

Removal of textile dye from aqueous solutions by nanofiltration process

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Abstract

The feasibility of employing nanofiltration (NF) in the decolorization of ionic (direct blue 86) and nonionic (disperse blue 56) dye aqueous solutions was investigated. The effects of feed concentration (60- 180 mg/l), pressure (0.5- 1.1 MPa) and pH (6- 10) were studied. Experiments were performed in a laboratory- scale set up by using a TFC commercial spiral wound polyamide nanofilter. The response surface method (RSM) was adopted in the experimental design to obtain the impact of the mentioned factors. It was found that increasing the dye concentration and pH enhances the removal efficiency up to 96 and 92% for direct and disperse dye, respectively. With an increase in pressure from 0.5 to 0.8 MPa the removal percentage increases and then, from 0.8 to 1.1 MPa decreases. The maximum dye removal efficiencies which were predicted at the optimum conditions by Design Expert software were 98 and 94% for direct blue 86 and disperse blue 56, respectively.

Key words: Textile, Wastewater, Reuse, Nanofiltration, Dye

Highlights

- Investigation of the effective parameters (concentration, pressure and pH) on the performance of commercial nanofilter for color removal and water reuse
- Assessment of the impact of dye charge on nanofiltration performance
- Prediction of the optimum condition and maximum dye removal efficiency by response surface method

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Introduction

Today, a wide variety of industries such as textile, pharmaceutical, paper and pulp are benefiting from dyes in their products (1). Textile industries produce high volumes of waste water and therefore contaminate the water resources. As an example, Textile wastewater may include different dyes, detergents, sulphide compounds, solvents and heavy metals. The majority of dyes are carcinogenic, toxic and highly stable (2). Hence, water treatment and reuse in textile industries seems necessary due to high rate of water consumption and environmental impacts (3). Various methods have been used for treatment of textile wastewater and dye removal. Traditionally, biological and physico-chemical treatments have been applied to textile effluents being discharged into the municipal sewage. Biological treatment is considered as a high efficient method for COD removal, but since it cannot degrade the non-biodegradable dyes completely due to the complex, stable and aromatic structure and has its limitations for water salinity removal, it is not much desirable (4). Physical-chemical treatment has advantages such as reducing dissolved, suspended, colloidal and nonsettleable materials from water by chemical coagulation followed by gravity settling which will cause the total elimination of colors. However, there are a few drawbacks such as experiencing some difficulty in managing the produced sludge, the high chemical costs, and the low soluble COD as well as salinity removal efficiency (5). Most recent methods such as

electrochemical treatment, chemical oxidation by ozone, or a combination of UV-radiation, ozone and H_2O_2 , have shown better results in the treatment of the textile wastewater; however these methods are still costly and cannot deal with water salinity (6). Another method which has gained popularity overtime is the utility of membrane separation as it has lower costs and easy to operate (7). This technology offers a realistic solution to meet the increasingly strict discharge limits and to reuse the textile wastewater by providing treated water with acceptable quality. Filtration methods require capital investments, but since they result in cost savings through recycling solutes and permeate reuse, they seem beneficial (8).

Ultrafiltration (UF) is widely applied in separation and purification processes to recover auxiliary chemicals and sizing agents or high molecular weight substances and insoluble dyes from the dyeing effluents. However, UF cannot reduce the conductivity and eliminate low molecular weight dyes (9 & 10). Nanofiltration or reverse osmosis can eliminate auxiliary matters and salts concentrations which Ultrafiltration fails to omit, so water purified by nanofiltration is suitable for being reused in textile industry (11 & 12).

Alcaina-Miranda et al. compared different NF membranes to investigate the impact of pH and transmembrane pressure (TMP) on Nanofiltration performance (13). Their results showed that change in pH did not have any effect on the permeate flux. However, it was observed that the salts rejection increased. At operating condition

of pH of 11 and pressure of 10 bar, salt rejection was reached to 71%. It was also found that pH variation from 5 to 11 caused an average increase in salts rejection of 43%. According to their work, the combination of UF and NF for treatment of waste water is a viable water treatment approach, though the effect of different parameters on color removal was not included in their study.

