



Comparison between nanofiltration and forward osmosis in the treatment of dye solutions

Abdalmunem Ammar^a, Ibrahim Dofan^a, Veeriah Jegatheesan^{a,*}, Shobha Muthukumaran^b, Li Shu^a

^aFaculty of Science, Engineering and Built Environment, Deakin University, Waurn Ponds Campus, Geelong, Victoria 3216, Australia, Tel. +61 3 5227 3403; email: jega.j@deakin.edu.au

^bInstitute for Sustainability and Innovation, College of Engineering and Science, Victoria University, Melbourne, P.O. Box 14428, Victoria 8001, Australia

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ABSTRACT

Colour removal and the flux behaviour of nanofiltration (NF—DOW FILMTEC—NF245) and forward osmosis (FO—a flat sheet cellulose triacetate membrane with a woven embedded backing support) membranes were investigated in this study. The NF membrane was employed to perform dye removal experiments with aqueous solutions containing 15 g/L of NaCl and different concentrations of Acid Green 25, Remazol Brilliant Orange FR and Remazol Blue BR dyes. The increase in dye concentration resulted in a decline in water permeability and an increase in colour removal. When the concentrations of dye solutions varied from 250 to 1000 mg/L, at 0.8 bar of trans-membrane pressure, the NF system exhibited a steady permeate flux of more 30 L/m²h and a colour removal of more than 99%; salt rejection was more than 20.0%. Furthermore, the FO system possessed high dye rejection efficiency (almost 100%), with low permeate flux of around 2.0 L/m²h, when using dye solutions as feed streams and seawater as draw stream. The mode of operation (either FO or pressure retarded osmosis (PRO)) did not change the flux significantly but PRO mode always produced higher fluxes than FO mode under the operating conditions used in this study. While both NF and FO can be used to reduce the volume of effluent containing dyes from textile industries, the energy spent in NF on applied pressure can be substituted by the osmotic pressure of draw solution in FO when concentrated draw solutions such as sea water or reverse osmosis concentrate are readily available.

Keywords: Dye; Flux; Forward osmosis; Nanofiltration; Textile effluent

1. Introduction

The textile industry uses a large quantity of water (usually 200–400 L to produce 1 kg of fabric) [1] and

generates various types of wastewater differing in magnitude and quality. The wastewater from printing and dyeing units in a textile plant are often rich in colour, containing residual of reactive dyes and chemicals, and needs proper treatment before releasing into

*Corresponding author.

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the environment. Most synthetic dyes have complex aromatic molecular structures which make them inert and non-biodegradable [2,3]. For example, the high intake of reactive dyes, mainly in the cotton industry, increases the environmental issues, due to their low degree of exhaustion (60–90%) [4,5].

As a result of water scarcity, the treated textile effluent, commonly discharged to the sewer in the past, is nowadays being recycled in industrial processes for use when water quality meets the requirement. Numerous physico-chemical processes, e.g. coagulation [6,7], flotation [8], chemical oxidation [9], biodegradation like use of activated sludge, etc. were reported for textile effluent treatment. These treatment processes are still inadequate to remove the coloured dyes from the effluent stream. Adsorption is a commonly used process for the decolourisation [10,11]; however, the process is very slow and its performance is limited by the equilibrium. Since the discharge regulations are becoming more stringent there is a growing trend and attention in the advanced treatment methods like ozonation, photocatalysis and membrane processes for a better treatment of the textile wastewaters [12]. A membrane process is an attractive alternative and it is gradually progressing as a technically significant and commercially feasible process in the treatment of textile wastewater. This is because they have the potential to either remove the dyes stuffs or allow reuse of the auxiliary chemicals used for dyeing or to concentrate the dyestuffs and auxiliaries and produce purified water.

Several investigations have been carried out on the treatment of the dye effluents using microfiltration [13,14], ultrafiltration [15–17], nanofiltration [18–20] and reverse osmosis membranes [21–23]. Microfiltration is suitable for removing colloidal dyes from the exhausted dye bath and the subsequent rinses. UF is effective as single step treatment of secondary textile wastewater. NF and RO allow the separation of low molecular weight organic compounds and salts. The NF process combines the advantages of ultrafiltration by using moderate pressure and those of reverse osmosis by separating solutions, makes it possible to recover 97–99% of the water from wastewater, with sufficient quality to recycle the permeate into the dyeing process [24]. The permeate from NF is usually colourless and low in total salinity [25,26]. Yazhen et al. [24] studied cross-flow NF for the treatment of pure dye solutions as well as an industrial dye solution and demonstrated that the potential of the NF treatment for textile dye plant effluent. On other hand, Yu et al. [27] used NF process for the desalting and concentrating of aqueous dye at a dye producing plant. They have demonstrated that the newly developed process using NF was continuous in operation, was not labour

