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Abstract: Carpet printing wastewater (CPW) was spiked with metal-complex dyes at concentrations of 10 and 30 mg/L to investigate the effect of feed color on separation performance of nanofiltration (NF). The rejection was excellent; 98–100% for color and COD under all spiking conditions. Although the flux decline increased with increasing dye concentration, the concentration polarization was the main cause of the flux decline. The effect of surfactants on NF separation performance was also investigated by preparing synthetic wastewaters with dyes and auxiliary chemicals. The presence of a non-ionic penetrant did not adversely affect the color rejection whereas the COD rejection was reduced from 100% to 91%. Furthermore, fouling became dominant when surfactants were used.

Keywords: Carpet printing wastewater, color, dye, nanofiltration, recovery, surfactant

INTRODUCTION

Manufacturing of synthetic carpets is a textile subcategory where acid dyes and metal-complex dyes are generally used. These dyes have fixation rates as high as 80–93% (1) and therefore lead to the generation of wastewaters

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with relatively less intense color contents. However, wastewaters containing these dyes are potentially more problematic due to their toxicity and carcinogenicity associated with the formation of aromatic amines and the presence of metals such as copper and chromium.

Printing is a widely applied method for dyeing of the synthetic carpets where auxiliary chemicals such as uniformity and antifoaming agents, and thickeners are used in addition to the dyes. These chemicals are surfactants and they are not consumed during the dyeing process, ending up in the wastewater stream causing complex characteristics such as increased organic content, toxicity, and foaming. Carpet printing wastewaters (CPW) are mainly generated from the print after-wash process, which is ranked as one of the highest water demanding unit processes with a water consumption rate of around 110 L/kg (2, 3). A typical range of 25–228 L/kg is also reported for print fixation and washing (4). Recalling the scarcity of the earth's fresh water supplies and growing problems of environmental pollution, reclamation of carpet printing wastewaters becomes an urgent need.

Color is an important parameter in the process of water recovery. In carpet dyeing the original process waters have a groundwater quality and the successful dyeing process requires that the process water is free of any color. Therefore the recovered water quality has to meet the criteria of being colorless (5), which directly relates to the performance of the technology used for water recovery.

Membrane separation technology has been widely applied for water recovery from textile effluents. Provided that an appropriate pre-treatment stage is applied, nanofiltration (NF) and reverse osmosis (RO) can produce permeates with excellent qualities for recovery purposes. High performances have been reported with almost complete removal of color and COD, and high rates of water recovery (6–14). However, one major drawback of membrane separation systems is the flux decline, which occurs due to concentration polarization and fouling. This means reduced productivity and a costly operation due to the need for frequent cleaning. Dye concentration has been reported to influence flux significantly, where flux decreased with increasing dye concentration (15). Similarly, surfactants have an adverse effect on flux as they are preferentially adsorbed on the membrane surface, leading to concentration polarization and/or fouling depending on their concentrations (16, 17). Therefore maintaining a high performance of the membrane systems, which is crucial in the implementation of the membrane technology, requires that the effects of wastewater components on the membrane performance are well understood. To this end, this study aims to investigate the effects of increased dye concentration, i.e., increased feed color and the presence of surfactants on the performance of NF for the recovery of process waters from printing stream of a carpet dye house. The broad changes of wastewater color in the industrial scale is associated with the use of different dyes at different concentrations. Therefore, the effect of dye concentration was studied by spiking the real wastewater with varying dye concentrations in order to simulate the real case. Because, in the carpet dyeing industry only the type and concentration of dyes are subject to change in order to obtain the desired color on the carpet whereas the auxiliary chemicals remain the same. To this end, the effect of surfactants on the separation performance and the flux decline was studied with synthetic wastewater, which enabled the investigation of their effects individually.

EXPERIMENTAL

Sample

Wastewater Sample

The wastewater sample was a chemically precipitated composite mixture of four individual carpet printing wastewaters, which was formed in order to compensate for the diverse characteristics of each sample. Chemical precipitation (CP) was applied at an optimum alum dose of 250 mg/L, which had been predetermined as the best pre-treatment process for CPW (18). The chemically precipitated CPW contained 418 ± 6 mg/L COD; 496 ± 3 mg/L total solids; 6.3 ± 0.4 NTU turbidity; 2.46 ± 0.05 UVA_{197} and 33.0 ± 0.1 mg/L total hardness as $CaCO_3$. The CPW samples were generated during the washing procedure of the carpets after they had been dyed with a print paste, which includes metallized acid dyes, sodium anhydrous sulphate and the auxiliary chemicals (Table 1).

