International Journal of Engineering

Journal Homepage: www.ije.ir

Influence of Operating Variables on Performance of Nanofiltration Membrane for Dye Removal from Synthetic Wastewater Using Response Surface Methodology

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PAPER INFO

Paper history:
Received 08 July 2016
Received in revised form 26 October 2016
Accepted 11 November 2016

Keywords:
Dye Removal
Textile Wastewater
Direct Red 16
Nanofiltration Membrane
RSM

ABSTRACT

The textile industry is a water intensive industry that produces a large amount of highly colored wastewater that must be properly treated before disposal or reuse. In the present study, to verify the possibility of reusing textile wastewater with nanofilteration (NF), an attempt was made to treat synthetic reactive dye aqueous solution by commercial nanofiltration membrane. Experiments were conducted based on a central composite face-centered design and analyzed using response surface methodology. Dye concentration (10-50 mg/l), operating pressure (3–6 bar), and initial pH of the dye solution (3–9) were selected as the operating variables to analyze, optimize and model the process. The results showed that alkaline and acidic conditions led to considerable increase in dye rejection and permeation flux that high quality of water could be recovered. This study clearly showed that response surface methodology was one of the suitable methods to optimize the operating conditions and maximize the dye removal. Also, the commercial NF membranes could be efficiently applied in the dye removal process in a one-stage process.

***doi**: 10.5829/idosi.ije.2016.29.12c.03*

1. INTRODUCTION

Since 1856, when the first synthetic dye was reported, the application of dyes in industries has increased significantly. There are more than 10,000 dyes available commercially and about 7×10^5 metric tons of dyestuffs are produced annually [1]. Therefore, the effluents from industry included dye component containing high concentration of inorganic and organic chemicals and are identified by residual COD and strong color [2, 3].

There are many dye categories such as azo, diazo, anthroquinone, triphenylmethane, phetalocyanine that are recalcitrant to treatment [4]. Among these, azo dyes have been used widely as coloring agents in the textile, paint, ink and plastic industries that large outturn of them remain in the effluent after the completion of dyeing process [5]. Also, azo dyes are stable compounds that are difficult to decompose by common treatment in a biological treating station [6].

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Considerable amounts of water are used in the textile industry during dyeing and finishing operations and colored wastewater from this industry are a major source of environmental contamination [7]. Also, more growing of dyestuff manufacturing industries during past decades has caused immense increase in the volume and complexity of the wastewater discharged to the environment. Therefore, the treatment of colored wastewater is important from two aspects [8].

First, from the environmental point of view, the dye component in colored wastewater may be toxic to ecosystem. In addition, the presence of dyes or their intermediates in water resources can cause many different human health disorders [9]. Even if these components in effluent of textile industries are non-toxic, such wastewater obstructs light penetration and, therefore leads to decreasing photosynthesis in aquatic plants and raising the chemical oxygen demand (COD) [10]. Therefore, stringent environmental legislation has been imposed on industries in order to treat their waste effluents before being released into the recipient streams [11].

Please cite this article as: H M. Moradi, A. A. Zinatizadeh, S. Zinadini, Influence of Operating Variables on Performance of Nanofiltration Membrane for Dye Removal from Synthetic Wastewater Using Response Surface Methodology, International Journal of Engineering (IJE), TRANSACTIONSC: Aspetcs Vol. 29, No. 12, (December 2016) 1650-1658

Second, water shortage is raising global concern. As textile industries consume large volumes of water, reuse of this kind of wastewater is an urgent consideration for the industry sustainability in terms of water demand [12]. Till this date, various processes such as biodegradation, chemical degradation, adsorption, coagulation, photocatalytic degradation have been examined for treatment of the colored wastewaters [13-17]. Variety of adsorbents have been used for adsorption processes and in most cases they are not cost effective [18-20]. One of the effective process decolorizing particulate dyes is coagulation process which requires chemicals [21-24]. As the most of the dye compounds are resistance to biological degradation, the conventional biological process is not efficient to degrade the compounds [25]. Ozonation and chemical oxidation using chlorine are the effective methods for destruction of dye component, however involving high oxidant requirements [18-24]. Due to high stability of the dye compounds to light, the photochemical degradation is slow.

