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ORIGINAL PAPER

Combining activated sludge process with membrane separation to obtain recyclable quality water from paper mill effluent

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Abstract The present study was carried out for the treatment of paper mill effluent using combination of activated sludge process and membrane separation. An integrated paper mill employing $OCE_{OP}HH$ sequence (oxidation, chlorination, alkali extraction re-enforced by oxygen and peroxide, hypo-1, and hypo-2) for the bleaching of hardwood pulp was selected for the study. The purpose of this work was to examine the water quality and membrane performance when combining activated sludge process with different membrane separation processes in series. Pollutant removal including adsorbable organic halides (AOX) was compared among different treatment combinations; (i) ASP + microfiltration (MF), (ii) ASP +MF + ultrafiltration (UF), (iii) ASP +MF + UF + nanofiltration (NF), and (iv) ASP +MF + UF + NF + reverse osmosis (RO) to select the optimal treatment scheme for water recycling in the paper mill. Different initial inlet pressures were used for the UF and NF (6.8, 10.3, and 13.7 bar) and for RO (10.3, 13.7, and 17.2) The retentate from each membrane was recycled back to the feed and retreated until the inlet pressure increased to the maximum cut-off pressure for each membrane. After separation, 100 % total suspended solids, total dissolved solids, color removal and 94.2 % chemical oxygen demand, and 86 % AOX removal was observed. This study suggests the potential application of the combination of membrane separation with activated sludge process for recycling water in the paper industry.

Keywords Activated sludge process · Adsorbable organic halides · Membrane separation · Paper industry · Paper mill effluent

Introduction

The pulp and paper industry uses huge amount of water in the papermaking process. Fresh water intake to the mills has decreased significantly during the last few decades, and the trend is today toward more closed water circulation systems in the mills (Shukla et al. 2013c). However, paper mills cannot operate without sufficiently clean water. Also, the profile of the raw wastewater is changed, and existing systems for water treatment are usually no longer sufficiently efficient to fulfill the standards for effluent discharge (Hernández-Sancho and Sala-Garrido 2009; Parthasarathy and Krishnagopalan 1999). For a few years, in the production of packaging paper, some mills have been running with a totally closed water system including different processes for water treatment in the internal water cycles, the so-called 'kidneys' (Hamm and Schabel 2007; Bulow et al. 2003). Some mills, with partially closed water system use only physico-chemical effluent treatment (Nassar 2003; Abou-Elela et al. 2008; Shukla et al. 2013b). Other mills, which use secondary (biological) treatment, have reported some operational problems and proposed remedy measures (Nandy et al. 2002; Abbasi and Abbassi 2004; Azbar 2004).

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Biologically treated effluents are not sufficiently clean for reuse in the production of most paper grades. This kind of water can only be reused for production of packaging paper (Bulow et al. 2003). Biologically treated effluents still contain significant amounts of color compounds, micro-organisms, recalcitrant organics, and small amount of biodegradable organics, as well as suspended solids. Biological treatment does not significantly reduce the inorganic content in the effluent, and advanced treatment is required before reuse of the effluents in the manufacturing processes (Mänttäri et al. 2006). To what amounts these impurities need to be removed before reuse of the water is not well known, but the higher the quality (i.e., brightness) of the paper produced, the cleaner the water should be (Mobius and Helble 2003). Specific purification technologies such as flotation, evaporation, and membrane filtration are used to concentrate and fractionate spent liquor, remove color, and treat bleach effluent (Pizzichini et al. 2005; Sattler et al. 2004; Liu et al. 2004; Shukla et al. 2010, 2013a). Membrane filtration is an attractive alternative to purify process waters and effluents for reuse (Shukla et al. 2010, 2013a; Paul and Sikdar 1998). Generally, most studies on purification of biologically treated effluents have been carried out with microfiltration (MF) and ultrafiltration (UF) membranes. Some studies have been done with nanofiltration (NF) and reverse osmosis (RO) membranes (Choi et al. 2002; Blöcher et al. 2002; Mänttäri et al. 2006; Pizzichini et al. 2005; Negaresh et al. 2012). In the paper industry, recent investigations are oriented toward improvement of end-of-pipe measures (effluent treatment): an electrochemical technique (Khansorthong and Hunsom 2009), membrane processes (Zhang et al. 2009), and other alternative wastewater treatment plant options for the pulp and paper industry (Buyukkamaci and Koken 2010). However, according to information available in the literature, NF and RO membranes have not been used to purify the effluent from the activated sludge process in the pulp and paper industry.