Spiking Test

Dyes were spiked into the CPW after chemical precipitation. Dye solutions were prepared by using concentrations of 10 mg/L and 30 mg/L of single

Table 1. Print paste contents

Chemical name	Function/specification
Yellow 2R (acid yellow) Red G (acid red) Grey G (acid black)	Metal-complex azo dyes, contain cobalt and chromium III, harmful to aquatic organisms, may cause long-term adverse effects in aquatic environment
Citric acid	pH adjustment
Nofome 1125	Silicone antifoaming agent
Tanaprint ST 160 Conc	High electrolyte-resistant synthetic thickener for sprey dyeing and printing of carpets with acid and metal-complex dyes, an anionic ammonium salt for adjusting the viscosity
Tanasperse CJ	Non-ionic penetrant, for increasing wetting and penetration and producing homogeneous paste

Table 2. Characteristics of real wastewater and synthetic wastewater

Type of wastewater	Parameter		
	COD (mg/L)	Color (Pt-Co)	Total solids (mg/L)
Real wastewater-chemically precipitated	418 ± 6	66 ± 1	496 ± 3
Real wastewater-spiked with dyes			
Dye: Y2R (10 mg/L)	446 ± 15	384 ± 4	501 ± 16
Dye: Y2R (30 mg/L)	465 ± 26	1044 ± 17	580 ± 6
Dyes: Y2R + RG + GG (30 mg/L)	488 ± 16	908 ± 9	666 ± 9
Synthetic wastewater-prepared with dyes and surfactants			
Dye: RG (10 mg/L)	18 ± 0	376 ± 12	[a]
Dyes: Y2R + RG + GG (30 mg/L)	25 ± 3	922 ± 0	[a]
Dye + Surfactant: RG + Tanasperse CJ (10 mg/L + 0.2 mg/L)	415 ± 0	382 ± 2	[a]
Dye + Surfactant: RG + Nofome 1125 (10 mg/L + 0.1 mg/L)	199	435	[a]

[a]Not measured.

and multiple LANASET dyes that had originally been present in the collected wastewater samples (Table 2). These dyes were namely Yellow 2R (Y2R), Red G (RG) and Grey G (GG). The selected dye concentrations correspond to the high color values aimed in the wastewater, i.e. approximately 500 Pt-Co and 1000 Pt-Co. The effects of single dye and multiple dyes were studied separately, and since these dyes belonged to the same class, Yellow 2R was chosen randomly as a single dye. The composition of the CPW after spiking is given in Table 2.

Synthetic Wastewater

Synthetic wastewaters (SW) were prepared in the laboratory by dissolving single dye (RG) and three dyes (Y2R, RG, GG) in distilled water (DW) (Table 2). The concentrations of dyes were chosen based on the spiking tests performed with real wastewater and the concentrations of the two auxiliary chemicals, namely Tanasperse CJ and Nofome were calculated based on their concentrations used in the actual dyeing process.

Nanofiltration

NF experiments were carried out by a lab-scale plate and frame module, LabStak M20 (product of DSS Company) in cross-flow and total recycle mode of filtration (TRMF) (Fig. 1). The NF membrane (NFT-50) consisted of three layers: an ultrathin polyamide barrier layer, a microporous polysulfone interlayer and a high strength polyester support. It was a hydrophilic membrane with a contact angle of 40° (Wilhemy Method) and $MgSO_4$ rejection $\geq$99%. The NF membrane had an effective area of 0.036 m^2 and it was tested under a trans membrane pressure (TMP) of 5.90 bar. The samples were fed to the system at a flow rate of 6 L/min. The permeates were analyzed for their COD, color, turbidity, and total solids contents.

Flux Declines

The permeate fluxes were monitored throughout the experiments in order to determine the flux declines. The permeates were collected in a graduated cylinder and the fluxes were calculated by dividing the permeate volume by the collection period and the effective membrane area. The flux declines were evaluated based on the calculations shown in Table 3. In this way, the extent of concentration polarization and fouling were determined. The flux measurements were performed in four steps:

1. Initial clean water flux (J_{cwi}): the first flux determined with the clean membrane, which was subjected to an initial chemical cleaning procedure by the manufacturer's recommendation,

