



Optimization of conditions in ultrafiltration treatment of produced water by polymeric membrane using Taguchi approach

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ABSTRACT

In this study, the ultrafiltration of produced water was studied using a two-stage ultrafiltration process. In the first stage, the influences of operating parameters, including transmembrane pressure, temperature, and cross-flow velocity on the amount of flux decline caused by membrane fouling, were investigated using a polymeric membrane. In order to design the experiments and optimize the experimental results, the Taguchi method was applied. L_9 (3^3) orthogonal array for experimental planning and the smaller-the-better response category was selected to obtain optimum conditions because the lowest flux decline was our aim. Analysis of variance was used to determine the most important parameters affecting the flux decline caused by membrane fouling. The optimum conditions were found at the first level of transmembrane pressure (1.5 bar), second level of temperature (40°C), and third level of cross-flow velocity (1 m/s). In the second stage, performance of ultrafiltration system by the polymeric membrane was investigated under the optimum conditions and 99% oil and grease, 100% TSS, 99% Turbidity, and 68% TOC removal was obtained. Also, the size of particles in feed decreased from the range of 200–800 nm to 1.5–3 nm.

Keywords: Polymeric membrane; Produced water; Taguchi; Ultrafiltration

1. Introduction

Petrochemical processing, petroleum purifying, natural gas, and oil production always generate large amounts of produced water, waste water, contents of oil, salts, heavy metals, and other organic components [1]. Their toxic nature and effects on the environment

necessitate a treatment before they are released, for which there are several methods such as biological treatment, adsorption, dissolved air flotation, etc. [2]. Traditional purification methods require high energy consumption or the application of many chemical materials, which decreases the efficiency and increases the costs. There are four filtration methods: microfiltration [3,4], ultrafiltration (UF) [4], nanofiltration, and reverse osmosis [5]. Membrane filtration processes are

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used in different produced water treatments because of their high removal efficiency, easy operation, and lower costs [6]. However, they have disadvantages such as fouling and concentration polarization causing flux decline, which can be controlled by adjusting the operating conditions like temperature, transmembrane pressure, pH, and velocity [7]. Reduction of concentration polarization and membrane fouling has been the main issue of many studies. Several methods have been developed in various applications of UF: the use of higher cross-flow velocities (CFVs), pulsed flow, use of modified membranes, feed oil concentration optimization, use of static turbulence promoters, operation under uniform transmembrane pressure (TMP), use of optimum salt concentration, feed temperature optimization, vibrating membranes, and more recently proposed high shear rotary UF [8,9]. Researches show that there is still an enormous interest in this field. Good studies are available, although they are limited in terms of factors variability. Membrane filtration is seemingly a complex process, disregarding the interaction of factors which may result in erroneous conclusions. A reliable solution to this problem would be to consider all the possible combinations of the levels of factors: the solution that is called one factor at a time, in short OFAT. Still, using this method is time-consuming and expensive, since the number of experiments rises rapidly as the input variables increase. Hence, the alternative way is to utilize a design of experiment method from which a smaller number of experiments with similar results can be achieved [10,11].

Taguchi method is a statistical technique introduced by R.A. Fisher in England in the 1920s, which is used for optimization. Taguchi method aims at finding optimum process conditions with a minimized sensitivity to noises. It is a type of fractional factorial design which uses an orthogonal array to study the influence of factors with fewer numbers of experiments [6]. Taguchi method-based designed experiments provide systematic approach to meet optimum conditions [12]. Hesampour et al. [13] carried out 18 experiments on the UF of emulsified oil in water under different operating conditions including: pH, oil concentration, feed flow rate, temperature, and salt (NaCl and CaCl₂) concentrations on the permeate flux of a model oil wastewater using the Taguchi method. Each factor was considered at three levels. The optimum conditions were obtained when salt and oil concentrations were at their lowest levels (CaCl₂=0 g/L, NaCl=0 g/L, and 0.5 (% v/v)); whereas temperature, transmembrane pressure, and flow velocity were at their highest levels ($T=40^{\circ}\text{C}$, $\text{TMP}=3.5\text{ bar}$, and flow velocity=3.2 m/s). Rezvanpour et al. [2] studied the effects of membrane type, transmembrane pressure,