In another study by Aouni et al., ultrafiltration and nanofiltration processes were used to treat synthetic reactive dyes aqueous solutions and a raw textile effluent supplied from rinsing baths of Spanish textile industry (5). In their work, the influence of the reactive dyes molecular weights and the effect of used membranes types and membrane cut-off on permeate flux were studied. Good conductivity rates (80%) and high COD and color retention rates (>90%) were obtained for both NF 200 and NF 270 membranes for all studied dyes solutions. But they did not investigate color removal under different operating conditions. In another effort, investigations were conducted to closewater cycle in dye-houses (14). Optimum operation conditions of a nanofilter membrane (DL membranes, Osmonics, USA) were determined. The effect of temperature, pressure and pH on filtration rate was investigated. Experiments were performed in a laboratory-scaled dyeing apparatus for different types of real wastewater. Their best results were obtained at pH range of 7-10 and at a temperature of below 60 although the pressure effect and color type were not considered in their work. E. Ellouze

et al. examined the performances of the nanofiltration (NF) as a post treatment after coagulation flocculation (CF) (6). In their study, they proposed nanofiltration process for the treatment of industrial textile effluent coming from a Tunisian factory in terms of permeates flux performances and pollutant retention. The best operating conditions were found at 10 bar for TMP and 40°C for operating temperature. However, they did not investigate the pH impact on color removal. In an investigation performed by Liu et al. reverse osmosis (RO) and nanofiltration (NF) were evaluated and compared for a textile effluent treatment in terms of COD removal, salinity reduction and permeate flux. However, color removal efficiency and the influential parameters had not been studied (4).

Response surface methodology was applied to find the optimum conditions. The main purpose of this work was to study the effects of pressure, concentration, pH and the type of color (direct, disperse) on color removal efficiency of a textile wastewater using nanofiltration

It was also tried to maximize the dye removal efficiency based on the obtained optimum operating conditions.

Material and Method

Materials: HCL (37%), NaOH were provided by Merck Company of Germany. The used colors in this investigation were supplied by Golnesar Woolen Co., Isfahan, Iran and were used without further purification, with the characteristics presented in Table 1.

Table 1- Characteristics of applied dyes used in the experiments

Commercial name	C.I.Name	Application	Color tone	Physical form	Powder pH	MW	Maximum wavelength (nm)	Molecular Formula	Charge
Direct Turquoise Blue G	Direct blue 86 (DB86)	Paper, leather, textile, fiber and paint industry	Bright Greenish Blue	Dark Reddish Blue Powder	8.5-10.5	780	660	$C_{32}H_{14}CuN_8Na_2O_6S_2$	2-
Tulasteron blue 2RX 100	Disperse blue 56 (DB56)	Textile	Blue	dark blue powder	4- 9	365	560	$C_{15}H_{13}BrN_2O_4$	0