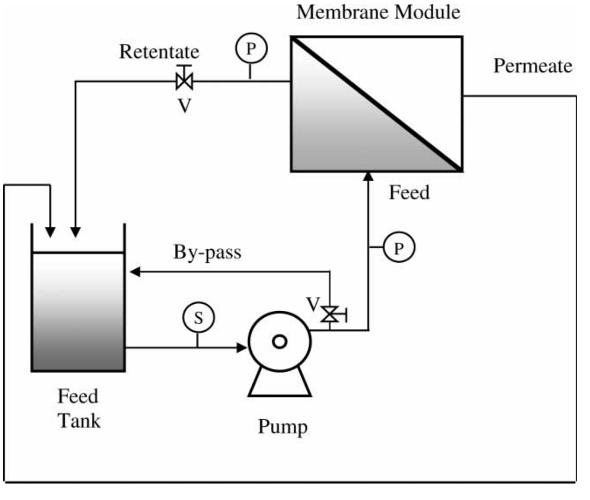

Figure 1. Experimental set-up for NF tests (P: pressure gauge, S: suction gauge, V: valve).

Table 3. Flux decline analysis

Calculation	Explanation
$(J_{cwi} - J_{ww})/J_{cwi}$	Total flux decline
$(J_{cwf} - J_{ww})/J_{cwf}$	Flux decline due to concentration polarization
$(J_{cwi} - J_{cwf})/J_{cwi}$	Flux decline due to fouling (irreversible + reversible)

2. Wastewater flux (J_{ww}): the wastewater flux stabilized with time during filtration,
3. Clean water flux of the fouled membrane (J_{cwf}): the clean water flux measured with the fouled membrane,
4. Clean water flux of the cleaned membrane (J_{cwc}): the clean water flux measured with the cleaned membrane after the cleaning procedure.

Membrane Cleaning

All the membranes used in filtration tests were cleaned by the clean-in-place (CIP) method where the membranes were exposed to a solution of nitric acid (pH 3) for 30 min followed by a solution of sodium hydroxide (pH 9–10) for 30 min while they were in the module. The cleaning procedure was applied in order to remove the organic and inorganic precipitates on the surface of the membranes and hence recover the fluxes.

Analytical Techniques

COD was measured using HACH DR-2000 Model spectrophotometer at wavelengths of 620 (high range) and 420 nm (low range), respectively. Color measurements were performed by the same instrument, which was already calibrated for color measurement in terms of Pt-Co at a wavelength of 455 nm. Ultra violet absorbance (UVA) measurements were performed by a Varian Cary 100 Model spectrophotometer at a wavelength of 197 nm, at which the highest absorbance values were obtained. Turbidity was measured with a HACH Model 2100A turbidimeter. Total solids content of the samples were determined by gravimetric analysis. All the analyses were performed according to the Standard Methods (19), except COD, which was measured following USEPA approved HACH Method 8000.

RESULTS AND DISCUSSION

Effect of Color on NF Performance

The original low color of the real wastewater was increased from 66 Pt-Co up to 1044 Pt-Co by spiking the dyes at concentrations of 10 mg/L and 30 mg/L.

During the membrane tests, the color was monitored continuously both in the feed and the permeate streams. Although the feed composition remains relatively constant in the total recycle mode of filtration, the feed color decreased significantly at 20–30% in the first 1 h of the experiments, indicating the accumulation of the dyes on the membrane surface (Fig. 2). This was also visually observed when the membranes were taken out of the module and the color on the membranes could not be removed completely by chemical cleaning.

The separation performance of the NFT-50 membrane for real wastewater and synthetic wastewater is given in Table 4. In real wastewater, when the single dye concentration was 10 mg/L, the color rejection was the same as compared to the condition of "no dye spiking", i.e. 100% removal efficiency remained the same when the feed color of 66 Pt-Co was increased to 384 Pt-Co. Similarly, the NF performance was very good when the single dye concentration was increased to 30 mg/L; a feed color value of 1044 Pt-Co was reduced to 4 Pt-Co in the permeate, yielding a removal efficiency of 99%. The performance of the NFT-50 membrane still remained high with a removal efficiency of 99% when the wastewater was spiked with three dyes at a total concentration of 30 mg/L. These results are expected since the dyes used in spiking tests belong to the same class (Table 1), leading to the same separation performance of NFT-50 membrane when the dye concentration was increased either with a single dye or a combination of three dyes at the same concentration. In case of synthetic wastewater, the color removal was similarly very high and the permeate color was as low as 8 Pt-Co at a feed color of 922 Pt-Co.

