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Nanofiltration process on dye removal from simulated textile wastewater

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ABSTRACT: Dyestuffs removal from industrial wastewater requires special advanced technologies, since dyes are usually difficult to remove by biological methods. In this study nanofiltration process was used for removal of different dyestuffs from solutions. The rate of dye removal by spiral wound nanofiltration membrane in film thin composite MWCO=90 Dalton, was evaluated for four classes of dyes acidic, disperse, reactive and direct in red and blue dyes medium. Dye absorbance was measured by spectrophotometric method (2120 Standard Method 1998). Effects of feed concentration, pressure and total dissolved solids concentration were also studied. Results showed that increasing dye concentration lead to higher color removal up to 98 % and at different pressures for acidic and reactive blue were up to 99.7 %. Different types of dyes had no effect on dye removal and permeate flux. During 2 h.of the operation time, permeate flux decline was increased. Permeate fluxes for different types of red dyes were from 16.6 to 12.6 (L/m^2/h.) and for blue dyes were from 16.6 to 10.45 (L/m^2/h.). Presence of sodium chloride in dye solutions increased dye rejections nearby 100 %. Chemical oxygen demand removal efficiencies for reactive blue, disperse blue, direct and disperse red dyes were also approximately 100 %.

Key words: *Membrane process, dyestuff, textile effluent, chemical oxygen demand, total dissolved solids*

INTRODUCTION

Textile industries produce large amounts of colored wastewater (Al Bastaki *et al.*, 2007). Textile effluents contain many chemical substances generating from resizing, dyeing, printing and finishing processes. Moreover, textile wastewater quality is variable with time and may include many types of dyes, detergents, sulphide compounds, solvents, heavy metals and inorganic salts, the concentration of these chemicals depends on the kind of process (Lopez *et al.*, 1999; Kim *et al.*, 2000).Textile wastewater can not directly be discharged because it has dramatic impacts on receiving water body (Van der Burggen *et al.*, 2001); hence, wastewater reuse and treatment in textile industries is necessary due to the high rate of water consumption and the environmental impacts (Papic *et al.*, 2004). Dyes from dyeing operations are the major source of color in textile effluents. As a result of the low biodegradability of most dyes and chemicals used in textile industry, biological treatment by activated sludge does not always meet with great success and in fact most of these dyes resist aerobic biological treatment and oxidizing agents (Moulin *et al.*, 2004).An advanced treatment technology is necessary, especially if reuse of treated wastewater and decolorization are objective (Rott and Minke, 1999). Therefore, membrane filtration can be an optimal solution to remove color, COD and salinity (DasGupta *et al.*, 2004; Chen *et al.*, 2002). Nanofiltration (NF) is a newly developed membrane technology for various water treatment and purification purposes (Ku *et al.*,

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2005; Tchobanoglous *et al*., 2003). Although, filtration techniques require high initial setup cost, reuse of salts and permeate recompense them. Many approaches have been studied to minimize membrane fouling and reducing the costs; these include pretreatment of feed water, hydrodynamic cleaning with high cross-flow velocity, optimization of chemical and operational conditions such as pH and recovery ratio and modification of the membrane surface (Lee *et al*., 2001; Laine *et al*., 1989; Jucker *et al*., 1994; Yuan *et al*., 1999; Hong *et al*., 1997). There are several appropriate techniques as pretreatments prior to nanofiltration process, including membrane process as microfiltration (MF) and ultrafiltration (UF). Many studies on decolorization of textile wastewater by nanofiltration processes have been carried out which some of them are as follows:

