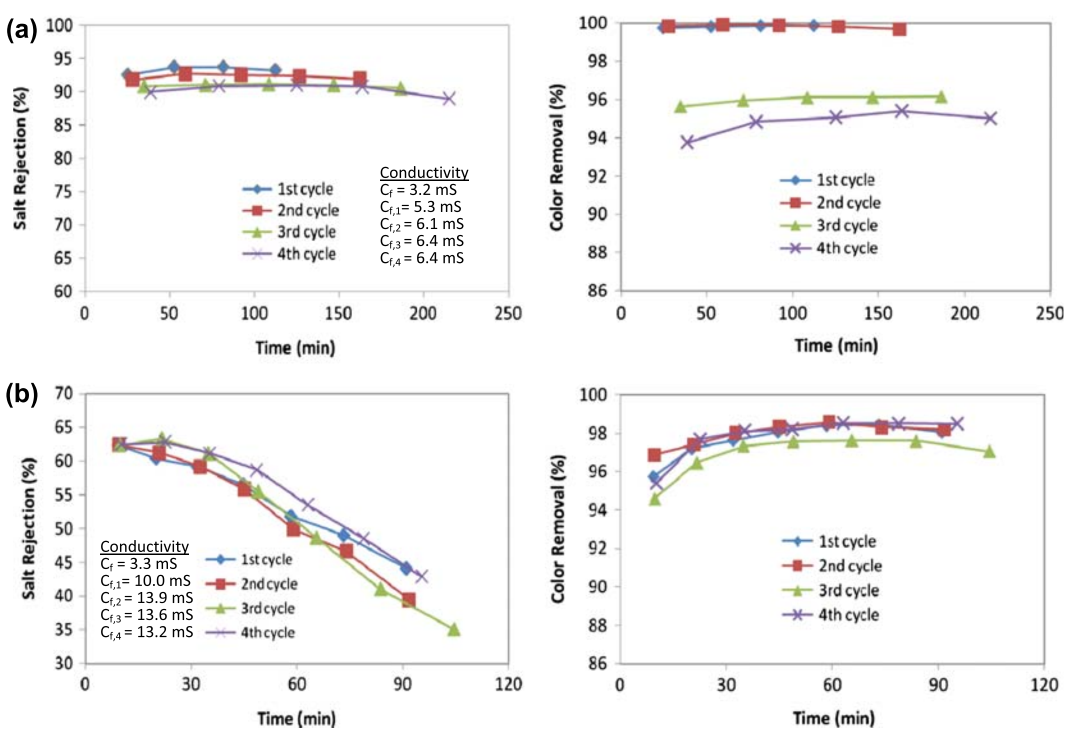

Fig. 5. Stability of NF membranes with respect to salt rejection and color removal, (a) NF90 operated at 12 bar and (b) NF270 operated at 15 bar.

increasing salt concentration at the feed side would play major role in diminishing the membrane electrostatic repulsion, making it less effective to reject salts. Nevertheless, it must be pointed out that though NF270 suffered significant loss in salt rejection as a function of time, its consistent separation performance in these four operation cycles revealing the decreased rejection is mainly due to the combination effects of increased salt concentration in the bulk and increased concentration polarization adjacent to membrane/feed solution interface, not the fouling problem due to solute attachment on the membrane surface. To verify the statement, a chemical cleaning process will be conducted to assess membrane fouling propensity and the findings will be presented in the following section.

With respect to color removal, it is found that NF90 suffered 4–5% and 1–2% loss in removal rate after second and third operation cycles, respectively. This decrease in separation efficiency can be explained by the fact that the membrane surface charge was affected following the adsorption of dye onto the certain portions of membrane surface which as a consequence decreased the effect of Donnan exclusion. In principle, the rejection of reactive dye is mainly governed by sieving effect, but the decreasing Donnan exclusion effect may play minor role in lowering the perfect rejection of dye component. This is mainly because of the negatively charged properties of RB 5 when it dissolves in aqueous solution as shown in Fig. 6. In addition to this, another possible explanation on the reduction in color retention could be due to the decreased membrane water flux going from operation cycle 1 to 4, leading to decreasing convective water flux over the time while the diffusion rate of dye molecules remained more or less constant.

