

Biological treatment and nanofiltration of denim textile wastewater for reuse

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Abstract

This study aims at coupling of activated sludge treatment with nanofiltration to improve denim textile wastewater quality to reuse criteria. In the activated sludge reactor, the COD removal efficiency was quite high as it was $91 \pm 2\%$ and $84 \pm 4\%$ on the basis of total and soluble feed COD, respectively. The color removal efficiency was $75 \pm 10\%$, and around 50–70% of removed color was adsorbed on biomass or precipitated within the reactor. The high conductivity of the wastewater, as high as 8 mS/cm, did not adversely affect system performance. Although biological treatment is quite efficient, the wastewater does not meet the reuse criteria. Hence, further treatment to improve treated water quality was investigated using nanofiltration. Dead-end microfiltration (MF) with 5 μm pore size was applied to remove coarse particles before nanofiltration. The color rejection of nanofiltration was almost complete and permeate color was always lower than 10 Pt–Co. Similarly, quite high rejections were observed for COD (80–100%). Permeate conductivity was between 1.98 and 2.67 mS/cm (65% conductivity rejection). Wastewater fluxes were between 31 and 37 L/m²/h at 5.07 bars corresponding to around 45% flux declines compared to clean water fluxes. In conclusion, for denim textile wastewaters nanofiltration after biological treatment can be applied to meet reuse criteria.

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Keywords: Textile wastewater; Water reuse; Activated sludge; Nanofiltration

1. Introduction

Textile industries are characterized by high water and chemical consumption due to dyeing, finishing, sizing processes and multiple washing and rinsing cycles, which caused significant amounts of colored wastewater with a high COD and inorganic loads [1–5].

Conventional treatment methods for textile wastewater are mainly physico-chemical [4,6,7] or biological [4,8,9]. Compared to chemical treatment, biological treatment methods are more dependable in meeting the required removal of organic matters [2]. In this context, Orhon et al. [2] reported that denim-processing wastewater contains high COD with a biodegradable fraction of 90%. Also, they noted that the COD of wastewater can be reduced from around 2000 mg/L to around 150–300 mg/L with the proper designed activated sludge process. Similarly,

Pala and Tokat [10] reported that around 95% COD removal from a textile industry wastewater can be achieved in an activated sludge reactor. Although high COD removal efficiency is possible with conventional wastewater processes, the color removal is not so effective and the main removal mechanism is indicated to be adsorption [8–10].

In Turkey, COD and BOD removal is the major objective in wastewater treatment for the textile industry as color removal is not yet a concern for discharge to sanitary sewer [11] or any receiving environment. Textile companies may face a shortage of available water sources due to water scarcity and limitations of ground water use. In the near future, many textile companies will have to improve wastewater quality to the fresh (ground) water standards for reuse purpose [12].

Hence, advanced treatment is necessary to reuse wastewaters from textile mills. For that purpose, coupling of biological treatment with advanced treatment methods, such as membrane filtration [1], ozonation [13,14] and adsorption [15], is necessary to upgrade treated wastewater quality to reuse criteria. Within advanced treatment alternatives, membrane separation seems to be one of the most promising methods, as although adsorption

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and ozonation can be effective in the removal of residual color and COD from activated sludge effluents; conductivity removal, hence the reuse, is only possible with the membrane separation [16].

In some studies on membrane treatment of textile mill effluents for reuse, the wastewater was directly filtered after a pretreatment to remove only coarse particles to reduce membrane fouling. However, it was reported that after nanofiltration (NF), a concentrated mixture of dyes and auxiliary substances remains, and the problem of its utilization has not yet been solved [1]. Additionally, filters selected for this kind of application should withstand high concentrations of organics and inorganics if any specific pretreatment before membrane is not applied [17].

Mänttari et al. [17], in their study with pulp and paper mill effluents, reported that a well operated activated sludge can be a good pretreatment alternative before NF operation as the high COD removal can be observed in activated sludge process and the resulting concentrate after NF operation will contain less amount of organic pollutant compared to NF operation without pretreatment. They also stated that another advantage of activated sludge process is that it can equalize the fluctuation in incoming wastewater, which makes operation of downstream membrane operation easier.

In this context, the present study aims at evaluating the feasibility of activated sludge process as a pretreatment for NF of real denim wastewater for reclamation.