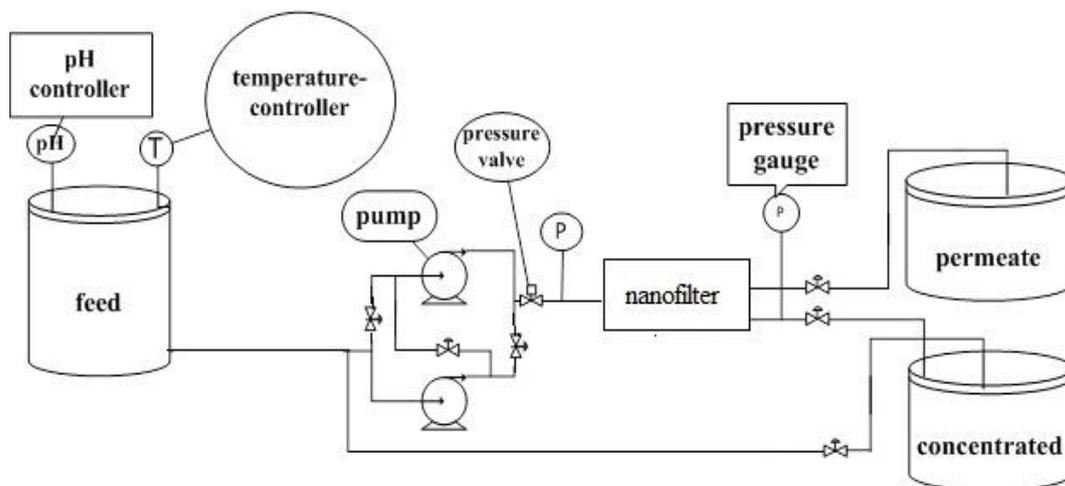


Fig.1- Schematic view of the pilot system

Experimental set- up: As schematically presented in Fig. 1, experiments were conducted cross- flow module using a polyamide spiral wound NF membrane. To have a constant flux experiment, the diaphragm pumps pressure was set to 8.5 bar to achieve a permeate flow rate of 1.6 liter per minute.

Membrane characterization: The commercial membrane NF (A Korean spiral wound polyamide membrane) was used in the nanofilter module. According to the manufacturer product specifications, these membranes are thin film composite which operate at pH of 2 to 11 and a maximum pressure of 20 bar. The membrane specifications are presented in Table 2.

Table 2- Commercial polyamide TFC membrane specifications

Provider	TFC company of Korea
Skin layer	Polyamide
Maximum tolerable pressure	20 bar
pH range	2- 11
Isoelectric point	4.5
Surface electrical charge Active surface (m ²)	Negative 0.35

Experimental procedure: Dye powders (direct blue 86, disperse blue 56) were dissolved and stirred in distilled water by a Bandelin HD 2200 model sonopuls in order to prepare synthesized solutions in three concentrations of 60, 120 and 180 mg/L. The pH of solution was adjusted by 0.1M HCl or 0.1M NaOH. The experiments were run at constant temperature of $20 \pm 2^\circ\text{C}$ with the recovery percentage of $75 \pm 3\%$ of

feed volume. All measurements were performed according to water and wastewater examination methods (15). The residue dye concentrations were obtained by measuring the absorbance at maximum wavelength of each dye and computing the concentration from calibration curve using a V- 570 spectrophotometer according to standard method No. 2120C. The dye decolorization percentage was calculated by using NF membrane rejection as below:

$$R(\%) = \left[1 - \frac{C_p}{C_f} \right] \times 100 \quad (1)$$

Where R is the rejection percentage, C_p and C_f is permeate and feed concentration, respectively.

Response surface methodology: The response surface methodology (RSM) is an effective method for the optimization of responses including different designs i.e. Central Composite, Box- Behnken, One Factor, D- Optimal and etc. (16). In this work, the Box- Behnken design was selected to optimize the responses. A full factorial design for three parameters of pH, pressure and concentration requires 27 runs while by using the design total number of experiments reduced to 15. The experiments were conducted randomly and also the confidence level (C.L) of 95% is considered to avoid possible errors due to systematic bias. In this study, the aim was to obtain maximum removal rate of DB86 and DB56, which were considered as the responses. The contour plots and the analysis of variance (ANOVA) evaluation are used to analyze the results. The contributory factors and their selected levels are presented in Table 3.

Table 3- Factors and selected parameters

Factors	Level 1	Level 2	Level 3
Dye concentration (mg/L)	2 ± 60	3 ± 120	5 ± 180
pH	0.1 ± 6	0.1 ± 8	0.1 ± 10
Pressure (MPa)	0.1 ± 0.5	0.1 ± 0.8	0.1 ± 1.1

Results

The experimental design and results of experiments for both of the colors are presented in Table 4 and 5. The ANOVA table for the dye removal efficiency of DB86 and DB56 is shown in Table 6 and 7, respectively.

The mathematical model based on actual values for DB56 and DB86 removal percentages are expressed through Eqs. (2) and (3) as follow, respectively:

$$R_1(\%) = 89.49 + 1.49 \times A + 1.08 \times B + 0.64 \times C + 0.36 \times A \times B + 0.095 \times A \times C$$

$$- 0.46 \times A^2 - 0.14 \times B^2 - 2.25 \times C^2 \quad (2)$$

$$R_2(\%) = 95.48 + 1.22 \times A + 1.48 \times B + 0.44 \times C + 0.98 \times A \times C + 0.34 \times B \times C + 0.13 \times A^2 - 0.72 \times B^2 - 2.27 \times C^2 \quad (3)$$

The regression parameter R^2 is applied to determine the agreement compared with the experimental responses to the ones estimated by Box- Behnken method. For DB56 and DB86 rejection, R^2 statistic parameter is 98.2 and 99.3%, respectively. Due to their proximity to unity, the proposed models are accurate and acceptable.