Based on the data from Fig. 5, it is true to say that at the initial operation cycle, NF270 was not as excellent as NF90 in achieving almost complete decolorization of wastewater, but its stable performance in color removal which varied between 94 and 98% throughout the studied period has proved it to be more reliable in long run in industry.

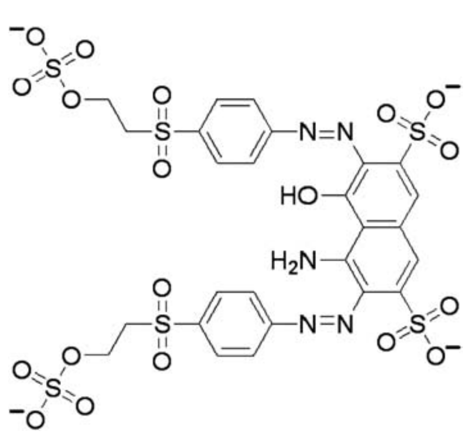

Fig. 6. Molecular structure of RB 5 dissolved in water solution.

3.5. Sustainability of NF

In order to assess the extent of fouling on the membrane surface, NF90 and NF270 used in Section 3.4 were subject to chemical cleaning process and the water flux of cleaned membranes was compared with their virgin state as shown in Fig. 7. The results revealed that the flux of NF90 was unable to be considerably recovered after cleaning process. Its PWF coefficient was dropped sharply from 5.43 l/m^2 h bar at virgin state to 1.74 l/m^2 h bar after cleaning process, indicating the significant changes on its characteristics as a result of irreversible fouling due to cake formation. In comparison to NF90, it is found that NF270 was less susceptible to fouling. With a simple chemical cleaning process conducted, as high as 90% of the original flux of NF270 could be retrieved. High recovery rate of NF270 can be strongly linked to its

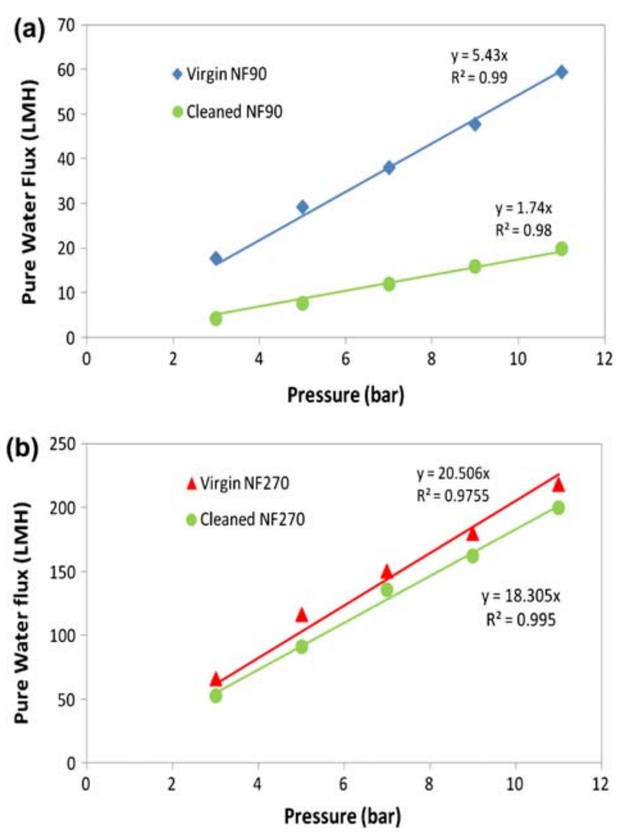

Fig. 7. Comparison between PWF of virgin NF membranes and cleaned NF membranes, (a) NF90 and (b) NF270.

relatively high surface charge density which in turn reduced the possibility of permanent dye particle penetration and/or pore blockage (i.e. irreversible fouling). Similar observation was also reported in our previously published work in which the dye adsorption could be potentially diminished with the use of strongly negatively charged NF membranes [3].