2. Materials and methods

The study consists of two successive steps. In the first step, effluent of a denim textile mill was treated in a bench scale activated sludge reactor (Fig. 1). The second stage was the NF of biologically treated wastewater to improve the treated wastewater quality for reuse. Before NF, dead-end MF was conducted with a nitrocellulose acetate membrane (pore size of 5 μm) to prevent NF from fouling.

2.1. Plant description

The plant, which was located in the Middle Anatolia region of Turkey, produces 20,000 tonnes of cotton fiber; 45 million

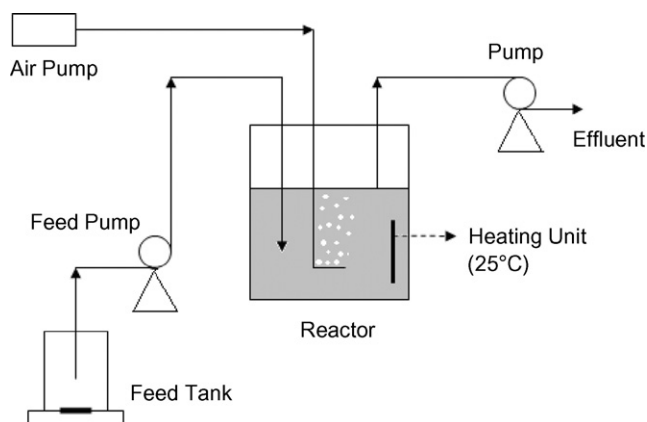


Fig. 1. Activated sludge reactor used in experiments.

meter of woven fabric and 12 million meter of denim per year with a daily water consumption of about 3500–5000 tonnes/day. This fact makes water reuse inevitable in the plant [18,19].

2.2. Bench scale activated sludge reactor

A bench scale activated sludge reactor (Fig. 1) was used to remove organic compounds from wastewater. The volume of the reactor was 10 L. The reactor was continuously fed and effluent was continuously withdrawn using two peristaltic pumps. The hydraulic retention time (HRT) (which is equal to sludge retention time (SRT) for a stirred tank reactor) was kept constant at 8 days throughout the operation.

The reactor was inoculated with a sludge mixture (1 L) from a membrane bioreactor receiving municipal wastewater and an activated sludge reactor receiving denim-processing wastewater. The purpose of having sewage sludge in the inoculum was to allow diverse microbial community development in the reactor. During acclimation period, the feed wastewater was diluted four times (WW1) and two times (WW2) between days 0–26 and 26–56, respectively. Then the reactor was directly fed with a real undiluted wastewater (WW3) between days 56 and 100.

The feed and the effluent of the reactor were preserved in a refrigerator at 4 °C. In the reactor, the temperature and oxygen concentration were maintained at 25 ± 0.5 °C and 5 ± 1 mg/L, respectively. The reactor was regularly sampled to monitor COD, color, pH, conductivity, mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS).

The Yield coefficient (Y) in the completely stirred tank activated sludge reactor (Fig. 1) was calculated using the formula given below;

$$Y = \frac{X}{(S_0 - S)} \quad (1)$$

where X = biomass concentration within the activated sludge reactor as mg/L MLVSS; S_0 and S are feed total COD and effluent soluble COD (mg/L), respectively.

2.3. Wastewater characteristics

Throughout the study, four different wastewater samples from the mill effluent were characterized. The composition was found to be highly variable with the average composition given in Table 1. Although COD of the wastewater was quite high, nitrogen and phosphorus concentrations were not adequate for biological treatment. Hence, NH_4Cl and KH_2PO_4 were added to obtain COD/N/P ratio as 100/5/2. Also, the pH of wastewater was adjusted to 6.8–7.3 with concentrated H_2SO_4 .

