



Recovery of caustic from mercerizing wastewaters of a denim textile mill

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ABSTRACT

The objective of this study was to evaluate caustic recovery from mercerizing wastewater originating from a denim textile producing plant using membrane technology. For this purpose, ultrafiltration (UF) and nanofiltration (NF) processes were considered. In the first stage, in an attempt to control the possible membrane fouling, pretreatment alternatives of flocculation, centrifugation, and microfiltration were evaluated. These pretreatment application alternatives were unsuccessful as they did not provide considerable color and solids removal. In the second stage, UF and NF processes were tested using a tight UF membrane (GR95PP, Alfalaval) and three NF membranes (NP010 and NP030, Microdyn Nadir, and MPF34, Koch Membranes) to accomplish the caustic recovery without applying any pretreatment. The best performance was obtained with NP010 NF in terms of permeate flux along with color and COD rejections. Then, for this membrane the effects of transmembrane pressure (4.03 and 6.23 bar), cross-flow velocity (from 0.40 to 1.40 m/s), and feed temperature (20 and 40°C) were investigated. Temperature positively affected the permeate flux without significant loss in recovery and rejections. Caustic stream produced had about 98–100% of NaOH in the feed at a concentration of 30–40 g/L and therefore was recyclable after a concentration process.

Keywords: Caustic recovery; Mercerizing wastewater; Membrane filtration; Textile industry

1. Introduction

Textile companies often face shortage of water resources not only because of water scarcity but also discharge limits [1,2]. These facts have shifted considerations toward water saving and recovery alterna-

tives for the textile industry [3–5] and implied that treating textile wastewaters in an end of pipe approach is unsuitable. In general, it is accepted that membrane processes are realistic solutions for the treatment of textile wastewaters targeting at recovery [6]. Traditionally used methods (e.g. biological degradation with activated sludge) are insufficient not only

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for obtaining the required water quality [1] but also for water and chemical recovery. In textile production, various chemicals and additives are used to process fibers for improving process efficiency or providing some features to the fibers. These chemicals make textile effluents complex. Basic chemicals used in textile processing are alkali, acids, salts, and solvents [1,7].

Mercerization is one of the major finishing processes of cotton textile production where caustic (NaOH) is the main chemical used. This process is generally applied to increase dye affinity, luster, and strength of fiber. Wherever it is applied, mercerization process is followed by an intensive hot water rinsing or washing process to remove excess caustic. Caustic is responsible for the high pH values (12–13). Beside this, because of strong alkali and high temperature, fiber ceases solids and dye absorbed in previous processes results in high dissolved solid content in the waste stream. Although these materials are not in a structure which contributes to BOD load much, produced strongly alkaline and colored effluent constitutes important environmental problems and also affects further treatment mechanisms negatively. As a result, a highly alkaline (60–70 g/L NaOH) and hot wastewater including various impurities is generated. This wastewater is problematic due to its high caustic content and necessitates neutralization. Its treatment is costly due to neutralization requirement that corresponds to the loss of caustic for textile mills.

At this point, the advantageous alternative for the disposal of caustic mercerizing effluents is the simultaneous reclamation of wastewater and recovery of caustic. Currently, there are two types of caustic recovery methods from mercerizing wastewaters. One of them is staged evaporation which has been applied since 1900. In this technique, with a multi-staged evaporation process, weak caustic in rinsing waters of caustification process can be concentrated up to reusable values. After concentrating caustic soda it should be purified [8]. However, this method is not widely used due to operational difficulties arising from broad range of chemicals applied in the textile processing. These problems have induced the development of the second method which is based on membrane filtration processes. Membrane filtration applications are considered as a good alternative for both reuse of wastewaters and recovery of caustic. Among membrane filtration processes, ultrafiltration (UF) is generally regarded as satisfactory for the removal of particles and macromolecules from textile effluents [9,10] but not ionic species. Further treatment by nanofiltration (NF) or RO is needed for the separation of low molecular weight organic compounds and divalent salts and hence for caustic recovery [10,11].