Membrane separation technologies have shown to be a promising practical method for dye removal from colored wastewaters [18-24]. Nanofiltration (NF) membrane is the most useful membrane for the complete treatment of the colored wastewater [11, 26-28]. There are many studies on textile dye bath wastewater treatment with nanofiltration membranes. Koyuncu and coworkers [29] used nanofiltration membranes to recycle the reactive black 5 (RB5), reactive orange 16 (R016) and reactive blue 19 (RB19). They investigated the effects of dye concentration, cross-flow velocity and pressure on the membrane performance in terms of the permeate flux and color removal. By increasing pressure, the permeate flux was increased and the permeate flux was decreased with an increase in dye concentration. In another work [30], synthetic PMIA nanofiltration membrane was examined for rejection of some of the synthetic dyes. Also, according to results of Koyuncua et al. [31], efficacy of cross-flow velocity on permeate flux was more noticeable at low concentration of sodium chloride. Alkaline conditions increased hydrophobicity of dye compound and lowered the permeate flux.

In recent years, many scientists have tried to improve morphology of the membrane and to analyze and optimize the filtration performance using statistical approaches [32, 33]. Before, the reported NF papers dealt with conventional and classical methods of experimentation that one of the parameters is varied maintaining the others constant at different levels. The conventional methods usually involve many experimental runs, which are time consuming, ignore interactions effects between the considered parameters of the process and lead to a low efficiency in optimization issues. Response surface methodology (RSM) has been employed to determine optimum operating conditions. By applying RSM, the limitations of the classical method can be avoided. This effective method has been used successfully in different areas of membrane technology [32-39].

The aim of our study was to treatment of synthetic colored wastewater containing direct red 16 via commercial nanofiltration membrane. The main emphasize in the current work was to study the direct and interactive effects of the influential factors on the membrane performance which is not much dealt with in the literature. A general factorial design was used for designing the experiments with three variables (initial pH, dye concentration, and operating pressure) and model the variation trends of two significant responses (dye rejection and flux permeation) using response surface methodology (RSM).

2. MATERIALS AND METHODS

2. 1. Materials All chemicals used in the experiments were of reagent grade. The Direct red 16, as an azo dye, $C_{26}H_{17}N_5Na_2O_8S_2$ (MW= 637.26) with purity of 99% was purchased from Alvan Sabet Co., Iran and used to prepare synthetic colored wastewater. The hydrophilic polyvinylidene fluoride (PVDF) microfiltration membrane with thickness of 125 µm and nominal pore size of 0.22 µm was obtained from Millipore Corporation. De-ionized water was used throughout this study.

2. 2. Apparatus The whole experiments in our study were carried out in a stainless steel dead-end cell (125 ml volume) with a membrane surface area of 12.56 cm^2 that was connected to a nitrogen gas line. To force the liquid through the membrane, pressurized nitrogen gas was used. The dye solution was stirred at a rate of 400 rpm to diminishing of concentration polarization effect. Each membrane was initially pressurized at 6 bar for 30 min to get a steady permeate flow, then the pressure was lowered to the operating pressure. Figure 1 shows schematic of the system used for filtration tests.