the content of oil in the feed, the flow velocity of the feed, and pH on the UF of an emulsion of kerosene in water. They found that membrane type is the most important factor affecting the UF to get the best flux, which at the optimum conditions of three bars, oil concentration of 3 (% v/v) and membrane type C30F was predicted as 108 L/(m²h). Using Taguchi method, Gonder et al. [6] aimed at estimating the best operational conditions in CIP, i.e. cleaning-in place, wastewater treatment from detergent industry by NF and proved that the neutral pH (7), first level of transmembrane pressure (12 bar) and low temperature (25°C), yielded the optimum conditions.

This paper describes a case study investigating the parameters affecting the permeate flux decline for produced water treatment by UF process using a polymeric membrane. Three factors, i.e. temperature, transmembrane pressure, and cross-flow velocity, were chosen for the experiments. The objective is to find a combination of levels that get the lowest flux decline. Optimum conditions are determined using Taguchi method by the signal-to-noise (S/N) ratio of experimental results. Analysis of variance (ANOVA) was used to determine the significance of the factors. Furthermore, a long-time experiment was done to remove oil and grease, total suspended solids (TSS), total organic components (TOC), and turbidity under optimum conditions using UF membrane to meet the expectations of one of the desalination units in Iran. Finally, as a demand for desalination unit, the size distribution of permeate must measure less than 100 nm in size of solutes.

2. Materials and methods

2.1. Membrane

A rectangular-sheet polymeric membrane (PAN350) with a surface area of 66.15 cm², purchased from Sepro Company (America), was used in this study. This membrane is highly hydrophilic. The characteristics of membrane are listed in Table 1.

Table 1
Characteristics of membrane used in experiments

Commercial name	PAN350
Material	Polyacrylonitrile (PAN)
MWCO (kDa)	20
Water flux (L/m ² h bar)	1,000
pH range	3–10
pH range (cleaning)	3–10
Max. temperature (°C)	100
Contact angle	44°

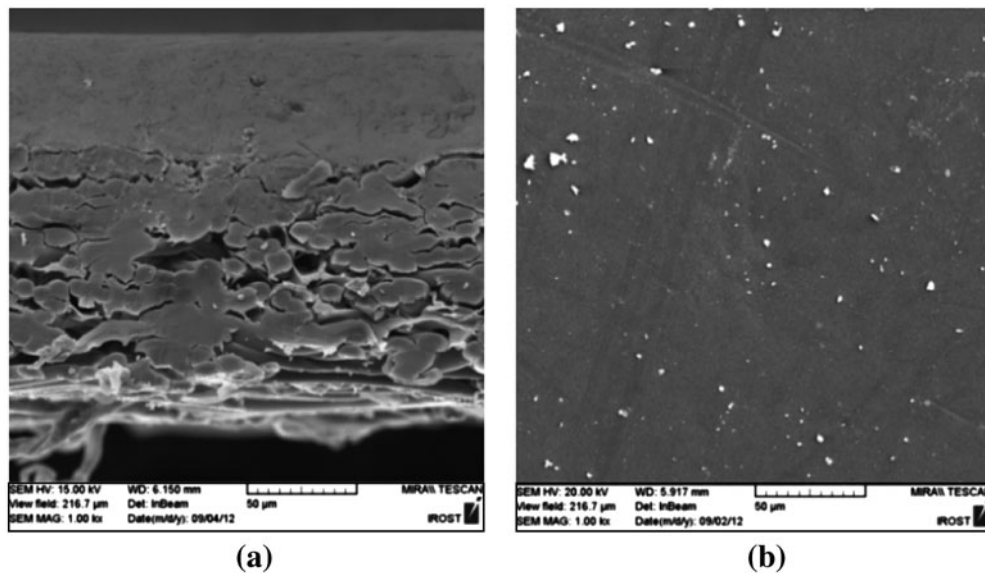


Fig. 1. SEM image of PAN350 as a fresh polymeric membrane, (a) cross section and (b) surface.