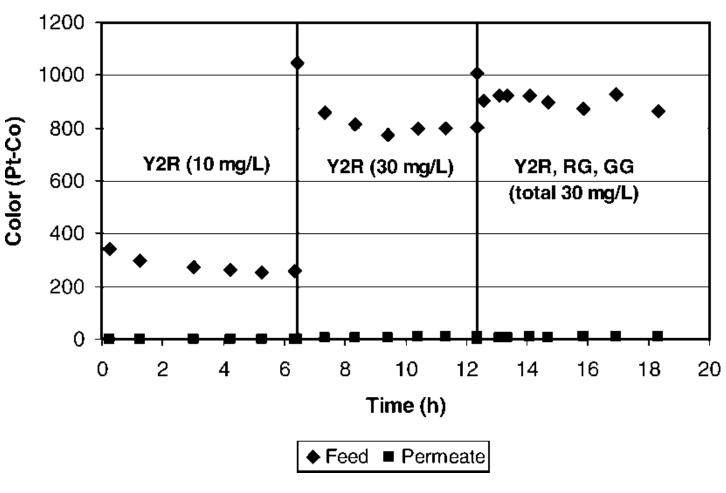

Figure 2. Effect of dye concentration on NF performance for spiked wastewater.

Table 4. NF performance for real wastewater and synthetic wastewater

Type of wastewater	NF permeate quality (Percent removal)		
	COD (mg/L)	Color (Pt-Co)	Total solids (mg/L)
Real wastewater-chemically precipitated	16 ± 2 (96)	0 ± 0 (100)	36 ± 7 (93)
Real wastewater-spiked with dyes			
Dye: Y2R (10 mg/L)	11 ± 3 (98)	0 ± 0 (100)	29 ± 1 (94)
Dye: Y2R (30 mg/L)	17 ± 2 (97)	4 ± 1 (99)	27 ± 7 (95)
Dyes: Y2R + RG + GG (30 mg/L)	19 ± 2 (96)	13 ± 1 (99)	86 ± 20 (87)
Synthetic wastewater-prepared with dyes and surfactants			
Dye: RG (10 mg/L)	0 ± 0 (100)	0 ± 0 (100)	[a]
Dyes: Y2R + RG + GG (30 mg/L)	0 ± 0 (100)	8 ± 1 (99)	[a]
Dye + Surfactant: RG + Tanasperse CJ (10 mg/L + 0.2 mg/L)	38 ± 0 (91)	2 ± 1 (99.6)	[a]
Dye + Surfactant: RG + Nofome 1125 (10 mg/L + 0.1 mg/L)	0 ± 0 (100)	1 ± 0 (99.8)	[a]

[a]Not measured.

The COD removal efficiency was also very high, i.e., 96–98%, and did not deteriorate due to the spiking of dyes in real wastewater. This is an expected result since the increase in the feed COD due to spiking of dyes was not high, which means that the addition of LANASET dyes did not cause a significant increase in the organic matter content of the wastewater. Similarly, increasing the dye concentration from 10 mg/L to 30 mg/L in synthetic wastewater did not reduce the COD rejection efficiency, which was 100% in both cases (Table 4). Meanwhile, the total solids content of the real wastewater increased from 496 mg/L to 666 mg/L via spiking of three dyes at a total concentration of 30 mg/L, resulting in a slight decrease of removal efficiency.

The NF permeate quality was compared to the original process water quality and the reuse criteria (Table 5). Although the NF permeate quality was slightly worse than that of the original process water in terms of color and organic matter, it was much better than the reuse criteria set for all the parameters. This comparison revealed that the NF permeate quality was acceptable for reuse in the dyeing process.

Table 5. Comparison of NF permeate quality with original process water and reuse criteria for real wastewater

Parameter	Original process water	NF permeates	Reuse criteria
Color (Pt-Co)	1	0–13	0–20 (20)
COD (mg/L)	[a]	11–19	8–40 (5, 8, 21, 22)
Total solids (mg/L)	446	27–86	500 (4)
Total hardness (mg/L)	11	0	60 (4)
Turbidity (NTU)	0.15	0.15–0.16	15 (4)

[a]Total organic carbon (TOC) = 2 mg/L.