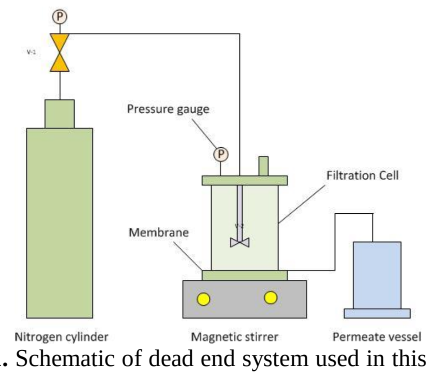


Figure 1. Schematic of dead end system used in this study for filtration test

TABLE 1. Experimental range and levels of the independent variables

Type of variables	Name of variables	Range and level		
		-1	0	+1
Numerical	A- Initial pH	3	6	9
	B- Dye concentration (ppm)	10	30	50
	C- Pressure (bar)	3	4.5	6

2. 3. Permeation and Rejection Tests To evaluate the nanofiltration performance for treating of synthetic wastewater, in each run, the flux of pure water and colored wastewater was calculated using the following equation:

$$J = \frac{m}{A\Delta t} \quad (1)$$

where, m (kg) is the weight of the permeates collected, A (m^2) the membrane area and Δt (h) the permeation time. Permeates were collected over a given period and weighed. The experiments were carried out at 25 ± 1 ^{o}C. The pure water flux (PWF), $J_{w,1}$, and flux of was initially measured and this value was used as a reference for the membrane permeability. In order to investigate the fouling, after filtration of colored wastewater during 60 min, the membranes were washed with distilled water for 15 min and the water flux of the cleaned membranes $J_{w,2}$ (kg/m^2h) was measured again. Finally, the flux recovery ratio (FRR) was calculated as follows:

$$\text{FRR}\ (\%) = \left(\frac{J_{w,2}}{J_{w,1}}\right) \times 100 \quad (2)$$

Also, the rejection (R) at any runs in the process is defined as follow:

$$\text{R}\ (\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100 \quad (3)$$

where, C_P the concentration of Direct red of permeate and C_f the dye concentration.

2. 4. Experimental Design and Mathematical Model In this research, the statistical method of factorial design of experiment was applied to describe the interactive effects of the variables studied on the process and eliminate systematic errors with an estimate of the experimental error and minimizes the number of experiments. Response surface method (RSM) designs as a factorial design help us quantify the relationships between one or more measured responses and the vital input factors and the central composite design (CCD) as the most popular RSM design was used in this study.

In order to describe the interactive effects of dye concentration, pH and pressure as a driving force on membrane, 20 experiments were conducted as dye concentration varied from 10 to 50 mg/l (these values are within the range of typical concentration in textile wastewaters [40]), initial pH varied from 3 to 9 and pressure varied from 3 to 6 bar. The range and levels of the variables in coded form and actual units are given in Table 1.

The flux (kg/m^2h) and rejection (%) were measured or calculated as response. The experimental conditions and results obtained are shown in Tables 2.

After conducting the experiments, the coefficients of the polynomial model were calculated based on Khuri and Cornell's equation (Equation (4)) [41]:

$$Y = \beta_0 + \beta_i X_i + \beta_j X_j + \beta_{ii} X_i^2 + \beta_{jj} X_j^2 + \beta_{ij} X_i X_j + \ldots \quad (4)$$

where, i and j are the linear and quadratic coefficients, respectively, and β is the regression coefficient. The model terms were selected or rejected based on the probability (P) value with 95% confidence level. The results were completely analyzed using the analysis of variance (ANOVA) by Design Expert software (Stat-Ease Inc, version 7.0.0). 3D graphs and contour plots were obtained based on the effect of the levels of the two factors and used to show of the simultaneous interaction of the two factors on the responses. The optimal region was also explored in the overlay plot based on the optimization criteria.

3. RESULTS AND DISCUSSION

Three process independent variables for treating of colored wastewater include initial pH, dye concentration of Direct red 16 (mg/l) and pressure (bar) were chosen in the experimental design used for response surface modeling. Subsequently, the adequacy of the model was tested by analysis of variance (ANOVA) and additional experiments. The optimum conditions of the operating factors could also be determined using the response models. The experimental conditions and results are given in Table 2.