The effect of dye concentration, pH and pressure: According to the presented results in Table 6 and 7, P - value of dye concentration, pH and pressure are less than 0.05. As the F - value increases, its impact on the response enhances. The factors of pH and dye concentration have the most significant effect on the responses of DB86 and DB56, respectively and the interactions among parameters are not important.

Table 4- Box- Behnken design results for DB86

Experiment No.	Dye Concentration (mg/L)	pH	Pressure (MPa)	Dye Removal Percentage
1	60 ± 2	8 ± 0.1	1.1 ± 0.1	91.5
2	120 ± 3	10 ± 0.1	1.1 ± 0.1	93.8
3	180 ± 5	6 ± 0.1	0.8 ± 0.1	93.2
4	60 ± 2	10 ± 0.1	0.8 ± 0.1	94.0
5	60 ± 2	8 ± 0.1	0.5 ± 0.1	90.9
6	180 ± 5	10 ± 0.1	0.8 ± 0.1	96.7
7	120 ± 3	8 ± 0.1	0.8 ± 0.1	94.4
8	120 ± 3	10 ± 0.1	0.5 ± 0.1	92.1
9	180 ± 5	8 ± 0.1	0.5 ± 0.1	93.0
10	180 ± 5	8 ± 0.1	1.1 ± 0.1	93.9
11	60 ± 2	6 ± 0.1	0.8 ± 0.1	91.2
12	120 ± 3	6 ± 0.1	0.5 ± 0.1	89.9
13	120 ± 3	8 ± 0.1	0.8 ± 0.1	94.9
14	120 ± 3	8 ± 0.1	0.8 ± 0.1	94.2
15	120 ± 3	6 ± 0.1	1.1 ± 0.1	90.3

Table 5- Box- Behnken design results for DB56

Experiment No.	Dye Concentration (mg/L)	pH	Pressure (MPa)	Dye Removal Percentage
1	120 ± 3	10 ± 0.1	1.1 ± 0.1	88.7
2	180 ± 5	6 ± 0.1	0.8 ± 0.1	89.2
3	60 ± 2	8 ± 0.1	0.5 ± 0.1	84.9
4	60 ± 2	6 ± 0.1	0.8 ± 0.1	86.4
5	120 ± 3	8 ± 0.1	0.8 ± 0.1	89.7
6	120 ± 3	6 ± 0.1	0.5 ± 0.1	85.5
7	60 ± 2	10 ± 0.1	0.8 ± 0.1	87.9
8	120 ± 3	10 ± 0.1	0.5 ± 0.1	87.6
9	180 ± 5	10 ± 0.1	0.8 ± 0.1	92.1
10	120 ± 3	8 ± 0.1	0.8 ± 0.1	93.9
11	120 ± 3	6 ± 0.1	1.1 ± 0.1	86.6
12	60 ± 2	8 ± 0.1	1.1 ± 0.1	86.2
13	120 ± 3	8 ± 0.1	0.8 ± 0.1	89.2
14	180 ± 5	8 ± 0.1	0.5 ± 0.1	87.1
15	180 ± 5	8 ± 0.1	1.1 ± 0.1	88.8

Table 6- Analysis of variance for direct blue 86 removal efficiency

Model terms	Mean square error	Sum of the error squares	Degree of freedom	F- value	P- value	Status
Model	5.65	50.83	9	78.35	<0.0001	Significant
A: dye concentration	10.7	10.7	1	148.36	<0.0001	Significant
B: pH	18.33	18.33	1	254.29	<0.0001	Significant
C: P	1.5	1.5	1	20.76	0.0061	Significant
B×A	0.14	0.14	1	1.95	0.2213	Not significant
C×A	0.036	0.036	1	0.5	0.5108	Not significant
C × B	0.45	0.45	1	6.23	0.0548	Not significant
A × A	0.017	0.017	1	0.23	0.6514	Not significant
B× B	2.21	2.21	1	30.60	0.0026	Significant
C× C	18.04	18.04	1	250.25	<0.0001	significant
Lack of fit	0.033	0.098	3	0.25	0.8583	Not significant
Pure Error	0.13	0.26	2	-	-	-

