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Nanofiltration performance in the removal of dye from binary mixtures containing anthraquinone dyes

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ABSTRACT

Textile industries produce a large quantity of wastewater containing different dyes which can impose a significant risk to environment. In this study, the performance of a commercial polyamide nanofilter membrane for the removal of binary mixture of dyes of reactive (ionic) and disperse (non-ionic) dyes was investigated. The synergic effects of both dyes on the removal performance of the membranes were also studied. The experiments were performed at different dye concentrations of 60, 120, and 180 mg/l for each dye, pH of 6, 8, and 10, and for constant feed pressure of 800 kPa. Results showed that increasing pH from 6 to 10 and also disperse concentration from 60 to 180 mg/l enhanced the removal efficiency of both dyes about 10%. Whereas, by increasing the reactive dye concentration, disperse removal efficiency decreased by 3%. Response surface methodology was also used to optimize the effect of experimental parameters on the dye removal percentage.

Keywords: Reactive blue; Disperse blue; Color removal; Nanofiltration; Textile wastewater; RSM

1. Introduction

Water is rapidly becoming scarce in many countries around the world because of its high consumption rate as a result of population growth and industrialization of developing countries. Textile industry is considered as one of the top heavy users of water, between 25 and 250 m^3 per ton of product, and is also the largest consumer of dyes [1]. Textile industries produce high volumes of wastewater and therefore contaminate the water resources. Studies indicated that 10% of synthetic dyes are released into the wastewater stream during dyeing and washing processes [2,3]. Dyes have been found to be the worst pollutants in the textile wastewater as they are able to absorb dissolved oxygen in water and thus undermine aquatic life by affecting the photosynthesis process [4]. Since the majority of dyes are carcinogenic, toxic, and highly stable, their removal from textile wastewater before releasing them into the environment seems to be an essential process [2]. Various methods have been

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used for the treatment of textile wastewater and dye removal, including membrane systems [5], chemical precipitation, adsorption, biosorption [6], electrocoagulation [7], and even ion exchange process [8].

Chemical precipitation needs treatment chemicals and also produces high quantities of sludge which needs extra separation stages [9]. Biological methods quite often are time-consuming process and cannot degrade dyes completely due to the complex, stable, and aromatic structure of synthetic dyes. There are also a few synthetic dyes which have inhibitory effects on micro-organism [1]. Chemical oxidation methods like using chlorine are slow and need reactive materials which are dangerous for transporting and storing. Advanced oxidation methods such as ozonation, photocatalyst, and photo-Fenton are costly and may produce toxic byproducts [4]. These disadvantages emphasized the need for further research employing more efficient and inexpensive methods consuming the least amount of chemicals to treat textile wastewater in order to reuse water [10].

Among these methods, pressure-driven membrane technologies including ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) have been found to be an effective advanced way to treat wastewater [5]. NF process is a lower pressure-needed technology than RO and subsequently has lower cost with higher permeate flux, while it still has a higher rejection efficiency than UF [11,12]. Size exclusion and Donnan exclusion are the separation mechanisms of NF process [13], which makes it useful to separate both charged in addition to uncharged organic solutes [14].

NF performance for dye removal from contaminated water has attracted attention of many researchers as a standard technique [15].

Reactive dye removal was experimented by Petrinic et al. using an NFT-50 NF membrane [16]. They studied membrane fouling, permeation flux and color, conductivity, and chemical oxidation demand (COD) removal as a function of operating pressure and feed concentration. Sahinkaya et al. [17] used a combination of membrane bioreactor (MBR) and NF to treat textile wastewater. Nataraj et al. [18] utilized NF and RO for the removal of color from a contaminated solution. Liu et al. [19] used and compared RO and NF in order to evaluate COD removal, salinity reduction, and permeate flux. Alcaina-Miranda et al. [20] compared different NF membranes to investigate the impact of pH and transmembrane pressure (TMP) on NF performance. The effects of electrolytic oxidation on the NF performance and the permeate flux for treating textile wastewater was evaluated by Xu et al. [21]. To investigate the feasibility of reusing textile wastewater with NF, three NF membranes with different pore sizes (NF90, NF200, and NF270 from Dow Filmtec) were studied under four different pressures by Gozálvez-Zafrilla et al. [22]. A thin-film composite (TFC) NF hollow-fiber membrane was used by Zheng et al. [4] for dye and COD reduction of biologically treated textile wastewater. The color removal and COD reduction were achieved 99 and 90%, respectively. Ellouze et al. [23], studied DK spiral-wound NF membrane performance under the pressure of 10 bar and temperature of 40℃. COD, color, and salinity removal were 57, 100, and 30%, respectively. Yu et al. [24] evaluated the effect of membrane properties (cellulose acetate membrane and TFC polyamide) on the reactive dye removal from dye/salt mixtures.