PAN350 is formed from two layers called top layer with 1- μm thickness, and sub-layer as support. These layers can be seen in Fig. 1(a). Surface of membrane before UF is shown in Fig. 1(b).

2.2. Process feed

The real produced water used in the experiments consisted of TDS (65,852 mg/L), Fe (0.49 mg/L), COD (440 mg/L as O_2), oil and grease (42 mg/L), TSS (108 mg/L), TOC (109 mg/L) with turbidity=90, and pH=8.5 had been generated at the outlet of skimmer unit in one of the desalination units operating in Iran. The range of size distribution of feed is 200–800 nm that suggests the presence of dissolved oils [14].

2.3. Filtration

Fig. 2 shows the diagram of filtration setup. The real produced water in the feed tank was pumped to a flat-sheet membrane module. The needed flow velocity and pressure were attained by controlling the input electromotor power and the back pressure valve after the membrane module. As temperature is one of the controlling factors, a cooling/heating system was employed to detect the required temperature. All experiments conducted in cross-flow operation were carried out in concentration mode of filtration (CMF) for 60 min. During the experiments, the weight of permeates were measured.

2.4. Measurements

The values of oil and grease, TSS, turbidity, and TOC of the feed and permeates were measured by special standard methods and devices which are listed in Table 2.

To measure the particle size distribution of emulsified oil droplets in the sample by light-scattering method, the LLS instrument (SEMA-633) was used. The measurement range of this instrument is from 0.4 to 10,000 nm.

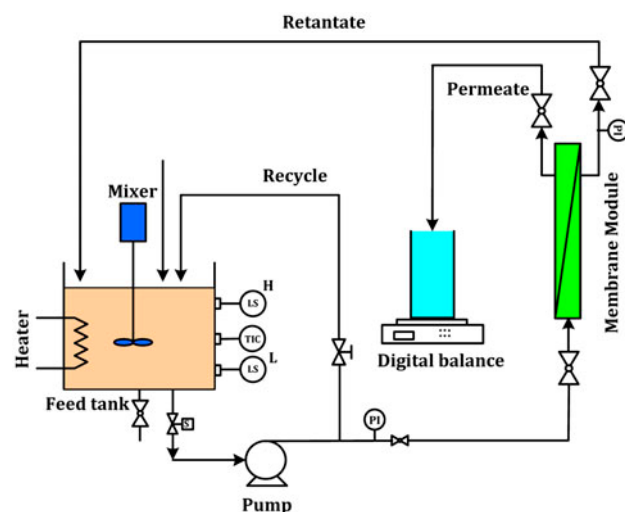


Fig. 2. Schematic diagram of the UF experimental set-up.

Table 2
Standard methods and devices used to measure properties of the samples

Parameter	Standard method	Device
Oil and grease	5,520 B	TOG/TPH analyzer, Infrical
TSS	2,540 D	–
Turbidity	1889 D	Turbidimeter, HACH, 2,100 A
TOC	5,310 C	TOC analyzer, Dohrmann, DC-190

2.5. Scanning electron microscope

The membrane fouling on the pores and the surface of membrane was observed with a scanning electron microscope (SEM). The scanning electron microscope was operated with maximum voltage of 30 kV (model: XL30, Philips company).

2.6. Experimental design based on Taguchi method

Taguchi method is based on several steps including planning the experimental matrix [13], conducting the experiments, using signal-to-noise (S/N) ratios to evaluate the results, analyzing variance, selecting the optimum levels of factors, and verifying the optimum process parameters through the verification step of the confidence interval [6].

The first step determines the number of factors [13]. In this study, three factors, i.e. temperature, TMP, and CFV, at three levels were chosen based on the literature [6,13]. Temperature, TMP, and CFV were fixed at 25, 40, and 55 °C, 1.5, 3 and 5 bar, 0.5, 0.75, and 1 m/s, respectively. Taguchi method employs fractional factorial experimental designs and orthogonal arrays to decrease the number of experiments. Using mentioned array, the number of experiments required to investigate the important effects can be reduced to 9; whereas full factorial experimentation requires $3^3 = 27$ experiments. $L_9 (3^3)$ orthogonal array of Taguchi design includes nine experiments for three factors at three levels. Suggested experimental plan for the $L_9 (3^3)$ is illustrated in Table 3 which has eight degrees of freedom (DOF) and each parameter has two DOF. If N is considered as the number of experiments and k_A is the number of levels of parameters, the total DOF is $N-1$, and DOF for each factor is k_A-1 .