Lebrun *et al.* investigated dye removal by electric field enhanced nanofiltration process by BQ01 and NF45 membranes. Results showed variations in dynamic permeability in the presence of electrolytes and according to the electrical potential applied. A 100 % dye rejection was obtained for both membranes tested (Lebrun *et al*., 2000). Fuchs *et al.* studied the performance of a membrane bioreactor (MBR) for the treatment of textile wastewater, and to investigate its capability to achieve a water quality meeting reuse criteria. COD removal was found to vary between 60 and 95 % and COD levels reduced at lower volumetric loading rates that tested. A distinct relationship between sludge growth and color removal was observed (Fuchs *et al*., 2006). Das Gupta *et al.* studied a nanofiltration with MWCO = 400 Dalton to treat the effluent from a textile plant. Reactive black and red dyes were used and separations with retentions up to 94 and 92 % of the two dyes were achieved respectively. COD removal was obtained up to 94 % in cross flow cell (Das Gupta *et al*., 2003). De *et al.* studied unstirred batch and cross flow nanofiltration with MWCO = 400 Dalton to separate dye from aqueous solutions. Using the experimental results, the model parameters i.e. the diffusivity of the solute (D) and real retention (Rr) of the membrane were evaluated by optimizing the experimental flux and permeate concentration profiles (De *et al*., 2004). Chaudhari *et al.* investigated the decolorisation of the commercially important azo dyes under anaerobic conditions in wastewater. Color removal was achieved up to 99 % in both the dye-containing reactors. COD removals of up to 95 %, 92 % 94 % were achieved in control, orange and black dye-containing reactors, respectively (Chaudhari *et al*., 2002). The aim of this investigation is to study the evaluation of color, COD and TDS removal by Nanofiltration of dye-salt mixtures solutions produced by textile industries.

MATERIALS AND METHODS

Experiments were carried out at the department of Environmental Engineering laboratory at the Science and Research Campus of Islamic Azad University in Tehran, Iran and ended in 2007. In this study, synthetic dye solutions were used for experiments. Four classes of dye with the highest usage rates in Iranian textile industries reactive, disperse, acidic and direct in blue and red, were purchased from Bayer Company. Characteristic of these dyes are presented in Table 1.

Chemical substances which were used for COD experiments were supplied by Merk Company in Germany. All the solutions were prepared with tap water. The experiments were preformed with a nanofiltration pilot plant as shown in Fig.1, without recycling of permeate. Two different microfiltration membranes as prefilter and one nanofilteration membrane were used simultaneously in the same unit. The commercial membranes NF 90 from Filmtec were used. According to the manufacturer product specifications, these membranes are thin film composite for operation at pH 2 to 11 and temperatures up to 45 ºC. The NF 90 membrane is reported in the literature as having a top layer of polyamide composition (Cabral Goncalves *et al*., 2005). Polyamide compounds have amide and carboxyl groups bound to the aromatic rings, which tend to reduce membrane hydrophobicity (Cho *et al*., 1999). Characteristics of the nanofiltration membrane used in this work are shown in Table (2). Two micro filters with pure size of 5 and 1 micron were used in the pilot, rolled as a prefilter for nanofilteration membrane 90 Dalton. Permeation experiments were run at constant temperature 20 ± 2 º C with transmembrane pressures, ranging from 7.5 to 12 bars and flow rate was 8 L/min. Schematic diagram of nanofiltration pilot plant is shown in Fig. 1.

Synthetic wastewater preparation

Dye solutions were synthesized in three concentrations of 5, 50 and 100 mg/L by mixing of every individual dyes powder direct, disperse, reactive and acidic in red and blue in tap water. In this investigation,

Table 1: Characteristics of applied dyes used in the experiments

Commercial name	Color index	Characteristics	Type of dye
		RNF 5 (300%)	Disperse blue
		B2R	Direct blue
Cyanine 5R	Blue 113	5 R	Acidic blue
	Red 60	KNB	Reactive blue
Red E3B	Red 12 b	E3B	Disperse red
Direct red 105			Direct red
Carmozin 206	Red88		Acidic red
		KE3B	Reactive red

Table 2: Characteristics of nanofilteration membrane

Type of membrane	Nominal Active Surface Area (ft^2/m^2)	Operating pressure (Psi / bar)	Product water flow rate (Gpd / m^3/d)	Stabilized salt rejection (%)
NF 90 4040	82 / 7.6	70 / 4.8	(2000)/(7.6)	>97.0

