

Recovery of Caustic Soda in Textile Mercerization by Combined Membrane Filtration

J. Yang^{*}, C. Park^{**}, D. Lee^{*}, S. Kim^{*}

^{*}Korea Institute of Industrial Technology, Chonan 330-825, South Korea, sykim@kitech.re.kr

^{**}Kwangwoon University, Seoul 139-701, South Korea, chpark@kw.ac.kr

ABSTRACT

This study is intended to find the optimum operational conditions for the recovery of caustic (NaOH) solution from mercerization in textile process. For this, the silt density index (SDI) of ceramic membranes, the fouling property of NF membranes, the optimum conditions for the membrane regeneration through chemical cleaning, the optimum removal conditions of total organic carbon (TOC), turbidity, color and the permeate flux through the membranes were investigated. As a result, a combined membrane process using ceramic membrane (first step) and polymeric membrane (second step) was found to be suitable for the removal of total suspended solid (TSS), residual organics, turbidity including color and the recovery of caustic solution from wastewater caustic stream in mercerization process. The permeated caustic solution can be reused, which could offer economical benefits through the reduction of chemical use and the cost of wastewater treatment.

Keywords: recovery, caustic soda, mercerization, ceramic membrane, combined membrane filtration

1 INTRODUCTION

General industrial textile processes consist of pretreatment, desizing, scouring, bleaching, mercerizing, dyeing, and finishing. They are not only heavy consumers of energy and water but also producers of large amounts of chemical pollutants. Their impact on the environment is mainly related to the exhaustion of non-renewable water resources and the production of wastewater, which requires an appropriate pretreatment before discharge into recipient water bodies. The caustic wastewater from textile mercerization is hot and alkaline, containing one to five percent sodium hydroxide, but it contains few fiber impurities since most of them are removed in the upstream processes [1]. Therefore, the membrane technology is suitable for the treatment of various textile effluent streams and the recovery of valuable chemicals from them. The polymeric nanofiltration/reverse osmosis (NF/RO) membrane can selectively permeate relatively small organic molecules and ions from the textile mercerization. Using

this type of membrane, the removal of fiber impurity and turbidity with color, the recovery of sodium hydroxide, and the recycle of process water from textile mercerization can be achieved. However, the NF/RO membrane filtration has problems of easy fouling, which often results in low flux and poor separation efficiency [2]. Therefore, a pretreatment step, such as the microfiltration/ultrafiltration (MF/UF) membrane process, is required.

2 MERCERIZATION

Mercerization is a process in which textiles (typically cotton) are treated with a caustic solution to improve properties such as fiber strength, shrinkage resistance, luster, and dye affinity. The caustic solution actually rearranges the cellulose molecules in the fiber to produce these changes. Higher-end fabrics may be double or triple mercerized for additional benefits. The fabric is first immersed in a caustic solution of about 18-25% strength and a relatively cool temperature of 16-32°C. A series of rollers (timing cans) are used to keep the fabric flat and smooth while controlling the time of caustic exposure. The fabric is then sprayed with rinse water and then washed with a neutralizing chemical before final drying (figure 1). In this study, a combined membrane process was applied, not only to improve the rejection efficiencies and flux recovery, but also to recycle the purified caustic solution back into the process.

3 EQUATIONS

The permeate flux of MF/UF can be expressed in terms of membrane resistances as in Eq. (1).

$$J \equiv \frac{1}{A} \frac{dV}{dt} = \frac{\Delta P}{\mu(R_m + R_c + R_p + \dots)} = \frac{\Delta P}{\mu(R_t)} \quad (1)$$

Where, μ is viscosity of the fluid, ΔP is transmembrane pressure, R_t is total resistance, R_m is intrinsic membrane pressure, R_c is cake layer resistance and R_p is pore plugging resistance.