intensive, and produced high quality water. In recent studies by Dhale and Mahajani [28] has reported a combined process of NF and wet oxidation to treat the dye bath waste stream and found up to 99% of colour and 97% of COD were removed from NF and retentate was treated by wet oxidation process. Although conventional nanofiltration and/or reverse osmosis membrane process requires very high levels of chemical pre-treatment of the wastewater to remove the synthetic and natural oils which foul membranes, resulting a chemical waste stream after the water is removed.

Forward osmosis (FO) has recently drawn attention as a novel method for wastewater treatment, and sea water and brackish water desalination [29,30]. FO process operates on the principle of osmotic transport of water across a semi-permeable membrane from a dilute feed solution into a concentrated draw solution. The high concentration solution has a higher osmotic pressure than the feed solution, to draw water from the feed across the membrane to itself. As FO does not require high pressure for separation and thus, consumes lesser energy to produce water than NF system. In this work, both NF and FO process were used to treat three synthetic reactive dyes aqueous solutions (using Acid green 25, Remazol Brilliant Orange FR and Remazol Blue BR). The performance of both the processes was evaluated with regard to the permeate flux and separation efficiency.

2. Materials and methods

2.1. Materials

Acid green 25 (acid dye, MW: 622.58), Remazol Brilliant Orange FR (reactive dye, MW: 729.03) and Remazol Blue BR (reactive dye, MW: 626.54) were used in the experiments. The chemical structure of these dyes is shown in Fig. 1. Distilled water and

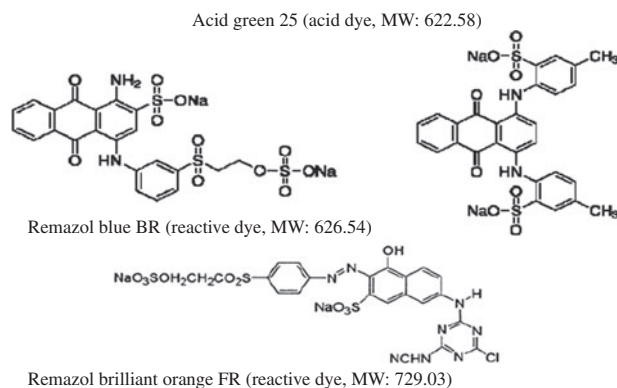


Fig. 1. Chemical structure of dyes.

sodium chloride (15 g/L) were also used in the preparation of dye solutions with different concentrations.

2.2. Apparatus

2.2.1. Nanofiltration

Nanofiltration experiments were performed in a dead-end mode laboratory scale NF unit with a DOW FILMTEC –NF245 membrane (Fig. 2(a)). The dye concentration in textile effluent can go up to 1,500 mg/L [31]; further, 15–50 g/L of NaCl concentration will be used in dyeing baths to get light to deep tone while dyeing cotton. Thus, we conducted our experiments with 15 g/L of NaCl and 250, 500, 750 and 1,000 mg/L of dye to represent textile effluent. Experiments were conducted at 200, 400, 600 and 800 kPa. Every experiment was run approximately for an hour and required measurements were taken every 15 min.

2.2.2. Forward osmosis

A flat sheet cellulose triacetate (CTA) membrane with a woven embedded backing support obtained from Hydration Technology Inc. USA and cross-flow membrane cell arrangement (Fig. 2(b)) were used in this study. The cross-flow rate of both the feed and the draw solutions was kept at 1.5 L/min. Experiments were conducted both in the FO and pressure retarded osmosis

(PRO) modes. In the FO mode, the feed solution was facing the active layer of the membrane and the draw solution was facing the support or the porous layer of the membrane. In the PRO mode, the feed solution was facing the support layer of the membrane and the draw solution was facing the active layer of the membrane. Experiments were carried out at $23 \pm 1^\circ\text{C}$ temperature. In all FO and PRO modes, the feed solution was prepared by using different concentrations (25, 500, 750 and 1,000 mg/L) of Acid Green 25 dye, Remazol Brilliant Orange and Remazol Blue for short-term experiments (2 h duration) and just one concentration (1,000 mg/L) of those dyes for long-term experiments (6 h duration). The salt (NaCl) concentration in the feed solutions was 15 g/L. The draw solution was sea water collected from Geelong beach which had more osmotic pressure than the feed solution.