In Taguchi method, “signal” and “noise” introduce the acceptable and unacceptable values for outputs, respectively, and their ratio (S/N) is used to transform the permeate flux to find the optimum conditions [13], which will be obtained by using the S/N ratio of the

Table 3
Orthogonal array by Taguchi method [15–17]

Trial no.	Factors		
	TMP	T	CFV
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

results of experimental data. The larger-the-better, the smaller-the-better, and nominal-the-better are three basic S/N ratios. For low flux decline, the “smaller-the-better” criteria were chosen. The performance characteristic was calculated using the following equation [15]:

$$\text{The smaller the better S/N} = -10 \log \left(\frac{1}{n} \sum_{i=1}^n Y_i^2 \right) \quad (1)$$

where n is the number of iteration for an experimental combinations and Y_i is the response (here, flux decline).

2.7. Analysis of variance

ANOVA was applied to determine the significance of the factors [6]. ANOVA’s results including: the sum of squares (SS), DOF, mean of square (MS), and error as well as two statistical parameters called F and P are gathered in Table 6. These values are calculated as follows [16,17]:

$$SS_A = \sum_{i=1}^{k_A} \left(\frac{A_i^2}{n_{A_i}} \right) - \frac{T^2}{N} \quad (2)$$

where k_A is the number of the levels of factor A, n_{A_i} is the number of all observations of factor A at level i , n_{A_i} is the sum of all observations of factor A at level i and T is the sum of all observations. Total SS is calculated using Eq. (3).

$$SS_T = \sum_{i=1}^{k_A} (Y_i^2) - \frac{T^2}{N} \quad (3)$$

where Y_i is the observation of i . SS of error can be calculated by the following equation:

$$SS_e = SS_T - (SS_A + SS_B + \dots) \quad (4)$$

MS is calculated by dividing the sum of squares by the degrees of freedom. F -ratio value is calculated as follows:

$$F_A = \frac{MS_A}{MS_e} \quad (5)$$

MS_e is the variance of error. Using F -ratios in an ANOVA is only useful for the qualitative calculation of factorial influences. Quantitative calculations are obtained using percentage contribution (%P) [6]. It is evaluated by dividing the source's net variation by SS_T , which is given as follows [16,17]:

$$P(\%) = \frac{SS_A - (DOF_A \times MS_e)}{SS_T} \times 100 \quad (6)$$

3. Results and discussions

3.1. Experimental results

The values of initial permeation flux (J_i), final permeation flux (J_f), TOC rejection, and turbidity rejection obtained from experiments are reported in Table 4. As it can be seen, the values of TOC and turbidity rejection are practically constant in the all experiments, therefore, just the values of permeation fluxes at the beginning and the end of the filtration were considered in order to find the optimum conditions by Taguchi approach.

3.2. Taguchi results

Fig. 3 shows the flux decline vs. time throughout the process of produced water UF for all experiments. As it is illustrated in Fig. 3, as well as Table 5, the highest point of flux decline (56%) occurred in trial 9, while the lowest one (28%) was in experiment 2. In Table 5, the values of initial fluxes (J_i) and final fluxes (J_f) of the permeates, the amounts of flux decline caused by membrane fouling, and the S/N ratios calculations are reported for each experiment. So as to specify the effect of each factor on flux decline, the S/N ratio must be calculated for each factor. In Taguchi method, S/N ratio is worked out just by averaging the S/N values in different levels of each factor. For instance, the mean S/N ratio values for TMP in the levels 1, 2, and 3 could be calculated by averaging the S/N ratios for the trials 1–3, 4–6, and 7–9, respectively.

