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Evaluation of effects of textile wastewater on the quality of cotton fabric dye

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Abstract. In this study, reuse of biologically treated wastewater of denim washing and dyeing industry has been evaluated by membrane technologies. After that experiments were carried out at laboratory scale in textile dyeing unit by using obtained permeate water samples on 100% cotton based raw fabric belonging to examined industry. During membrane experiments, two different UF (UC100 and UC030) and two different NF (NP010 and NP030) were evaluated under alternative membrane pressures. In permeate water obtained on selected samples, conductivity at the range of 1860-2205 µS/cm, hardness at the range of 60 to 80 mg/L, total color at the range of 2.4 to 7.6 m-1 and COD at the range of 25-32 mg/L was determined. The following analyzes were performed for the dyed fabrics: perspiration fastness, rub fastness, wash fastness, color fastness to water, color fastness to artificial light, color measurement through the fabric. According to analysis results, selected permeate water have no negative impact on dyeing quality. The study showed that membrane filtration gave good performance for biologically treated textile wastewater, and NF treatment with UF pre-treatment was suitable option for reuse of the effluents.

Keywords: cabric dyeing; nanofiltration; reuse; textile industry; ultrafiltration

1. Introduction

Textile industry requires plenty of water and fresh water consumption. Physico-chemical and biological treatment methods are used to reduce or eliminate the pollutant concentrations for the textile industry wastewater. However, this type of treatment does not allow the re-use of water at any stage of the process. Because treated water is still a significant amount of pollutants after the biological treatment. These are suspended solids, COD, BOD, high pH and very strong color (Lopes, Petrus, *et al.* 2005, Marcucc, Ciardelli i *et al.* 2002, Fersi, Gzara *et al.* 2005, Gozálvez-Zafrilla, Sanz-Escribano *et al.* 2008, Ong, Li *et al.* 2014). Day by day, because of declining groundwater levels and stringent environmental legislation during the adaptation process to the

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European Union, reuse of wastewater has become a topical issue besides the treatment of wastewater (Florio, Giordano *et al.* 2005, Dasgupta, Sikder *et al.* 2015, Vajnhandl and Valh 2014).

Membrane technologies are the kind of methods that are proven in water and wastewater recycling, compelling and requiring more research and development (Shen, Xiao *et al.* 2012). With this system, process water need can be met and the wastewater discharge can be reduced (Lopes, Petrus *et al.* 2005, Babursah, Çakmakci *et al.* 2006, Akbari, Remigy *et al.* 2002). According to the studies made by some researchers, ultrafiltration (UF) process can be used for water recycling and for removing high molecular weight and insoluble dies (indigo, dispers), auxiliary chemicals (polyvinyl alcohol), on the other hand it cannot be used for removing low molecular weight and isoluble dies (asidic, direct, reactive and basic etc.) (Debik, Kaykioglu *et al.* 2010). The permeate water obtained by UF cannot be reused. However it meets the pre-treatment need of nanofiltration (NF) and reverse osmosis (RO) (Akbari, Remigy *et al.* 2002, Debik, Kaykioglu *et al.* 2010, Tang and Chen 2002, Barredo-Damas, Alcaina-Miranda *et al.* 2006). Most of RO membranes show efficiency above 90% for ionic species and it provides a permeate obtained with high quality. The textile wastewater containing dyes and auxiliary chemicals can be removed by reverse osmosis. However high osmotic pressure difference limits the RO applications (Kocaer and Alkan 2002). On the other hand high stratification problem occurs in the use of RO and this leads to low flux and removal efficiency (Tang and Chen 2002, Ong, Li *et al.* 2014). NF membranes separates low molecular weight organic compounds (200-1000 g/mol) and divalent salts (effective in softening) successfully (Debik, Kaykioglu *et al.* 2010). NF membranes have a wide range of acceptability on dyed wastewater treatment. In usage of NF, type of dye and operating conditions are effective for the removal of dye from wastewater and more than 98% color removal can be achieved (Ku, Lee *et al.* 2005, Petrinic, Andersen *et al.* 2007, Qin, Oo *et al.* 2007). Lopes, Petrus *et al.* (2005) have performed spiral-wound membrane types DK 1073 for textile industry wastewater and they achieved about 99% color removal and 87% COD removal.

Flux reduction, in membrane processes, may be due to the concentration polarization and accumulating on the surface of the material and decrease in the solvent activity. Prevention of decrease, in the permeate stream, plays a key role in the selection of pre-treatment system (Babursah, Çakmakci *et al.* 2006, Akbari, Remigy *et al.* 2002). Fouling of the membrane and damage of modules can be prevented by pre-treatment (Marcucci, Ciardelli *et al.* 2002, Tang and Chen 2002). Significant pre-treatment methods are; the biological degradation, coagulation-flocculation, microfiltration (MF) and UF (Uzal, Yılmaz *et al.* 2006).

Membrane systems, when combined with many conventional methods such as activated sludge and chemical oxidation, can give good results (Bruggen, Kim *et al.* 2004). Choo, Choia *et al.* (2007) have implemented coagulation and UF system, and have achieved 97% turbidity removal for reuse of textile wastewater. Membrane bioreactors can also be used for the treatment and reuse of textile wastewater (Badani, Ait-Amar *et al.* 2005, Schoeberl, Brik *et al.* 2005). Baburşah *et al.* (2006) have suggested to use sand filter, UF and RO at the discharge of the biological treatment plant for the reuse of textile wastewater. It was observed that this system provides the removal efficiency of over 90% for the studied parameters (COD, TSS, conductivity, turbidity, hardness, sulfides, sulfates, color). Marcucci, Ciardelli *et al.* (2002) have used NF and MF membranes for the reuse of textile wastewater from biologically activated sludge plant. As a pretreatment for NF, sand filters and MF have been used. Use of the sand filter and MF have played an important role in the removal of suspended solids and turbidity, on the other hand its effectiveness was 30% in the removal of COD. After NF process application, COD has been removed completely (100%) and color has been removed at the rate of 81%. It was observed that permeate of NF, containing very small amounts of dye, have the qualities to be re-used in all production steps of the textile industry.