Effect of Surfactants on NF Performance

In an attempt to distinguish the effects of dyes and surfactants on the NF performance, four sets of TRMF experiments were run with SW prepared in the laboratory. The non-ionic penetrant (Tanasperse CJ) caused the highest organic matter content in the SW (Table 2) and the removal performance of the NFT-50 membrane is presented in Table 4. The presence of surfactants in addition to the dye RG had a negligible adverse effect on color rejection. However, COD rejection reduced from 100% to 91% due to the presence of non-ionic penetrant (Tanasperse CJ), which created the highest organic load in the SW. On the other hand, the antifoaming agent (Nofome) was ineffective (Table 4). This may be due to the fact that the concentration of the antifoaming agent was half of that of the non-ionic penetrant. This may also indicate a possible difference in the molecular sizes of these two surfactants, causing different rejection performances.

Effect of Dyes and Surfactants on Flux Declines, Concentration Polarization, and Fouling

The effect of increasing the feed color on the permeate flux is depicted in Fig. 3 for real wastewater. The flux decline was 17% when the CPW was not spiked with dyes. In spiking tests, the increase of Y2R concentration from 10 mg/L to 30 mg/L caused the flux decline to increase from 14% to 24%. On the other hand, introducing three dyes at a total concentration of 30 mg/L resulted in a flux decline of 27%. These results indicate that Y2R, RG and GG had similar influence on the flux decline and increasing the concentration rather than the number of dyes played a role in reducing the permeate flux.

The changes in flux declines were also monitored for SW (Fig. 4). The flux declines were 3%, 5%, 10%, and 7% for single dye, three dyes, single dye + non-ionic penetrant, and single dye + antifoaming agent, respectively.

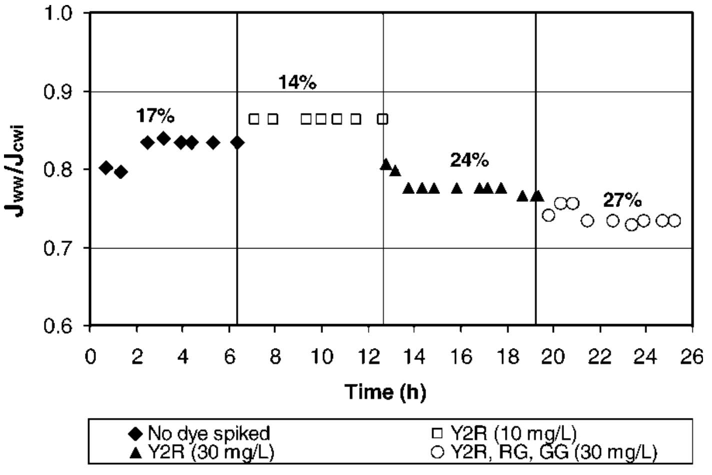

Figure 3. Effect of dyes on flux decline of real wastewater.

The flux declines were quite low in SW as compared to the real wastewater (Fig. 3), which increased from 3% to 5% only by increasing the dye concentration from 10 mg/L (single dye) to 30 mg/L (three dyes). These results clearly indicate that the effect of the dye concentration on the flux decline is more pronounced in real wastewaters. This is quite expected since the real wastewater contains both dyes and auxiliary chemicals, which have combined effects on the flux declines.

The presence of a surfactant in addition to the dye resulted in increased flux declines in SW, although the increase was not high (Fig. 4). The

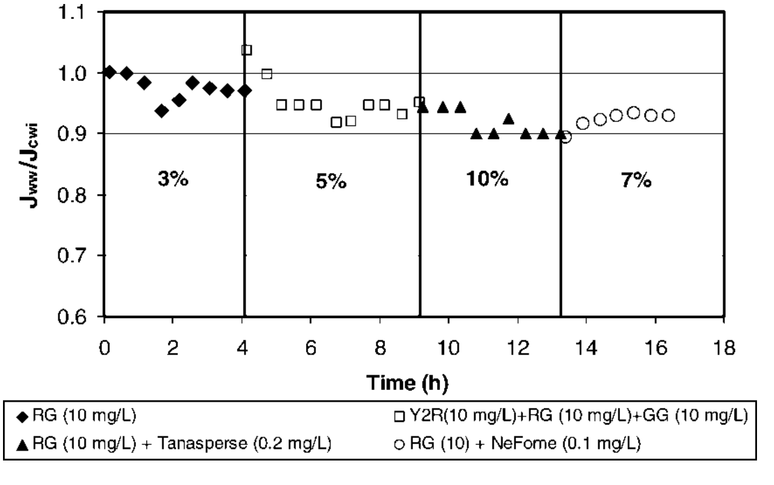

Figure 4. Effects of dyes and surfactants on flux decline of synthetic wastewater.

