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Separation of Metal Ions and Color from Wastewater of Dyed Polyester by Using Nanofiltration

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Abstract: The investigation presented herein is focusing on an Egyptian textile dyeing industrial wastewater which is highly variable depending on the dyestuff type. The performance of a nanofiltration membrane separation process is demonstrated by treating a dye bath waste stream containing Scarlet red and FB disperse dyes used in dyeing polyester fabrics. The nanofiltration module used was hollow fiber membrane. Two sets of experiments were carried out by using synthetic and real wastewater, at different operating pressures. The contaminants removal performance was evaluated as the difference between two main indicators, namely total dissolved salts (TDS) and the chemical oxygen demand (COD).The obtained results showed a slight difference in TDS removal percent between synthetic and real solutions (32.2% and 31.6% respectively), while large difference in COD rejection of contaminants was observed (47.1% and 30.2%).The mass transfer coefficient for synthetic and real solutions was evaluated. The permeate recovery, salt passage and salt rejection were nearly the same for both solutions. For real solution the removal percent of the monovalent, divalent and trivalent ions were measured. All permeates were colorless using synthetic solutions, while in case of real wastewater solutions it succeeded to remove only 91.2% of the color at 16 bar.

Key words*:* Color removal, disperse dye, nanofiltration; polyester; wastewater.

INTRODUCTION

Textile industries use huge amounts of water and chemicals for finishing and dyeing processes. The composition of wastewater from dyeing and textile processes varies greatly from day to day and hour to hour, depending on the dyestuff, fabric and concentration of fixing compounds which are added [Al-Degs, *et al*.,2000]. Dyeing wastewater contains large amounts of dyestuff together with significant amounts of suspended solids, dispersing agents, salts and metals and consequently can cause serious environmental problems. Unfixed dye releases high doses of color to effluent and is considered as one of the most important hazards in textile effluents, which needs to be treated [Forgacs, *et al*., 2004], because the presence of dyes in water reduces light penetration, precluding the photosynthesis of aqueous flora [Robinson, *et al*., 2002]. Besides that, some dyes may cause allergy, dermatitis, skin irritation, and cancer to humans [Bhatnagar, *et al*., 2005] in addition to being mutagenic [Gong, *et al*., 2005]. The risk, which dyes represent in wastewaters, depends on their chemical structure, physical properties, concentration and exposure time [Reife,*et al*., 1996, Robinson,*et al*., 2001]. Among the available dyes, the use of disperse dye has been continuously increasing in the textile industry after the discovery of synthetic fibers and owing to their chemical properties, they can be applied to most of the synthetic fibers such as polyester, nylon, acetate, cellulose and acrylic [Dominguez,*et al*.,2005; Arslan,*et al*.,2001; Akabari,*et al*.,2002] using simple immersion techniques. Disperse dyes are structurally classified as mainly an azo and anthraquinone chromophoric system with colloidal dispersion, small molecular size and low aqueous solubility [Sirianuntapiboon,*et al*.,2007]. Conventional removal techniques for coloring substances include biological processes [Peralta-Zamora,*et al*.,1999] adsorption techniques [Malik,2004; Benefield,*et al*1982; El-Geundi, 1991; Janos,*et al*.,2003; Meshko,*et al*., 2001; Daneshvar, *et al*.,2003], photo-degradation [Vlyssides,*et al*.,1999] chemical oxidation [Magara,*et al*., 1998]and coagulation-flocculation [Allegre,*et al*.,2004; Anouzla,*et al*.,2009]. Although these physical and advanced chemical treatment processes might be effective for color removal, yet they generate chemical wastes and use more chemicals and energy yielding their relatively high costs.

Moreover, as dyeing processes consume large quantities of water, textile industries often face a shortage of available water sources so that one will have to look for alternative water sources, such as purified wastewater. In Egypt, this strategy to ``purify and reuse`` is also dictated by the fact that governmental regulations and penalties are forcing textile companies to be responsible for their own wastewater treatment and its adaptation to standard norms for the discharge in water courses.