Fersi, Gzara *et al.* (2005) stated that use of MF process, for the pre-treatment of treated textile wastewater by the biologically active sludge method, would be an appropriate approach. In comparison with the direct UF and MF+UF processes applications, they emphasized that use of MF+UF process increases the quality of permeate highly. Debik, Kaykioglu *et al.* (2010) have evaluated the reuse of raw textile wastewater and aerobic and anaerobic pre-treated textile wastewater after being processed by UF and NF membranes. It is expressed that the appropriate permeate water quality has been obtained by UF+NF process application after aerobic treatment under different operating pressures. As a result of comparison with the process water quality parameters specified in the literature, the quality of permeate water has been expressed to be suitable for reuse in the process.

As a result of literature research, UF and NF membrane processes are applied to the biological wastewater treatment plant discharge of the textile industries. However the reuse of obtained permeate water at the process is not evaluated in many studies. For that reason, studies are needed to be made on that subject. In previous studies, application possibilities in the process of membrane permeate water obtained from the process was evaluated compared with the literature. In this study, reuse of biologically treated denim washing and textile dye industry wastewater by means of membrane technologies (UF, NF and UF+NF (NF application after UF pre-treatment)), has been evaluated with testing of obtained permeate water at the laboratory scale dyeing unit.

2. Material and method

2.1 Wastewater origin

In the study, aerobical treatment plant effluent, which is currently available in textile factory engaged in denim dye and washing, has been utilized. The average monthly water consumption is 40800 tons in the factory. Industrial and domestic wastewater are treated by physical, biological (aerobic) and sand filtration. The capacity of wastewater treatment plant is 1500 m^3/day.

Pumice stone, pumice flour and coarse-grained fiber components are passed through the fine mesh and are subject to a preliminary sedimentation process. Then floating fiber fragments that cannot be kept in the pumice stone pool and in the pre-sedimentation basin are put through static screens with a range of 0.5 - 0.25 mm and subsequently it is transferred to the equalization tank. After a mixture is obtained the equalization tank by jet aerator, it is transferred to the neutralization tank and is raised to pH 9.5 with lime dosing and is subsequently taken to aeration tank. Suspended solids spilling from sluice of the aeration tank, are processed to a sand filtration accuracy of 20 μ in the multiple filter medium. Suspended solids, kept in the bed by back flushing process, are recycled to the equalization tank through drainage. After being collected in the clean water tank, the treated water from the filtration system is discharged into the Corlu Stream. Discharged water meets Water Pollution Control discharge limit values specified in the Regulations. Composite samples gathered for 24 hours were used as feed water in the membrane process.

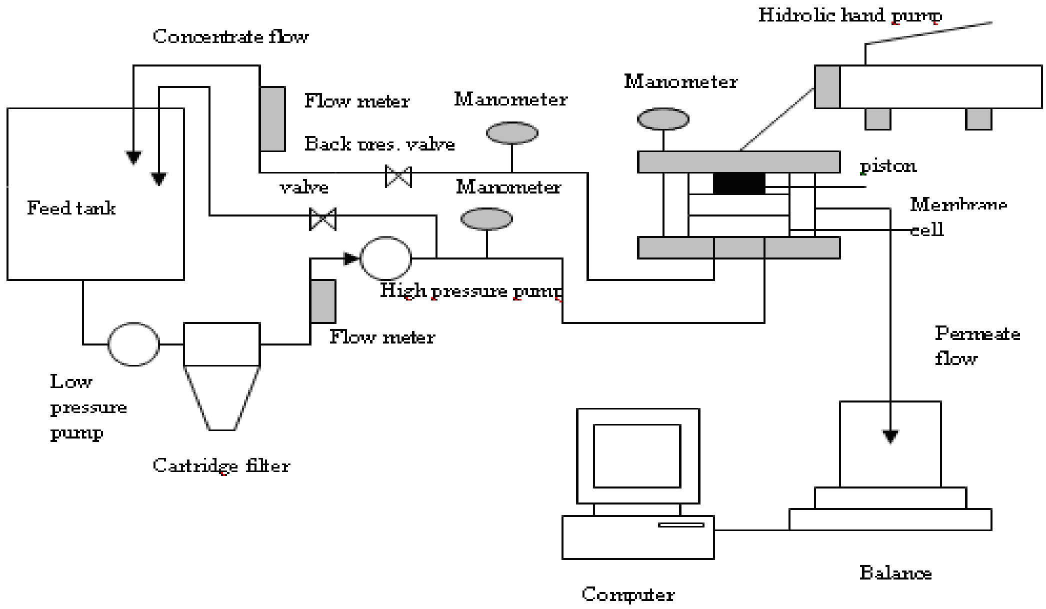

Fig. 1 Schematic diagram of membrane process (Debik, Kaykioglu *et al.* 2010)

Table 1 Characteristics of the nanofiltration and ultrafiltration membranes used in experiments

Memb. type	Manufacturer	Material	Membrane property	MWCO[a], kDa	M.O.B.[b] bar	M.O.T.[c], C°
NP010	Macrodyn® Nadir	Poliethersulfone	Hidrophilic	NA[d]	40	95
NP030	Macrodyn® Nadir	Resistant Poliethersulfone	Hidrophilic	NA[d]	40	95
UC100	Macrodyn® Nadir	Cellulose	NA[d]	100	3	55
UC030	Macrodyn® Nadir	Cellulose	NA[d]	30	3	55

[a]Molecular weight cut-off, [b]Maximum operation pressure, [c]Maximum operation temperature, [d]Not available.