Among the dying colors, reactive dyes are heavily used in the textile industries for cellulose and cotton fiber dyeing. This significant attention is due to the dying simplicity procedure, brightness, and also as high water solubility [25,26]. Nonbiodegradable reactive dyes are the most problematic as their degree of fixation to cellulose fibers is low and approximately 10–40% of them are released in to the sewage stream [27]. Disperse dyes are used in textile industry for dyeing the polyester fibers. Since the production of polyester fibers has increased rapidly since 1940s, a significant increase in the consumption rate of disperse dyes has been observed [28]. Thus, these two types of dye removal were investigated in this study. The optimization of operational factors can significantly increase the removal efficiency. In this study, two dyes, reactive blue 19 and disperse blue 56, were fed into an NF system as a binary mixture. The dye concentrations and pH were considered as the experimental parameters. Response surface methodology (RSM) was applied to find the optimum condition. The main purpose is to maximize the dye removal applying the optimum operating conditions.

2. Materials and methods

2.1. Chemicals

Experiments were conducted using two anthraquinone dyes, reactive blue 19 containing two anonic sulfonate groups and disperse blue 56 (non-ionic). The chemical structures are as shown in Fig. 1. Dyes were supplied by Golnesar Co., Isfahan, Iran and were used to prepare simulated textile wastewater without purification necessity. The characteristics of both dyes are pointed in the Table 1. All other chemicals to adjust pH (HCl (37%), NaOH) were purchased from Merck Company of Germany.

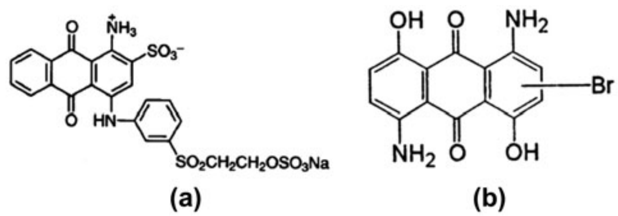

Fig. 1. Chemical structures of reactive blue 19 (a) and disperse blue 56 (b).

2.2. Laboratory NF plant

As shown in Fig. 2, experiments were conducted in a cross-flow module using a polyamide spiral-wound NF membrane. According to the manufacturer, the NF membrane is a TFC membrane with pH range of 2–11 and allowable maximum tolerable pressure and temperature of 15 bars and 45˚C, respectively. Its isoelectric point and active surface area is 4.6 and 0.35 m^2, respectively (Table 2). The NF experimental setup was equipped with diaphragm pumps and a feed tank with 60 L capacity to storage and supply of effluent to the system (see Fig. 2).

2.3. Preparation of dye solution

Dye powders were dissolved and stirred in distilled water by a Bandelin HD 2200 model sonopuls in order to prepare synthesized solutions with concentrations of 60,120, and 180 mg/l of each dye. The pH of solution was adjusted by 0.1 M HCl and 0.1 M NaOH and was measured using a Metrohm 827 pH Lab meter. All experiments were performed according to the American Public Health Association water and wastewater examination methods [29], at a constant temperature (25˚C) and pressure of 800 kPa. The recovery percentage was 75 ± 3% of feed volume. The permeated concentration of dyes were determined by measuring the absorbance values of samples at two maximum wavelengths of 592 and 560 nm related to the reactive blue 19 and disperse blue 56, respectively. Measurements were conducted by a V-570 model spectrophotometer. The below equation was used to calculate the color removal efficiency:

$$R\,(\%) = \left[1 - \frac{C_P}{C_f}\right] \times 100 \qquad (1)$$

where R is the removal efficiency, C_P and C_f are the permeate and feed concentrations, respectively.