Table 7- Analysis of variance for disperse blue 56 removal efficiency

Model terms	Mean square error	Sum of the error squares	Degree of freedom	F- value	P- value	Status
Model	5.54	49.90	9	30.51	0.0008	Significant
A: Dye concentration	17.73	17.73	1	97.58	0.0002	significant
B: pH	9.27	9.27	1	51	0.0008	Significant
C: P	3.33	3.33	1	18.32	0.0079	Significant
B×A	0.51	0.51	1	2.81	0.1543	Not significant
C×A	0.036	0.036	1	0.2	0.6744	Not significant
C × B	1.000E- 004	1.000E- 004	1	5.5E- 004	0.9822	Not significant
A ×A	0.78	0.78	1	4.28	0.0935	Not significant
B× B	0.071	0.071	1	0.39	0.5591	Not Significant
C× C	18.71	18.71	1	102.99	0.0002	Significant
Lack of fit	0.26	0.79	3	4.56	0.1850	Not significant
Pure Error	0.058	0.12	2	-	-	-

Effect of dye concentration: Adding more dyes from all types into the feed solution resulted in a higher concentration and consequently higher removal efficiency due to increase in screening effect. The results indicate that among the tested dyes in this investigation direct blue 86 removal efficiency is 5% more than disperse blue dye. Furthermore, increase in the concentration of low solubility dyes (blue 56) has shown a greater effect on the rejection percentage as they could induce concentration polarization and thus fouling on the membrane surface following the pore plugging mechanism (17- 18). The results are shown in Fig. 2a and 3a. Another possible mechanism could be the cake or gel layer formation as the NF membranes usually have very narrow pore size therefore the possibility of cake layer formation is higher than the pore constriction. From contour plots can be observed that changing in parameters at the same time didn't affect on the final results.

Effect of pH: The Korean TFC membrane commercial NF membrane has the isoelectric point of 4.5. Therefore, as the pH range is above these points, the membrane is negatively charged. Also with an increase in pH, the membrane thin polyamide layer start to swell, and thus its

pores begin to shrink [18]. It is revealed from the Fig. 2b and 3b that pH increase from 6 to 10 has a positive effect on nanofiltration performance. Having the most electrical repulsive force between the membrane and the direct blue 86 dye (for ionic dyes as both the membrane and dye are negatively charged) and pores shrinkage at pH 10, the experiments show that pH 10 was a suitable operating pH for both dyes. The pH effect was more significant on the colors of greater capacity. Similar behavior was observed by Mahmoudi et al. on remediation of contaminated water from nitrate and diazinon by nanofiltration process (18).

Effect of pressure: The results showed that dye removal efficiency increases with an increase in pressure from 0.5 to 0.8 MPa as membrane processes are under pressure, thus increasing the pressure increases efficiency. It was found that further increase in pressure had negative impact on nanofiltration performance and dye removal efficiency as it causes the dye molecules to pass through the membrane pores. The observed results are consistent with other researchers (5 & 6). Therefore, the optimum pressure was defined at 0.8 MPa as shown in Fig. 2c and 3c.