The primary objective of this study was to examine the pollutants removal and membrane performance when combining activated sludge process with different membrane separations (MS) processes in series. A special focus on adsorbable organic halides (AOX) removal is given in this study as ASP is not very efficient to remove AOX (Bajpai 2012). Potential application of MS in removing AOX was also explored. In this study, we assessed several MS combinations with ASP for end pipe treatment of paper mill effluent to get high quality water, so that it could be reused in the paper manufacturing process. For this, we have evaluated several treatment combinations, i.e., ASP +MF; ASP +MF + UF; ASP +MF + UF + NF; and ASP +MF + UF + NF + RO. Furthermore, on the basis of findings, we tried to assess the applicability of ASP + MS as an alternative to recycle water from the already established paper mill wastewater treatment plants.

Materials and methods

An integrated paper mill producing writing, printing, packaging and absorbent grade paper, employing Kraft pulping and an $OCE_{OP}HH$ (oxidation, chlorination, alkali extraction re-enforced by oxygen and peroxide, hypo-1, and hypo-2) sequence for the bleaching of hardwood pulp, was selected for the present study. At present, specific water consumption and effluent generation in the mill are 28,890 m^3/day and 27,865.5 m^3/day, respectively. Activated sludge process treatment was carried out in the mill itself and effluent of treatment plant was brought to the laboratory to carry out membrane experiments. Experiment was conducted as per the scheme shown in Fig. 1.

Activated sludge process

Activated sludge process was used as biological treatment process. Details specification and operating parameters of the process are given in the Table 1.

Bag filtration and microfiltration

Effluent coming from ASP was passed through bag filter and micro filter (pore size 2 micron). Micro filtered water was collected in a tank and was fed to UF membrane. The specifications of the three membranes are given in Table 2.

Characterization of raw/treated effluent

Effluent samples were collected from outlet of mill and also from each and every inlet and outlet points of the treatment stages for chemical analysis. After biological treatment 200 L effluent was taken to conduct membrane treatment. The qualities of the raw and pretreated effluent were assessed using (APHA and AWWA 2005) methods. The parameters analyzed included pH, total dissolved solids (TDS), chemical oxygen demand (COD), closed reflux, titrimetric method), color (spectrophotometric method), and AOX. AOX were analyzed using an AOX analyzer model ECS 1200 employing the column method (Instrument based on coulometric determination). Further, feed wastewater, retentate, and the permeate samples from the UF, NF, and RO were collected in clean and dry canisters.

Membrane experiments

Membrane treatment experiments were performed in the batch concentrations mode, such that the retentate of each