Table 3: Operating condition of NF 90

Parameters	Description
Membrane type	Polyamide Tin film composite
Maximum operating temperature	113 ºF (45 ºC)
Maximum operating pressure	600psi (41 bar)
Maximum feed flow	16gpm (3.6 m^3/h.)
Maximum pressure drop	15 psi (1 bar)
pH range , continuous operation	(2-11)
pH range, short term cleaning (30 min.)	(1-12)
Free chlorine tolerance	<0.1 ppm

to evaluate the effect of total dissolved solids (TDS) on dyes removal by nanofiltration membrane, sodium chloride was added to the solutions in order to produce TDS concentration from 1000, 2000 and 3000 mg/L.

Experimental

Nanofiltration experiments were carried out in different steps:

• In the first step, experiments were carried out in 3 different concentrations 5,50 and 100 mg/L of each dyes, and the nanofiltration consist of single concentration step and solutions had TDS concentration as a solvent (TDS of tap water = 400 mg/L).

• In second step, the nanofiltration experiments were carried out at different pressure 7.5, 10 and 12 bars, acidic and reactive blue dye was used in this stage. Dye concentrations in all experiments were 50 mg/L.

• In the third step, different TDS concentrations were investigated. In order to prepare the dye-salt mixture solutions, sodium chloride was added to tap water to produce TDS concentrations of 1000, 2000, 3000 mg/L. In this step, the temperature was kept constant, between 20-25 ºC, dye concentration was 50 mg/L and transmembrane pressure was 10 bars in all runs.

In all steps, samples were collected for analysis every 15 minutes within 2 h. All experiments were carried out in 2 h. to reach the steadystate condition (Koyuncu and Topacik, 2002).

Sample analysis

Samples of permeate and raw wastewater were collected during experiments. All the experiments were carried out based on Standard Method. COD was determined through open reflux method (5220 Standard Method). The color was determined spectrophot ometrically at a dominant wave length by spectrophotometric method No. 2120 Standard Method (Clesceri *et al*., 1998), using a Shimadzu UV-Vis spectrophotometer (UB-1201 PC). The conductivity for NaCl solution was measured by Horiba DF-H conductivity meter. pH of samples was measured by Horiba pH meter. Retention factor (R) of each species is calculated as:

$$R = \left(1 - \frac{C_P}{C_R}\right) * 100 \quad (1)$$

Where R is retention factor (%) , Cp is concentration in the permeate (mg/L) , C_R isconcentration in the raw dye solutions (mg/L) (Moulin *et al*., 2004).