2.3. Analytical methods

The colour of the feed and permeate samples was analysed with a Spectrophotometer (PHARO300 Photometer) at the following wave lengths: 612 nm for Acid green 25, 489 nm for Remazol Brilliant Orange and 577 nm for Remazol Blue. The pH was measured by a pH meter (PH 315i) and the conductivity was measured using a conductivity meter (Cond 3210). TOC was measured using Shimadzu. The dye rejection was calculated as follows:

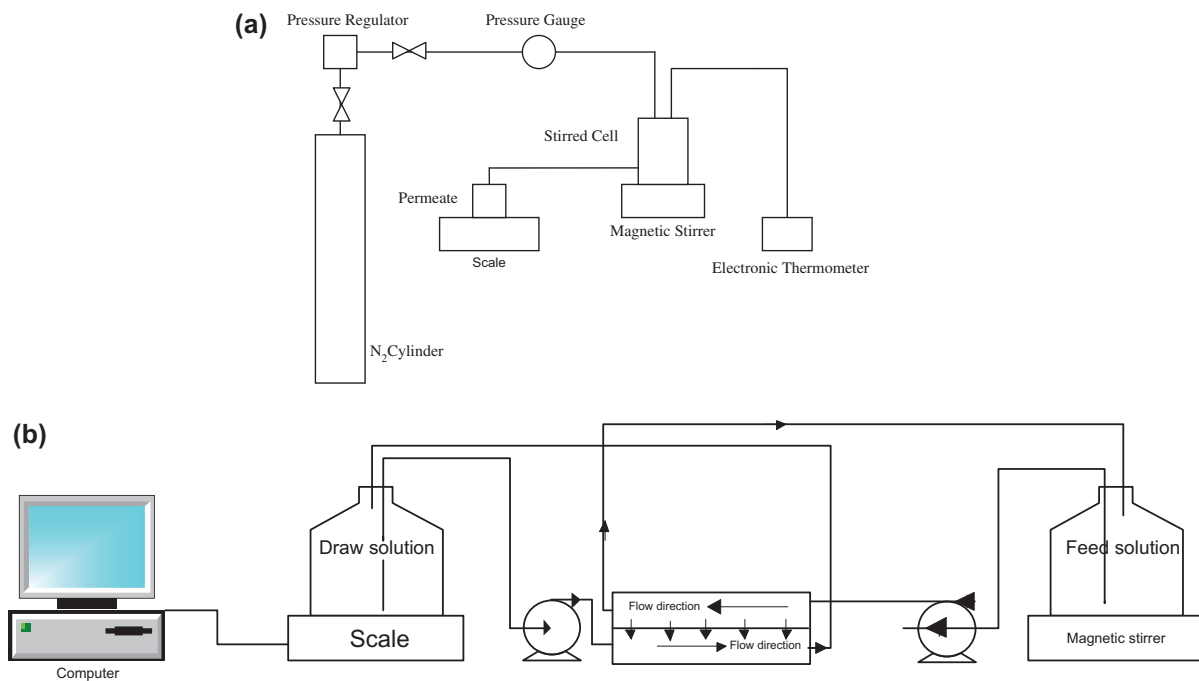


Fig. 2. Experimental set-up (a) Nanofiltration; (b) FO.

$$R(\%) = \left[1 - \left(\frac{C_P}{C_F} \right) \right] \times 100\% \quad (1)$$

where C_P and C_F are the dye concentrations in permeate and the feed solution, respectively.

Experimental water flux ($J_{w,e}$) was determined by:

$$J_{w,e} = \frac{(m_2 - m_1)/\rho}{A * t} \quad (2)$$

where m_2 is the final mass of the permeate collected after t minutes of nanofiltration and m_1 is the initial mass before t minutes of nanofiltration. Similarly, in FO, m_2 is the mass of the draw solution after t minutes of filtration and m_1 is the mass before the t minutes of filtration. The time interval t was 3 min for nanofiltration and 30 min for FO. The ρ is the density of the permeate in the nanofiltration ($= 1.015 \text{ kg/L}$) and in FO it is the water flowing from the feed side to the draw side which was considered as 1.00 kg/L and the A is the effective area of the membrane which was 0.00216 m^2 for NF and 0.0071 m^2 for FO membrane. The salt rejection by NF is low and therefore, the NF permeate will contain significant amount of salt and thus will have higher density. In order to incorporate this effect, the density of 1.015 kg/L is used in flux calculations related to NF. However, in the FO process, the passage of salt is low and it can be assumed that only the water is passing from the feed side to the draw side. Therefore, the density of water (1.0 kg/L) is used for flux calculations in the FO process.