2.2 Equipment

2.2.1 Membrane process

The membrane system installed in Environmental Engineering laboratory at Yildiz Technical University was supplied by Osmonics® Inc. and the membrane cell was supplied by a GE Sepa™ CF2. As illustrated in Fig. 1, the concentrate stream flowed back to the feed vessel, during collecting the permeate stream separately. Before the membrane cell, a cartridge filter (10 μm pore size) was used as a pre-filter to remove coarse particulates from the wastewater. All membrane experiments were performed at 25°C, with a heat exchanger located in the feed vessel. UF and NF membranes were used to filtrate aerobically pre-treated textile wastewaters under pressures of 3, 4, 6, 8 and 10 bar. A flat sheet type membranes were used in the experiments utilized NF and UF membranes supplied by Macrodyn® Nadir. A new membrane sheet was used for each experiment. The characteristics of the membranes are given in Table 1. Membranes were pressurized by distilled water in order to get to the stable flux of the membranes used in experiments. Flowmeter is set at 300 L/h.

Table 2 Prepared water samples and codes

Code	Water samples
PW	Process water
N30	NP030 permeate water
N10	NP010 permeate water
UN30	UC100 + NP030 permeate water
UN10	UC100 + NP010 permeate water

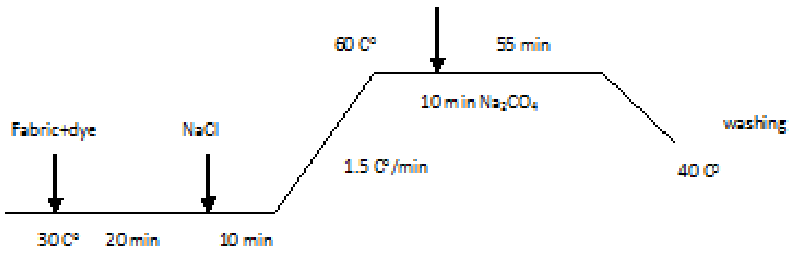

Fig. 2 Dyeing process carried out at laboratory conditions

2.2.2 Textile dyeing unit

In the study, textile dyeing experiments were carried out at laboratory scale unit to determine the reusability of the treated water obtained from the application of the membrane process, in the textile industry. The receipt conditions of the plant were formed and prewashing and dyeing were applied to raw fabric. Dyeing was realized in the Thermal Company HT laboratory type dyeing device according to the padding method (in the long run and high bath ratio (more than 1: 2)). The fabric used in the dyeing process was the raw material utilized in the production of the examined plant. This raw fabric is 100% cotton, pre-treated (desizing, bleaching, mercerization), has the number of warp and weft wire, 21 ends / cm 1/1 plain weave, and made by 30/1 carded yarn. In order to simulate exactly the plant conditions, chemicals and their amount are taken into account specified in the recipe being used in cotton fabric finishing of the examined plant. Water, used in dyeing process, was prepared by mixing of permeate water (P) and process water (PW, softened with ion exchangers) in 5 different ratios (100% PW, 75% PW+25% P, 50% PW+50% P and 25% PW+75% P). Prepared samples and the amount of chemicals are named as shown in Table 2.

Firstly 100% CO (cotton) plain woven fabrics which will be dyed were kept for 24 hours under standard laboratory conditions (65% relative humidity, 20±2^{o}C). The fabrics were applied pre-washing using Rudolf-duraner Gmbh 2 g/L of lube anticreasing rucola, Eurody to-CTC s.a. Company of 2 g/L Cross serial wetting and 0.25 g/L Merck CH_3COOH (98%) and were rendered hydrophilic. In order to simulate the dyeing process in the investigated industry, pre-washed fabric samples were colored according to 1% color intensity using chemicals of Everlight (Everzole Yellow 3RF H/C (ColourIndex Reactive Yellow 145), Everzole Red 3BS (ColourIndex Reactive Red 239), Everzole Blue BRF (ColourIndex Reactive Blue 221)) containing vinylsulfone and bifunctional group with NaCI (40 g/L), Na_2CO_4 (22 g/L) chemicals supplied by Merck. 0.5 g/L Merck CH_3COOH (98%) and, Eurody A-CTC sa. Company of 2 g/L Cross super dispersants were used for the rinsing operation. In the dyeing process, sample fabrics were processed at 60^{o}C for 65 minutes by adding auxiliary chemical substances and dyestuff at above-mentioned 1% color intensity into the 30°C 100 mL dye bath at ratio of 1:20. Excess fabric dyes were removed through the washing process for fastness as a result of dyeing process (Fig. 2).

2.2 Analytical methods and experimental studies

2.3.1 Membrane processes

Permeate waters, obtained as a result of membrane experiments, were collected to be analyzed. Conductivity, color, hardness, COD, Mn^{+2}, Zn^{+2}, Cu^{+2}, Fe^{+2} and HCO_3 were measured for aerobically treated effluent and permeate waters. All parameters are determined according to Standard Methods except for COD (ISO 6060) and color (ISO 7887). pH and conductivity parameters were determined using WTW pH 315i/set and WTW Cond. 3210 Set 1 brand appliance, respectively. Color and metal parameters were analyzed by using Thermospectronic Aquamate brand spectrophotometer in the permeate and the feed samples. Membrane performance was expressed as flux and removal efficiency. Permeate flux was determined gravimetrically. The time-dependent flow and pressure values measured in the experiment were calculated as follows;
Flux (Jv, $L/m^2.h$)=permeate weight in terms of g and in 5 minutes x density of the water / membrane area.

Removal efficiency was determined by the following equation using the relationship between the obtained permeate stream concentration and the feed stream concentration.

$$Ro(\%) = \frac{C_f - C_p}{C_f} = 1 - \frac{C_p}{C_f} \quad (1)$$

where; R_o, the observed removal efficiency, C_p and C_f represents the concentration of the feed solution and the permeate stream, respectively (Koyuncu 2004, Kural 2000, Ellouze, Tahri *et al.* 2012).

2.3.2 Textile dyeing process

Performed tests applied to dyed fabrics were carried out in R&D Laboratories of the Department of Textile Engineering of Namik Kemal University and of Denge Kimya Firm. A Color i7 benchtop spectrophotometer (X-Rite, America) were used to measure the color yield values of dyed fabrics. Before color yield measurements, dyed fabrics were conditioned by using a Macbeth Color Eye 7000A spectrophotometer (Macbeth Division of Kollmorgen, Newburgh, NY) under D65 illuminant. Color yield was defined as a K/S value within the visible spectrum range from 400 nm to 700 nm with a 20-nm interval. The K/S values were calculated using the below equation:

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (2)$$

where; K is the absorption coefficient depending on the dye concentration, S is the scattering coefficient caused by dyed fabric, and R is the reflectance of dyed fabric.