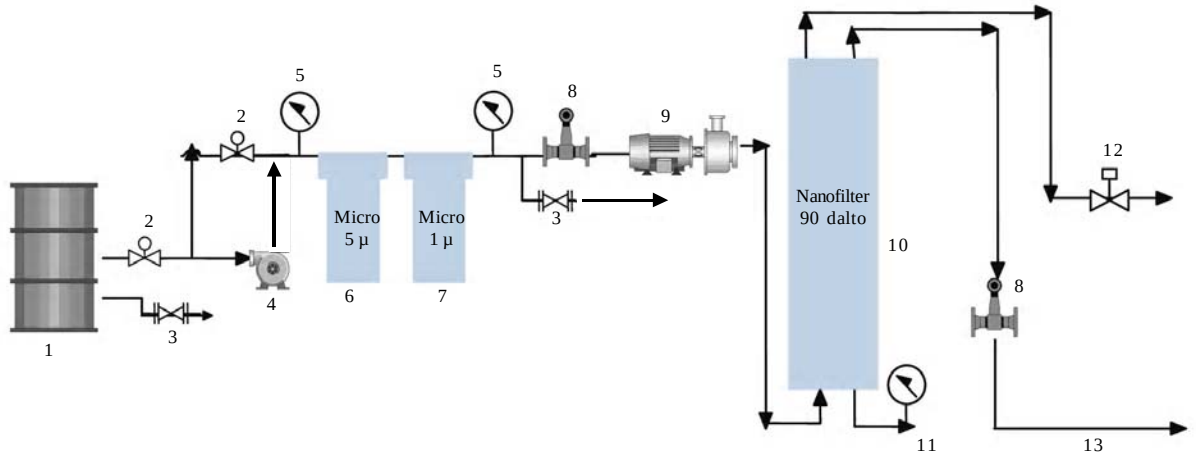
$$J_W = \frac{Q_P}{A} \quad (2)$$

Where J_w is the permeate flux ($L/m^2/h.$), L/ h./ unit of membrane surface area, Q_p is permeate flow per h; A is active surface area of membrane (m^2) respectively. (Ahmad *et al*., 2006).

RESULTS AND DISCUSSION

Effect of dye concentration

The results are shown in Figs. 2 and 3.which the percentage of dye removal in different dye concentration 5. 50 and 100 mg/L were obtained as follow: The dye



1. Feed tank (V= 300 µL) 2. by pass valve 3. Outlet valve 4. Low pressure pump 5. Gage pressure of microfilter 6. Microfilter membrane 5µ 7. Microfilter membrane 1µ 8. Flow meter 9. Centrifuge pump (- Flow = 8 L/min - Pressure head = 5-60 m -RPM = 1425-1725) 10. Nanofilter membrane housing 11. gage pressure of nanofilter membrane 12. Outlet valve (concentrate) 13. Permeate valve

Fig. 1: Schematic diagram of nanofiltration pilot plant

removal percentage for acidic red was 98.33, 99.61, and 99.36 %; disperse red 93.57, 97.24, 99.26 %; reactive red were 99.07, 99.48, 99.74% and direct red 97.5, 99.46, 99.68 %. The results for blue dye were respectively : acidic blue 96.51, 99.74 , 99.8 %; disperse blue 97.54 , 99.24 , 99.58 %; reactive blue 99.07 , 99.15 ,99.62% and direct blue 98.59 , 99.52 , 99.64%. As the results, with increasing of dye concentration, color removals slightly were increased. Different types of dyes did not have any effect on dye removal but color removal for disperse red was increased more than the other because of low solubility of dye in solution. Permeate flux was decreased with increasing dye concentration as a result of increasing the osmotic pressure and the polarization on the membrane surface (Al Bastaki *et al*., 2007; Koyuncu, 2002; Akbari *et al*., 2002). Figs. 4 and 5 show the effects of different types of dyes on permeate flux decline. Permeate fluxes for all types of red dyes were obtained between 16.6-12.6 (L/ $_m$ ^{2}/h), but permute flux for all types of blue dyes were decreased between 16.6-10.45 (L/m^2/h.). Results in Fig. 4 and 5 concluded that permeate flux decline for blue dyes obtained were higher than the red dyes. The Lowest permeate fluxes were obtained for direct and disperse blue dyes (L/m^2/h.) because of low solubility characteristics of these dyes which resulted in membrane fouling in the end of operation time. Furthermore, permeate flux decline were increased during the operation time due to membrane fouling in each run. Membrane fouling may be caused by the dye adsorption on the membrane surface observed at the experimental runs, which was indicated by the presence of color on membrane after filtration, and is in accordance with findings in other studies (Koyuncu, 2002). In this step TDS of dye mixtures without salt addition was measured in all feed samples and permeate samples. TDS rejection was obtained in permeate flow by nanofilteration process 90 Dalton up to 97 % for all experiments and different dyes concentrations and types of dye did not have any effect on TDS rejection.The results of measurements for all experiments were the same, therefore two of them are shown in Figs. 6 and 7.