2.4. Membrane filtration

Biologically treated effluent from the activated sludge reactor was collected as three different samples. The collected effluent from activated sludge reactor was allowed to settle around 1 h to remove suspended biomass. Then, the settled effluent was filtered with dead-end MF using filters of 5 μm pore size before preserving the effluent in a refrigerator at 4 °C. The pre-filtered

Table 1
Wastewater characteristics

Parameters	Concentration
Total COD (mg/L)	2300 ± 800
Soluble COD (mg/L)	1350 ± 500
Color (Pt–Co)	2347 ± 1200
TSS (mg/L)	150–300
TDS (mg/L)	4000–8000
Total nitrogen (mgN/L)	20 ± 3
NH ₄ -N (mgN/L)	3.5 ± 0.5
Total P (mg/L)	0–3
pH	9–13
Conductivity (ms/cm)	9 ± 3

samples were subjected to NF in a laboratory-scale cross-flow module (DSS LabStak M20) equipped with a circular flat-sheet NF membrane giving a total membrane surface area of 0.036 m² (Fig. 2). The NF 270, made from piperazine and benzenetricarbonyl trichloride, is a negatively charged, hydrophilic membrane and has a smooth surface with a MWCO of 200–300 Da [20,21]. NF 270 membrane (Dow Filmtec, USA) was used at a trans-membrane pressure of 5.07 bars and temperature of 18 ± 2 °C.

The trans-membrane pressure for NF ranges between 5 and 20 bar [22]. We have chosen the minimum pressure (5.07 bar) applicable to NF to make process cost-effective. Fresh membrane was used for each experiment.

The volume of the feed solution was 6–6.5 L, including 1.5 L in the tubes of the installation. The wastewater was fed to the membrane at a flow rate of 6 L/min, which create cross-flow velocity of 0.62 m/s. Experiments were conducted in total recycle mode (the concentrate and permeate were completely recycled to the feed tank) for 6.5 h to evaluate performance of membrane at steady-state conditions.

Permeate and feed were regularly sampled to monitor COD, color, pH and conductivity. All the experiments were conducted until steady-state operation was reached in terms of flux and permeate quality. The membrane pure water fluxes were measured before and after wastewater experiments to evaluate the filtration performance of the membrane.

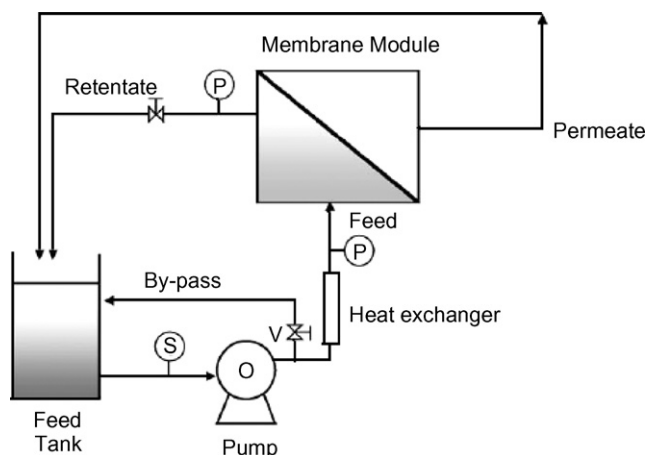


Fig. 2. Membrane system used in experiments (adapted from Capar et al. [28]).

2.5. Analytical techniques

COD measurements were carried out using Hach COD vials according to the EPA approved reactor digestion method [23]. In this method, after 2 h digestion, COD values of samples were directly read using Hach Spectrophotometer (Model No 45600-02, Cole Parmer Instrument Co., USA). Soluble COD were measured after filtration of samples through 0.45 µm filter paper. Activated sludge reactor effluent was always filtered through 0.45 µm filter paper to remove suspended biomass before COD measurement. Color measurements as Pt–Co were performed with the same instrument at 455 nm. Conductivity and pH measurements were conducted using Hach Sension 378 pH, Conductivity, Dissolved Oxygen meter. In the measurement of total suspended solids (TSS), total dissolved solids (TDS), nitrogen and phosphorus, standard methods [24] were followed.

Physical removal of colour by adsorption or precipitation was determined by alkaline extraction of dye from biomass samples. For this purpose, around 50 mL of biomass samples were centrifuged at 5000 rpm for 10 min. The liquid portion was removed and dye adsorbed on biomass was extracted with 0.5 M NaOH. After extraction of dye, the biomass was removed again by centrifugation. The pH of liquid was adjusted to around 7.5 with 0.5 N H₂SO₄ and colour was measured in liquid portion as Pt–Co.