3. 1. ANOVA for the Factors Studied For both flux and rejection, the quadratic model was selected as suggested by the software. The final empirical models in terms of coded factors after eliminating the insignificant terms for flux and rejection are shown in Table 3. The model quality developed was assessed using the correlation coefficient value. Lack of fit, R^2, and adjusted R^2 are some of the statistic parameters presented in the ANOVA results. Lack of fit was used to determine whether the constructed model was adequate to describe the observed data. Accordingly, the insignificant lack of fit is desired. The model had sufficient potential for explaining response variation. In general, R^2 and adjusted R^2 indicated the model suitability to represent the real relationship among the selected factors and the percentage of the variability of the optimization parameter.

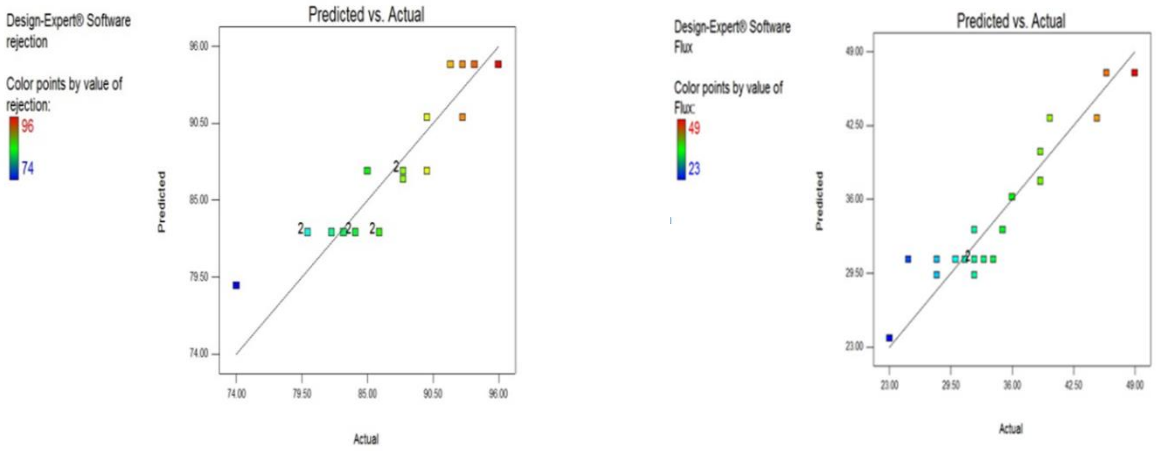
From the obtained equation for flux, the variables of pH and pressure have significant effect on response. The significant ranking in this study for permeation flux was (A): first order effect of pH, C: first order effect of pressure, and A^2: quadratic effect of pressure. It is obvious that there was no major interaction between the factors (AC). The results indicated that the increasing effect of initial pH (A) was more than the other variable on response. Also, the equation for prediction rejection illustrated that the increment effect of A^2 was more than decreasing effect of C. As shown in Table 3, the empirical model of permeation flux showed good validity and reliability as shown by the value of R^2 (0.89) and Adj R^2 (0.873) which were reasonable close to 1. Also, for rejection prediction, these values were 0.83 and 0.81 for R^2 and Adj R^2, respectively.

TABLE 2. Experimental conditions and results obtained

Run	Variables			Responses	
	Factor 1 A: pH	Factor 2 B: Dye concentration (ppm)	Factor 3 C:Pressure (bar)	Response 1 Flux (kg/m2h)	Response 2 Rejection %
1	6	30	4.5	32	80
2	6	30	4.5	28	84
3	9	50	6	40	88
4	9	30	4.5	36	90
5	9	10	6	45	85
6	6	30	4.5	31	83
7	3	50	6	46	90
8	9	50	3	28	93
9	6	50	4.5	33	86
10	6	30	4.5	34	80
11	6	30	4.5	30	82
12	9	10	3	32	92
13	6	30	3	23	88
14	6	30	4.5	25	86
15	3	10	3	35	94
16	6	10	4.5	32	84
17	3	30	4.5	39	93
18	3	10	6	49	88
19	6	30	6	39	74
20	3	50	3	32	96

TABLE 3. ANOVA for the regression equations obtained for the flux and rejection.