Fig. 4 shows the averages of S/N ratio for each factor at three levels. As it can be seen, the slopes of the trends of different levels are not the same for the TMP and temperature factors. Therefore, they would have different influences on flux decline and membrane fouling. The flux decline during the experiments was less in the temperature range of 25–40°C than the one in 40–55°C. It means that the membrane fouling rose by increasing the temperature in the range of 40–55°C, which results in an overall decline of flux. On the other hand, researches have shown that an increase in temperature causes a rise in the amount of permeate flux [18,19]. This increase usually is explained through the viscosity of solvent, solvent diffusion coefficient in the membrane, and the extent of thermal expansion in the membrane substance. The increase of temperature causes a reduction in the viscosity of solvent and, hence, an increase in the solvent diffusion coefficient. Furthermore, the high

Table 4
Experimental results

Trial no.	J_i (L/m ² h)	J_f (L/m ² h)	TOC of permeate (mg/L)	TOC rejection (%)	Turbidity of permeate (NTU)	Turbidity rejection (%)
1	110	57	38	65	0.34	99.62
2	114	82	37	66	0.25	99.72
3	139	92	36	67	0.43	99.52
4	159	78	37	66	0.42	99.53
5	146	95	36	67	0.51	99.43
6	206	99	41	62	0.55	99.38
7	213	103	36	67	0.58	99.35
8	174	101	39	64	0.25	99.72
9	286	126	40	63	0.47	99.47