Reclamation of highly alkaline wastewaters and caustic recovery using membrane filtration has found applications in a wide range of industrial fields. Zhao and Xia [12] used a pilot-scale stainless steel UF coupled with an NF system for the recovery of sodium hydroxide from wastewater discharged after caustic treatment of shrimp residue in chitin industry. This investigation showed that UF coupled with NF can recover the NaOH solution efficiently (96%). In another study, reuse of cleaning-in-place caustic cleaning solutions in the dairy industry was investigated and NF reported to provide a high caustic recovery yield (>97%); and therefore to be an efficient membrane separation for the recovery of caustic solutions. A permeate from NF was reported to be a clear cleaning solution with low soluble COD (0.2–3.5 g/L) which could be successfully exploited owing to its cleaning potential [13]. In another study, about caustic recovery from soda (CIP) solution in dairy plants, the microfiltration (MF) operation reported to be the more appropriate operation as it retained surfactants at a lower cost. But, COD content of MF effluents were relatively high compared to values obtained with UF and NF [14]. In a few of the recent investigations, NF has been applied to recover caustic from mercerizing process effluents from both cotton and polyester fabric manufacturing [10,15]. Choe et al. [15] operated a pilot scale NF system (The SelRo, MPT-34 PES) for the recovery of NaOH and concentration of disodium terephthalate (DST) from wastewater discharged after the caustic treatment of polyester fabrics. The NF operation resulted in a concentrated DST solution and in a permeate of caustic soda solution containing 84% of the feed NaOH. Although successful caustic recovery efficiency was reported, membrane performances were not assessed in full and no data was provided in terms of COD or color removal. In fact, the recovered caustic solution can only be used if its color and organic matter content are sufficiently low.

As presented above, the information available in the literature in the field of caustic recovery from mercerizing effluents is still very limited. Mercerizing wastewaters are very high in color, suspended solids, COD, and also temperature. As mercerizing wastewaters are typically produced at 80–90°C, the consideration of high temperature as an operational parameter is vital. Our primary intent in this paper is to evaluate caustic recovery from mercerizing wastewaters by membrane processes with the goal of producing a caustic permeate stream that can be reused. The removal of suspended solids by MF and UF prior to NF was assessed. In the light of results from pretreatment tests, NF process was studied. Membrane performance was monitored in terms of permeate flux and

caustic retention. Meanwhile, color and COD rejections of the membranes were also followed as the reuse of the recovered caustic solution requires low color and low COD. Bearing in mind real operating conditions of mercerization operations, the effect of elevated temperature on NF were also evaluated along with the effects of cross-flow velocity (CFV) and transmembrane pressure (TMP).

2. Materials and methods

2.1. Wastewater

Caustic mercerization wastewater samples used throughout the study were obtained from the mercerization line of a denim textile mill (Fig. 1). As the mercerization line employs seven post-rinsing tanks in-line, caustic effluents are generated from each of these post-rinsing tanks. All these wastewaters flow into a channel and form the mixed wastewater from the mercerizing process. Five weekly samples were collected from each rinsing tank effluent, characterized, and used in the experiments (Table 1). The

constituents' levels presented in Table 1 are the averages for five samples collected.

As can be noted from Table 1, the concentration of wastewater constituents decrease considerably from the first to the last post-rinsing stage of the mercerization line. Considering that the wastewater flow rate from each rinsing stage is almost the same (200–300 L/min), it is calculated that more than 85% of NaOH is discharged from the first two rinsing stages of the mercerizing line. Based on this fact, the wastewater mixture from the first two rinsing tanks (with 1/1 volume ratio), was subjected to the treatment for alkali recovery, COD, and color removal.

In MF experiments, wastewater that is pre-filtered using 500 µm metallic strainer was used using a dead-end conventional vacuum filtration experimental system.