Response	Modified equations with significant terms	Probability	R^2	Adj.R^2	Adeq precision	S.D	CV	PRESS	Probability for lack of fit
Flux	$+30.70-2.00A+6.90C+7.50A^2$	< 0.0001	0.89	0.873	21.31	2.44	7.10	144.84	0.90
Rejection	$+82.70-3.80C+8.20A^2$	< 0.0001	0.83	0.81	16.94	2.41	2.77	139.13	0.50



(a) (b)

Figure 2. Predicted vs. experimental data for responses (a) flux and (b) rejection

Figure 2 represents the predicted values versus the actual date for the responses studied. From the figure, the predicted values obtained were in very good agreement with the experimental data, indicating good correlation between the filtration variables and the two responses.

3. 2. The Effects of Factors on Membrane Performance In this part, the performance of commercial nanofiltration membranes (NF-CSM) for the removal of color (Direct red 16) from the synthetic wastewater by variation of different parameter is discussed. The contour and surface plots of the response models are beneficial in understanding both the main and the interaction effects of the variables. These plots can be obtained by computations using the developed response models in Design Expert software.

3. 2. 1. Effects of Factors on Permeate Flux All the data and analysis from RSM are presented graphically in order to get a clear view of the interactions between the factors and responses. The regression equation presented for the response in Table 3 illustrates that dye concentration did not show any significant effect on the response. Figure 3 shows the 3D response surfaces for membrane permeability as a function of initial pH and pressure. Driving force for the permeate flux was increase by increasing the operation pressure. A maximum flux about 45 Kg/m^2.h was obtained at acidic pH and highest pressure (6 bar).

pH is very important factor for the flux permeation of the NF membrane in colored wastewater treatment. The pH values of 3, 6 and 9 were applied to the batch runs. From the Figure 3, the lowest flux values were obtained at the neutral pH conditions for the given pressures. At acidic and alkaline conditions, the repulsion between membrane surface and dye molecules was increased that led to reduction of membrane fouling and increasing of permeability. In the present study, the results showed that the transport of the direct red 16 through the nanofiltration membrane is controlled by adsorption interaction and diffusivity. The interactive effect of AC on the response is represented in Figure 3c. The parallel lines in the graph implies on no interaction between the variables.

3. 2. 2. Effects of Factors on Dye Rejection Figures 4a and b show the three-dimensional and contour plots that represents the effect of the two factors on color rejection. From the figure, the NF rejection factor increases to 92 % with the acidic and alkaline feed condition (pH of 3 and 9) and also at low pressure (3 bar). The NF rejection decreased when the pressure increased to 6 bar, and also initial pH=6. It is reported in the literature that by increasing of pressure, driving force behind the membrane surface will be increased and lead to diffusion of direct red molecules through membrane and therefore, reduction of rejection [42]. The initial pH value of feed showed to be an important parameter that affects the nanofiltration performance significantly. This could be explained by electrostatic interaction between the membrane surface and the dye molecules under different pH values. When thin film nanocomposite nanofiltration membrane was placed in contact with feed solution at acidic or alkaline conditions, the association and dissociation of functional group on the surface NF membrane leads to the formation of charges. Thus, the charge generated can be related to the ionization of functional group existing on the membrane surface, which strongly depends on pH. At acidic and alkaline conditions (pH=3 and 9), a repulsion strength with dye molecules appears and leads to increasing of dye rejection [43]. From Figure 4c, similar to membrane permeability, A-C did not show any interaction.