3. Results and discussion

3.1. Nanofiltration experiments

3.1.1. Effect of dye concentration on dye removal

Fig. 3 shows the variation of dye rejection with dye concentration in the feed for different concentrations of Acid Green 25, Remazol Blue BR and Remazol Brilliant Orange FR. Very high percentage of dye rejections (more than 90%) were observed at all concentrations of dyes indicating the effectiveness of the molecular weight cut-off of pores of NF membrane (200 Da) that are smaller than the molecular weights of all three dyes (MW of Acid Green 25 = 622.58; MW of Remazol Blue BR = 626.54 and MW of Remazol Brilliant Orange FR = 729.03) tested. Further, as can be seen from Fig. 3, when the concentration of dye increased the removal of dye also increased for all type of dyes but not significantly. This is possibly due to the increase in concentration polarisation layer that was formed on the surface

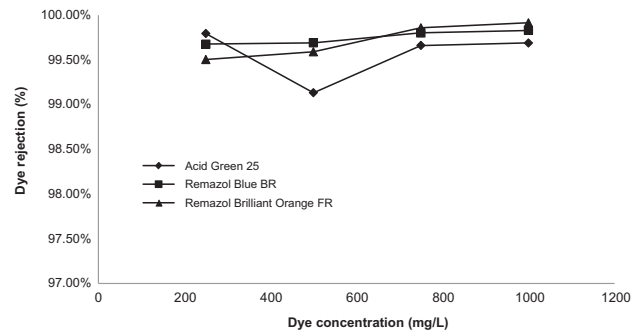


Fig. 3. Effect of concentration of dye on the rejection of dye during nanofiltration.

of NF membrane at higher concentrations of dyes in the feed solution. In addition to the above, increasing the dye concentration in feed will also lead to higher osmotic pressure in the feed side in the concentration

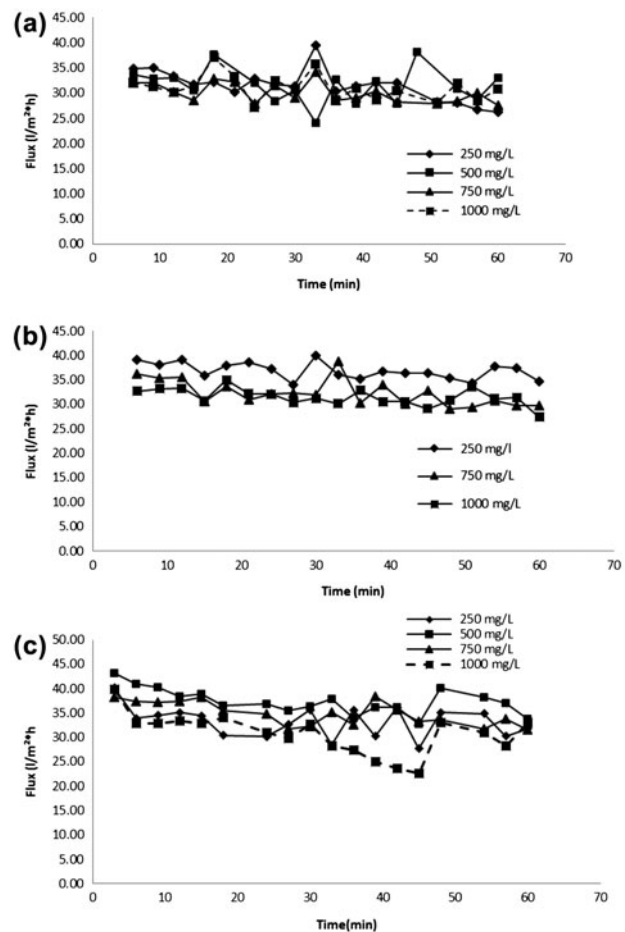


Fig. 4. Temporal variation of flux during nanofiltration (800 kPa) at various concentrations of dye (a) Remazol Blue BR; (b) Remazol Brilliant Orange FR; (c) Acid Green 25.