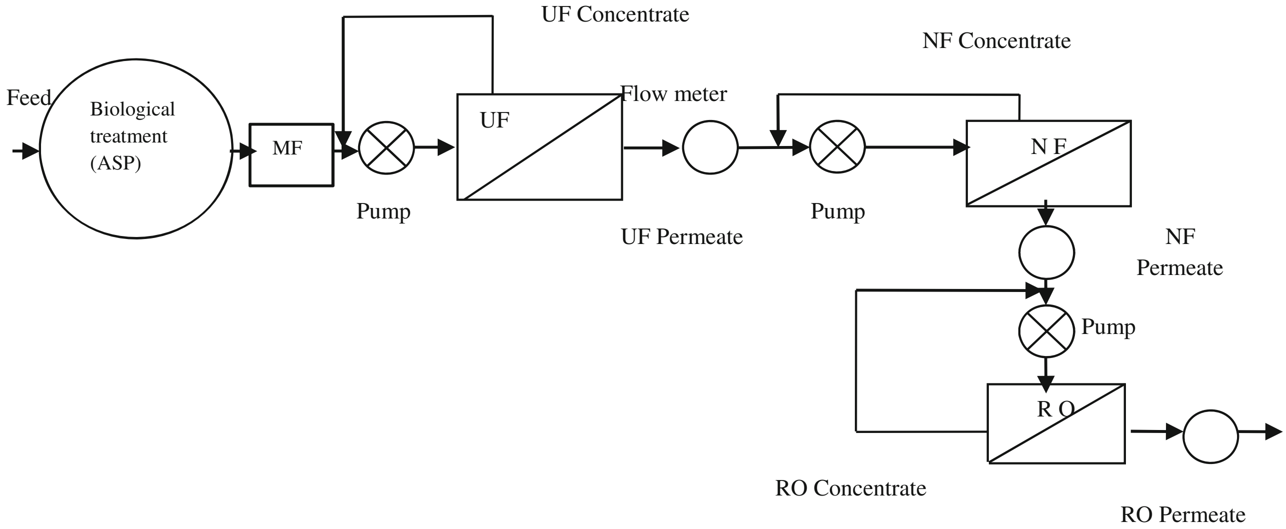

Fig. 1 Scheme of biological and membrane treatment plant

Table 1 Specifications of activated sludge process

Sections	Capacity (m^3)	HRT
Primary clarifier	10,000	12 h
Aeration chamber	18,000	24 h
Secondary clarifier	6,000	8 h

Flow rate; 18,000 m3/day; *MLSS* mixed liquor suspended solids 3,400–3,500 mg/l; *MLVSS* mixed liquor volatile suspended solids 2,700 mg/l; *SVI* sludge volume index 100–120 mg/l; *HRT* hydraulic residence time

experiment was recycled back into the feed and retreated until the inlet pressure increased to the maximum cut-off pressure for each membrane indicated by the manufacturer. This was done to look at the possibility of system closure. Three different initial inlet pressures were taken for each membrane, i.e., 6.8, 10.3, and 13.7 bar for the ultra- and NF, with 10.3, 13.7, and 17.3 bar used for the RO treatment. The removals of pollutants in terms of

TDS, COD, color, and AOX were assessed with the range of pressures for each membrane mentioned above. The performance of each membrane was assessed over time at each of the initial inlet pressures based on the variations in three parameters; namely, the trans-membrane pressure (TMP), permeate flux (PF), and fouling index (Shukla et al. 2010). The UF permeate was fed to the NF, and subsequently to the RO, as shown in Fig. 1, in the respective batch modes. Each membrane was stabilized with fresh water for 30 min) before treatment.

$$\text{Trans} - \text{membrane pressure is given by (TMP) in (bar)} = [(Pi + Po)/2] - Pp, \quad (1)$$

whereas Pi, Po, and Pp are inlet pressure, outlet pressure, and permeate pressure, respectively.

$$\text{Permeate Flux in}\left(\text{L m}^{-2}\text{h}^{-1}\right) = \text{Flow rate of permeate in the given time/Membrane area} \quad (2)$$

$$\text{Fouling index } J_t = J_0 e^{-bt}, \quad (3)$$

where b is the fouling index (min^{-1}), J_0 is the initial permeate flux ($\text{L m}^{-2}\text{ h}^{-1}$), and J_t is the permeate flux at time t ($\text{L m}^{-2}\text{ h}^{-1}$).