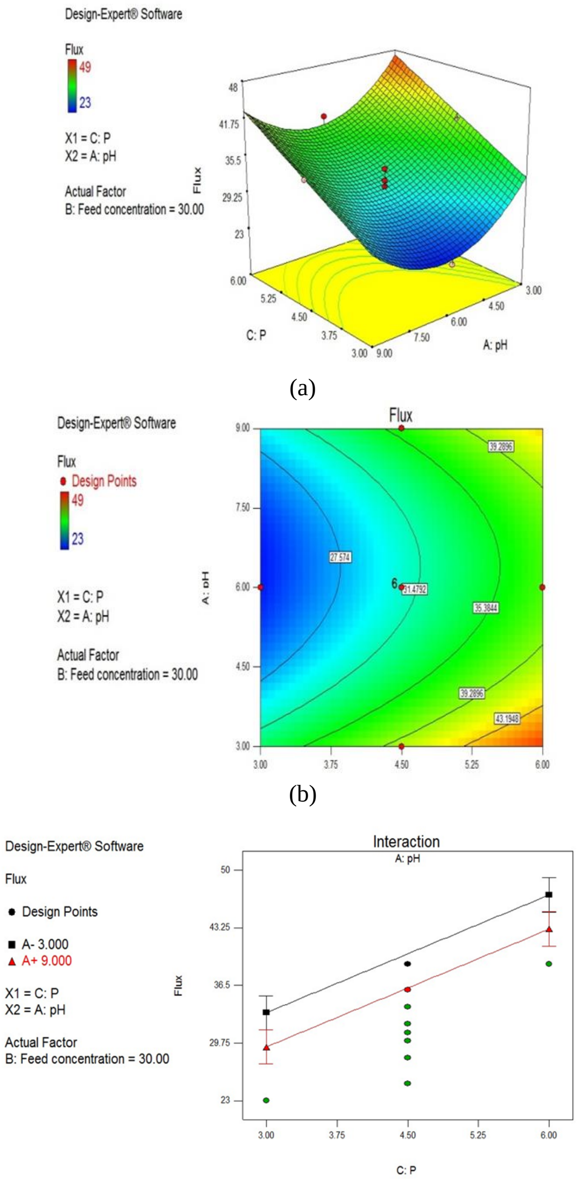


Figure 3. (a) 3D plot, (b) counter plot and (c) AC interaction for membrane permeability