Table 1

Average fluxes and salt rejections during nanofiltration (at 800 kPa) for different concentrations of three dyes

	Acid green 25 (MW = 622.58)				Remazol brilliant orange FR (MW = 729.03)				Remazol blue BR (MW = 626.54)			
	250	500	750	1,000	250	500	750	1,000	250	500	750	1,000
Dye concentration (mg/L)	250	500	750	1,000	250	500	750	1,000	250	500	750	1,000
Average flux (L/m ² h)	33.26	37.87	35.23	30.37	36.86	–	33.41	31.64	31.17	32.01	30.47	32.19
Salt rejection (%)	16.75	21.31	15.17	11.33	19.75	–	19.25	19.35	16.37	14.87	21.37	18

polarisation layer which in turn will reduce the flux that has been discussed in the next section. However, we neither determined the concentration polarisation layer nor the osmotic pressure.

3.1.2. Permeate flux

Fig. 4(a)–(c) show the temporal variation of the flux at a constant operating pressure of 800 kPa. It is evident from those figures that the permeate flux decreases with the time of operation. As discussed above, the concentration polarisation increases the osmotic pressure in the feed side. This in turn reduces the net effective applied pressure which is important to maintain the flux. At low feed concentrations, the decrease in the permeate flux values are not substantial. Further, as can be seen from Table 1, the average flux generally decreases with the concentration of the dye in the feed solution; however, it is not significant. At lower concentration of dye (250 mg/L), Remazol Brilliant Orange FR gave higher flux (36.9 L/m²h) followed by Acid Green dye 25 (33.3 L/m²h) and Remazol Blue BR (31.2 L/m²h). However, at higher concentration of dye (1,000 mg/L) the average fluxes of all three dyes were very similar (30.4–32.2 L/m²h).

Salt rejection was computed using the increase in the conductivity of the feed solution. As can be seen

from Table 1, salt rejection was less than 20% and which indicated the removal of salt by nanofiltration is not efficient.

3.1.3. TOC removal during NF experiments

Table 2 represents the removal of TOC with time for the two different dyes (Remazol Orange and Remazol Blue) at four different concentrations and at the same operating pressure of 800 kPa. As TOC is directly proportional to the concentration, the profile of the removal of TOC shows the same trend that was shown by the profile of the removal of dye for both dyes. The maximum removal of TOC was up to 98 and 90% for Remazol Brilliant Orange and Remazol Blue respectively, after 60 min of nanofiltration. Thus concentrated rejectant in terms of dyes and TOC can be obtained through nanofiltration.

3.2. FO experiments

3.2.1. Effect of concentration on flux

Fig. 5(a) and (b) show the temporal variation of flux for the short-(2 h) and the long-(6 h) term FO experiments for Acid Green dye under FO mode. Similar profiles of Remazol Brilliant Orange FR are shown

Table 2

TOC concentration in the permeate during NF experiments

Time	Remazol brilliant orange FR				Remazol blue BR			
	250 mg/L	500 mg/L	750 mg/L	1,000 mg/L	250 mg/L	500 mg/L	750 mg/L	1,000 mg/L
Feed TOC	32.24	75.59	113.4	154.7	41.32	84.21	129.9	171.4
Permeate TOC after 15 min	4.906	5.243	5.874	3.311	10.64	11.66	14.24	17.97
Permeate TOC after 30 min	3.713	0.0*	2.579	2.366	7.065	9.876	13.85	18.3
Permeate TOC after 45 min	2.442	0.0*	12.84	2.266	6.306	9.876	14.26	18.64
Permeate TOC after 60 min	2.276	0.0*	2.053	3.867	8.988	10.95	14.45	19.16

*Negative TOC values were obtained while measuring and therefore they were changed to 0.0.

in Fig. 5(c) and (d) and of Remazol Blue (BR) are shown in Fig. 5(e) and (f). Fig. 5(g)–(i) show the temporal variation of flux for the short-term (2 h) FO experiments under PRO mode for Acid Green, Remazol Brilliant Orange FR and Remazol Blue (BR) dyes, respectively. It can be seen from Table 3 that PRO mode gave higher fluxes at all concentrations for all dyes. However, the flux starts to decline at a higher

rate in the PRO mode for Acid Green 25 when the concentration was increased. Thus, there is a possibility that the flux can be higher in FO mode when the concentration of Acid Green 25 dye is large (Fig. 6). Lower molecular weight of Acid Green could introduce higher diffusion of dye molecules which in turn could saturate the porous layer of the membrane under PRO mode when the concentrations are large.