Table 6. Flux decline analysis for real wastewater and synthetic wastewater

Type of wastewater	Flux ($L/m^2/h$): Clean water, Initial (J_{cwi})	Clean water, Final (J_{cwf})	Clean water, Cleaned (J_{cwc})	Wastewater or syntetic water (J_{ww})	Flux decline (%): Total $(J_{cwi} - J_{ww})/J_{cwi}$	Concentration polarization $(J_{cwf} - J_{ww})/J_{cwf}$	Fouling $(J_{cwi} - J_{cwf})/J_{cwi}$
Real wastewater-chemically precipitated	34.8	33.6	—	29.0	16.7	13.7	3.4
Real wastewater-spiked with dyes							
Dye: Y2R (10 mg/L)	33.6	34.3	31.8	29.0	13.7	[a]	[a]
Dye: Y2R (30 mg/L)	31.8	29.7	33.1	24.3	23.6	18.2	6.6
Dyes: Y2R + RG + GG (30 mg/L)	31.3	27.7	32.8	23.0	26.5	17.0	11.5
Synthetic wastewater-prepared with dyes and surfactants							
Dye: RG (10 mg/L)	29.3	28.1	29.3	28.5	2.9	[b]	[b]
Dyes: Y2R + RG + GG (30 mg/L)	29.7	28.8	32.5	28.0	5.7	2.8	3.0
Dye + Surfactant: RG + Tanasperse CJ (10 mg/L + 0.2 mg/L)	27.4	25.2	28.3	24.7	9.9	2.0	8.0
Dye + Surfactant: RG + Nofome 1125 (10 mg/L + 0.1 mg/L)	27.3	25.3	27.8	25.3	7.2	0	7.2

[a]Could not be calculated since the final clean water flux was higher than the initial clean water flux.
[b]Could not be calculated since the final clean water flux was less than the synthetic wastewater flux.

presence of non-ionic penetrant caused higher flux decline (10%) as compared to the antifoaming agent (7%). This result is in agreement with the removal performance, as the COD removal efficiency was reduced only by the presence of the non-ionic penetrant.

The fractions of flux declines, namely concentration polarization and fouling were also compared (Table 6). Concentration polarization, which leads to a reversible flux decline, is responsible for greater fraction of the total flux decline for real wastewater when spiking was not performed. This is also valid for the spiking of single dye Y2R at a concentration of 30 mg/L where the flux declines due to concentration polarization and fouling were 18% and 7%, respectively. The effect of fouling increased from 7% to 12% when three dyes were spiked at the same concentration 30 mg/L. On the other hand, the concentration polarization remained at 17–18% by changing the type of dyes although the total concentration was kept constant at 30 mg/L. This result may indicate that the dyes Yellow 2R, Red G, and Grey G, although belong to the same class, have different characteristics in terms of their fouling potential.

In case of synthetic wastewater prepared with three dyes, the concentration polarization and fouling had almost equal effects on the total flux decline (Table 6). The flux decline due to concentration polarization was only 2.8% in SW when three dyes were added at a total concentration of 30 mg/L. This clearly indicates the significant role of surfactants on flux decline. The addition of surfactants in SW lead to increased fouling. At a total flux decline of 10%, the flux decline due to fouling was 8% and the fraction of concentration polarization was only 2% for the non ionic penetrant (Tanasperse CJ). In case of the antifoaming agent (Nofome 1125) all the flux decline was totally due to fouling (Table 6). The surfactants preferentially adsorb to the membrane surface, leading to fouling, whereas the dyes often build up near/on the membrane surface, creating a concentration polarization, which has a reversible effect on the flux. These results reveal that the more complex the wastewater composition becomes, the higher is the flux decline with more severe fouling.

CONCLUSIONS

- The NF permeate quality was very good and it was acceptable for reuse purpose.
- The highest flux decline occurred due to the addition of three dyes and flux decline increased when the dye concentration increased in real wastewater.
- The presence of a non-ionic penetrant in synthetic wastewater caused a reduction in COD removal efficiency. The non-ionic penetrant had a worse effect than the antifoaming agent on the flux decline of synthetic wastewater.

- The spiked metal-complex dyes had a pronounced effect on flux decline of the real wastewater due to their combined effects with auxiliary chemicals. Increased complexity resulted in an increased flux decline.
- The concentration polarization was dominant in real wastewater spiked with dyes; however, fouling increased with increased number of dyes. When the auxiliary chemicals were added together with the dye, fouling became very dominant.

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