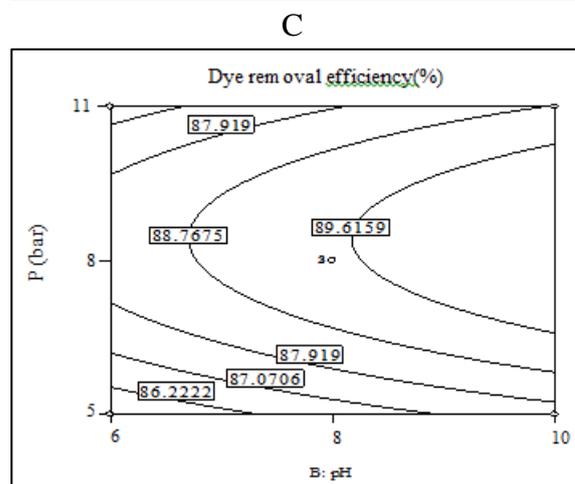
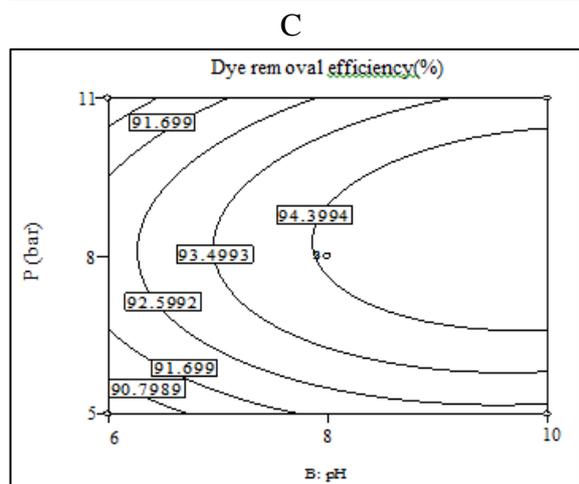
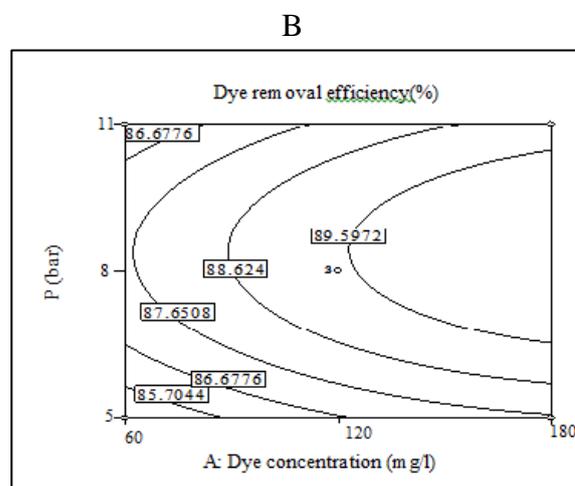
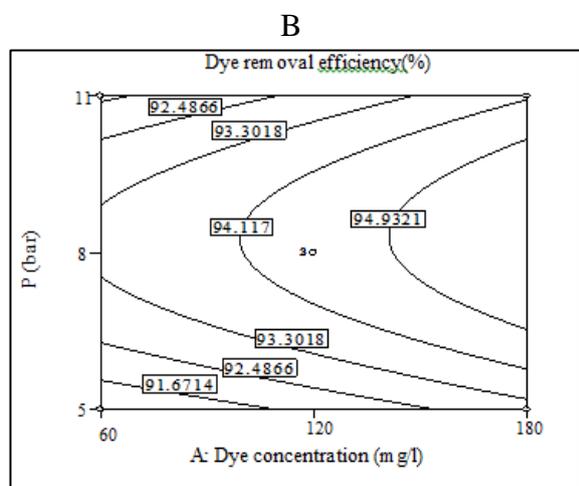
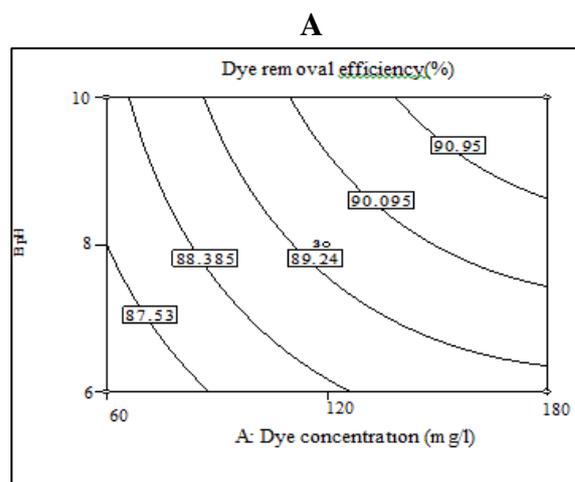
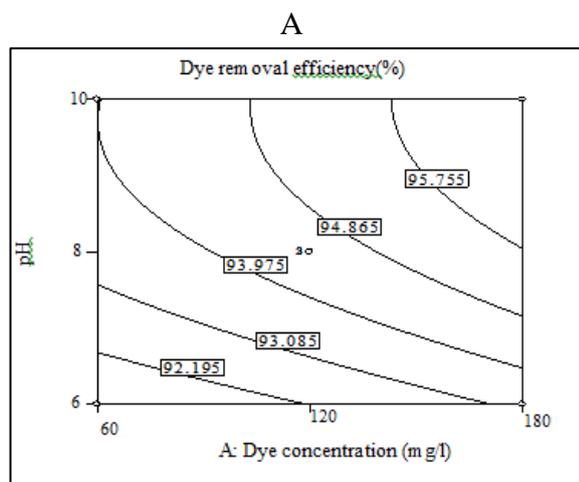