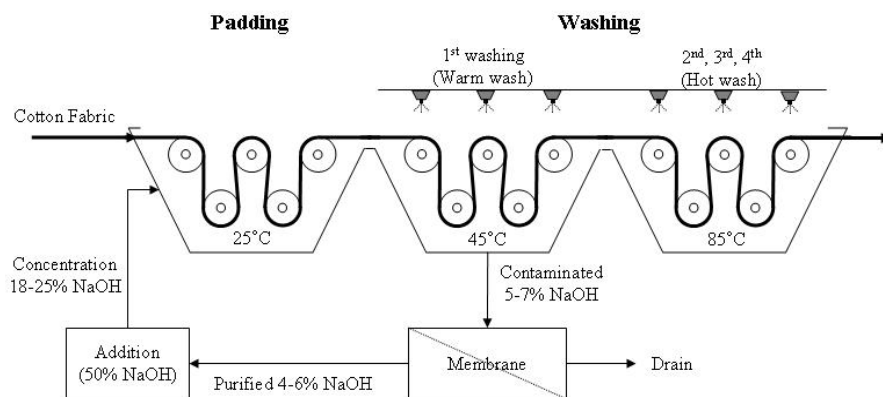


Figure 1: Caustic recovery process of mercerization effluent.

4 MATERIALS AND METHODS

4.1 Membrane

Three ceramic membranes (MF with $0.5\mu\text{m}$ pore size, MF with $0.1\mu\text{m}$ pore size and UF with $0.05\mu\text{m}$ pore size) manufactured by pall Co., USA, and three polymeric membranes (UF with MWCO of 1k Da, NF with MWCO of 200 Da and RO with MWCO of 50 Da) manufactured by Osmonics Inc. (USA) were used in this study. The characteristics of the membranes are summarized in Table 1.

Item	Ceramic membrane (Pall Co., USA)		Polymeric membrane (Osmonics Inc., USA)		
	MF	UF	UF	NF	RO
Active layer	Zirconia	Titania	Polyamide thin film (TF)		
Support layer	α -Alumina		Polysulphone		
Module type	Tubular		Plate		
pH range	1-14		2-12		
pressure	1-4 bar		1-40 bar		
temperature	1-100°C		1-50°C		
SDI	-		< 5	< 5	< 3
Filtration area	0.005 m ²		0.0136 m ²		

Table 1: Characteristics of the membranes used in this study

4.2 Combined membrane process

The dual-membrane process was composed of MF/UF and NF/RO for the recovery of caustic solution (Figure 2). A ceramic membrane unit of tubular type was used for the pretreatment process. It was designed for a product flow rate of 25.2 m³/h and TMP of 1 to 4 bar in the lab scale. A polymeric membrane unit of plate type was used for main process. It was designed for a product flow rate of 7.5 m³/h and TMP of 1 to 40 bar in the lab scale. The hybrid system was composed of a 6 L feed tank, where the caustic wastewater was circulated at a constant speed, circulating pump and crossflow flow membrane module.

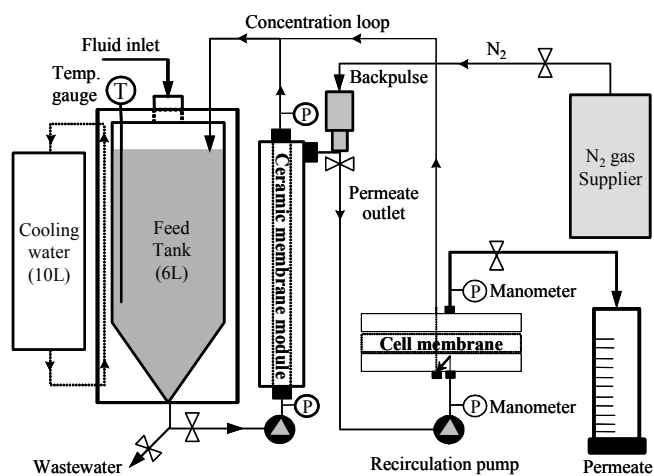


Figure 2: Schematic flow diagram of the experimental system.

4.3 Analysis

SDI, TSS, TOC, turbidity, conductivity, NaOH concentration and color were measured to analyze the sample. Representative samples were collected daily for the analysis. The permeate samples were taken from permeate valve located after membrane units (Figure 2). SDI value was measured following ASTM D4189-45. TSS value was measured using the Standard Methods 2540 D. The concentration of organic compounds was measured using a TOC analyzer (Multi N/C 3000, Analytikjena, Germany). Turbidity value was determined using a turbidity meter (Orion AQUAfast, Thermo Electron Co., USA). Conductivity value was measured using a conductivity meter (Multi 340i, WTW Inc., Germany). The concentration of NaOH was measured using a sodium electrode (Orion 4 star, Thermo Electron Co., USA). The decolorization rate was measured using a UV-Vis spectrophotometer (UNVIKON XS, BIO-TEC Ins., Italy).