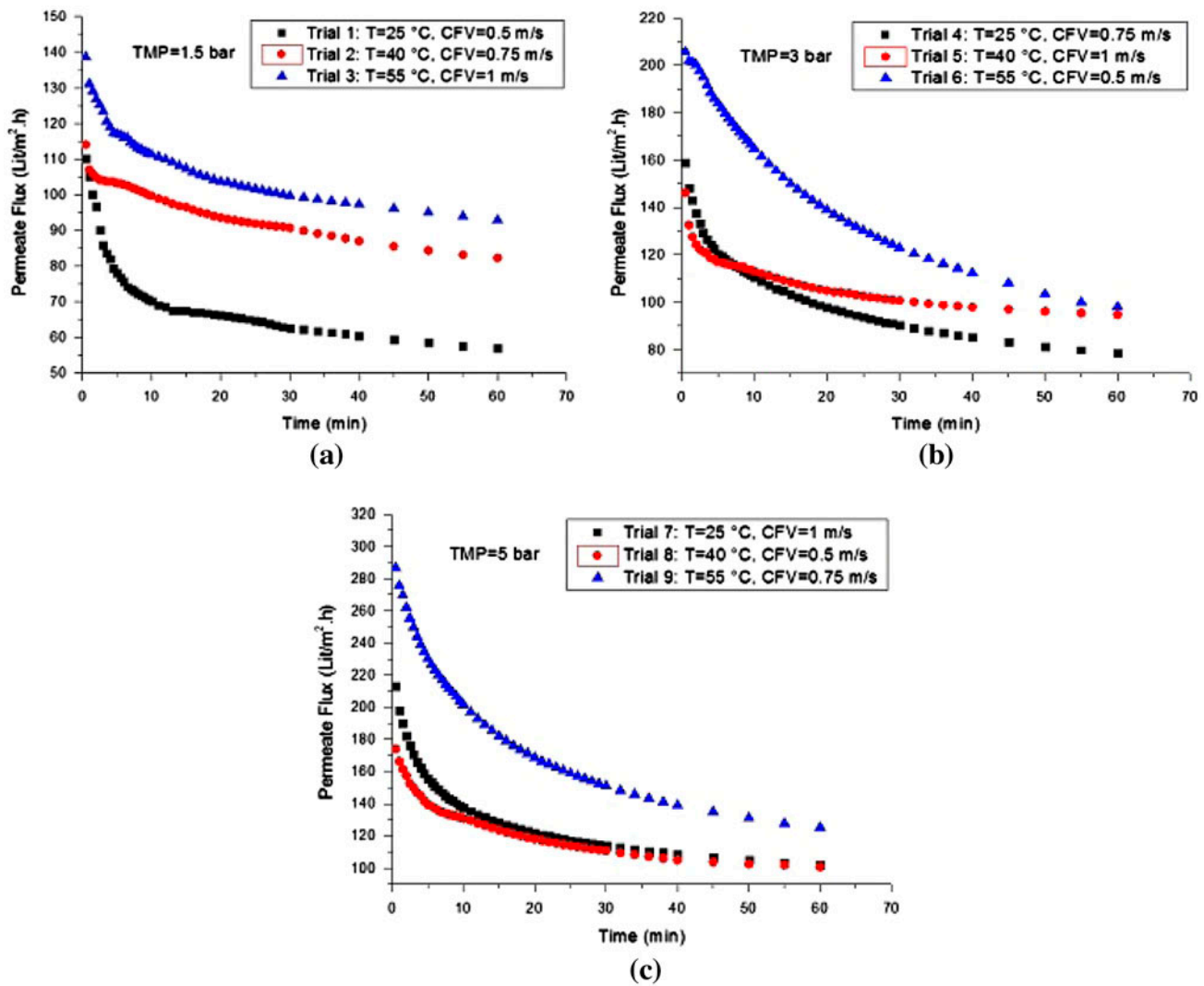


Fig. 3. Permeate flux variation vs. time for all experiments at (a) TMP = 1.5 bar, (b) TMP = 3 bar, (c) at TMP = 5 bar.

Table 5
Initial and final permeate fluxes, flux decline, and S/N ratio in experiments

Trial no.	Permeate flux (L/m ² .h)		Flux decline (%) $(J_i - J_f) / J_i \times 100$	S/N ratio
	J_i	J_f		
1	110	57	48	-33.6248
2	114	82	28	-28.9432
3	139	92	33	-30.3703
4	159	78	50	-33.9794
5	146	95	35	-30.8814
6	206	99	52	-34.3201
7	213	103	52	-34.3201
8	174	101	42	-32.4650
9	286	126	56	-34.9638

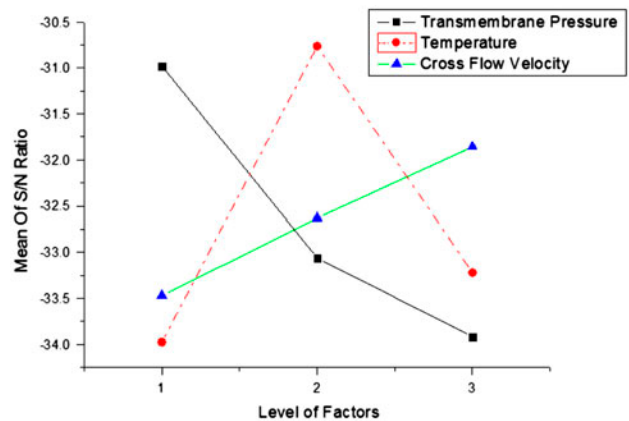


Fig. 4. Main effect curves for S/N ratios of flux decline for three factors at three levels.

temperature may cause the structure of membrane to expand facilitating the permeation of solutes [20,21]. As a result, in the range of 40–55°C, increase in the permeate flux decline can be due to the pore-plugging process as well as the membrane surface which allows the solute particles to pass at higher temperatures more easily. As it is shown in Fig. 4, the lowest level of flux decline occurred at 40°C.

In membrane processes, TMP is one of the most important factors in investigating the existence and kind of fouling phenomenon as well as the flux decline [6]. Fig. 4 shows that an increase in TMP caused a decrease in the ratio of S/N; hence, the lowest level of fouling occurred at the lowest TMP (1.5 bar). According to Darcy's law, the pressure difference at two membrane sides brings about an increase in flux, although the effects of fouling limit this increase [22,23]. The flux decline caused by fouling increased due to the rise in TMP. By this increase, the concentration polarization phenomenon occurred on membrane surface; thus, the concentration difference between two membrane sides increased [4,24]. Consequently, the diffusion driving force increased and more particles crossed the membrane and pore-plugging occurred by oil droplets that resisted the permeation flow [6]. This means that pore-plugging is more likely to occur at higher levels of TMP. Increasing TMP makes the sediments compacted on the surface of membrane (Fig. 5), and as a result, they block the membrane pores [22,23].