A recent technology that may offer such issues is membrane separation, mostly nanofiltration. The nanofiltration membrane is a low-pressure physical removal process by which organic compounds of low molecular weight, divalent ions or large monovalent ions, such as dyeing auxiliaries, are separated from the water as it is forced through a membrane[All`egre,*et al*., 2006].In most published studies concerning dye house effluents, the concentration of mineral salts does not exceed 20 g/l.[Tang,*et al*.,2002] and the concentration of

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dyestuff 1.5 g/l. The treatment of dyeing wastewater by nanofiltration thus represents one of the newly accessible applications possible for the treatment of solutions with highly concentrated and complex solutions [Peuchot,*et al*.,1997; Knauf,*et al*.,1998; Rossignol,*et al*., 2000; Freger,*et al*.,2000; Kelly,*et al*., 1995].

In this investigation, nanofiltration (NF) membrane process was examined for the treatment of colored wastewater from dispersed dyeing processes in order to be reused. Experiments were first performed using pure dye solutions to get a better insight into the process, and results were then compared with actual disperse dye bath waste effluent resulted from dyeing processes of polyester fabrics at Al-Alamia Company for Dyeing Textile Industry, El-Obour in Egypt.

2. Materials and Analytical Methods:

2.1. Materials:

Two disperse dyes FB and Scarlet red and their dye bath effluent samples used in this study were kindly provided from AL-Alamia Company for Dyeing Textile Industry. The reagents for analysis and other chemicals were used of A.R. grade and used as delivered. The COD and TDS are analyzed according to standard methods [Standard Methods for examination of water and wastewater,2005].

2.2. Instruments:

A digester reactor model HACH and the Ultrameter II^{TM} Myron L Company Model 6P II CE were used for COD and conductivity-TDS measurements respectively. Anions Cl^{-1} and SO_4^{-2} were determined by ion chromatography using a metrolhm 761 Compact IC with conductivity detection. The anion chromatography measurements with chemical suppression were carried out using a metroseanion dual column (4.6×75mm) with particle diameter of 6nm.

2.3. Analytical Methods:

The determination of Ca^{+2} and Mg^{+2} concentrations were performed by atomic absorption spectroscopy by means of an analytical ASS Vario Spectrometer. Na^{+1} and K^{+1} were analyzed by atomic emission spectroscopy using Genway PFP 7 spectrometer, other metals ions were measured by using Spectrophotometer DR2800 HACH. Feed and permeate samples were also analyzed for color by means of a Perkin Elmer Spectrophotometer, the color intensity was recorded using the integral of the absorbance curve in the whole visible range (λ = 400-800).

Experimental Apparatus and Procedure:

Filtration experiments were carried out using a bench scale nanofiltration (NF)unit of capacity 1.68m^3/day as shown in Fig.1. The system consists of a feed tank of 100 liter capacity, a feed pump multi-stage vertical pump of capacity 0.5m^3/hr with 25m head and a 5 micron pretreatment filter. The nanofiltration module is a thin film hollow fiber membrane (TFM) of mol. wt. cut-off of 150 – 300 Da and of active area 2.1 m^2. The module dimensions are 53.3 cm length and 6.4 cm diameter. The membrane side pressure could be varied in the range of 4-16 bar with a maximum pressure of 20 bar. A typical operating flux is ranging between 15–45 l./m^{2}h with a maximum operating temperature of 50°C. The system is very well equipped with permeate and retentate receiving tanks and necessary auxiliaries such as regulators, flow meters, manometers,…..etc.

Two sets of experiments were carried out using synthetic solutions and real dyeing wastewater effluents. Synthetic spent dispersed dye bath was prepared according to that prepared in the company consisting of the following mixture as indicated in Table 1.

Fig. 1: Bench Scale Nanofiltration Unit.

Table 1: Components of typical synthetic spent disperse dye solution.