Effect of pressure

With increasing the pressure, flux is expected to increase accordingly due to solution – diffusion model. The increase in feed pressure will increase the driving force, overcoming membrane resistance (Chen *et al*., 2002; Gholami *et al*., 2003). The effect of transmembrane pressure on dye removal in permeate flux shows a quasi-liner increase in flux with increasing the pressure (Koyuncu, 2002). In this investigation, icreasing the pressure from 7.5 to 10 and 12 bars leads to an increase in the permeate flux. Higher flux values were obtained at 12 bars for applied dyes. The results are shown in Figs. 8 and 9. In this step, reactive blue and acidic blue dyes were used for all experiments. After 2 h., operation time of each run, permeate flux decline was increased due to concentration polarization. The mass transfer coefficient remains constant at low pressure and increased rapidly at high pressure. Both these

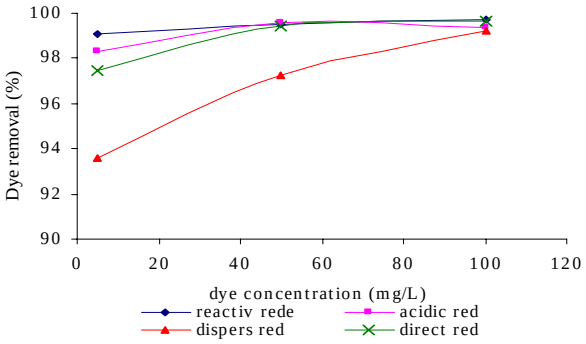


Fig. 2: Variation of dye removal at different concentration of red dye (Dye concentration = 5, 50, 100 mg/L, p=10 bar, T = 20-25 °C, TDS =400 mg/L)

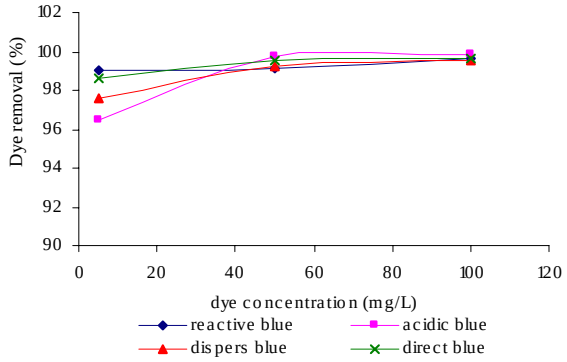


Fig. 3: Variation of dye removal at different concentrations of blue dye (Dye concentration = 5, 50, 100 mg/L, p=10 bar, T = 20-25 °C, TDS =400 mg/L)

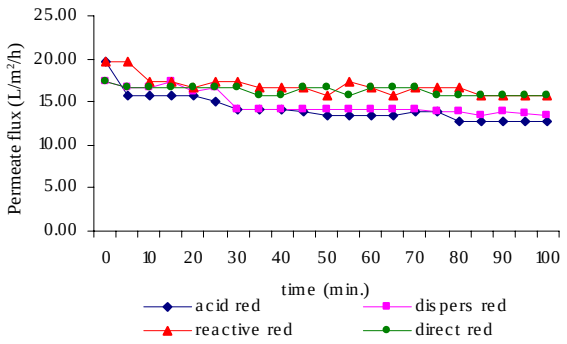


Fig. 4: Variation of permeates flux with time for different type of red dye (Dye concentration = 50 mg/L, p=10 bar, T = 20-25 °C, TDS =400 mg/L)

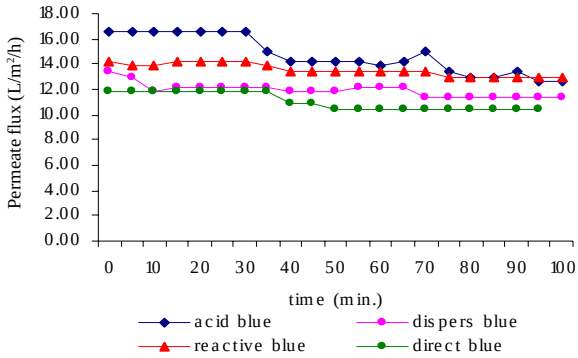


Fig. 5: Variation of permeates flux with time for different type of blue dye.Dye concentration = 50 mg/L, p=10 bar, T= 20-25 °C, TDS =400 mg/L)