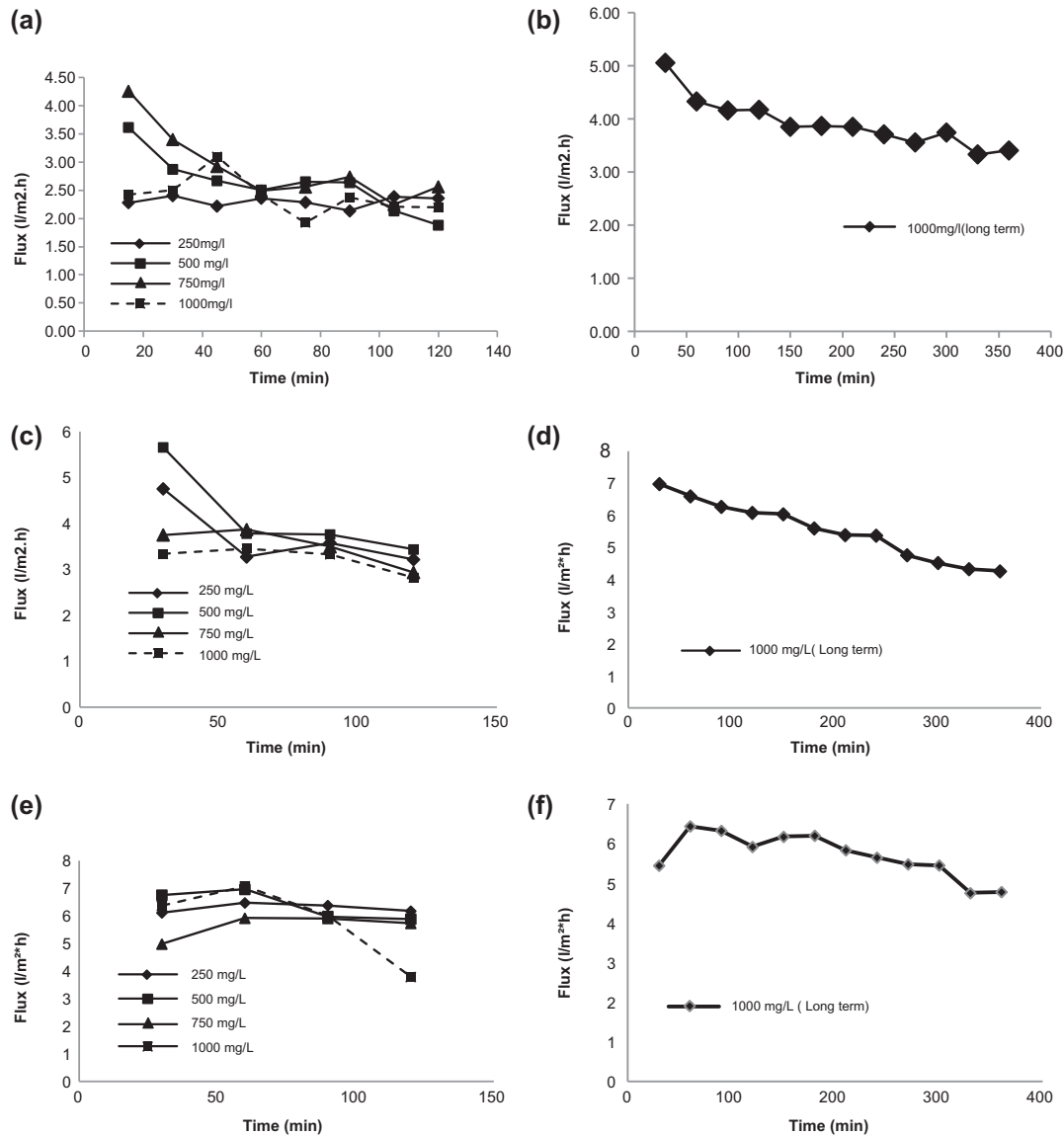


Fig. 5. Temporal variation of flux in FO experiments (a) Acid Green 25 (from 250 to 1,000 mg/L), FO mode and short-term experiment (2 h); (b) Acid Green 25 (1,000 mg/L), FO mode and long-term experiment (6 h); (c) Remazol Brilliant Orange FR (from 250 to 1,000 mg/L), FO mode and short-term experiment (2 h); (d) Remazol Brilliant Orange FR (1,000 mg/L), FO mode and long-term experiment (6 h); (e) Remazol Blue BR (from 250 to 1,000 mg/L), FO mode and short-term experiment (2 h); (f) Remazol Blue BR (1,000 mg/L), FO mode and long-term experiment (6 h); (g) Acid Green 25 (from 250 to 1,000 mg/L), PRO mode and short-term experiment (2 h); (h) Remazol Brilliant Orange FR (from 250 to 1,000 mg/L), PRO mode and short-term experiment (2 h); (i) Remazol Blue BR (from 250 to 1,000 mg/L), PRO mode and short-term experiment (2 h).