Table 2 Specifications of the membranes used in the study

Module	Trade code	Membrane material	MWCO	Area (m^2)	Initial inlet pressure	Manufacturer
UF, spiral bound	AP-01	Thin film polyamide/polysulphone blend	1,000 Da	2.51	6.8, 10.3, 13.7	Aastropure, India
NF, spiral bound	AP-02	Thin film polyamide/Polysulphone blend	300 Da	2.51	6.8, 10.3, 13.7	Aastropure, India
RO, spiral bound	AP-03	Thin film Polyamide	50 Da	2.51	10.3, 13.7, 17.2	Aastropure, India

Results and discussions

Pollutants removal

Pollutants removal during different sets of treatment is presented in Fig. 2. During ASP treatment, 8.45 % TDS, 60.33 % TSS, 49.9 % COD, 60.7 % Color, and 34 % AOX removal were observed. TDS removal efficiency in the ASP was very low as ASP is not considered effective for TDS removal (Sumathi and Hung 2006). Being a combined effluent it contains chlorination stage effluent too which might have led to TDS generation (Patoczka 2006). Raj et al. (2007) reported 52–78 % COD removal by some identified lignin-degrading bacterial strains in 6 days of incubation, which is similar to the observation in this work. Pizzichini et al. (2005) stated that biological treatment does not remove the organics which contribute to color or chlorine demand in the treated effluent, contrary to them, our study confirms that biological treatment do remove color-causing organics. AOX removal in this study is analogous to the reported study, Ataberk and Gokcay (1997) reported 21 % AOX removal efficiency during lab-scale experiment. Gergov et al. (1988) found about 48–65 % AOX removal in the activated sludge process. Bajpai (2012) reported, AOX removal efficiencies ranging from 14 to 65 % in the ASPs. In the case of ASP and MF, 19.18 % TDS, 85.2 % TSS, 50.80 % COD, 66.3 % Color, and 41.31 % AOX removal were observed. MF is basically used for TSS removal and a significant TSS removal was observed. While ASP + MF + UF treatment case pollutant removal increased significantly and TSS was 100 % removed, rest of the pollutants removal was 31.8 % for TDS, 73.9 % for COD, 88.50 % for color, and 65.76 % for AOX. Karthik et al. (2011) observed 93 % TSS and 91.7 % COD removal after UF treatment (operating conditions; maximum flow 4.5 m^3/h, inlet pressure 1.3–5.17 bar, membrane material polyethersulfone, MWCO 100,000 Da, pretreatment was applied), we achieved almost same removal with maximum 90 % COD removal and 100 % TSS removal, although they used chemical pretreatment before membrane and we did not use. ASP + MF + UF +NF treatment case pollutants removal was even more effective, TDS 67.65 %, COD 84.67, Color 100 %, and AOX 78.5 % removal were observed. Beril Gönder et al. (2011) found 92 % COD removal by NF in the biologically treated effluent (Best performing operating conditions; inlet pressure 12 bar, temperature 25 °C, MWCO 1,000 Da, material polyethersulfone), we also achieved maximum 91.5 % COD removal (with average 84.67 %) which is very closed to their study. At last, ASP + MF + UF +NF + RO treatment scheme was applied and almost all pollutants were 100 % removed except COD 94.2 %, AOX 86 %. RO results in this study were better than previous studies: Zhang et al. (2009) reports average 91.7 % COD removal by RO (Operating conditions; Feed pressure 0.4 MPa, Temperature 25–34 °C). If we compare our pollutant removal results with membrane bioreactor (MBR) technology, our results are better than MBR, Galil and Levinsky (2007) reports 86 % and Zhang et al. (2009) 92.1 % COD removal (operating conditions; feed pressure < 0.03 MPa, pore size 0.1 micron, material hydrophilic polyethersulfone) by MBR.

AOX removal is very difficult during biological treatment (Bajpai 2012) so membrane could provide an efficient option for AOX removal. With 1.22–2.11 mg/l AOX concentration in the permeate of RO, while AOX concentration after ASP is 7.6–9.7 mg/l. Water recovery through membrane separation was 89.13–91.11 % in UF, 90–91 % in NF, and 89–90 % in RO. Numeral values of all investigated parameters for each inlet and outlet streams are given in supplementary material (please refer to Table. A.1).