relationships imply that mass transfer is not a limiting factor with regards to the high operating pressure. It also signifies the under-development of the concentration polarization layer, placing little importance on this phenomenon. However with high salts concentration in the dye bath, flux dose not increases as rapidly as does normal water flux (Chen *et al*., 2002). Fig. 10 shows the effect of pressure on dye removal in nanofilteration membrane. Dye removal increased with increasing pressure. Dye removal was increased up to 99 % for both reactive and acidic blue dye and it increased slightly up to 100 % at 12 bars. Dye removal for acidic blue was obtained higher than reactive blue. Feed dye concentration was set at 50 mg/L in all experiments in this step.

Effect of salt concentration

Figs. 11 and 12 show the variation of permeate flux with time in dye-salt mixture solutions. The nature of the membrane material, the type and concentration of solute and pH are the parameters that can affect the dye removal in different TDS concentrations. In each case, different electrostatic interactions take place between dye and NaCl and membrane (Koyuncu *et al*., 2003). In the three runs, TDS of solutions concerns to NaCl concentration was set at 1000, 2000 and 3000 mg/L respectively for both reactive and acidic blue dye. The concentration of dye was fixed at 50 mg/L and pressure was fixed at 10 bars. All the experiments were carried out for 2 h.. to reach the steady state conditions. As expected, increasing the salt concentration resulted in a lower permeate flux (Al Bastaki *et al*., 2007). Permeate flux is directly related to the feed pressure and osmotic pressure differences. Osmotic pressure increases with increasing salt concentration which leads to decreases of permeat flux. Flux decline at high salt concentration was lower than the flux decline at low salt concentration (Koyuncu *et al*., 2003). Fig. 11 shows the results of variation permeate flux decline with salt concentration.

Although flux values were high for low salt concentrations, it decreased with time (Koyuncu *et al*.,

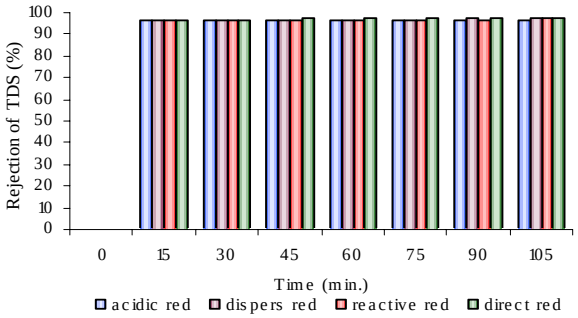


Fig. 6: Variation of TDS removal with time for different type of red dye in dye solutions without salt(Dye concentration = 50 mg/L p=10 bar, T = 20-25 °C, TDS =400 mg/L)

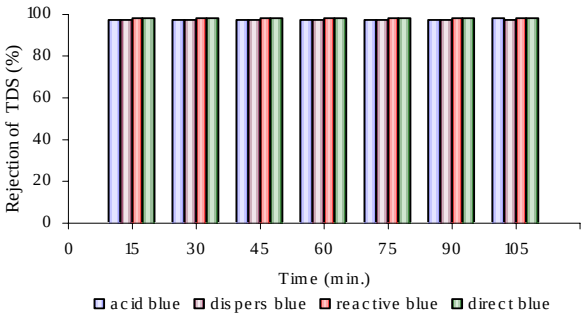


Fig. 7: Variation of TDS removal percent with time for different type of blue dye (Dye concentration = 50 mg/L p=10 bar, T = 20-25 °C, TDS =400 mg/L)