Component	Component Concentration gm/l
Scarlet GS	0.225
Red FB	0.44
Acetic Acid	0.50
Oil removal soap	0.15
Dispersed agent	0.50
Sodium Chloride	2200

The synthetic solution is allowed to pass through the NF unit with a feed flow of 480 l/h. at room temperature (28^{o}C). The applied pressure over the nanofiltration membrane was varied between 5 and 16 bar. The actual wastewater collected from polyester dyeing processes was pre-treated using $FeCl_3$ at 600 mg/l concentration as optimum coagulant dose in order to reduce the suspended solids (SS) and the COD as was stated and recommended in previous work of the authors(Hafez *et al*.,2011). The pre-treated wastewater was then fed to the NF unit using the conditions described above. In all experiments, the pressure at inlet and outlet of the module was continuously monitored and kept constant. The operating time was established according to steady state conditions (about 10-15 min). Permeate (flux) was measured by means of a pre- calibrated Rotameter, while the retentate was recorded by measuring the discharge volume in specific time. The membrane was cleaned periodically with tap water at the end of each run.

The experimental measured parameters were: flow rates of feed, permeate concentrations, color and applied pressure. The analyses parameters were: S.S., TDS, COD, metal ions such as Na^{+1}, K^{+1}, Mg^{+2}, Ca^{+2},Pb^{+2}, Fe^{+3}, Ni^{+2} and color. The calculated parameters were: water flux (F_w), salt flux (J_s), salt transport (Q_s), permeate recovery (R), salt rejection (S_r) and salt passage (S_p) percentages.

Basic Membrane Equations:

The basic membrane equations used in this study are:

-Water permeate flux:

F_w, l./ m^{2}h. $\quad = K_w\ (\Delta p - \Delta\pi)$ (1)

- The transport salt:

Q_s, l./ m^{2}h., $\quad = (C_m - C_p) \times K_s \times S/d$ (2)

- Salt fluxL:

J_s,mol/m^{2}h $\quad = Q_s/S = \beta\ (C_m - C_p)$ (3)

- Permeate recovery percentage:

R% $\quad = Q_p/ Q_f \times 100$ (4)

- Salt rejection percentage:

S_r % $\quad = (C_f - C_p/ C_f) \times 100$ (5)

- Salt passage percentage:

S_p% $\quad = (C_p / C_{avg}) \times 100$ (6)

RESULTS AND DISCUSSION

5.1. *Synthetic Solution NF Experiments:*

The analysis of the initial prepared synthetic wastewater feed indicated 2200 mg/l. of TDS, 420 mg/l. COD, and remarkable color. The experimental results of the synthetic solution after passing through NF unit at room temperature (28 ^{0}C) and the permeate flux are presented in Table 2.

Table 2: Experimental results and permeate flux of polyester synthetic solution at 28^{0}C.

pressure bar	Effective pressure $\Delta p - \Delta\pi$, bar	Permeate flow rate, l./h.	TDS conc. in permeate, mg/l.	COD conc. in permeate, mg/l.	Permeate flux, l./ m^{2}h.
5	4.95	40	1822	398	19.05
7	6.96	50	1765	387	23.81
9	8.96	70	1698	286	33.33
11	10.96	84	1632	198	40.00
13	12.96	90	1598	156	42.86
16	15.96	95	1549	121	45.24

Feed flow rate:480 l./h.; TDS conc. in feed:2200 mg/l.; COD conc. in feed:420mg/l.

5.1.1.Dependence of TDS and COD Concentrations in Permeate on Applied Pressure:

Figure 2 shows the TDS and COD concentrations in the permeate flow after nanofiltration treatment on varying the applied pressure. It can be observed that increasing the transmembrane pressure produced a remarkable COD and TDS reduction equivalent to 71.2% and 32.2% at 16 bar respectively from initial concentrations. The low %removal of TDS referred to the pore size of the NF membrane.