Membrane performance

Figure 3 shows the changes in the TMP across the ultrafiltration membrane and the PF during the treatment over time at the different initial inlet pressures. It can be seen from the figure that the TMP is almost stable at the low pressure of 6.8 bar, but increased slowly at higher pressures (10.3 and 13.7 bar). Also flux is behaving in a similar way- almost steady at low pressure and decreases at higher pressures. Although PF is decreased comparatively faster at a higher pressure than the lower, it was always observed higher than the PF at a lower pressure. Increase in the TMP and decrease in the PF at a higher pressure might have occurred due to the fact that the complete recycling of the retentate leads to the rapid increase in the concentrations of the pollutants in the feed.

Figure 4 shows the changes in the TMP across the NF membrane and the permeate flux during the treatment over time at the different initial inlet pressures. It can be seen from the figure that the TMP is almost stable at the low pressures of 6.8 and 10.3 bar, while it increase slowly at the maximum pressure of 13.7 bar. It can be seen in the figure that almost stable permeate fluxes were observed at initial inlet pressures of 6.8 and 10.3 bar, while at an initial inlet pressure of 13.7, a slow decrease in the PF was observed. Mänttäri et al. (1997) reported that during the nanofiltration treatment of paper mill effluent, the critical pressure was approximately 10 bar, as at this pressure, the permeate flux was stable, but above this pressure, the permeate flux decreased slowly. This may have been due to the fact that the pressure increased the concentration polarization layer, with the subsequent decrease in the permeate flux.