2.2. MF experiments

In MF experiments, a dead-end filtration device, in which filtrate is collected in a chamber placed on top

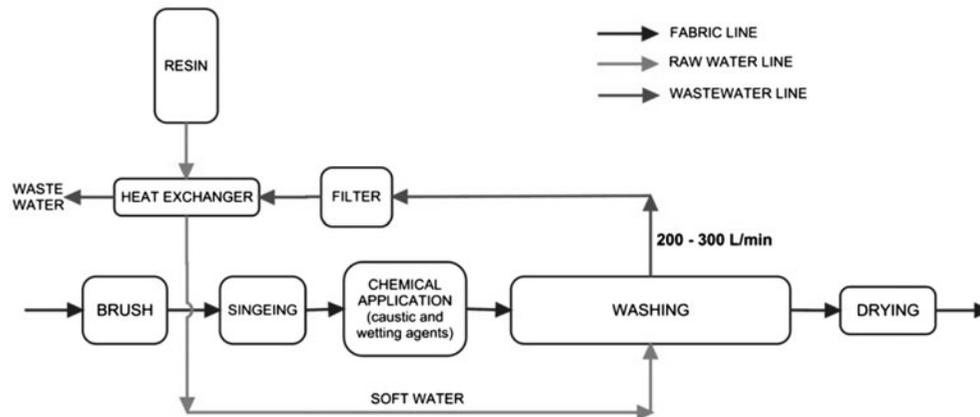


Fig. 1. General flow diagram of a denim mercerizing process.

Table 1
Mercerizing effluent characteristics

Parameter	Wastewater from post-rinsing tank #						
	1	2	3	4	5	6	7
pH	12.3	12.1	11.8	11.7	11.4	11.1	8.9
Conductivity, mS/cm	150	60	20	13	9	5	1
TSS, mg/L	1,632	793	248	172	144	131	40
Color, Pt-Co	10,113	7,059	3,689	3,651	3,568	3,078	844
Turbidity, NTU	879	584	534	561	406	379	138
COD, g/L	8.5	5.2	2.3	2.1	2.3	2.0	0.5
NaOH, g/L	67.5	34.7	6.3	2.8	1.9	1.4	–

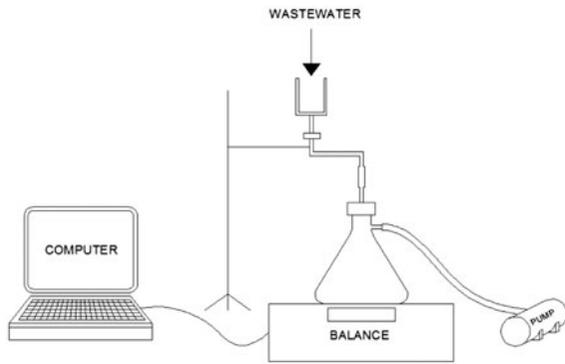


Fig. 2. Computerized dead-end MF unit.

Table 2
General specifications of the MF membranes

Filter	Chemistry	Pore size (µm)	Maximum operating temperature (°C)
Millipore SVLP04700	Hydrophilic PVDF	5	85
Millipore LCWP04700	Hydrophobic PTFE	10	260
GE S50WP320F5	Hydrophilic PES	5	135
GE S99WP320F5	Hydrophilic PES	10	135

of a balance, that is connected to a computer for continuous monitoring of the filtrate rate was used (Fig. 2). In these tests, different 5 and 10 µm MF membranes that can withstand high temperatures and extreme pH conditions were tested. The specifications of the MF membranes used throughout the study are summarized in Table 2. In order to increase the reproducibility of the results, MF tests with each membrane were repeated 6–7 times under the same conditions and the filtrate rate was monitored.

2.3. UF and NF experiments

UF and NF experiments were performed using a cross-flow plate-and-frame module (DSS LabStak M20) with a total membrane area of 0.036 m² in total recycle mode at constant temperature (Fig. 3). One UF and three NF membranes with the specifications presented in Table 3 are tested.