2.4. Experimental design

The statistical design of experiments (DOE) is a structured and systematized method of experimentation in which all factors vary simultaneously over a set of experimental runs in order to determine the relationship between the operating factors and the

Table 1
Characteristics of applied dyes in this study

Color index name	Commerical name	Color index number	Molecular weight (g/mol)	Chemical formula	Maximum wavelength (nm)	Valence
Reactive blue 19 (RB19)	Remazolbreliant blue	61200	626.55	$C_{22}H_{16}O_{11}N_2S_3Na_2$	592	-2
Disperse blue 100	Tulasteronblue2RX	63285	365	$C_{15}H_{13}BrN_2O_4$	560	0

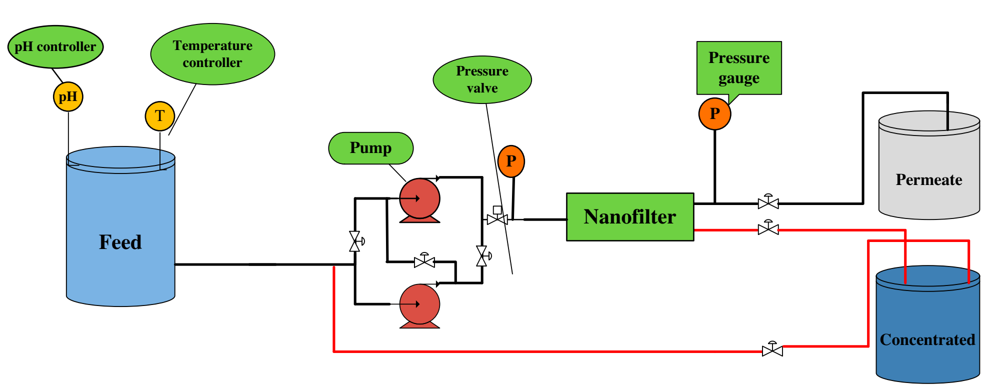

Fig. 2. Schematic of the NF experimental apparatus.

Table 2
Commercial polyamide TFC membrane specifications

Provider	CSM company of Korea
Skin layer	Polyamide
Maximum tolerable pressure	20 bar
pH range	2–11
Isoelectric point	4.5
Molecular weight cut off (MWCO)	300 Da
Surface electrical charge	Negative
Active surface (m^2)	0.35

Table 3
Experimental factors and their corresponded selected levels

	Levels		
Factors	−1	0	1
Concentration of reactive dye	60.0	120.0	180.0
Concentration of disperse dye	60.0	120.0	180.0
pH	6.00	8.00	10.00

response. The RSM was used to determine the optimal experimental conditions for the maximum dye removal. The Box–Behnken design consisting of 15 runs with 3 factors and 3 levels was applied in this study. The experimental parameters were pH, reactive blue 19 and disperse blue 56 concentrations. The experimental parameters and their levels for the experimental design are presented in Table 3.

3. Results and discussions

The RSM has been applied to find out the relation between the dye removal and the operating factors according to the experimental results (based on the Box–Behnken design) shown in Table. Each experiment is repeated twice apart from itself, which means there are three runs for each experiment to measure the experimental error.