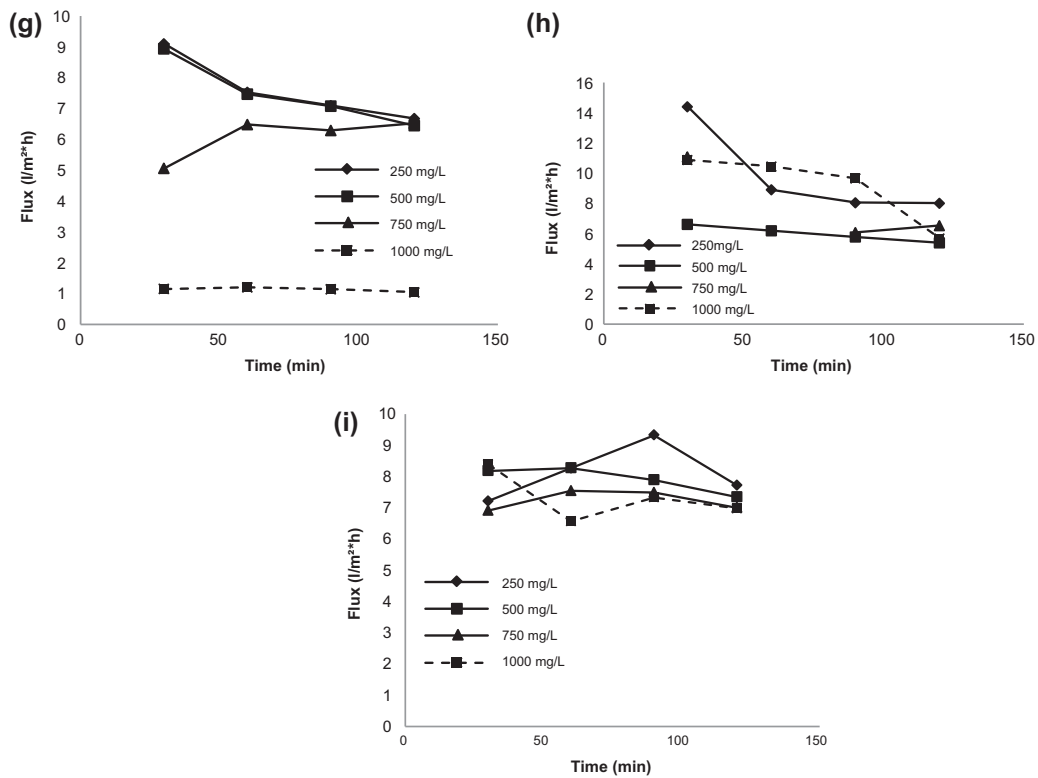


Fig. 5. (Continued)

This in turn will increase the concentrative internal concentration polarisation and reduce the flux. The above trend was less significant in the other two dyes as the decline in flux with the increase in concentration was minimal.

The NF can be used to recover water from the textile effluent discharged by the factory; the water that is recovered from NF process can be used in the appropriate textile processes. The selection of textile process where the water can be reused will depend on the

Table 3
Initial and average fluxes in short- and long-term experiments under FO and PRO modes in FO

	2 h experiment	2 h experiment	2 h experiment	2 h experiment	6 h experiment
Dye concentration (mg/L)	250	500	750	1,000	1,000
Acid green 25 FO mode initial flux (L/m ² h)	1.63	2.55	3.00	1.71	2.11
Average flux (L/m ² h)	1.62	1.85	2.04	1.69	2.76
Acid green 25 PRO mode initial flux (L/m ² h)	3.20	3.15	1.78	–	–
Average flux (L/m ² h)	2.67	2.63	2.14	–	–
Remazol brilliant orange FR FO mode initial flux (L/m ² h)	1.67	1.99	1.32	1.17	2.46
Average flux (L/m ² h)	1.68	1.87	1.62	1.49	1.94
Remazol brilliant orange FR PRO mode initial flux (L/m ² h)	5.08	2.33	3.91	3.84	–
Average flux (L/m ² h)	3.47	2.12	2.21	3.23	–
Remazol blue BR FO mode initial flux (L/m ² h)	2.15	2.38	1.75	2.24	1.92
Average flux (L/m ² h)	2.21	2.25	1.98	2.04	2.01
Remazol blue BR PRO mode Initial flux (L/m ² h)	2.54	2.88	2.44	2.96	–
Average flux (L/m ² h)	2.87	2.79	2.55	2.58	–