Throughout UF and NF experiments, permeate flow was recorded with respect to time until a steady permeate flux rate was reached. When the fluxes were stabilized, permeate and feed samples were collected and analyzed for the parameters of COD, color, pH, electrical conductivity, and Na content.

NF experiments were run at different TMPs, CFVs, and temperatures in order to investigate their effects. Attempted operational conditions relative to membrane type are summarized in Table 4. In the

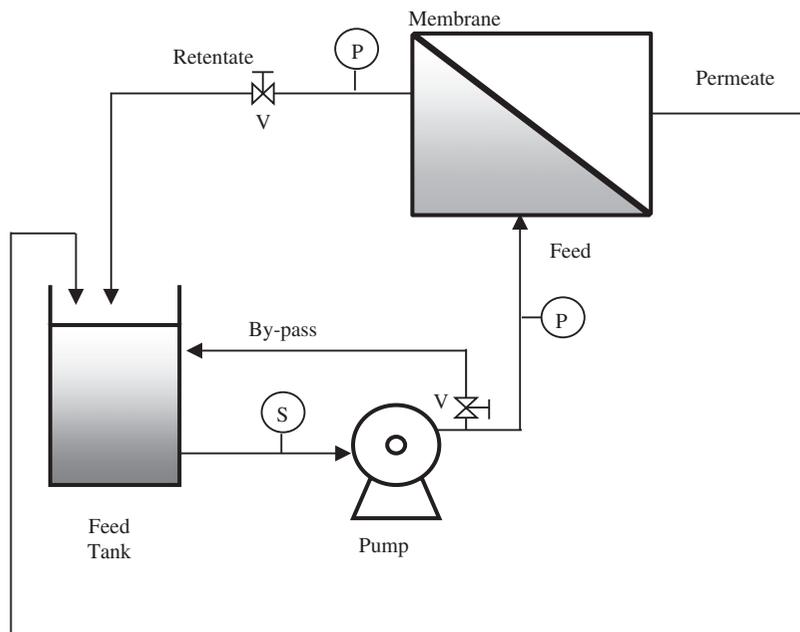


Fig. 3. Experimental set-up for UF and NF tests (P: pressure gage, S: suction gage, V: valve).

Table 3
Specifications of the UF and NF membranes

Membrane	Type	Membrane material	Max. temp (°C)	Nom. MWCO* (g/mol)	Max. pres. (bar)	pH range
DSS GR95PP	UF	PES on polypropylene	75	2,000	10	1–13
Microdyn-Nadir NP010	NF	PES hydrophilized	95	1,000	40	0–14
Microdyn-Nadir NP030	NF	PES hydrophilized	95	500	40	0–14
Koch MPT-34	NF	PES on polypropylene	70	300	35	0–14

*Nominal molecular weight cut-off.

Table 4
Applied operational conditions during UF and NF studies

Membrane	TMP (bar)	CFV (m/s)	Temperature (°C)
GR95PP	4.03	0.79	20 ± 2
NP030	4.03	0.79	20 ± 2
NP010	4.03	0.79	20 ± 2
NP010	4.03	0.79	40 ± 2
NP010	4.03	0.40	40 ± 2
NP010	4.03	0.40	20 ± 2
NP010	4.03	1.40	20 ± 2
NP010	6.23	0.79	20 ± 2
MPT-34	4.03	0.79	20 ± 2

experiments at 40°C, feed temperature was adjusted using a heater in the feed tank of the module.

2.4. Analytical methods

Electrical conductivity and pH measurements were conducted using Hach Sension 378 pH-conductivity-dissolved oxygen meter. Color and COD measurements were performed using a HACH DR-2000 model spectrophotometer following USEPA approved HACH method #8000. Turbidity was measured using HACH Model 2100A turbidimeter. For the determination of total suspended solids (TSS) content, gravimetric analysis was applied. In order to measure NaOH content of the samples, Na content was measured according to standard methods using Jenway Flame Photometer [16]. Since there are no other Na compounds except NaOH in the wastewater, caustic content was calculated directly from the measured Na concentration.