The final empirical models obtained based on the Box–Behnken design in terms of actual parameters for reactive and disperse removal percentages are as follows:

$$\begin{aligned}\text{Reactive removal} = {} & 92.307 \\ & + 0.026\ \text{Reactive concentration} \\ & + 1.3\,\text{pH} + 9.6 \\ & \times 10^{-3}\ \text{Disperse concentration} \\ & - 0.124\ \text{pH}^2\end{aligned} \quad (2)$$

$$\begin{aligned}\text{Disperse removal} = {} & 79.64 \\ & + 0.1\ \text{Disperse concentration} \\ & - 8.9 \\ & \times 10^{-3}\ \text{Reactive concentration} \\ & - 1.24 - 0.174\,\text{pH}^2\end{aligned} \quad (3)$$

where pH is between 6 and 10, reactive and disperse concentrations are between 60 and 180 mg/l. The regression factors R^2 used to distinguish the agreement of the experimental response values and the calculated values by the models were 0.98 and 0.99 for

Table 4
Experimental responses based on the Box–Behnken design

Run	*A*: Disperse concentration (mg/l)	*B*: Reactive concentration (mg/l)	pH	Disperse removal (%)	Reactive removal (%)
1	120.0	180.0	10.0	86.41 ± 0.5	98.22 ± 0.8
2	180.0	60.00	8.00	89.30 ± 0.4	96.33 ± 0.9
3	180.0	120.0	10.0	88.90 ± 0.7	98.01 ± 0.3
4	60.00	60.00	8.00	84.14 ± 0.1	94.28 ± 0.2
5	120.0	120.0	8.00	86.33 ± 0.5	96.10 ± 0.7
6	120.0	60.00	6.00	86.35 ± 0.6	93.85 ± 0.6
7	120.0	180.0	6.00	83.08 ± 1.1	95.32 ± 0.5
8	120.0	60.00	10.0	88.66 ± 0.6	96.26 ± 0.6
9	180.0	120.0	6.00	86.12 ± 0.2	95.95 ± 0.4
10	120.0	120.0	8.00	86.15 ± 1.4	96.38 ± 0.9
11	60.00	180.0	8.00	81.97 ± 0.9	95.91 ± 1.0
12	120.0	120.0	8.00	86.67 ± 0.3	96.20 ± 1.5
13	180.0	180.0	8.00	86.16 ± 0.6	97.96 ± 0.0
14	60.00	120.0	10.0	84.77 ± 1.2	96.72 ± 1.3
15	60.00	120.0	6.00	81.44 ± 0.8	94.06 ± 0.1

Table 5
ANOVA for disperse removal rate

Model terms	Mean square	Sum of squares	Degree-of-freedom	*F*-value	*p*-value	Status	Factor effect
Model	8.530	76.78	9	200.37	<0.0001	Significant	
A: Disperse concentration	41.31	41.31	1	970.31	<0.0001	Significant	Positive
B: Reactive concentration	14.61	14.61	1	343.06	<0.0001	Significant	Negative
C: pH	17.26	17.26	1	405.32	<0.0001	Significant	Positive
$A \times B$	0.230	0.230	1	5.3000	0.0696	Not significant	
$A \times C$	0.076	0.076	1	1.7800	0.2401	Not significant	
$B \times C$	0.260	0.260	1	6.1100	0.0564	Not significant	
$A \times A$	3.000	3.000	1	70.500	0.0004	Significant	
$B \times B$	0.026	0.026	1	2.6300	0.1658	Not significant	
$C \times C$	0.110	0.110	1	2.6300	0.1658	Not significant	
Lack-of-fit	0.024	0.073	3	0.3500	0.7975	Not significant	
Pure error	0.070	0.140	2	–	–	–	

reactive removal and disperse removal, respectively. This hints that more than 97.87 and 99.23% of the data can be explained by these empirical models. Therefore, the model can be considered adequate for the predictions and optimization of response value.

Analysis of variance (ANOVA) for the RSM model shown in Tables 4 to 6 implies high *F*-values of disperse concentration and pH for disperse and reactive removal, respectively. It should be pointed out here that as *F*-value increases, its effect on the response increases.

3.1. Influence of experimental parameters

The corresponding contour plots for reactive blue 19 and disperse blue 56 removals in the mixture of these two dyes are presented in Figs. 3–6. It should be noted that one of the factors is held constant at the center point.