L-histidine monohydrochloride monohydrate (Merck), sodium chloride (Merck), and disodium hydrogen orthophosphate (Merck) were used and adjusted to pH 8 with 0.1 mol/L sodium hydroxide (Merck) for testing colorfastness to basic perspiration, L-histidine monohydrochloride monohydrate (Merck), and sodium dihydrogen phosphate dihydrate (Merck) were used and adjusted to pH 5.5 with 0.1 mol/L sodium hydroxide (Merck) for testing colorfastness to acidic perspiration. According to the following standards of ISO 105 E01, ISO 105 E04 (performed on a

Prowhite perspiration fastness device in the acidic and basic forms), ISO 105X12 (performed on a Prowhite manual crockmeter device in the wet and dry forms), and ISO 150 C06 (performed on a Gyrowash, Gyrowash 815, James, H Heal & Co Ltd, Halifax, England), respectively, colorfastness to water, perspiration, crocking and washing of dyed fabric samples were tested. All the colorfastness tests were evaluated on a gray scale of 1 (worst) to 5 (best) for fading (color alteration) over fabrics and staining over adjacent fabrics or multifiber test fabrics.

3. Results and discussion

3.1 Membrane experiments

UF and NF membranes were applied sequestered (UF or NF) and combined (UF+NF) to biologically treated textile effluents. The performance of membranes was determined by the permeate flux and permeate water quality under different operating pressures.

3.1.1 Permeate flux

Permeate flux is an important parameter in terms of design and economic feasibility area in membrane processes. Permeate flux reduces due to the deposition of organic and inorganic molecules on the membrane surface. It is thought that this case occurred due to the formation of osmotic pressure between feeding solution and filtrate near the membrane surface (Debik, Kaykioglu *et al.* 2010). Flux results obtained in different membrane pressure for 60 minutes, in the study, were shown in Table 3.

The effect of osmotic pressure that occurs between the feed solution and the filtrate close to the membrane surface is reduced by increasing the pressure (Debik, Kaykioglu *et al.* 2010, Ellouze, Tahri *et al.* 2012). It can be seen in the Table 4 that permeate flux is reduced by increasing the pressure.

UC030 is a membrane with a smaller pore diameter compared to UC100. For this reason the amount of flux, (117.8 L/m^2.h) obtained from UC030, was less than the amount of flux (137.6 L/m^2.h) obtained from UC100. The highest flux values, obtained with NP030 and NP010 membranes were determined as 51.8 L/m^2.h and 83.7 L/m^2.h at 10 bar pressure, the lowest flux

Table 3 Membrane flux results obtained in this experiment

Membrane	Pressure (bar)				
	3	4	6	8	10
	Flux (L/m^2.h)				
UC030	117.8				
UC100	137.6				
NP030		22.5	31.7	44.3	51.8
NP010		50.3	62.3	78.1	83.7
UC030+NP010		49.2	61.9	78.8	83.9
UC030+NP030		27.8	35.3	46.7	54.5
UC100+NP010		56.2	66.6	79.2	84.8
UC100+NP030		25.5	34.0	45.5	51.7

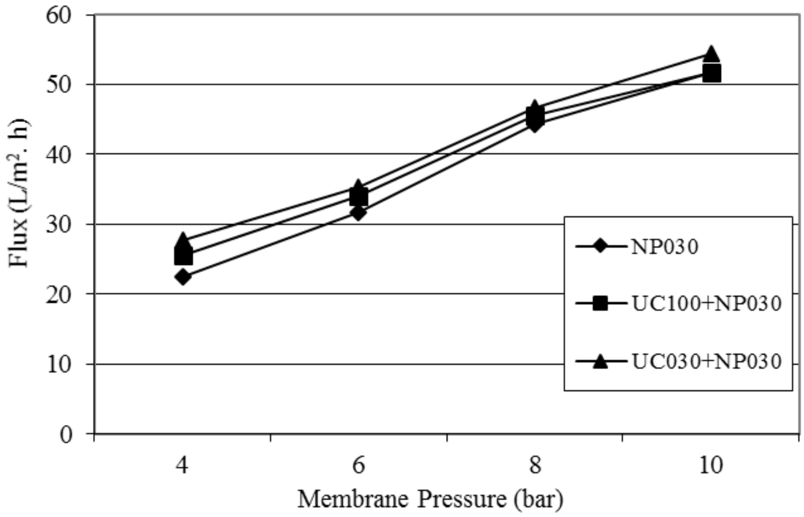

Fig. 3 Flux variation of NP030, UC100+NP030 and UC030+NP030 membranes applications under different membrane pressures

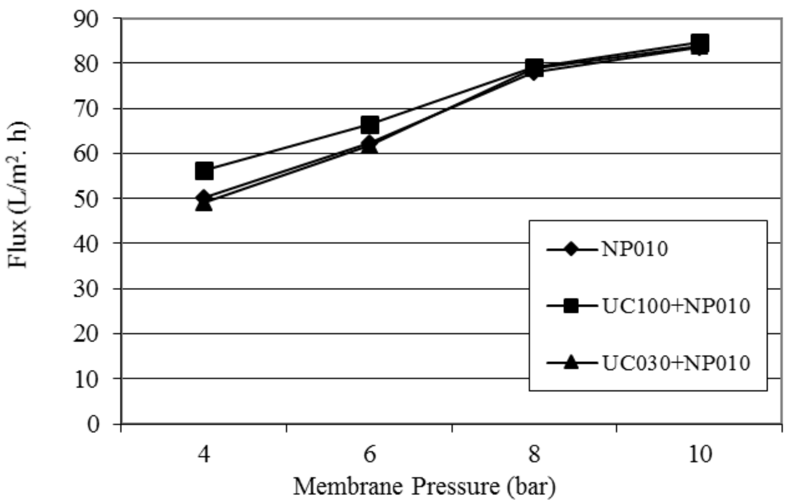

Fig. 4 Flux variation of NP010, UC100+NP010 and UC030+NP010 membranes applications under different membrane pressures

values were determined as 22.5 L/m^2.h and 50.3 L/m^2.h at 4 bar pressure, respectively. According to these results, it was observed that higher flux was obtained by NP010 than that of NP030. High flux values were the important point for the determination type of membrane utilized due to cost of energy, membrane regeneration, need of membrane changing. A significant positive impact has been identified on flux values when NP030 and NP010 were used after using UC100 and UC030 process as a pre-treatment (Figs. 3-4). As a result of UC100+NP010 application, an increase of 11% approximately is observed, but fluxes have become equal for both applications as pressure increases. Likewise, an increase of approximately 24% was observed under 4 bar pressure with