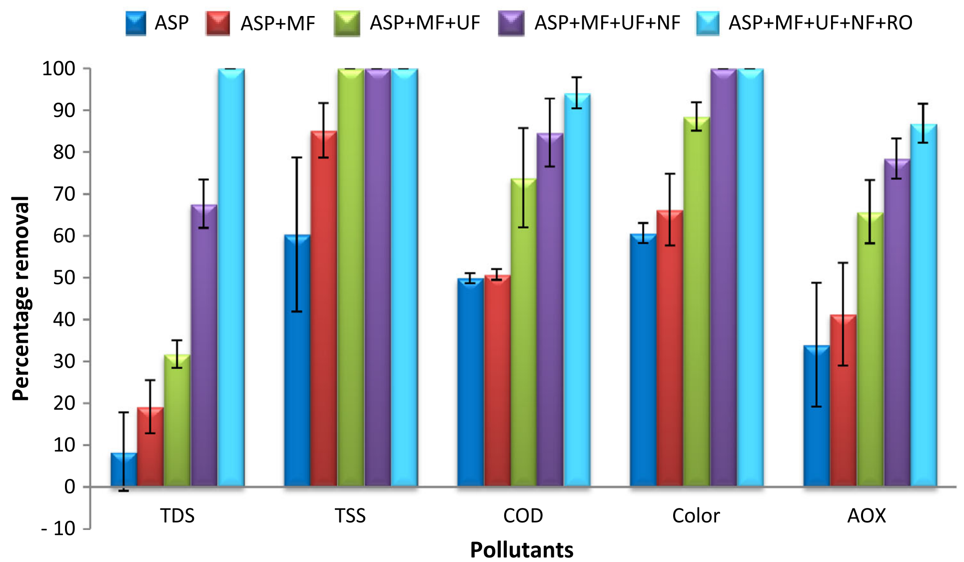

Fig. 2 Variation in pollutants removal in different stages

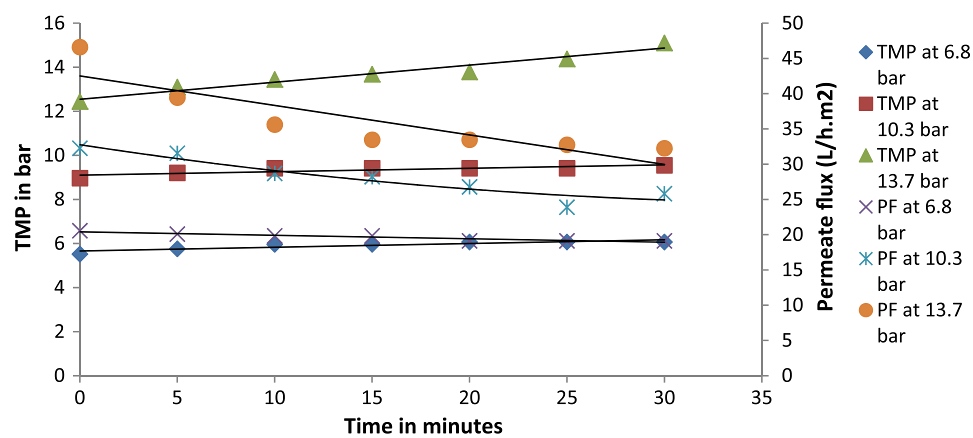

Fig. 3 Patterns of the TMP and permeate flux at the different initial inlet pressures during the ultrafiltration treatment

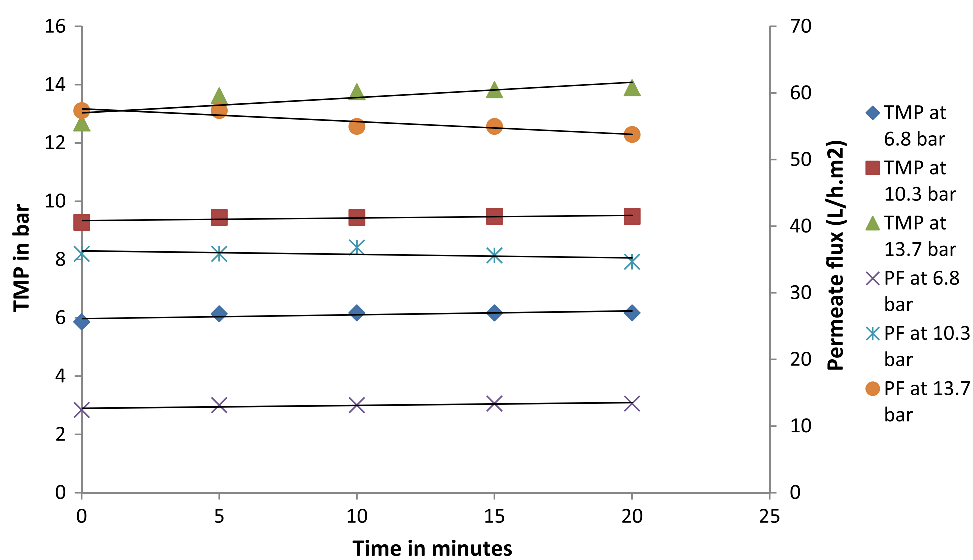

Fig. 4 Patterns of the TMP and permeate flux at the different initial inlet pressures during the nano-filtration treatment

Figure 5 shows the changes in the TMP across the RO membrane and the permeate flux during the treatment over time at the different initial inlet pressures. During reverse osmosis, comparatively rapid increase in the TMP was observed at the higher pressures of 13.7 and 17.2 bar, but a slow increase in the TMP was observed at the low pressure