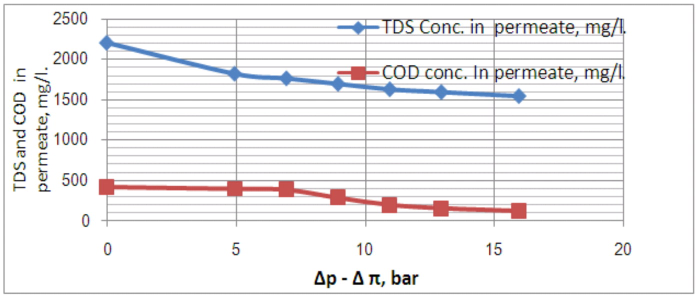

Fig. 2: Effect of applied pressure on COD and TDS removal (synthetic solution).

5.1.2.Transport Properties:

The influence of operating pressure on NF efficiency was analyzed.

5.1.2.1.Water Flux:

The relationship between the overall water flux (F_w)and applied pressure (Δp - $\Delta\ \pi$) is illustrated in Fig.3. Permeate flux increases proportionally with the pressure drop within the pressure range studied due to the enhancement in the driving force, which indicates that the operation is in the pressure–controlled region. It is expected that an increase in feed concentration results in an increase in surface concentration and therefore osmotic pressure increases and thereby leading to decrease in driving force and reduction in flux[Chakraborty,*et al*.,2003].The slope of the line in the figure represents the mass transfer coefficient (K_w) which is equal to 2.9327 l/h.m^{2}bar.

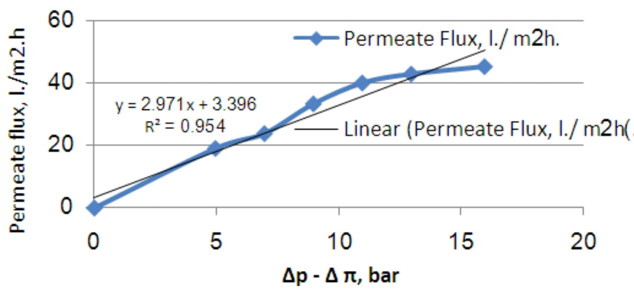

Fig. 3: Relationship between overall water flux and applied pressure for synthetic solution.

5.1.2.2. Salt Transport Constant:

Salt flux (Js) in l/h.m^2 and the driving force ($C_m - C_p$) in mol/l. were calculated by applying Eq. (2) and Eq. (3) at different operating pressure. The relationship between the salt flux (Js) and driving force ($C_m - C_p$) is illustrated in Fig. 4. From the slope of the line, the salt transport constant (β) was found to be equal to 4.815 l/h.m^2. The increase of ionic retention time with water flux is a general phenomenon.

Physically, this occurs because an increase in transmembrane pressure causes higher water flux, where as the salt flux is electrically Donnan exclusion and electro-migration effect and sterically hindered.

5.1.2.3. Permeate Recovery, Salt Passage and Salt Rejection:

The permeate recovery (R), salt passage (S_p) and salt rejection (S_r) percentages were determined via eqs (4-6) and presented in Table 3. It can be observed that with increasing the applied pressure the membrane permeability increases in terms of permeate recovery and salt rejection percent, the latter represents the monovalent metal ions rejection. Nearly 20% permeate recovery and 32.2% salt rejection was achieved at 16 bar. On the contrary, with increasing the pressure the salt passage decreased from 82.2% to 67.9% in the range of pressure studied.

Table 3: Dependence of permeate recovery (R), salt passage (S_p) and salt rejection (S_r) on applied pressure (synthetic solution).

Pressure (P),bar	$R = (Q_p/Q_f) \times 100\%$	$S_p = (C_p/C_m) \times 100\%$	$S_r = \frac{C_f - C_p}{C_f} \times 100\%$
5	8.33	82.2	17.2
7	10.42	79.13	19.8
9	14.58	75.72	22.8
11	17.50	72.22	25.8
13	18.75	70.40	27.4
16	19.79.	67.93	32.2