3.1.1. Effect of pH

The plots in Figs. 3 and 4 show the effect of pH on dye removal in the mixture. Higher pH values result in an increase in the removal efficiency for both dyes. It should be noted that the effect of pH on the reactive removal is more significant than disperse removal as seen in Table 5. As previously mentioned, the isoelectric point of used NF membrane is at pH 4.5 above which the membrane becomes negatively charged, resulting in an enhancement in the electrical repulsive

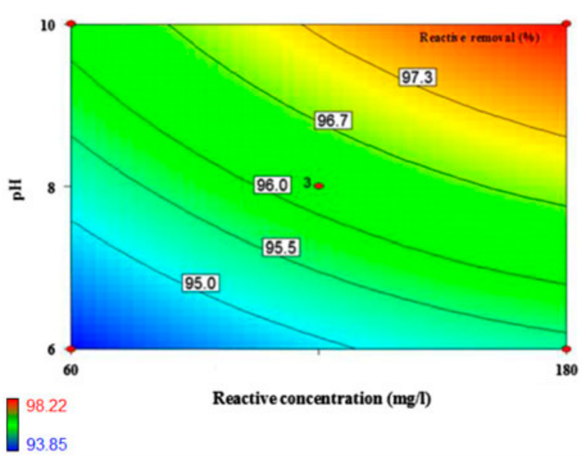

Fig. 3. Contour plot of reactive removal factor as a function of pH and reactive concentration at fixed disperse concentration of 120 mg/l.

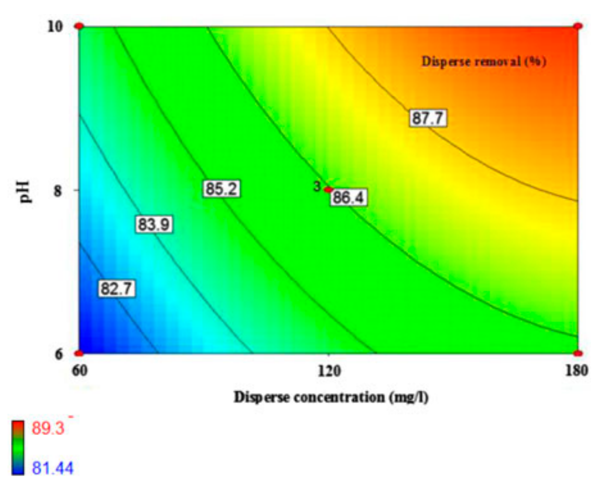

Fig. 4. Contour plot of disperse removal factor as a function of pH and disperse concentration at fixed reactive concentration of 120 mg/l.

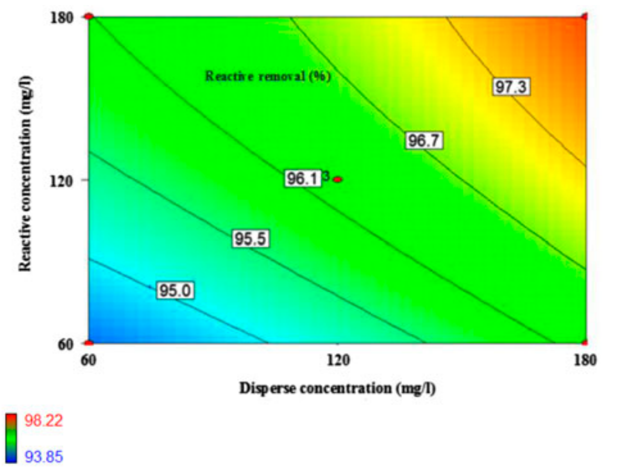

Fig. 5. Contour plot of reactive removal factor as a function of disperse concentration and reactive concentration at fixed pH 8.