Likewise Fig. 4, illustrates that as the level of CFV increased, the S/N ratio also rose. Therefore, the lowest level of flux decline occurred at the highest velocity (1 m/s). At CFV of 0.5 m/s, because of the low level of turbulence, a cake layer was formed faster and, hence, it increased the flux decline. Conversely, as the CFV rose, because of more turbulence, the permeate flux increased and the level of flux decline decreased. Increasing CFV increases mass transfer coefficient in the concentration boundary layer and also increases the extent of mixing over the membrane surface [25]. At higher velocities, a part of created layer was detached from the membrane surface and returned to the liquid mass as a result of hydrodynamic effects of the flow. Accordingly, the thickness of formed layers is less at higher velocities, so there was a decrease in the level of fouling resistance and flux decline [22–28].

The optimum conditions for flux decline caused by fouling were chosen based on the levels which gave out the highest S/N ratios for the factors. As it is illustrated in Fig. 4, the combination of TMP = 1.5 bar, $T = 40^\circ\text{C}$, and CFV = 1 m/s is the optimum operating condition for the process of UF which minimizes the

amount of flux decline caused by membrane fouling. A more detailed look into Table 3 shows that optimum conditions are not employed in experiments carried out in this study.

3.3. ANOVA results

ANOVA was employed to see whether or not the controlling parameters of the process are statistically significant. Results of ANOVA are listed in Table 6. The values reported as errors arose from uncontrollable factors (noises), which should be less than 50%; otherwise, the results would not be reliable [6]. As it reported in Table 5, the value of estimated errors is 6% which is sufficiently far from the limit. This means that errors only had insignificant effects on the results.

F-ratio is a tool to demonstrate whether or not a parameter has a significant effect on the flux decline caused by fouling. When the F-ratio of a factor is at the highest value, it has the most effect on flux decline. F-ratio values were calculated based on the results of the experiments using Eq. (5). F-ratio has been employed in ANOVA only to facilitate qualitative measurement of contributing factorial effects. For quantitative calculations, percentage contribution (%P) was used which was obtained from the Eq. (6) [6].

The percentage contributions of all factors are shown in Fig. 6. As it is shown, temperature is the most effective factor followed by TMP and CFV.

3.4. Long-time experiment

So as to conduct the second stage which is to investigate the performance of PAN350 membrane under optimum conditions, the process of prolonged UF was performed on a feed for 8 h. The change in the permeate flux from membrane as a function of time is shown in Fig. 7. The flux decline is a result of pore-blocking, concentration polarization and cake-layer formation [29,30], which occurred in the first 2 h of the process; then, permeate flux from membrane

Table 6
ANOVA results for flux decline

Factors	DOF	SS	MS	%P	F-ratio
TMP	2	292.67	146.33	33.18	8.96
Temperature	2	378	189.00	43.93	11.57
CFV	2	82.67	41.33	16.62	2.53
Error	2	32.67	16.33	6.27	
Total	8	786.00		100	