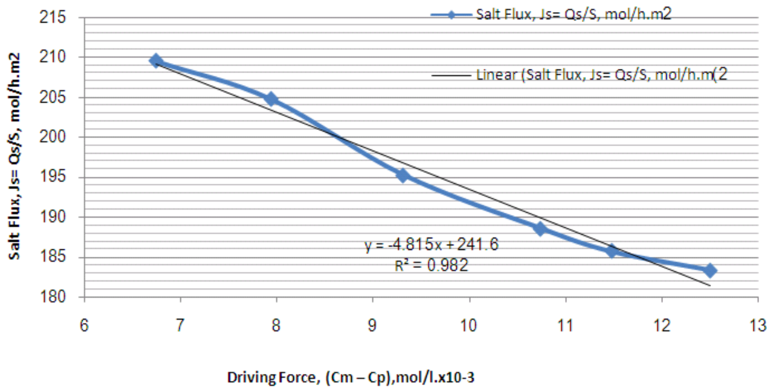

Fig. 4: Relationship between salt flux and driving force for synthetic solution.

5.2. Dyeing Bath Wastewater Treatment:

5.2.1. Pretreatment Experiments:

In order to avoid membrane fouling, which would shorten the membranes lifetime dramatically, the real wastewater solution is allowed to be pretreated, where ferric chloride with 600 mg/l. as suitable coagulant was used [Hafez *et al*.,2011], to reduce SS and COD contained in the textile effluent. The pre-treatment result is depicted in Table 4. The removal % of S.S, COD and TDS were 40%, 31.5% and 0% respectively.

5.2.2. NF Experiments:

The experimental and the permeate flux results of the treated real solution after passing to NF unit at room temperature (28 ^{0}C) are presented in Table 5 at feed flow rate, 480l./h, TDS 2080 mg/l and COD 700 mg/l

5.2.2.1. Dependence of TDS and COD Concentrations in Permeate on Applied Pressure:

The effect of applied pressure on TDS and COD concentration in the permeate is presented in Fig.5. It is noticed that the TDS decreases from 2080 to 1322 mg/l. i.e. 31.6% and the COD decreases from 700 to 488 mg/l.i.e.30.2% as applied pressure increases from 5 to 16 bar.

The TDS removal percent was slightly high in case of synthetic solution when compared with real one, while the COD removal percent was high in case of synthetic solution than real solution, this can be anticipated to the existence of auxiliaries and additives in dye bath effluents.

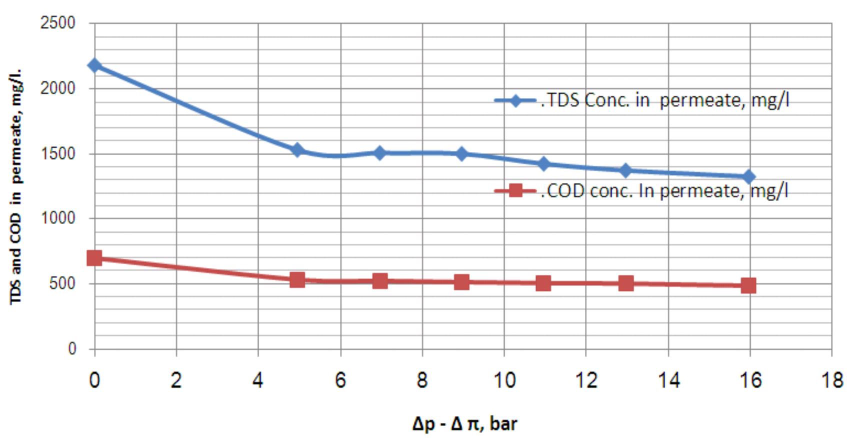

Fig. 5: Effect of applied pressure on COD and TDS removal (Real solution).

Table 4: Results of real wastewater pretreatment experiments.

	TDS in feed mg/l	SS in feed mg/l	COD in feed mg/l
Before treatment	2080	750	921
After treatment	2080	344	700

Table 5: Experimental results and calculations of polyester dyeing bath solution at 28^0C.

Pressure, bar	Effective pressure $\Delta p - \Delta \pi$, bar	Permeate flow rate, l./h.	TDS conc. in Permeate, mg/l.	COD conc. in permeate, mg/l.	Permeate flux, l./ m^2h.
5	4.95	40	1528	535	19.05
7	6.96	50	1504	525	27.62
9	8.96	70	1498	516	33.33
11	10.96	84	1452	508	40.00
13	12.96	90	1440	504	42.86
16	15.96	95	1422	498	45.24

Table 6: Dependence of permeate recovery (R), salt passage (S_p) and salt rejection (S_r) on applied pressure (real solution).