It must be noted that inappropriate chemical cleaning process may alter properties of membrane permanently. In view of this, assessment on the membrane separation efficiency after cleaning process was also performed and the results are presented in Fig. 8. It is interesting to note that both NF membranes demonstrated very similar dye removal rate compared to their performance (the fourth cycle of operation as shown in Fig. 5) prior to cleaning process. The salt rejection rate of NF, however, was slightly decreased after cleaning process and this indicated the decrease in electrostatic repulsion force. On the other hand, it must be pointed out that though the NF90 was still able to perform in removing salt and dye components from textile wastewater, its deteriorated water flux due to the irreversible fouling as shown in Fig. 7 confirmed its low sustainability for industrial applications.

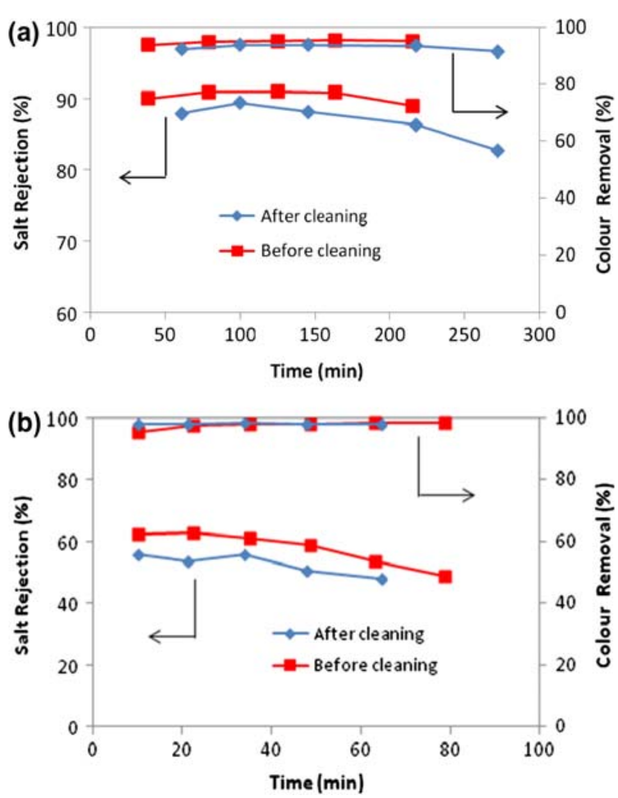

Fig. 8. Separation performance of NF before and after cleaning process, (a) NF90 and (b) NF270.

4. Conclusions

Two types of NF membranes with different properties have been selected in this study to treat the synthesized dyeing wastewater consisting of RB 5, NaCl, Na_2SO_4, and PVA. The results obtained in this study have led to the following conclusions:

(a) NF of dyeing wastewater showed almost complete decolorization of wastewater, mainly due to its significant difference between membrane pore size and size of dye molecules used. The efficiency of salt rejection, however, was strongly dependent on the properties of NF used. It is found that the mechanism involved in separating salt in NF90 was mainly due to sieving effect, whereas it was Donnan exclusion effect in NF270.

(b) NF270 which having higher surface charge density seemed to be favorable to reduce membrane fouling tendency due to the adsorption of dye components. It was recorded around 8.9% flux decline after four operation cycles of dyeing wastewater treatment in comparison to more than 36% decline in NF90.

(c) Simple chemical cleaning process was able to effectively retrieve the water flux of NF270. Results showed that as high as 90% of the water flux of NF270 could be recovered compared to only 32% in NF90. High recovery rate of NF270 can be strongly linked to its relatively high surface charge density which in turn minimized the extent of irreversible fouling. Besides, it is found that the chemical agent used during cleaning process was gentle to membrane structure because only a slight decrease in membrane salt rejection and insignificant change in color removal were experienced.

Based on these findings, it is showed that NF270 is more reliable and sustainable to be used in treating dyeing wastewater, though its efficiency in removing dissolved salts is not as high as NF90. It must be noted that the high water productivity of NF270 coupled with its high fouling resistance may outweigh its relatively low rejection rate of salt in industrial applications.

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