3. Results and discussion

3.1. Biological treatment of denim textile effluents

The performance of activated sludge reactor is depicted in Fig. 3. The wastewater was diluted four and two times on day 0–26 and 26–56, respectively, to acclimate the microorganisms. Between day 56 and 100, wastewater was fed to the reactor without dilution. The COD loading rates at steady-state operation were 0.23 and 0.75 mg COD/mg MLVSS.d on day 24–56 and 56–100, respectively. The effluent soluble COD concentrations averaged 118 ± 67 mg/L corresponding to 91 ± 2% and 84 ± 4% removal on the basis of total and soluble feed COD, respectively. The color removal efficiency was 75 ± 10% and extraction results revealed that around 50–70% removed color was adsorbed on biomass or precipitated within the reactor. The observed yield coefficient (*Y*), although it showed great variation, averaged at 0.34 ± 0.15 mg MLVSS/mg COD. These results showed that activated sludge treatment is quite efficient in removing the organic compounds from denim wastewater as the effluent soluble COD (filtered on 0.45 µm filter) was always lower than 270 mg/L, despite feed COD was as high as 2500 mg/L. It is important to note that although feed color concentration showed great variation between day 56 and 100, the effluent color concentration did not change so much. Hence, as noted by Mänttari et al. [17], one of the most important advantages of activated sludge process as a pretreatment before membrane filtration is that it can stabilize the fluctuations in incoming wastewater and the feed to the membrane unit will be more stable than when process waters are filtered directly. Also, both color and COD concentration can be appreciably decreased, which lessens the fouling of membrane.

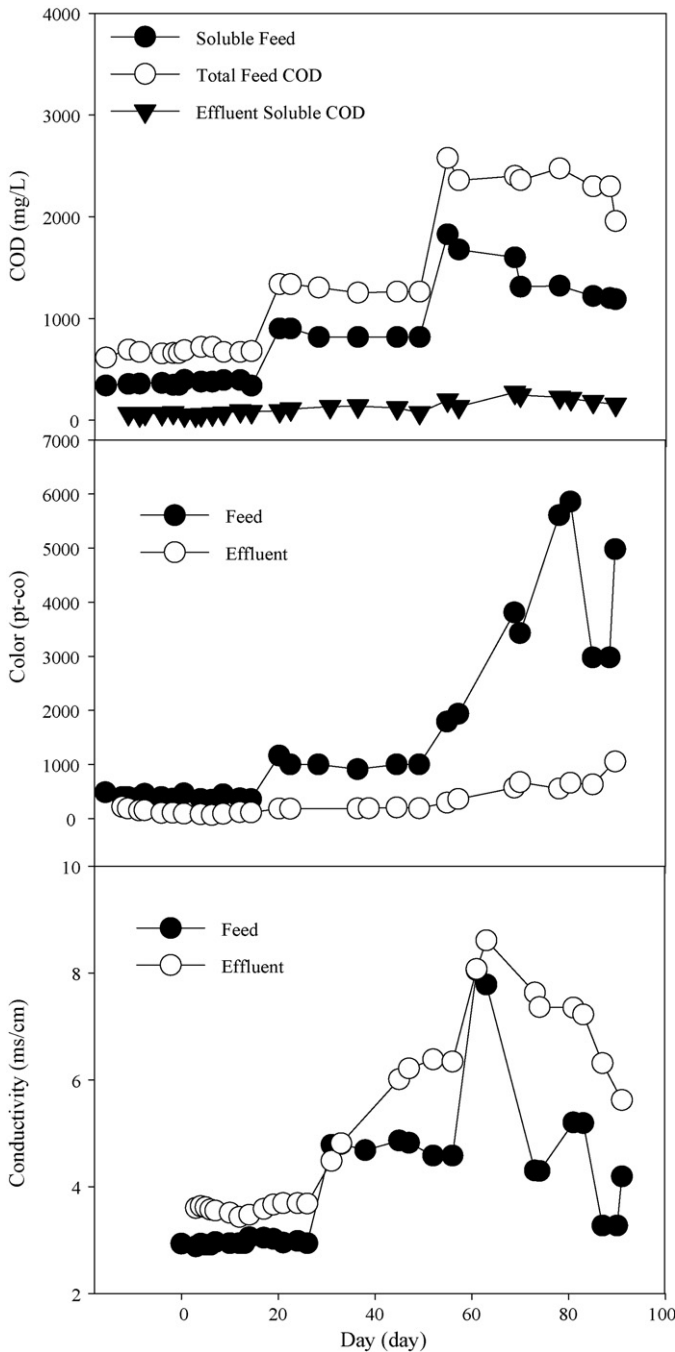


Fig. 3. Performance of lab-scale activated sludge reactor.