3. Results and discussion

3.1. Pretreatment

At the first stage of pretreatment studies, MF membranes with 5 µm (PVDF) and 10 µm (PTFE) pore size were tested. With each filter, seven parallel runs were

conducted and the time variation of permeate flux was monitored. Fig. 4 presents the results with 5 µm filter. As can be seen, permeate flux decreased very rapidly at all runs, and reached to a steady state within 25 s. Suspended solids in the wastewater clogged the membranes quickly within a very short time interval (about 10–30 s) resulting in very rapid flux declines. Thus, although these membranes are durable to alkaline conditions they were found to be inefficient for pretreatment.

Subsequently, polyethersulfone (PES)-based membranes which are hydrophilic and resistant to very high temperatures (135°C) were tested. For this purpose, two different pore sized (5 and 10 µm) PES-structured MF membranes were applied. As being different from previous MF experiments, clean water fluxes were also evaluated before and after filtrating wastewater in order to assess fouling behavior. These filtration tests were run at a pressure difference of 0.925 bar and the results are presented in Table 5. At the beginning of the filtration tests, clean water fluxes from 5 to 10 µm virgin membranes reached to their steady values of about 25 and 23 L/m²/h, respectively, within a similar short time period of about 20–30 s. However, after being used for wastewater

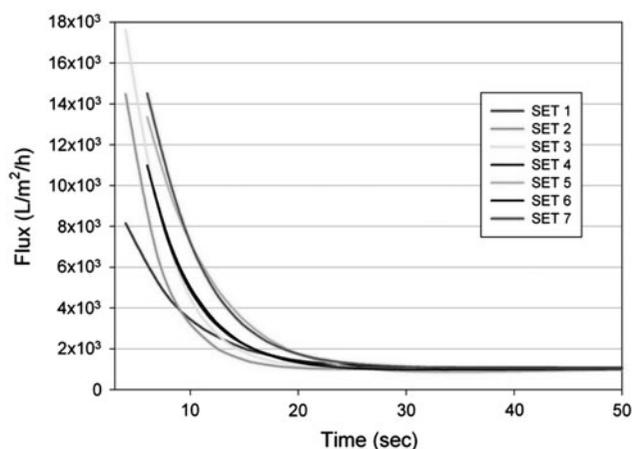


Fig. 4. Permeate flux with 5 µm PVDF filter.

Table 5
Clean water fluxes and flux declines of 5 and 10 μm PES membranes

Membrane (μm)	Clean water flux ($\text{L}/\text{m}^2/\text{h}$)			% Decrease	
	Test 1	Test 2	Test 3	After	After
				1st WW filtration	2nd WW filtration
5	24.8	14.5	6.2	42	57
10	22.8	19.7	13.5	14	31

filtration, these clean water fluxes decreased to 14.5 and 19.7 $\text{L}/\text{m}^2/\text{h}$ for 5 and 10 μm membranes, respectively. These results indicated that clogging or fouling of 5 μm membrane is more severe as compared to 10 μm membrane. The total decrease in clean water flux value of the 5 μm PES membrane after its two consecutive usage was about 75%, while that for 10 μm membrane was only 40% (Table 5). These values designate the serious clogging problems with this wastewater. Nevertheless, the clear finding from this series of filtration tests along with the previous MF trials is that MF pretreatment is not a successful option for the pretreatment of finishing wastewater.

Getting ineffective results from MF studies have induced investigation of other pretreatment options and in this framework flocculation was considered as the second pretreatment alternative. However, because of the presence of high variety of inorganic and organic constituents originating from sizing, spinning, weaving, desizing, dyeing, or scouring operations applied before mercerization of textile, no stability was reached. As can be seen from Table 1, mercerizing wastewaters are very high in suspended solids content and heavily loaded with organics and inorganics. This complex nature prevents flocculation to provide solids removal. After testing the applicability of flocculation as a pretreatment alternative and finding it unsuccessful, centrifugation was considered as the third alternative. However, even 30 min of centrifugation at a rotational speed of 2,500 rpm was not satisfactory and

did not provide any settlement. Looking at the consequences of applied pretreatment alternatives, it was decided to continue with the evaluation of caustic recovery directly with UF and NF treatment.