Pressure (P),bar	$R=(Q_p/Q_f)\times100$ %	$S_P=(C_p/C_m)_{\times}10$ %	$S_r=$ $((C_fC_p)/C_f)\times100$ %
5	8.33	72.6	21.5
7	12.08	71.8	27.7
9	14.58	70.30	28.0
11	17.50	66.04	30.5
13	18.75	62.63	33.1
16	19.79.	60.79	31.6

Transport Properties:

a- Water Flux:

The relationship between the overall water flux (F_w)and applied pressure ($\Delta p - \Delta \pi$) is illustrated in Fig.6 as it was expected, a trend similar to that achieved using the synthetic solution was observed, resulting in an equal mass transfer coefficient (K_w),i.e. 2.9327 l/h.m^2bar.

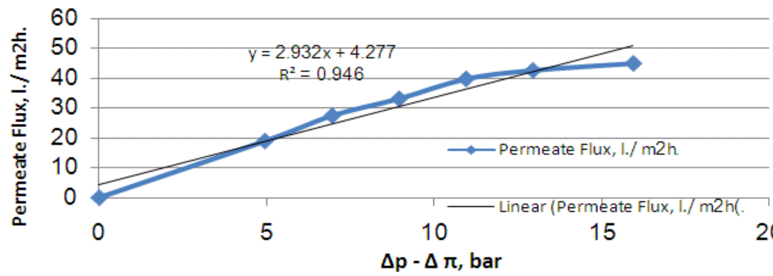

Fig. 6: Relationship between permeate flux and applied pressure (real solution).

b- Salt Transport Constant:

Salt flux (Js) in l/h.m^2 and the driving force ($C_m - C_p$) in mol/l. were determined as above at different operating pressure. The results are depicted in Table 6 and the relationship between the salt flux (Js) and driving force ($C_m - C_p$) is shown in Fig.7. From the slope of the line, the salt transport constant (β) was found to be equal to 4.952 l/h. m^2.

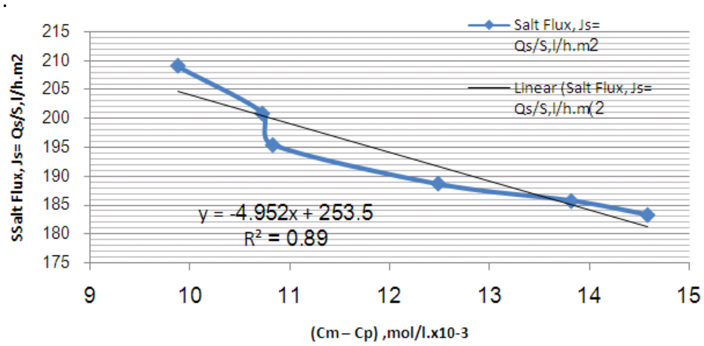

Fig. 7: Relationship between salt flux and driving force (real solution).

c- Permeate Recovery, Salt Passage and Salt Rejection:

As mentioned above, the permeate recovery (R), salt passage (S_p) and salt rejection (S_r) percentages were determined via equations (4-6) and the results are shown in Table 6.

Again, similar behavior is noticed as above for the three parameters, with some difference in the percentages values as depicted in Table 7 which represents the summary of the treatment of synthetic and real dye bath wastewater effluents using NF technique.

It can be seen that only the COD removal and the salt passage percentages exhibited a remarkable differences between the two treated solutions. This can be attributed to the existence of additives and auxiliaries in the company effluent which may affect the separation performance.

Table 7: Results of the NF membrane separation experiments for synthetic and real solutions.