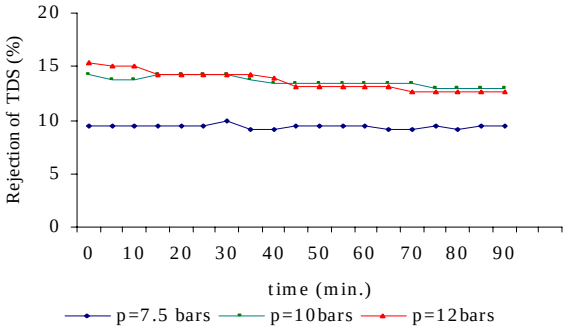


Fig. 8: Variation of permeate flux with time for reactive blue dye in different pressure (Dye concentration = 50 mg/L T = 20-25 °C, TDS =400 mg/L)

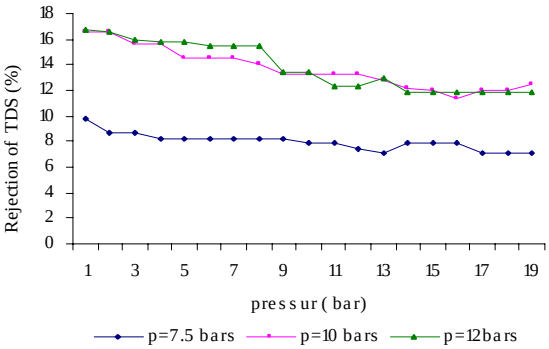


Fig. 9: Variation of permeate flux with time for acidic blue dye in different pressure (Dye concentration = 50 mg/L, T = 20-25 °C, TDS =400 mg/L)

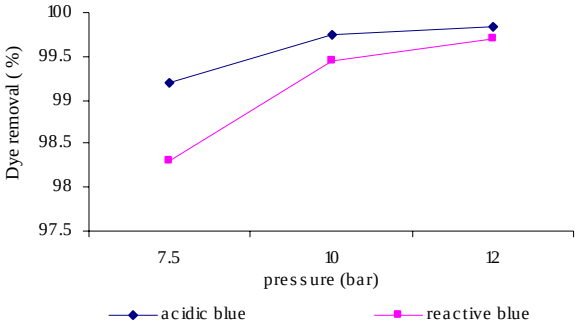


Fig. 10: Variation of dye removal with different pressure for acidic and reactive blue dyes (Dye concentration = 50 mg/L, T = 20-25 °C, TDS =400 mg/L)

2003). One of the reasons for flux decline in high salt concentrations was decreasing of dyes solubility resulted in increasing of dye aggregation (Koyuncu *et al.*, 2003). However, there was almost no variation in flux decline with time for the high NaCl concentrations. Dye removals in all experiments were obtained up to 99 %. It was evident that the presence of NaCl has resulted in a higher color removal; this might be due to the concentration polarization layer formed by the salt that acts as an additional barrier to the passage of the color together with the barrier formed by the dye concentration polarization layer (Al Bastaki *et al.*, 2007). In spite of previous studies, Fig. 13 shows that, with increasing NaCl concentration, dye removal percentage did not change in all the experiments and most of the results were obtained up to 99 %.

COD removal

Results were shown in Fig.14, COD removal for blue dye were obtained higher than red dye and results showed that COD could be removed completely by nanofilteration membrane for some dyes reactive and disperse blue, disperse and direct red. It is concluded that nanofiltration system has been proven to be well suited for the treatment of high dye and salt concentration of textile wastewater and the parameters in steps were optimized. The process allowed the production of permeate with great reutilization possibilities and permeate fluxes can be considered suitable for possible reuse or further polishing. Dye

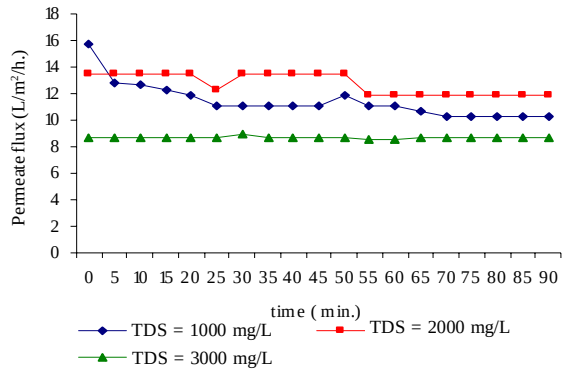


Fig. 11: Variation of permeate flux with time for acidic blue dye in different TDS concentration (Dye concentration = 50 mg/L, T = 20-25 °C, TDS =400 mg/L)