3.2. UF and NF experiments

Cross-flow UF and NF tests were carried out for the caustic recovery from denim mercerizing wastewaters. In UF and NF experiments, non-pretreated but coarsely (500 μm metallic strainer) filtered wastewater samples were used due to getting ineffective results from pretreatment studies.

UF and NF experiments were performed in total-recycle mode using a cross-flow plate and frame module with GR95PP UF and NP010, NP030, and MPF34 NF membranes. Table 6 presents the performances of UF and NF membranes tested at the same operational conditions at steady state. As can be depicted, these membranes were equal in performance in terms of NaOH recovery. The NaOH content of the concentrate was the same; 32 g/L from GR95PP (UF), NP030 (NF), and NP010 (NF) membranes. The other NF membrane tested (MPF34); however, provided a better caustic recovery with a permeate NaOH content of 41 g/L. With regard to COD and color retentions, as expected, all three NF membranes were superior to UF membrane and they provided 90.5–97.2% COD retention and 97.7–99.7% color retention. Among the NF membranes; MPF34 was better in performance; it provided 97.2% COD retention with a feed having 41 g/L NaOH.

Nevertheless, when evaluating membrane performances and selecting the most appropriate membrane, permeate flux is obviously of the primary concern. Time dependent permeate flux data for the tested NP010, NP030, and MPT 34 membranes and also GR95PP membrane is presented in Fig. 5. As stated before, during all these NF and UF tests, the operational conditions were kept constant.

After finding out that NP010 is the most appropriate membrane for caustic recovery, the effect of operational parameters on the performance of this membrane was

Table 6
Comparison of UF and NF membranes (TMP = 4.03 bars, CFV = 0.79 m/s, $T = 20 \pm 2^\circ\text{C}$)

Membrane	COD retention (%)	Color retention (%)	Flux ($\text{L}/\text{m}^2/\text{h}$)	Permeate NaOH content (g/L)	NaOH recovered in the permeate (%)
GR95PP (UF)	83.8	93.9	13.6	32	99
NP030 (NF)	90.5	98.5	6.0	32	99
NP010 (NF)	92.4	97.7	20.6	32	99
MPF34 (NF)	97.2	99.7	3.6	41	100

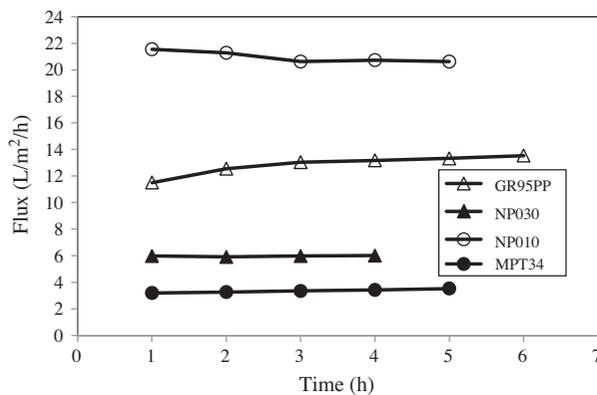


Fig. 5. Flux comparisons of GR95PP (UF), NP010 (NF), NP030 (NF), and MPT34 (NF) membranes at 4.03 bar TMP and 0.79 m/s CFV at $20 \pm 2^\circ\text{C}$.