	%TDS removal	%COD removal	Mass Transfer coefficient K_w	Salt transport constant β	R%	S_r%	S_p%
Synth.soln.	32.2	47.1	2.933	4.815	20	32.2	82-68
Real soln.	31.6	30.2	2.933	4.952	19	31.6	72.6-60.8

5.2.2.2. Effect of Time on Permeate Flux at 16 Bar:

The variation of flux value with time for specific feed concentration for real wastewater at 16 bar operating pressure is illustrated in Fig.8. It is evident from the figure that the permeate flux decreases with the operating time because during the experiments, the membrane surface concentration goes on increasing due to the Concentration Polarization phenomenon (CP) which refers to the built up of solute species within a thin boundary layer adjacent to the membrane surface. The accumulation of species at the membrane surface adversely affects the membrane performance.

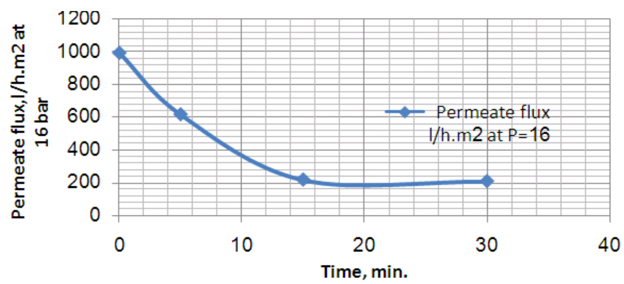

Fig. 8: Effect of time on permeate flux.

The fouling index of the feed to nanofiltration was determined via the following formula [Pizzichini,*et al*.,2005].

$$F_t = F_o e^{-bt} \tag{7}$$

Where b is the fouling index, F_o and F_t are the initial permeate flux and the permeate flux at time t respectively. For calculation purpose, it is desirable to represent the data on a semi-log paper as illustrated in Fig.9. A straight line represents the data fairly well and the slope of the line representing the fouling index b was found by linear regression to be 0.098 min.$^{-1}$ with a correlation factor equals 0.98. The fouling index value is low, thus ensuring good performance characteristics with time.

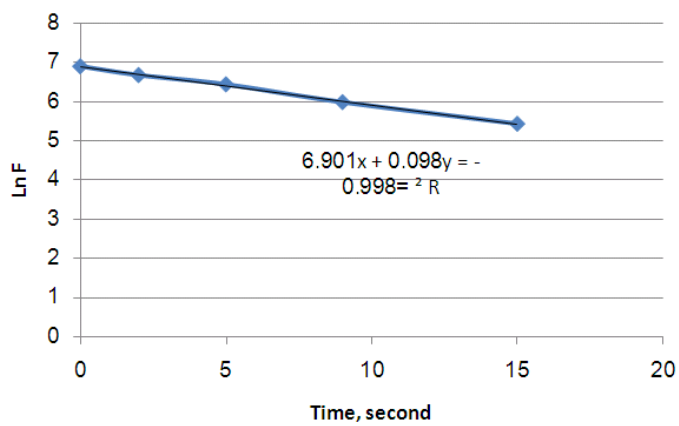

Fig. 9: Determination of the fouling index.

5.2.2.4. Metal Ions Removal in Real Wastewater:

Table 8 represents the metal ions removal percentages for real solution by using nanofiltration separation technology. It is noticed that the monovalent ions were removed by 38-68% while divalent ions were removed by 65-99.9% and trivalent ions were nearly completely removed.

As shown, a significant removal of Ca^{2+} (about 65%) and Mg^{2+} (about64%) were observed. This result indicates a strong interaction between Ca^{2+} and Mg^{2+} and other molecules, contained in the wastewater, forming complexes with high molecular sizes which minimize the incorporation of divalent ions through the NF membrane. This phenomenon can be attributed to intermolecular bridging by divalent ions which associates the COO^- functional groups on the textile wastewater.

Table 8: Metal Ions removal by using NF for real wastewater of polyester dyeing process.