UC030+NP030 process application regarding only NP030 process, but increase percentage in fluxes has been reduced for both process applications while pressure increased.

In this case, usage of UC100 and UC030 membranes, as pre-treatment, are concluded not to have any important contribution to the reuse of flux. On the other hand, pre-treatment with UF membrane before NF membrane process would be more useful due to the increase in operating costs depending on the frequency of membrane replacement, decrease in water quality parameters, potential operating problems because of the rapid clogging of the membranes (Ellouze, Tahri *et al.* 2012).

As in previous studies, use of UF membranes have been discussed and were considered a suitable technique to be a pre-treatment alternative before performing NF process for textile wastewater (Debik, Kaykioglu *et al.* 2010, Barredo-Damas, Alcaina-Miranda *et al.* 2006, Koyuncu, Topacık *et al.* 2004, Sostar-Turk, Simonic *et al.* 2005).

3.1.2 Permeate quality

UF, NF and UF + NF membrane process permeate water and membrane feeding water quality have been determined to assess the possibility of reuse of wastewater of denim washing and dyeing textile industry (Table 4). Conductivity, color, hardness, COD, Mn^{+2}, Zn^{+2}, Cu^{+2}, Fe^{+2} and HCO_3^- parameters are taken into account in determining the permeate water quality. In Table 5, characteristics of the process water according to the studied textile plant, the textile engineering lecture notes and the literature and process water characteristics officially requested were shown. According to Table 5, the process water must have clear, odorless, colorless, and have maximum in the range 6.5 to 7.5 pH, at 50 mg/L of COD, at 2200 µs/cm of conductivity, at 90 mg/L of total hardness, 50 mg/L of SS, 0.3 mg/L Fe^{+2}, 0.05 mg/L Mn^{+2}, 0.07 mg/L Cu^{+2}, 50 mg/L nitrate, 5 mg/L nitrite, 200 mg/L bicarbonate and 200 mg/L total alkalinity values.

Table 4 Analysis results of permeate water samples obtained with membrane process experiments

WASTEWATER	**Conductivity, µs/cm**	**Hardness, mg/L**	**436 nm, RES m^{-1}**	**525 nm, RES m^{-1}**	**620 nm, RES m^{-1}**	**COD, mg/L**	**Mn^{+2}, mg/L**	**Zn^{+2}, mg/L**	**Cu^{+2}, mg/L**	**Fe^{+2} mg/L**	**HCO_3, mg/L**
Aerobically pre-treated wastewater (pH= 6.5)	2843	135	39.8	37.7	31.4	350	4.5	5.6	1.81	0.88	-
MEMBRANE PERMEATE WATER	**Conductivity, µs/cm**	**Hardness, mg/L**	**436 nm, RES m^{-1}**	**525 nm, RES m^{-1}**	**620 nm, RES m^{-1}**	**COD mg/L**	**Mn^{+2}, mg/L**	**Zn^{+2}, mg/L**	**Cu^{+2}, mg/L**	**Fe^{+2}, mg/L**	**HCO_3, mg/L**
UC100	2509	101	20.1	14.3	8.7	92	2.49	2.96	0.43	0.55	49
UC030	2781	129	21.1	16.6	9.2	105	3.47	3.05	0.47	0.42	54
NP010 (4 bar)	2325	85	5.0	3.8	2.7	102	1.47	2.12	0.11	0.08	44
NP010 (6 bar)	2304	83	4.8	3.0	2.0	45	1.18	2.03	0.08	0.04	37
NP010 (8 bar)	2205	80	3.5	2.6	1.5	32	0.96	1.89	0.07	0.04	39
NP010 (10 bar)	2190	81	2.4	1.6	1.1	30	0.85	1.73	0.06	0.03	39
NP030 (4 bar)	2284	72	3.3	2.1	1.3	80	1.02	2.53	0.12	0.05	46

Table 4 Continued

NP030 (6 bar)	2190	68	3.1	2.0	1.2	40	0.88	2.43	0.09	0.05	41
NP030 (8 bar)	2062	60	2.8	1.9	1.1	28	0.84	2.15	0.05	0.02	39
NP030 (10 bar)	2162	66	1.9	1.3	0.9	20	0.33	2.05	0.04	0.01	41
UC100+NP010 (4 bar)	1952	78	3.8	2.6	1.5	80	1.01	1.52	0.07	0.05	37
UC100+NP010 (6 bar)	1940	80	3.3	2.2	1.3	45	0.78	1.33	0.06	0.02	34
UC100+NP010 (8 bar)	1910	76	1.9	1.3	0.4	30	0.66	1.17	0.05	0.02	34
UC100+NP010 (10 bar)	1920	74	1.9	1.2	0.5	30	0.42	1.11	0.28	0.01	32
UC100+NP030 (4 bar)	1974	72	2.0	1.4	0.7	60	1.00	1.93	0.09	0.05	37
UC100+NP030 (6 bar)	1886	70	1.9	1.2	0.4	42	0.79	1.90	0.07	0.04	34
UC100+NP030 (8 bar)	1860	64	1.2	0.8	0.4	25	0.79	1.75	0.06	0.03	32
UC100+NP030 (10 bar)	1750	61	1.3	0.9	0.4	22	0.62	1.50	0.04	0.02	29
UC030+NP010 (4 bar)	2353	82	4.3	2.9	1.9	93	1.37	1.82	0.09	0.03	46
UC030+NP010 (6 bar)	2374	86	5.0	3.3	2.1	41	1.25	1.78	0.07	0.03	44
UC030+NP010 (8 bar)	2361	83	4.2	2.8	1.9	30	1.06	1.33	0.07	0.03	44
UC030+NP010 (10 bar)	2347	81	2.8	1.9	1.2	28	1.06	1.31	0.06	0.02	41
UC030+NP030 (4 bar)	2275	75	3.4	2.3	1.6	75	1.07	1.87	0.12	0.05	49
UC030+NP030 (6 bar)	2129	70	3.2	2.2	1.6	40	0.99	1.85	0.10	0.04	46
UC030+NP030 (8 bar)	2280	66	3.4	2.2	1.4	23	0.90	1.78	0.06	0.03	44
UC030+NP030 (10 bar)	2154	64	2.5	1.6	1.2	23	0.88	1.63	0.05	0.02	41