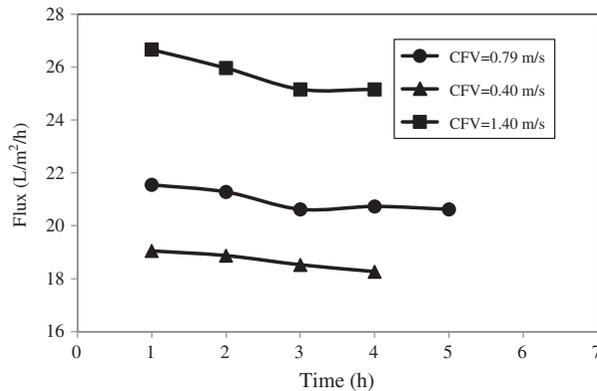


Fig. 6. Effect of CFV on NF permeate flux when TMP = 4.03 bar at $20 \pm 2^\circ\text{C}$.

evaluated. Because rejection and permeate flux are the two most important performance parameters [17], the effect of TMP and CFV on rejection and permeate flux were evaluated for this membrane. A lower and a higher CFV than the previously tested CFV of 0.79 m/s was maintained and permeate flux data presented in Fig. 6 were achieved. As shown, with an increase in CFV from 0.40 to 1.40 m/s, steady-state permeate flux value increased from 18.3 to 25.2 L/m²/h. In other words, with a threefold increase in CFV, there was about 50% increase in permeate flux. In the meantime, increasing CFV from 0.40 to 0.79 m/s, caused only a 9% increase in permeate flux; permeate flux increased from 18.3 to 20.6 L/m²/h.

It is known that higher velocities are the main reason for the distortion of filter cake that forms on membrane surface during filtration operation. Therefore permeate flux increases by an increase in CFV by this

reducing effect on the cake resistance [17]. In agreement with this fact, Koyuncu et al. [18] has reported that dye deposition and concentration polarization are one of the main reasons for flux declines at low CFVs. Based on the results obtained under different CFV conditions, it can be concluded that NF using NP010 membrane at a high CFV is useful to extend the membrane cleaning cycles and hence to provide a longer membrane service life.

TMP is another important operational parameter that determines membrane performance. In order to evaluate how TMP affects the flux behavior of NP010 membrane, the system was operated at the TMP's of 4.03 and 6.23 bar and the flux decline was followed. As can be depicted from Fig. 6, although the flux decline trend was quite similar, permeate flux at 4.03 bar TMP was greater than at 6.23 bar. In general higher permeate fluxes are expected at higher TMP; as increasing pressure difference affect filtration driving force through the membrane or increase it [19]. However the results are contrary to this general expectation (Fig. 7). As can be seen, steady-state permeate flux at 4.03 bar TMP is about 20.6 L/m²/h higher than that at 6.23 bar. Similarly, initial flux observed at 4.03 bar TMP (21.5 L/m²/h) is well above that observed at 6.23 bar (19.8 L/m²/h). This unexpected result was attributed to the particle deposition on the membrane surface. Satyanarayana et al. [20] reported that deposition mechanisms on membrane surface come into existence faster at higher pressures and the retained solutes on the membrane surface reduce flux by forming a second boundary layer as a result of concentration polarization.

Under real conditions of mercerizing processes in textile mills, rinsing operations after caustic treatment are applied at very high temperatures (80–90°C) to remove impurities coming from previous production

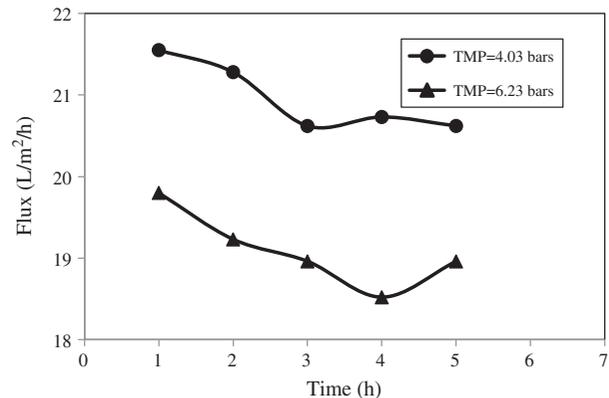


Fig. 7. Effect of TMP on NP010 membrane permeate flux when CFV = 0.79 m/s at $20 \pm 2^\circ\text{C}$.