Metal ions	Initial conc. mg/l	Pressure ,bar							
		7		9		11		13	
		Conc.	%R.	Conc.	%R.	Conc.	%R.	Conc.	%R.
Na^+	349	195	44	200	43	195	44	195	44
K^+	6.81	6.81	38	3.84	43.6	3.4	43.6	3.84	43.6
Ca^{+2}	27	13.5	50	9.5	65	9.5	65	9.1	66.3
Mg^{+2}	11	4.5	59	4	63.6	4	63.6	3.5	68.2
Ni^{+2}	0.01	nail		nail		nail		nail	
P_b	0.01	nail		nail		nail		nail	
Fe^{+3}	0.15	0.08	46.7	0.09	40	0.09	40	0.09	40

5.3. Color Removal:

The removal of dye from dispersed dye bath was determined calorimetrically by using the spectrophotometer. The absorbance values of dye before and after treatment were measured at corresponding maximum wavelength 605 nm. Table 9 represents the color removal percent for synthetic and real solutions of polyester dyeing wastewater after NF separation for different applied pressure.

It is remarkable that the difference in color removal percent of synthetic solution and real solution of polyester wastewater dyeing process are more pronounced at elevated pressure indicating 100% and 91.2% respectively than that at lower pressure which may be due to higher molecular weight of the auxiliaries and additives.

Table 9: Results of color removal percent for synthetic and real solutionsafter NF.

Synthetic solution				
Initial dye wastewater Conc., mg/l.	P_c. mg/l. at P = 9	$P_c$mg/l. at P = 11	$P_c$mg/l. at P = 13	$P_c$mg/l. at P = 16
81.0	8.3	8.1	0.0	0.0
Removal % after NF	90%	90%	100%	100%
Real solution				
Initial dye wastewater Conc., mg/l.	P_c mg/l. at P = 9	$P_c$mg/l. at P = 11	P_c mg/l. at P = 13	$P_c$mg/l. at P = 16
91	8.3	8.1	8.0	8.0
Removal% after NF	90.8%	91%	91.2%	91.2%

P_c: Permeate concentration.

6. Conclusion and Recommendation:

- It is remarkable that the TDS removal, the recovery and the salt rejection percentages salt were nearly similar for both synthetic and real solutions while the COD removal percent was higher in case of synthetic solution than for real solution due to the presence of auxiliaries and additives in real solutions.

- For real solution, the monovalent ions were removed by 38-68% while divalent ions were removed by 65-99.9% and trivalent ions were nearly completely removed.

- Color is removed completely for synthetic solution and the removal percent for real solution was 91.2% by using NF separation at 16 bar.

It is recommended to reuse the treated water of real solution in the early rinsing stage of the dye bath. But for using water in all dyeing stages, it is recommended to use low pressure reverse osmosis (LPRO) membrane in series with the NF for the removal of the rest of monovalent ions and color.

Due to the limitations imposed by CP (concentration polarization) and membrane fouling, it is recommended to identify the effects of the controlling parameters in the process at pilot scale.

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Abbreviations:

β Temperature dependent gradient and represent a unique constant for each membrane type, it is known as the salt transport constant
C_{avg} Mean salt concentration, mol/l = $(C_f + C_b)$ /2, mol/l.
C_b Brine concentration, mol/l.
C_f Feed concentration, mol/l.
C_m Salt concentration at the membrane surface = $(C_f + C_b)/2 = Q_s/ Q_W$, mol/l..
C_p Salt concentration in the permeate, mol/l.
CP concentration polarization.
d Membrane thickness.
F_o The initial permeate flux, l./m^2h.
F_w Water flux = permeate flow/ membrane area, l./m^2h.
F_t The permeate flux at time *t*, l./m^2h.
Js Salt flux , mol/m^2h,
Ks The membrane permeability coefficient for salt , l./m^2h.
K_w Mass transfer coefficient, l./m^2.h.bar.
Q_f Feed water flow, l./h.
Qp Product water flow (permeate), l./h.
Q_s Flow rate of salt through the membrane, l./h.
Q_s/S Salt flux J_s, l./m^2h.
Q_w Flow rate of water through the membrane, l./h.
R Permeate Recovery %
S Membrane area, m^2
S_r Salt Rejection
S_p Salt Passage

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