Similar to our results, Orhon et al. [2] reported that around 90% of COD in two different denim-processing wastewaters are biologically degradable and with the proper designed activated sludge reactor, quite high removal efficiencies can be attained. They also reported that soluble slowly degradable COD is predominant in denim-processing wastewaters and the soluble COD concentration at the effluent of activated sludge reactor can be reduced to quite low levels with high HRTs. In our study, we have directly used activated sludge reactor in which HRT and SRT were 8 days. Hence, one of the reasons of obtaining high COD and color removal efficiencies may be high HRT

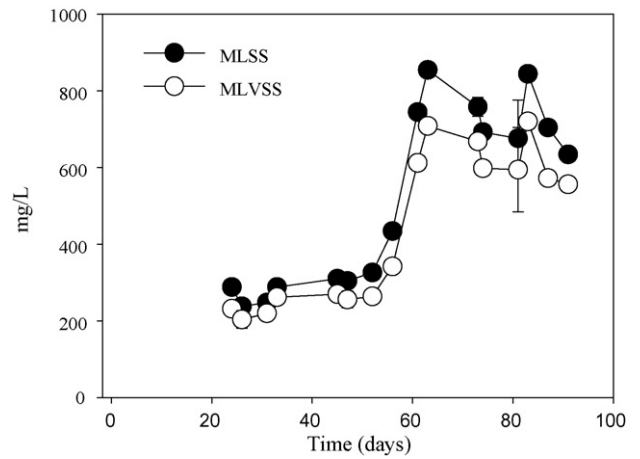


Fig. 4. Time course variations of MLSS and MLVSS.

in our experimental system. Although we have attained 75% color removal, Pala and Tokat [25] reported around 36% color removal efficiency in an activated sludge reactor. They also reported quite high Y of 0.76 mg MLSS/mg COD compared to that observed in this study (0.34 ± 0.15 mg MLVSS/mg COD). The different results may be due to different characteristics of incoming wastewater and differences in the experimental systems. Although they observed lower color removal efficiency, similar to our study, they reported quite high COD removal efficiency (94%). In order to improve color removal performance, they added activated carbon to the aeration tank of activated sludge reactor, which improved color removal performance to around 86% at 400 mg/L activated carbon dosage.

The conductivity of incoming wastewater was also quite variable and reached to around as high as 8 mS/cm on day 60. The conductivity of the wastewater was quite high compared to literature values [26,27] and it is quite significant to note that COD removal efficiency was not adversely affected from high conductivity values. The obtained low Y value in our study compared to the value of Pala and Tokat [25] may be due to quite high conductivity values of the wastewater used in our study. After biological treatment the effluent conductivity slightly increased (Fig. 3), probably due to increased inorganic content of wastewater during the biological activity.

The MLSS and MLVSS concentrations during the operation of the reactor were given in Fig. 4. As the feed COD increased, both MLSS and MLVSS increased, as expected. The ratio of MLVSS/MLSS throughout the study averaged 0.85 ± 0.04 .

Although biological treatment is quite efficient, the wastewater does not meet the reuse criteria given by British Textile Technology Group, which is one of the most commonly used reuse criteria for textile wastewater (Table 2). Hence, further treatment to improve treated water quality is necessary. Although several methods (e.g. ozonation, chemical precipitation, and adsorption) can be quite effective in the removal of color and COD and they do not produce reject stream, they are not effective to decrease conductivity of treated wastewater. Thus, the reuse of treated wastewater is not possible [16]. In this context, the use of membrane processes is necessary to meet the reuse criteria.