stages and to provide a resistant silk shine and good handle features to the fabric. In the mercerizing process of the denim textile plant considered, mercerizing rinsing waters are generated at the temperature of 80–90°C and then cooled down to 40–45°C by passing through heat exchangers. Thus, in the present study, on account of this high temperature application, the performance of NP010 NF membrane was investigated at the temperature of 40°C for a better evaluation of caustic recovery under real operating conditions of the textile mill. The tests were carried out at two different CFVs (0.79 and 0.40 m/s) at the same TMP of 4.03 bars at 40°C. Comparison of the permeate flux behavior of NP010 membrane at the temperatures of 20 and 40°C is shown in Fig. 8. As shown, increasing the feed temperature from 20 to 40°C has improved the permeate flux for both CFVs. There occurred about 60 and 45% increase in permeate flux when the temperature was increased from 20 to 40°C at the CFV of 0.79 and 0.40 m/s, respectively. Due to decreasing effect of higher temperature on viscosity, increasing in permeate flux is an expected phenomena [21]. Schlesinger et al. [22]

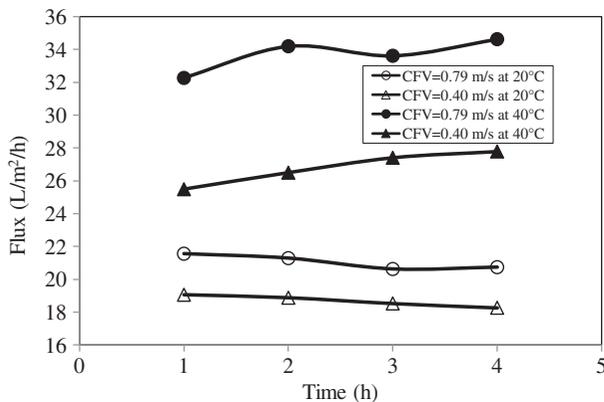


Fig. 8. Effect of temperature on permeate flux at TMP = 4.03 bar for CFV = 0.79 and 0.40 m/s.

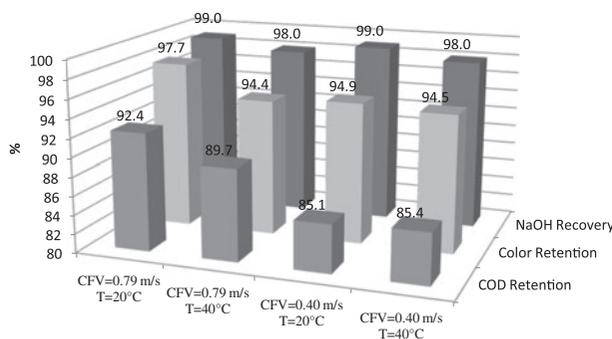


Fig. 9. Effect of temperature and CFV on color and COD retentions by NP010 NF (TMP = 4.03 bar).

reported a strong influence of temperature on NF permeate flux of various NF and UF membranes such that flux is doubled by increasing temperature from 20 to 40°C.

Temperature has also an effect on retention mechanism of membrane processes. Effect of temperature on COD and color retentions can be compared by Fig. 9. As can clearly be seen, the optimum performance is at the temperature of 20°C at a CFV of 0.79 m/s when the flux and retention parameters are considered together.

4. Conclusions

Based on the results obtained in this study; following conclusions were drawn:

- (1) Among the UF and NF membranes tested, NP010 NF membrane was best providing a highest permeate flux along with 97.7% color and 92.4% COD removals. The permeate flux was further improved with the application of higher temperature, higher CFV but not TMP.
- (2) NF retains almost all the dissolved and suspended substances in the caustic mercerizing wastewater from denim textile production and produces a highly purified caustic stream. This caustic stream contains 98–100% of NaOH in the feed at a concentration of 30–40 g/L and can therefore be recycled back to the mercerization process after an evaporation step as the mercerization of cotton yarn or fabric requires a solution of concentrated caustic soda at a concentration of 270–300 g/L NaOH.

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