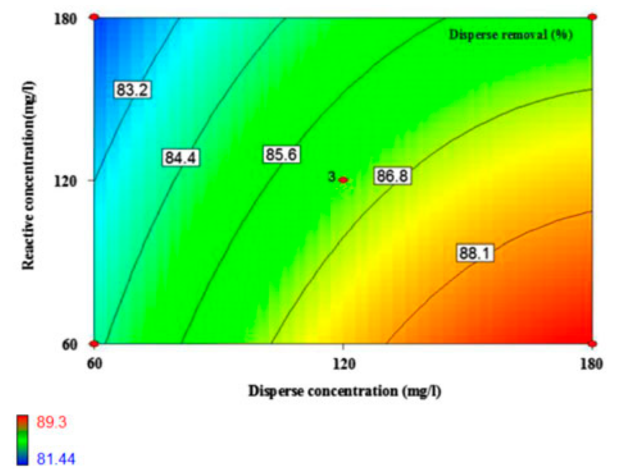

Fig. 6. Contour plot of disperse removal factor as a function of disperse concentration and reactive concentration at fixed pH 8.

force between the membrane and reactive dye [30]. Also, pH was found the second important parameter for disperse removal (Tables 4 to 6) despite being free of charge, as by increasing the pH, the membrane pores shrink, resulting in an increase in the disperse removal. Similar behavior was observed by other researchers [31–34].

3.1.2. Effect of dye concentration

From Figs. 4 and 6, it can be observed that increasing the disperse concentration resulted in higher removal efficiency (about 10%) for both dyes due to the increase in exclusion mechanism and space prevention [35]. Whereas, enhancing the reactive dye concentration decreased the disperse removal efficiency up to 3%. Reactive blue 19 molecules are larger than the disperse ones and are also negatively charged. Therefore, by increasing reactive concentration, the repulsion force between the membrane and the reactive dye increases. Since a limited number of molecules are able to pass through the pores, in a competition to cross the membrane, more disperse dye molecules are able to pass through the membrane and so its removal efficiency decreases [33].

The main objective of this study is to determine the optimum operating conditions in order to maximize the dye removal factor. The maximum efficiency

Table 6
ANOVA for reactive removal rate

Model terms	Mean square	Sum of squares	Degree-of-freedom	*F*-value	*p*-value	Status	Factor effect
Model	2.800	2.800	9	72.360	<0.0001	Significant	
A: Disperse concentration	6.620	6.620	1	171.46	<0.0001	Significant	Positive
B: Reactive concentration	5.590	5.590	1	144.79	<0.0001	Significant	Positive
C: pH	12.58	12.58	1	325.46	<0.0001	Significant	Positive
$A \times B$	0.000	0.000	1	0.0000	1.00	Not significant	
$A \times C$	0.090	0.090	1	2.3300	0.1875	Not significant	
$B \times C$	0.060	0.060	1	1.5500	0.2678	Not significant	
$A \times A$	0.025	0.025	1	0.6600	0.4545	Significant	
$B \times B$	0.130	0.130	1	3.4300	0.1230	Not significant	
$C \times C$	0.057	0.057	1	1.4800	0.2776	Not significant	
Lack-of-fit	0.051	0.150	3	2.5300	0.2957	Not significant	
Pure error	0.020	0.040	2	–	–	–	

Table 7
Results under the optimum conditions.

Dye type	Predicted value	Experimental value
Disperse removal	90	89.63 ± 0.1
Reactive removal	99	98.89 ± 0.4

for dye removal from contaminated water was estimated 99 and 90% for reactive blue 19 and disperse blue 56, respectively by Box–Behnken method, which was attained under the optimum conditions of 180 mg/l disperse dye, 166 mg/l reactive dye, and pH of 10.The experimental results under the optimum conditions are presented in Table 7. The results are obtained by the design expert software, version 8.0.1 and the predictions are estimated according to the experimental results.

4. Conclusions

The results of this study indicate that NF process using a commercial spiral-wound polyamide nanofilter has an effective efficiency in dye removal from contaminated water. The factors of pH and disperse dye concentration have the most significant effect on the response of reactive blue 19 removal and disperse blue 56 removal, respectively. The results also revealed that with an increase in pH and disperse blue 56 concentration, the removal efficiency of both dyes in the mixture increased, whereas increasing the reactive concentration only increased its removal efficiency and decreased the disperse removal efficiency. The DOEs using response surface method can be considered as a good choice for optimizing the number of experiments and the results analysis.

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