Table 2
British Textile Technology Group reuse criteria for textile wastewaters [30]

Parameter	Reuse criteria
COD (mg/L)	80
TSS (mg/L)	5
TDS (mg/L)	500
Hardness (mg/L as CaCO ₃)	60
Conductivity (μS/cm)	1000
Alkalinity (mg/L as CaCO ₃)	–
Color (Pt–Co)	20
pH	6–8
Turbidity (NTU)	1

3.2. Nanofiltration of biologically treated denim wastewater

Three different biologically treated effluents were collected at different feed dilutions (WW1, WW2 and WW3) to study the effect of increased COD and color on membrane removal efficiency and flux. Before NF, wastewater was pre-filtered with 5 μm MF to remove bacteria and other suspended solids, which may cause the fouling of the nanofiltration membrane. As Capar et al. [28] noted although MF, ultrafiltration (UF), chemical coagulation, sand filtration, and ozonation are the most commonly adopted pretreatment processes for textile effluents, MF has been gaining a wider acceptance as it is more economically feasible than the conventional methods.

In order to determine the COD and color removal performance of MF, the treated effluent was allowed to settle for around 1 h to simulate a settling tank in an activated sludge system. Then, the color and COD measurements before and after MF were compared. MF caused 0–15 and 4–19% color and COD removals, respectively after biological treatment. Wastewater characteristic after MF is summarized in Table 3.

After NF the color rejection was almost complete and permeate color was always lower than 10 Pt–Co (Fig. 5). Similar to color, quite high rejection was observed for COD. For WW2 and 3, almost complete COD rejections were achieved and average COD values were lower than 5 mg/L. For WW1, however, the COD rejection remained relatively low at 80% and the average permeate COD was 43 mg/L. Hence, slight increase in the feed COD and color did not adversely affect membrane performance. Although slightly higher COD concentration was obtained after NF for WW1, the value is quite lower than reuse criteria, 80 mg/L (Table 2). The reason of observing slightly high permeate COD for WW1, is not clear, but it can be attributed to the differ-

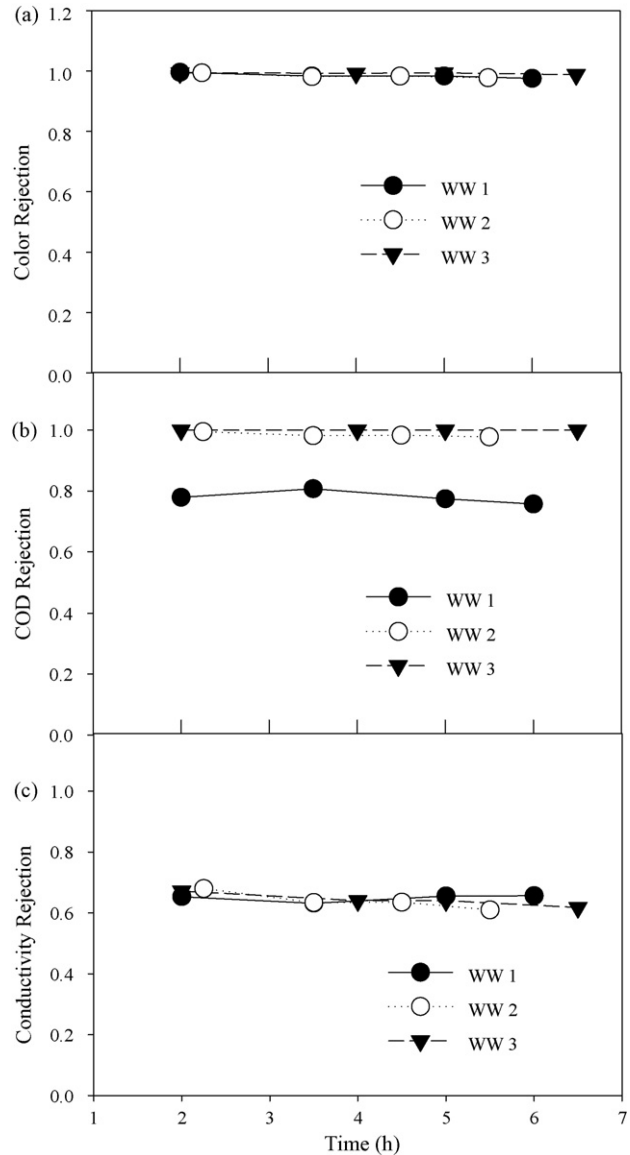


Fig. 5. Color (a), COD (b) and conductivity rejection performances of NF process.

ent composition and molecular mass distribution of biologically treated effluent. Mänttari et al. [17] postulated, in their study with pulp and paper wastewaters, that the activated sludge process effectively degrades small molar mass organic compounds; thus, relatively high molar mass components remain in the effluent of activated sludge process and these high molar mass organics can be effectively retained in NF.