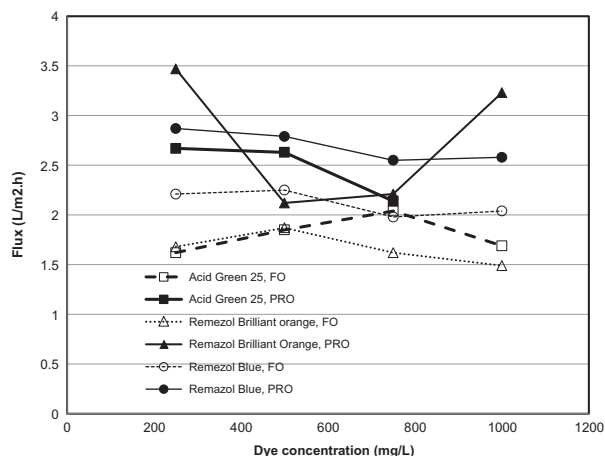


Fig. 6. Comparison of FO and PRO modes in producing fluxes at different concentrations of dye solutions.

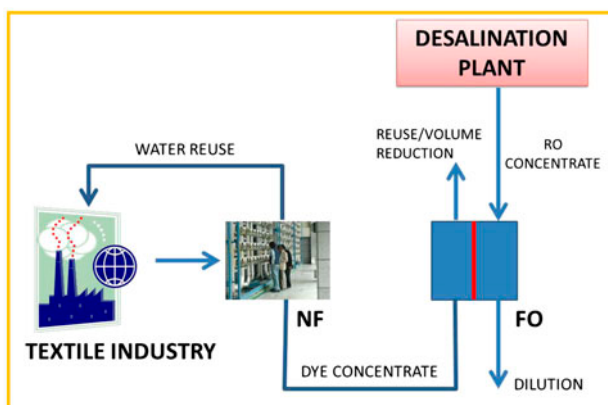


Fig. 7. Schematic of a hybrid NF and FO system to recover water and reuse or reduce the volume of dye concentrate.

water quality requirements of that process. This might need additional treatment to the NF permeate. The FO process can become effective to further concentrate textile effluent that is emerging from nanofiltration; this is possible when there is a concentrated stream with higher osmotic pressure (such as sea water or RO concentrate) is readily available to be used as draw solution. This has another added advantage as the RO concentrate will be diluted which can be either sent back to the RO process stream or discharged back to receiving bodies at a diluted state. A typical process flow diagram of such a concept is shown in Fig. 7.

4. Conclusions

Removal of three dyes (Acid Green 25, Remazol Brilliant Orange FR and Remazol Blue BR) by nanofiltration and FO and corresponding permeate fluxes

were investigated in this study. The following can be concluded which will be very useful in order to apply those systems at full scales in textile mills to recover dyes and reuse water.

The NF membrane (DOW FILMTEC –NF245) was employed in the experiments with aqueous solutions containing 15 g/L of NaCl and different concentrations of Acid Green 25, Remazol Brilliant Orange FR and Remazol Blue BR dyes. When the concentrations of dye solutions varied from 250 to 1,000 mg/L, at 0.8 bar of transmembrane pressure, the NF system exhibited a steady permeate flux of more 30 L/m²h and a colour removal of more than 99%; salt rejection was more than 20.0%. Increase in the concentration polarisation layer with the increase in the concentration of dye. Therefore, the increase in dye concentration generally resulted in a decline in water permeability and an increase in colour removal.

Furthermore, the FO system (a flat sheet CTA membrane with a woven embedded backing support) possessed high dye rejection efficiency (almost 100%); with low permeate flux of around 2.0 L/m²h. Dye solutions were used as feed streams and seawater was used as draw stream. The mode of operation (either FO or PRO) did not change the flux significantly but PRO mode always produced higher fluxes than FO mode under the operating conditions used in this study. While both NF and FO can be used to reduce the volume of effluent containing dyes from textile industries, the NF permeate flux was 15 times higher than FO permeate flux. But the performance of FO in long-term experiments was better. Hybrid NF and FO system could be used to recover water and reuse dyes in textile mills. Reducing the volume of dye effluent (if it is considered as waste) will save cost associated with transport and disposal; discharging the diluted RO concentrate back into the sea will reduce the adverse effect on the marine flora and fauna.

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