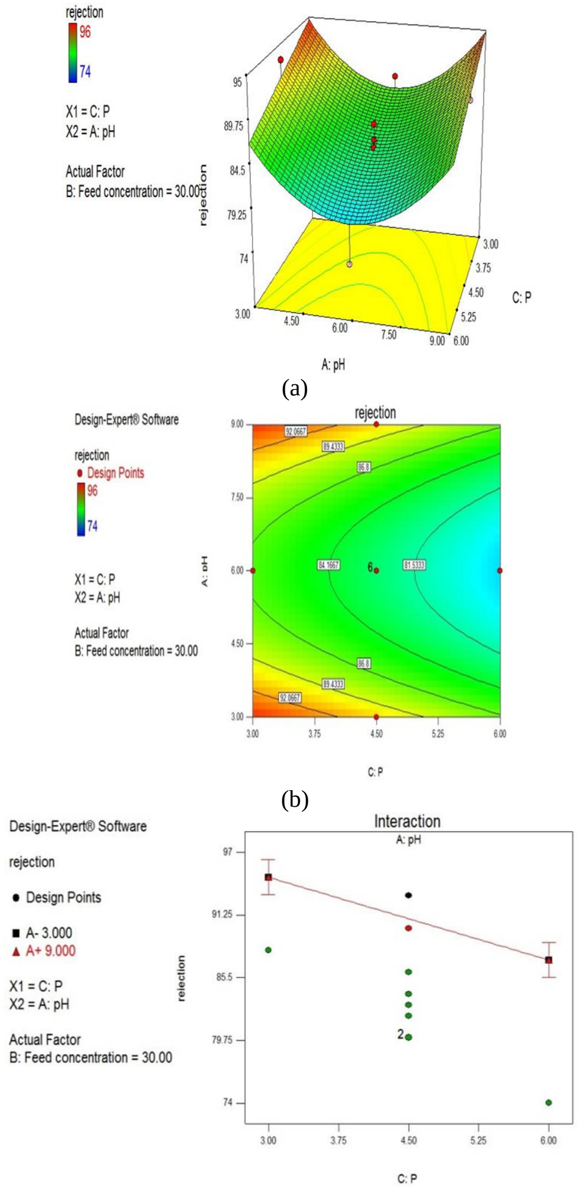


Figure 4. (a) 3D plot, (b) counter plot and (c) AC interaction for dye rejection

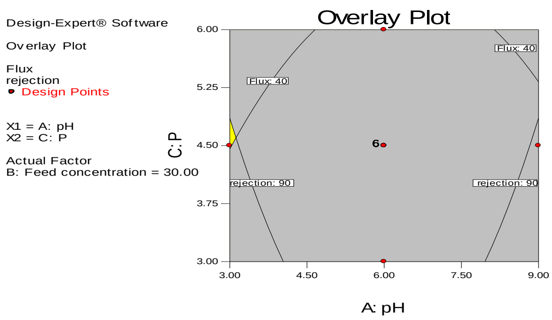
3. 3. Process Optimization The graphical optimization results allow visual inspection to choose the optimum values for the effective variables. The optimum conditions can visually be explored by overlaying response contours on a contour plot. The graphical optimization of the separation process is shown in Figures 5a-c, which displays the area of feasible response values (shaded portion) in three separation conditions according to Table 4 as the optimization criteria. The shaded area on the overlay plots in these figures is the regions that meet the proposed criteria which are flux permeation (kg/m^{2}h) and dye rejection as presented in Table 4.

From the Figure 5a, the optimum condition for membrane separation is acidic pH and the pressure about 4-5 bar for meeting the criteria condition of No 1 in Table 4. Also, by considering lower quality and flux in separation (No 2 and 3), as shown in Table 4, the optimum region will be increased as depicted in Figures 5b and c.

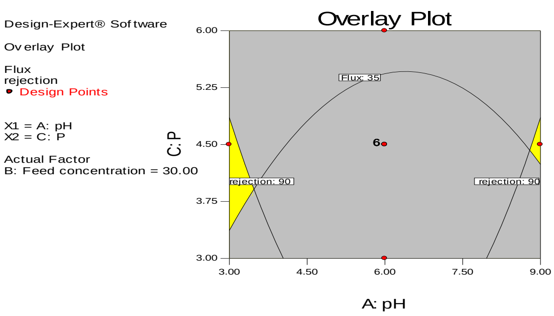
3. 4. Long Term Dye Removal Filteration Performance At the final stage, in order to determine the reproducibility of the membrane performance and the durability of antifouling property, five cycles of dye removal experiments were performed at the optimum conditions of pH= 3, P= 3.5 bar and C= 30 mg/l for the unfilled commercial nanofiltration membrane. Figure 6 demonstrates that the FRR values in those five cycles were 93.5%, 89.3%, 89.2%, 88.9% and 89.1% related to first water flux for commercial membrane.

TABLE 4. The criteria for process optimization

	Response	Limits	Unit
No. 1	Flux	>40	Kg/m^{2}h
	Rejection	>90	%
No.2	Flux	>35	Kg/m^{2}h
	Rejection	>90	%
No.3	Flux	>35	Kg/m^{2}h
	Rejection	>85	%



(a)