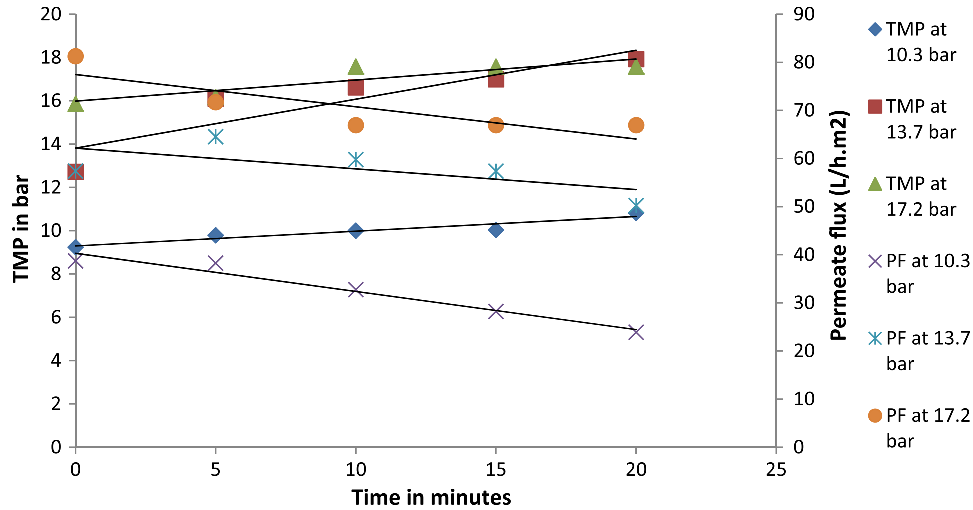

Fig. 5 Patterns of the TMP and permeate flux at the different initial inlet pressures during the reverse osmosis treatment

of 10.3 bar. It can be seen in the figure that the flux rapidly decreased at the higher pressures of 13.7 and 17.2 bar, but only a slow decrease was observed at the low pressure of 10.3 bar. As the RO membrane had a small MWCO of 50 Daltons, and the system was run in the concentration recycling mode with 100 % TDS removal, a rapid increase in the TMP and comparatively rapid decrease in the permeate flux resulted. Mänttäri et al. (1997) reported that the reductions in the flux of pure water were greater for the RO than the NF membranes.

Table 3 Fouling indices (min^{-1}) for each membrane at each initial inlet pressure

Module	Initial inlet pressure in bar			
	6.8	10.3	13.7	17.2
UF, AP-01	0.24×10^{-2}	0.74×10^{-2}	1.22×10^{-2}	–
NF, AP-02	0.0	0.16×10^{-2}	0.32×10^{-2}	–
RO, AP-03	–	2.4×10^{-2}	0.66×10^{-2}	1.29×10^{-2}

ASP activated sludge process, *MF* microfiltration, *UF* ultrafiltration, *NF* nanofiltration, *RO* reverse osmosis

Fouling indices

The fouling indices were calculated (using Eq. 3) for each membrane at each of the three inlet pressures, and shown in Table 3. The fouling indices were found to be higher at higher pressures for all three membranes. At a higher pressure more effluent will pass through the membrane in a given time, which will increase the concentration polarization on the membrane surface, with a higher fouling index observed. Among all membranes, fouling index was less in case of NF membrane. Tansel et al. (2000) stated that the flux decline during membrane filtration can be attributed to concentration polarization, the adsorption of contaminants within the membrane structure, pore blockage and the formation of a gel layer. The flux decline due to concentration polarization can be reversed by changing the process parameters and rinsing with water.

If we compare this study with our previous studies on the membrane separation of various paper mill effluents (Shukla et al. 2010, 2013a), we find that membrane separation of effluent after biological treatment is better than the direct application of membrane. We can also save the cost of coagulants because there is no need of coagulants while treating biological effluent through membrane. Also, these days membrane bioreactor is becoming popular for effluent treatment, however, in the current study, we found that combination of ASP and membrane filtration is superior than MBR (Galil and Levinsky 2007; Zhang et al. 2009) in terms of pollution reduction. In some cases, people are using RO and ozone treatments even after MBR (Zhang et al. 2009), especially in those cases ASP + MS will be very useful for recycling effluent in the process.

Conclusion

Membrane separation was found to be suitable for the treatment of biologically treated effluent to achieve high purity of recyclable water. During the study, best membrane performance was obtained at low pressure in case of UF, while NF and RO work better at higher pressures. Amongst the membranes tested, the NF membrane exhibited the most stable water flux and TMP; whereas, the RO membrane exhibited the least stable water flux and TMP. On comparing the performance of the three membranes at each of the three pressures, the fouling indices were found to be higher at higher pressure in all cases. The removals of pollutants by the membrane treatments were found to depend on the membrane pore size, concentration of pollutants in the feed, and the operating pressure. During the study, we found that membrane treatment of effluent after biological treatment is better alternative than the direct application of membrane. We also observed that membranes are performing better and we can save cost of coagulants. Biological treatment is not enough for AOX removal; however, combination with MS could be a viable option.

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