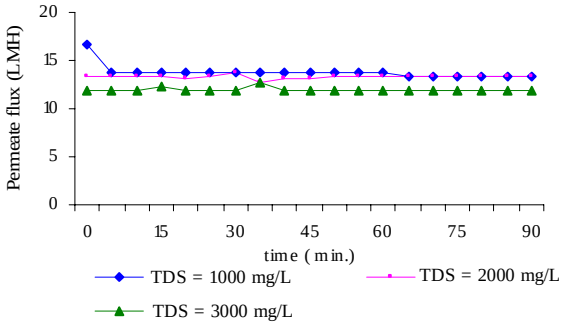


Fig. 12: Variation of permeate flux with time for reactive blue dye in different TDS concentration (Dye concentration = 50 mg/L, T = 20-25 °C, TDS =400 mg/L)

removals in different dye concentrations were obtained up to 98% for all types of dyes. As a result, with increasing dye concentration, color removal slightly increased. Different type of dyes did not have any effect on dye removal. Flux depended on osmotic pressure effects, thus with increasing dye concentration, permeate flux was decreased. Increasing the pressure leads to increasing the permeate flux. Higher flux values were obtained in pressure 12 bars for applied dye. Dye removal was increased with increasing the pressure and maximum dye removal percentage was obtained nearby 100% in 12 bars. Dye removal for acidic blue was higher than reactive blue. As a result, the presence of NaCl had resulted to a higher color removal that concerns to the concentration polarization layer and in the most of experiments, dye removal percent was obtained up to 99 %. Permeate flux was declined (L/m²/h.) in high salt concentrations due to dye aggregation so that minimum permeate flux, 11.9 and 8.3 (L/m²/h.), was obtained for both of reactive and acidic blue dye in TDS concentration of 3000 mg/L respectively. Chemical oxygen demand was completely removed by nanofilteration membrane for reactive blue, disperse blue, disperse and direct red dyes.

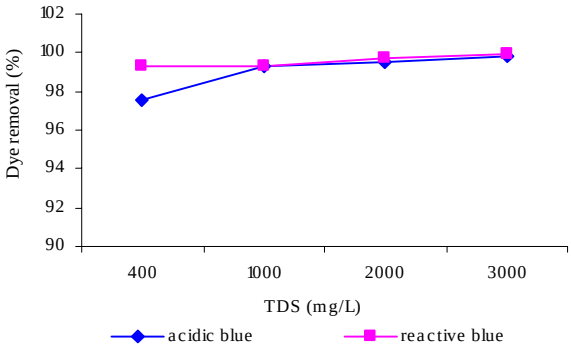


Fig. 13: Variation of dye removal with different TDS concentration for acidic and reactive blue dyes (Dye concentration = 50 mg/L, T = 20-25 °C, p =10

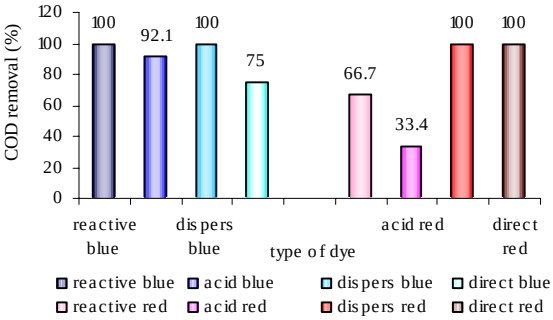


Fig. 14: Variation of COD removal at different classes of dye (Dye concentration = 50 mg/L, P= 10 bar, T= 20 – 25 °C, TDS = 400 mg/L)

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