The analysis results of permeate water showed that obtained removal efficiencies with only UF membranes were lower than NF and UF+NF for all experiments. The quality parameter values of UC100 and UC030 permeate under pressure of 3 bar conductivity was 2509 and 2781 µs/cm; hardness was 101 and 129 mg/L; total color was 43.1 and 46.9 m^{-1}; COD was 92 and 105 mg/L, respectively. These values were well above the limit values given in the literature, especially in terms of conductivity parameters, as can be seen in Table 5. Mn^{+2}, Zn^{+2}, Cu^{+2}, Fe^{+2} and HCO_3^- parameters have been determined that they are above limit values.

During the experiments of using of only NF , results from NP030 were negligibly better than NP010, but permeate water quality has increased by increasing the pressure (Debik, Kaykioglu *et al.* 2010). However, similar results were obtained in the experiments performed under 8 and 10 bar

Table 5 Textile industry process water quality parameters

Parameter	Odour	pH	COD mg/L	Cond. µS /cm	Color	T. hardness mg $CaCO_3$/L	SS mg/L	Fe^{+2} mg/L	Mn^{+2} mg/L	Cu^{+2} mg/L	NO_3^- mg/L	NO_2^- mg/L	HCO_3 mg/L	T. Alkalinity mg/L
Characterization of used process water	-	7.65	-	380	-	10	<10	3	-	0.07	3.5	-	-	60
Recovery water criterias for dye process required by industry authorities	Odourless	6.5-7	<20	-	-	0-3 German Hardness (0-5 French Hardness)	<50	<0.1	0.02	0.005	<50	<5	< 200 mg/l	-
Textile Engineering course notes	Odourless	6.5-7.5	<20	-	-	0-30	<50	<0.1	<0.02	<0.005	<50	<5	-	-
Li and Zhao (1999)	-	6.5-8	0-160	800-2200	-	0-100	0-50	0-0.3	< 0.05	-	-	-	-	50-200
Hoehn (1998)	-	6.5-7.5				90	<500	0.1	0.05	-	-	-	-	-
Rozzi, Malpei *et al.* (1999)	-	-	30	1800	0.01 (426 nm)	-	-	-	-	-	-	-	-	-
Rozzi, Malpei *et al.* (1999)	-	-	<10	<40	Colorless	-	-	-	-	-	-	-	-	-
Goodman and Porter (1980) (Uzal 2006)	-	-	178-218	1650-2200	20-30 Pt-Co	-	-	-	-	-	-	-	-	-
Brik, Schoeberl *et al.* (2006)	-	-	<30	<1800	$<1m^{-1}$ (426 nm)	-	-	-	-	-	-	-	-	-
Gozalvez-Zafrilla, Sanz-Escribano *et al.* (2008)	-	-	<20	<500	-	-	-	-	-	-	-	-	-	-
Lu, Liu *et al.* (2009)	-	-	<50	-	-	-	-	-	-	-	-	-	-	-
Comodo, Masotti *et al.* (1993)	-	-	40	2000	0.02 (426 nm)	-	-	-	-	-	-	-	-	-
Ciardelli, Corsi *et al.* (2000)	-	-	34	35	0.002 (420 nm)	-	-	-	-	-	-	-	-	-
Marcucci, Ciardelli *et al.* (2002)	-	-	8-10	330-2350	-	-	-	-	-	-	-	-	-	-
Bes-Pia, Iborra-Clar *et al.* (2005, 2003)	-	-	100	1000	-	-	-	-	-	-	-	-	-	-
Vajnhandl and Valh (2014)	-	6.5-7.5	20-50	-	Non-visible	90	-	0.1	-	0.005	-	-	-	-

pressure. Experiments performed under 8 bar, with NP010 and NP030 conductivity was 2205 and 2062 μs/cm; hardness was 80 and 60 mg/L; total color was 7.6 and 5.8 m^{-1}; COD was 32 and 28 mg/L, respectively. If permeate water quality results obtained with NF membranes under 8 bar, are compared to the results process water quality mentioned in literature, it was proven that obtained permeate water can be used in textile industry as a process water. Yet, Mn^{+2} parameter values (0.96-0.84 mg/L), obtained with permeate water of NP010 and NP030 membranes, were determined as above the required limit values (0.05 mg/L), respectively.

In the NP010 and NP030 applications where UC100 was used as pre-treatment, similar results were obtained and under 8 bar pressure conductivity was 1910 and 1860 µs/cm; hardness was 60 and 76 mg/L; total color was 3.6 and 3.6 m^{-1}; COD was 28 and 30 mg/L, respectively. Only Mn^{+2} parameter (0.66-0.79 mg/L) was measured above the limit value as measured when only NF membrane process was used.

In the NP010 and NP030 membrane process applications, where UC030 membrane process was used as pre-treatment, conductivity was 2361 and 2280 µs/cm; hardness was 83 and 66 mg/L; total color was 8.9 and 7.0 m^{-1}; COD was 30 and 23 mg/L, respectively. Mn^{+2} parameter (1.06 and 0.90 mg/L), was measured above the limit values as measured in all membrane experiments.