Table 3
Feed and permeate characteristics of NF process

	WW	COD (mg/L)	Color (Pt–Co)	Cond. (mS/cm)	pH	Flux, L/m ² /h (flux decline)
Feed	1	191	266	6	7	–
	2	259	606	6.9	7.38	–
	3	291	592	7.4	6.24	–
Permeate	1	43	5	1.98	5.74	33.33 (43%)
	2	<5	≤10	2.52	3.25	31.25 (46%)
	3	<5	6	2.67	3.24	37 (44%)

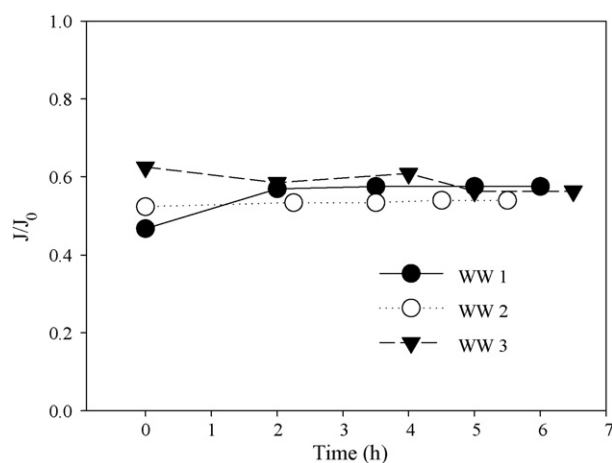


Fig. 6. Normalized water fluxes with time during NF process.

In our study, the conductivity rejection was around 65% for all wastewaters. The permeate conductivities of WW 1, WW 2 and WW 3 were 1.98, 2.52 and 2.67 mS/cm, respectively. Although quite high conductivity rejections (65%) were attained, the permeate conductivity values were equal or slightly higher than the reuse criteria, 1 mS/cm (Table 2). Hence, second NF or reverse osmosis membrane may be required to decrease conductivity further. Although the obtained conductivity values are higher than the reuse criteria given in Table 2, depending on the type of dyeing or process, the conductivity of the water to be reused can be in the wide range of 0.065–2.2 mS/cm [29].

Another important observation is that the pH of permeate was considerably lower than that of the feed especially for WW2 and WW3 (Table 3). A possible rejection of some negatively charged basic organics and inorganics could be a reason for this decrease considering a quite low feed alkalinity (<50 mg/L CaCO₃). Although the permeate pH is quite low (Table 3), the acidity (≤30 mg/L CaCO₃) is also quite low, hence, the permeate pH can be easily increased to neutral values, prior to reuse.

The normalized water fluxes for all studied wastewaters are depicted in Fig. 6. Fluxes changed between 31 and 37 L/m²/h (Table 3) corresponding to around 45% flux declines compared to clean water fluxes before the experiment. The specific fluxes were between 6.3 and 7.4 L/m²/h/bar. After completing filtration experiments, clean water fluxes were measured to determine the degree and type of fouling. The clean water flux values after wastewater filtration were between 88 and 100% of the clean water flux values before wastewater filtration. Hence, it can be concluded that concentration polarization was the predominant reason of flux decline and no irreversible fouling was observed.

4. Conclusion

High conductivity of the wastewater (as high as 8 mS/cm) did not adversely affect the performance of activated sludge process as COD removals were 91 ± 2% and 84 ± 4% on the basis of total and soluble feed COD, respectively. The color removal efficiency in activated sludge process was 75 ± 10% and around 50–70% of removed color was adsorbed on biomass

or precipitated within the reactor. Although biological treatment is quite efficient, the wastewater does not meet the reuse criteria. The color rejection of NF after biological treatment was almost complete and permeate color was always lower than 10 Pt–Co. Similar to color, quite high COD rejection (80–100%) was observed with NF. In NF, the conductivity rejection was around 65% and permeate conductivity was between 1.98 and 2.67 mS/cm. Wastewater fluxes at 5.07 bars were between 31.25 and 37 L/m²/h corresponding to around 45% flux decline compared to clean water fluxes.

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