5 RESULTS AND DISCUSSION

5.1 Membrane permeability

Permeate flux is an important factor to be considered during process design. In this study, two main parameters, membrane pore size and TMP, were examined for their effect on the permeate flux of pure water and caustic wastewater. The permeate flux values of pure water and caustic wastewater at different operation pressures were measured and plotted in Figure 3. When the same membrane was used, the average permeate flux of pure water was higher than that of caustic wastewater. This result is due to the decreased permeate flux by higher total resistance and viscosity of the caustic wastewater. As for the caustic wastewater, the permeate flux through the 0.5 μm pore size membrane was higher than that through the 0.1 and 0.05 μm pore size membranes. The difference of the permeate flux might be due to the different characteristics of the cakes formed on the membrane. When the cross-flow velocity is identical, the permeate flux is dependent on the size of deposited particles. The initial flux of 0.5 μm pore size membranes was higher than those of 0.1 and 0.05 μm pore size membranes, so the particle size of cake that formed on 0.5 μm pore size membranes surface might be larger and the cake resistance was smaller than those of 0.1 μm and 0.05 μm pore size membrane [3].

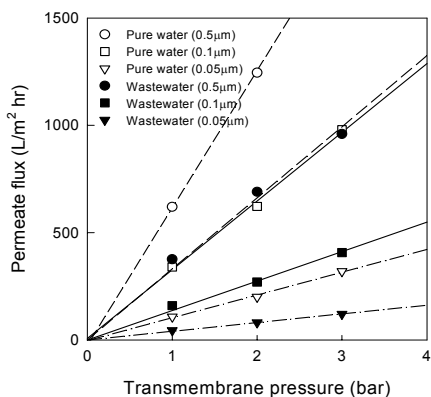


Figure 3: Dependence of the permeate fluxes of pure water and caustic wastewater on TMP (Temperature 30°C, velocity 4.4 m/s, initial volume 6 L).

5.2 Membrane selectivity

The NF/RO filter was required not only to remove large suspended solids but also to remove smaller suspended solids and colloidal substances. Membrane fouling index is an important parameter in the design of the integrated NF/RO membrane process. SDI values of 0.5 μm , 0.1 μm and 0.05 μm pore size membranes were measured to be 11.8, 5.8 and 4.3, respectively. The colors (absorbances) of the

permeates were 0.78, 0.63 and 0.43, respectively. In this study, UF ceramic membrane of 0.05 μm pore size was selected for NF/RO process was, since, in generally, the SDI values of pretreated permeate prior to NF/RO are no more than 5.

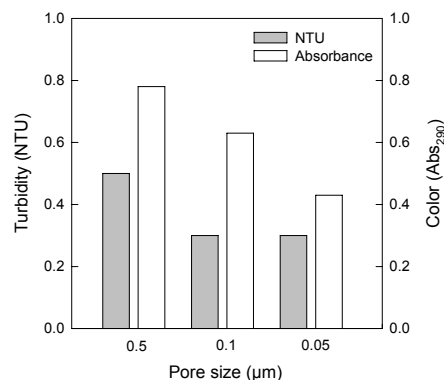


Figure 4: Pore size dependent properties (turbidity and color) of permeates obtained through ceramic membrane filtration (temperature 30°C, initial volume 6 L, TMP 2 bar).

5.3 Effect of temperature

Temperature is an important factor that affects the permeate fluxes of membranes. As shown in Figure 4, the steady flux increased when the temperature was higher than 10°C. Increase of the flux with temperature was consistent with the decrease in viscosity of the fluid in this temperature range. This means that the increase of flux with temperature was due to the decrease in viscosity of caustic wastewater [3]. Such relationship can be derived from Eq. (1). Permeability flux of the caustic wastewater through UF ceramic membrane increased by 2.4% when the temperature increased by 1°C.