As a result, it was seen that the best efficiency is accomplished using NP030. It is identified that pre-treatment with UF and subsequently NP030 has not any significant effect on the removal efficiency as well as flux. As a result of the experiments, the quality of the permeate water has also demonstrated some increase depending on the pressure increase in the permeate water sample. Experiments performed under 8 and 10 bar pressure do not have any significant difference regarding permeate water quality. Therefore samples from NP010, NP030, UC100+NP010 and UC100+NP030 applications studied under 8 bar pressure were used for dyeing textile fabric.

3.2 Textile dyeing

The dyed fabrics, achieved by dyeing performed with permeate water obtained after membrane

Table 6 Color measurement values of the dyed fabrics

		Color	L^*	a^*	b^*	%R	nm	K/S
PROCESS WATER	**PW 100%**	**B***	53.99	-3.94	-37.07	11.08	620	3.57
		Y*	95.61	32.08	99.21	7.23	440	5.95
		R*	57.64	78.46	9.08	5.23	550	8.59
Dyed Fabric Codes		**Color**	**L^***	**a^***	**b^***	**%R**	**nm**	**K/S**
N10 (NANO 10*)**	**N1025**	**B***	68.72	-81.31	-33.46	20.62	620	3.53
		Y*	99.01	28.64	97.11	9.28	440	4.43
		R*	57.24	77.39	8.63	5.28	550	8.51
	N1050	**B***	55.05	-3.96	-37.72	10.65	620	3.75
		Y*	96.75	32.63	99.04	7.79	440	5.46
		R*	57.89	78.39	8.99	5.43	550	8.24
	N1075	**B***	54.00	-3.94	-37.43	10.20	620	3.95
		Y*	96.03	34.07	100.13	7.23	440	5.95
		R*	57.21	77.95	9.67	5.24	550	8.57
	N10100	**B***	55.51	-4.44	-37.04	10.53	620	3.63
		Y*	95.97	34.05	100.37	7.00	440	6.18
		R*	57.91	78.43	8.74	5.37	550	8.34

Table 6 Continued

UN10 **(ULTRA** +NANO 10***)**	**UN1025**	**B***	57.56	-4.84	-36.25	12.32	620	3.12
		Y*	97.21	31.29	99.57	7.74	440	5.50
		R*	57.65	77.89	9.10	5.40	550	8.29
	UN1050	**B***	56.71	-4.66	-38.04	11.24	620	3.50
		Y*	96.42	32.21	98.56	7.70	440	5.53
		R*	58.13	77.81	7.46	5.64	550	7.89
	UN1075	**B***	58.82	-5.89	-35.22	13.12	620	2.88
		Y*	97.95	29.97	97.22	8.69	440	4.80
		R*	57.90	77.97	8.53	5.48	550	8.15
	UN10100	**B***	56.92	-4.80	-37.12	11.61	620	3.36
		Y*	97.42	31.08	96.46	8.80	440	4.73
		R*	59.51	76.74	6.82	6.62	550	6.69
N30 **(NANO 30****)**	**N3025**	**B***	60.42	-6.13	-35.55	14.08	620	2.62
		Y*	96.66	32.87	99.98	7.42	440	5.78
		R*	57.99	78.37	8.16	5.57	550	8.00
	N3050	**B***	54.72	-4.07	-37.53	10.44	620	3.84
		Y*	97.49	30.92	99.07	7.79	440	5.46
		R*	57.03	77.34	8.02	5.29	550	8.48
	N3075	**B***	61.80	-6.32	-36.27	14.79	620	2.45
		Y*	97.84	31.04	99.23	8.13	440	5.19
		R*	58.53	78.02	7.19	5.75	550	7.72
	N30100	**B***	57.03	-4.81	-37.37	11.65	620	3.35
		Y*	96.51	32.93	99.83	7.44	440	5.76
		R*	57.99	78.21	7.80	5.44	550	8.22
UN 10 **(ULTRA**+ NANO 30****)**	**UN3025**	**B***	57.82	-5.11	-36.52	12.31	620	3.12
		Y*	98.01	30.35	97.32	8.78	440	4.74
		R*	59.55	78.35	6.63	6.11	550	7.21
	UN3050	**B***	58.59	-5.07	-37.33	12.70	620	3.01
		Y*	97.84	30.58	98.66	8.27	440	5.09
		R*	60.20	78.01	5.10	6.40	550	6.89
	UN3075	**B***	58.48	-5.27	-36.86	12.67	620	3.10
		Y*	98.44	29.88	97.99	8.68	440	4.80
		R*	58.24	77.95	6.89	5.55	550	8.04
	UN30100	**B***	57.78	-5.08	-36.58	12.29	620	3.13
		Y*	97.43	31.64	99.27	7.95	440	5.33
		R*	57.20	76.69	8.01	5.52	550	8.09

*B: Blue. *Y: Yellow. *R: Red. **ULTRA: UC100. ***NANO 10: NP010. ****NANO 30: NP030

experiments, were evaluated in terms of measurement and color fastness tests, respectively after standing for 24 hours under standard atmospheric conditions.

CIE Lab and K/S color yield values, resulting with color measurement applied to the dyed fabric, are given in Table 6. K/S color yield values were calculated by using these CIE Lab values according to the Kubelka-Munk equation and assessments were carried out for each color dyestuff.

In all wavelengths, membrane permeate water (P) and process water (PW) used (softened in ion exchange resin) in industry were mixed in different proportions, (100% PW; 75% PW+ 25% P; 50% PW+50% P; 25% PW+75% P) to be used for dyeing process made in the dye bathroom containing 1% concentration of the dyestuff (the color intensity). All of mentioned dyed (in all mixing ratios) fabric samples have provided optimum values in terms of dye efficiency, while the highest color yield value was obtained by dyeing with "red" color dyestuff.

Respectively, color fastness test to sweat in acidic and alkaline media, color fastness to rubbing by krokmeter, color fastness to washing, color fastness to water and color fastness to artificial light tests were performed to the dyed fabrics. Results are illustrated on the Table 7.