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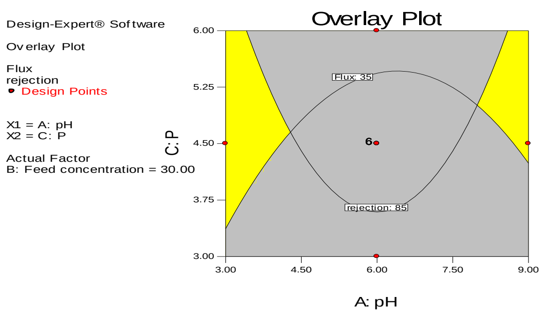

(c)

Figure 5. Overlay plots for the process optimization at different criteria as presented in Table 4; (a) No 1, (b), No 2 and (c) No. 3

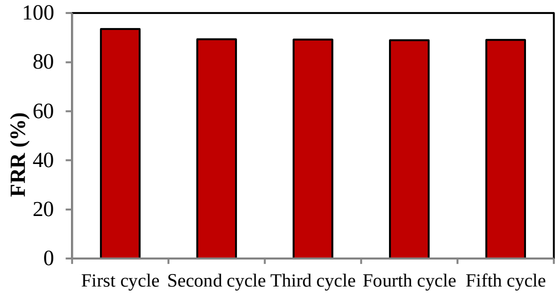

Figure 6. Reproducible characteristic of commercial membrane during five dye removal filtrations at optimum conditions (pH= 3, P= 3.5 bar and C= 30 mg/l)

Therefore, the dominant phenomenon in total fouling was reversible fouling as hydraulic cleaning maintained high efficiency after five cycles.

4. CONCLUSION

Nano filtration technology could be a promising and reliable method for complete treatment of colored industrial wastewater. Response surface methodology was an effective tool to model and optimize the nanofiltration process removing dye from a synthetic wastewater with minimum experiments. The most important variables were found to be initial pH and operating pressure. The conditions with acidic/alkaline pH and a pressure higher than 4 bar were found as optimum condition in terms of flux (>35 Kg/m^2.h) and rejection (>90 %). As a conclusion, no adverse impact was found on the membrane performance during long term filtration under acidic conditions.

5. ACKNOWLEDGEMENT

The authors would like to acknowledge Razi University for the full financial support provided for this research work.

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Influence of Operating Variables on Performance of Nanofiltration Membrane for Dye Removal from Synthetic Wastewater Using Response Surface Methodology

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PAPER INFO

Paper history:
Received 08 July 2016
Received in revised form 26 October 2016
Accepted 11 November 2016

Keywords:
Dye Removal
Textile Wastewater
Direct Red 16
Nanofiltration Membrane
RSM

چکیده

صنعت نساجی یک صنعت پرمصرف در آب است که مقدار زیادی فاضلاب شدیدا" رنگی تولید می‌کند که قبل از دفع یا استفاده مجدد می‌بایست به طور صحیح تصفیه شود. در مطالعه حاضر، به منظور بررسی امکان استفاده مجدد از فاضلاب نساجی با استفاده از نانوفیلتراسیون، تصفیه محلول رنگی آبکی توسط غشاء نانوفیلتراسیون تجاری انجام شد. طراحی آزمایشات بر اساس طرح مرکزی مرکب انجام و با استفاده از روش پاسخ سطحی تحلیل گردید. غلظت رنگ (۱۰-۵۰ میلی گرم بر لیتر)، فشار عملیاتی (۳-۵ بار) و pH اولیه محلول رنگی (3-9) به عنوان متغیرهای عملیاتی به منظور تحلیل، مدل سازی و بهینه سازی فرآیند انتخاب شدند. نتایج نشان داد که شرایط اسیدی و قلیایی سبب افزایش در حذف رنگ و شار عبوری گردید. این مطالعه نشان داد که روش پاسخ سطحی یک روش مناسب برای بهینه سازی شرایط راهبری بود. همچنین، غشاءهای تجاری نانوفیلتراسیون می‌تواند به طور کارآمد حذف رنگ در یک فرآیند یک مرحله‌ای را انجام دهد.

doi*: 10.5829/idosi.ije.2016.29.12c.03*