Fig. 2- Contour plots of the DB86 removal efficiency; (a): the effect of dye concentrations and pH on the removal efficiency of DB86 at constant pressure. (b): the effect of pressure and dye concentration on the removal efficiency at constant pH (c): the effect of pH and pressure on the removal efficiency at constant dye concentration.

Fig. 3- Contour plots of the DB56 removal efficiency; (a): the effect of dye concentrations and pH on the removal efficiency of DB56 at constant pressure. (b): the effect of P and dye concentration on the removal efficiency at constant pH (c): the effect of pH and pressure on the removal efficiency at constant dye concentration.

The optimum conditions for dye removal from contaminated water based on Box-Behnken method, are estimated at the maximum efficiency of 98.5% at the concentration levels about 180 mg/l of dye, pH of 10 and pressure of 0.84 MPa for DB86, also are estimated at the maximum efficiency of 94.83% at the concentration levels of 178 mg/l of dye, pH of 10 and pressure about 0.85 MPa for DB56, that have a reasonably good agreement with the experimental results. These results are obtained through design expert software, version 8.0.1 and the predictions are validated by the experimental results.

Discussion and Conclusion

The results of this study indicate that NF process by using a commercial spiral wound polyamide nanofilter (TFC) has an effective efficiency in dye removal from contaminated water sources. The factors pH and dye concentration have the most significant effect on the response of DB86 and DB56, respectively. The results indicated that with an increase in pH and dye concentration, the removal efficiency of both contaminants was increased. Also, data showed that the operating pressure effect under the optimum conditions can have a significant impact on the dye removal efficiency. The design of experiments 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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حذف رنگ‌های نساجی از محلول‌های آبی با استفاده از فرآیند نانوفیلتراسیون

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چکیده

هدف از پژوهش حاضر بررسی عملکرد غشاهای نانوفیلتراسیون در حذف رنگ‌های یونی (دایرکت آبی ۸۶) و غیر یونی (دیسپرس آبی ۵۶) صنایع نساجی از آب‌های آلوده است. در این پژوهش، با توجه به نمونه فاضلاب‌های واقعی، تأثیر غلظت خوراک (در محدوده ۶۰ تا ۱۸۰ میلی گرم بر لیتر)، فشار (۵۰۰ تا ۱۱۰۰ کیلو پاسکال) و اسیدیته (۶ تا ۱۰) مطالعه شد. آزمایش‌ها در یک سامانه آزمایشگاهی و با استفاده از غشای ماریچی تجاری ساخت کشور کره و از جنس پلی آمید (TFC) انجام شد. همچنین، به منظور بررسی تأثیر شاخص‌های یاد شده از طراحی آزمایش‌ها به کمک روش سطح پاسخ استفاده شد. نتایج نشان داد که با توجه به ساختار شیمیایی آلاینده، بار الکتریکی، غلظت رنگ و اسیدیته فاضلاب، راندمان حذف آلودگی‌ها می‌تواند به حدود ۹۶ و ۹۲ درصد به ترتیب برای رنگ دایرکت و دیسپرس افزایش یابد. همچنین، با توجه به ساختار غشا و ماهیت آلودگی‌ها، فشار بهینه عملیاتی حدود ۸۰۰ کیلو پاسکال به دست آمد. ماکزیمم بازده حذف توسط روش سطح پاسخ در شرایط بهینه فرآیندی حدود ۹۶ درصد پیش‌بینی شد و با داده‌های تجربی انطباق خوبی داشت.

واژه‌های کلیدی: نساجی، فاضلاب، استفاده مجدد، نانوفیلتراسیون، رنگ

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