Table 7 The color fastness of dyed fabrics

Dyed Fabric Codes		Color	Rubbing Fastness		Washing Fastness		Perspiration Fastness		Water Fastness	Light fastness
			Dry	Wet	Staining	Fading	Acidic	Alkaline		Blue Scale
PROCES WATER	PW 100%	**B***	4-5	4	3-4	4	4-5	4	3-4	6
		Y*	5	4-5	4	4	4-5	4	4	6
		R*	5	4	4	5	4-5	4	4	6
N10 (NANO 10***)	N1025	**B***	5	4	3-4	4-5	4-5	4	3-4	5
		Y*	5	4	4	5	4-5	4	4	5
		R*	5	4	4	4	4-5	4	4	5
	N1050	**B***	4-5	4	4-5	4	4-5	4	4-5	5
		Y*	5	5	4-5	4-5	4-5	4	4-5	5
		R*	5	5	5	5	4-5	4	5	5
	N1075	**B***	5	4	3	3-4	4-5	4	3	5
		Y*	5	4	4	4	4-5	4	4	5
		R*	5	4	4	4	4-5	4	4	5
	N10100	**B***	5	4	3	3-4	4-5	4	3	5
		Y*	5	4	3-4	4	4-5	4	3-4	5
		R*	5	4	4	4	4-5	4	4	5
UN10 (ULTRA** +NANO 10***)	UN1025	**B***	5	4	3-4	4	4-5	4	3-4	5
		Y*	5	4-5	4	4-5	4-5	4	4	5
		R*	5	4	4	5	4-5	4	4	5
	UN1050	**B***	4-5	4	3-4	4	4-5	4	3-4	5
		Y*	4-5	4-5	4	4	4-5	4	4	5
		R*	5	5	4-5	5	4-5	4	4-5	5
	UN1075	**B***	5	4	4	4-5	4-5	4	4	5
		Y*	4-5	4-5	4	5	4-5	4	4	5
		R*	4	4-5	4	5	4-5	4	4	5
	UN10100	**B***	4-5	4	4	5	4-5	4	4	5
		Y*	4	4-5	4-5	5	4-5	4	4-5	5
		R*	5	4	4	5	4-5	4	4	5

Table 7 Continued

N30 (NANO 30****)	N3025	**B***	5	4	3-4	4-5	4-5	4	3-4	5
		Y*	5	4	4	5	4-5	4	4	5
		R*	5	4	4	4	4-5	4	4	5
	N3050	**B***	4-5	4	4-5	4	4-5	4	4-5	5
		Y*	5	5	4-5	4-5	4-5	4	4-5	5
		R*	5	5	5	5	4-5	4	5	5
	N3075	**B***	5	4	3	3-4	4-5	4	3	5
		Y*	5	4	4	4	4-5	4	4	5
		R*	5	4	4	4	4-5	4	4	5
	N30100	**B***	5	4	3	3-4	4-5	4	3	5
		Y*	5	4	3-4	4	4-5	4	3-4	5
		R*	5	4	4	4	4-5	4	4	5
UN 30 (ULTRA**+ NANO 30****)	UN3025	**B***	5	4	3-4	4	4-5	4	3-4	5
		Y*	5	4-5	4	4-5	4-5	4	4	5
		R*	5	4	4	5	4-5	4	4	5
	UN3050	**B***	4-5	4	3-4	4	4-5	4	3-4	5
		Y*	4-5	4-5	4	4	4-5	4	4	5
		R*	5	5	4-5	5	4-5	4	4-5	5
	UN3075	**B***	5	4	4	4-5	4-5	4	4	5
		Y*	4-5	4-5	4	5	4-5	4	4	5
		R*	4	4-5	4	5	4-5	4	4	5
	UN30100	**B***	4-5	4	4	5	4-5	4	4	5
		Y*	4	4-5	4-5	5	4-5	4	4-5	5
		R*	5	4	4	5	4-5	4	4	5

*B: Blue. *Y: Yellow. *R: Red. **ULTRA: UC100. ***NANO 10: NP010. ****NANO 30: NP030

The fabrics dyed in standard dyeing process by using examined industry's PW (100%) and PW with P mixed in different ratios were compared for evaluation of the color fastness test. As a result of the comparison, dyed fabrics have met color fastness values defined in 1% color intensity by Everlight Company and have met color fastness values (3-4: good and better) stipulated in the dyeing results of all dyestuffs (blue, yellow, red) for all dyeing process.

4. Conclusions

In this study, reuse of biologically treated denim washing and textile dye industry wastewater by means of membrane technologies (NF and UF+NF), has been evaluated with testing of obtained permeate water at the laboratory scale dyeing unit. In the comparison of permeate water quality results, obtained with NF membranes under 8 bar, with the results process water quality mentioned in literature; it was proven that obtained permeate water can be used in textile industry as the process water. Yet, Mn^{+2} parameter values (0.96-0.84 mg/L), obtained with permeate water of NP010 and NP030 membranes, were determined as above the required limit values (0.05 mg/L), respectively.

All of mentioned dyed (in all mixing ratios) fabric samples have provided optimum values in terms of dye efficiency. The highest color yield value was obtained by dyeing with "red" color dyestuff. The dyed fabrics have met color fastness values defined in 1% color intensity by Everlight Company and have met color fastness values (3-4: good and better) stipulated in the dyeing results of all dyestuffs (blue, yellow, red) for all dyeing process.

As a result, it was found that clearly possible to reuse of permeate water obtained with NF membrane process systems by means of UF membrane process pre-treatment of dyeing industry wastewater and biologically pre-treated denim washing wastewater. Thus, an alternative source of water has been created for the textile industry which requires large amounts of clean water. The results of this study will be guiding in terms of reusing water. This study brings a new scientific approach to the Best Available Techniques (BAT) applications in textile industry characterized by intensive water consumption by "Integrated Pollution Prevention and Control Notification in Textile Industry" went in effect in Turkey in December 2011.

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