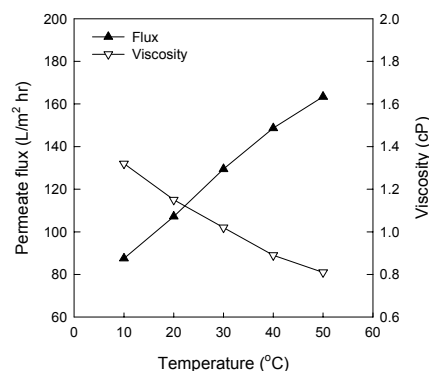


Figure 4: Effect of temperature on steady-state flux and permeate viscosity (velocity 4.4 m/s, initial volume 6 L, TMP 1 bar).

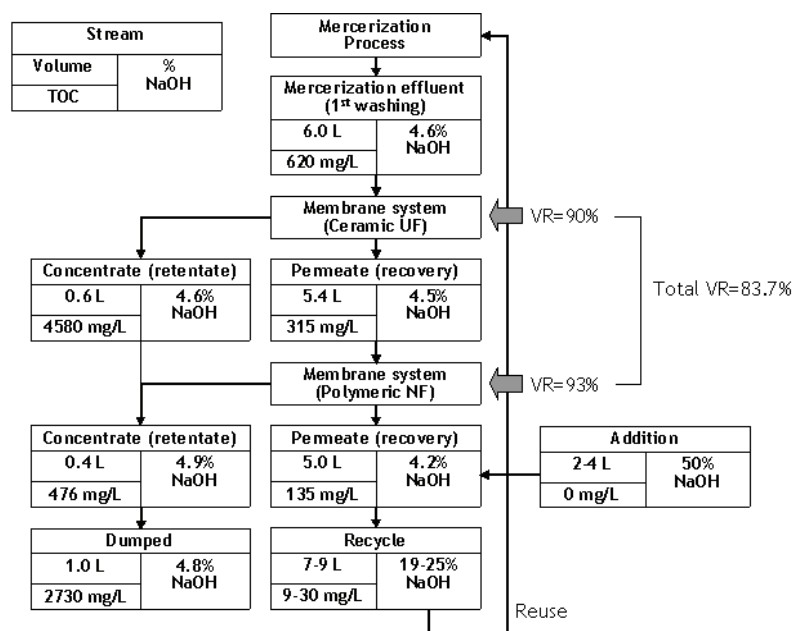


Figure 5: Mass balances for sodium hydroxide recovery and recycling by combined membrane system.

5.4. Combined membrane process

Table 2 shows the effects of combined process of ceramic UF and polymeric NF on rejection efficiencies of caustic wastewater from mercerization process. Only for the UF membrane, the rejection of TSS and turbidity was more than 99.0%, while the color and TOC rejection were about 74.7% and 49.2%, respectively. The combined membrane process of UF and NF membranes was found to treat the caustic wastewater effectively. This combined membrane process showed >99.9% TSS and turbidity, 87.7% color, 78.2% TOC removals.

Item	Caustic wastewater	UF Permeate water	NF permeate water
NaOH	4.6 w/w %	4.5 w/w %	4.2 w/w %
Conductivity	184 mS/cm	175 mS/cm	165 mS/cm
pH	13<	13<	13<
Absorbance ($\lambda_{max_{290}}$)	1.7	0.43	0.21
TSS	140 mg/L	0 mg/L	0 mg/L
TOC	620 mg/L	315 mg/L	135 mg/L
Turbidity	30 NTU	0.3 NTU	0.0 NTU

Table 2: Removal efficiencies of unit process (UF) and combined process (NF) by optimum conditions.

5.5 Mass balances

Figure 5 shows the caustic solution and total organic carbon mass balances for a single regeneration under the optimum conditions of the combined process. The

combined membrane process showed 91.3% sodium hydroxide recovery with 83.7% volumetric recovery.

6 CONCLUSIONS

In order to recover caustic solution from caustic wastewater, a combined membrane process was used. As a result, clean caustic solution was obtained with high purity, which can be recycled back into the process. It is expected that this system also can be applied to other processes like polyester caustification.

ACKNOWLEDGMENTS

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