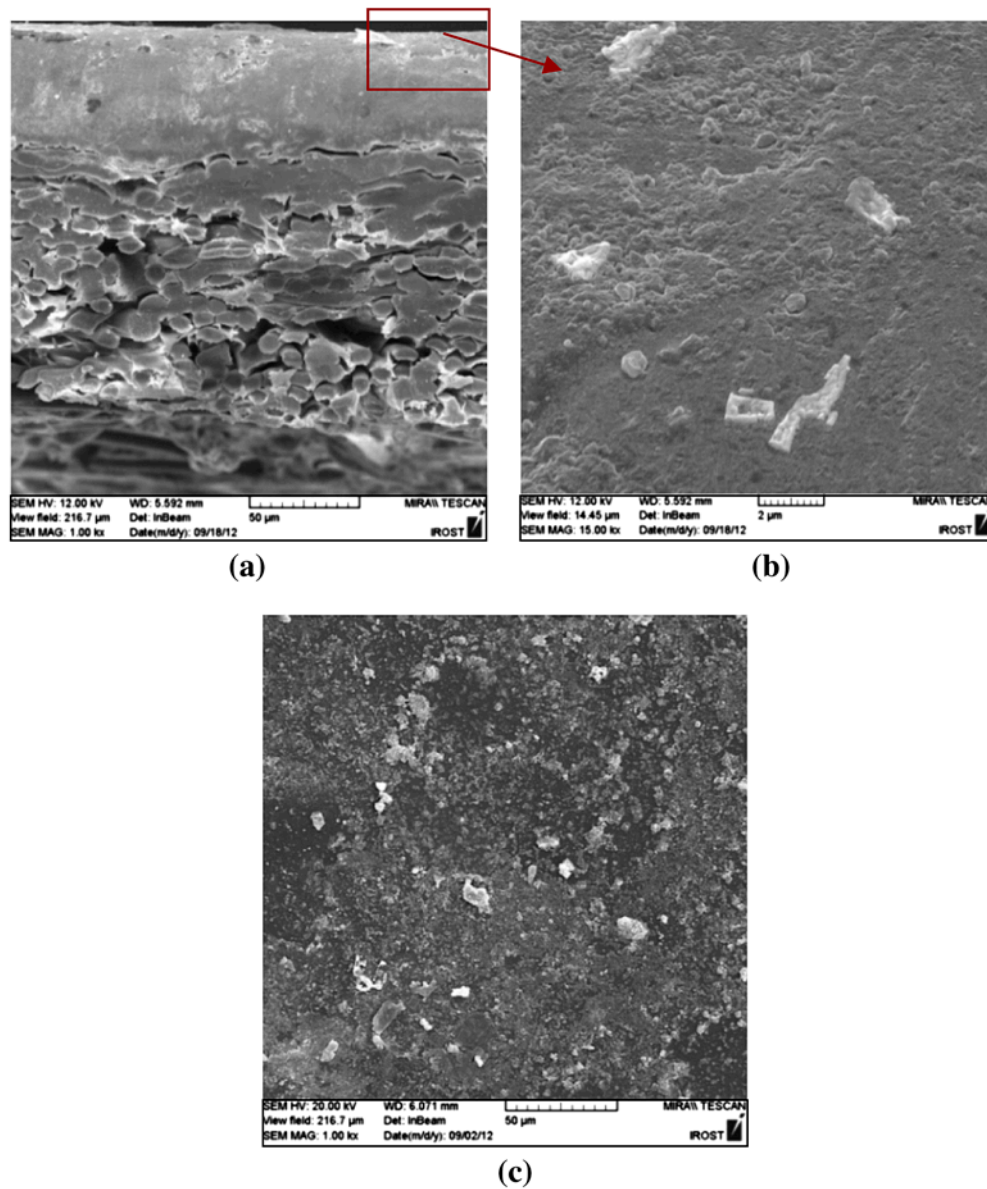


Fig. 5. SEM image of PAN350 after ultrafiltration process, (a) cross-section, (b) sediments in the pores of membrane, (c) compacted sediments and cake layer on the membrane surface.

decreased gradually to the point that the system reached a steady state. At the end, the level of flux was almost constant, so in the next 6 h there was only 11% decline in the flux. The percentage of flux decline in three time spans is illustrated in Fig. 7. The final amount of flux during the 8-h time was finally $54 \text{ L/m}^2 \text{ h}$.

The flux decline is related to the membrane fouling, which includes the combined effects of the absorption and accumulation of colloidal particles on the membrane surface and pore walls. Almost all parts of feed, to a certain extent, caused fouling of membrane. The

nature and amount of sediments depend on several things, such as the kind of pore size distribution, concentration of solutes material, flow hydrodynamic, properties of membrane surface and the interactions between membrane and dissolved material.

The UF process performance of the PAN350 membrane at optimum conditions is given in Table 7 and Fig. 8. In addition, all rejections of oil and grease, TSS, turbidity and TOC were calculated according to feed concentrations, and composite permeate concentrations obtained at the end of the long-time experiment in order to evaluate the overall performance of the

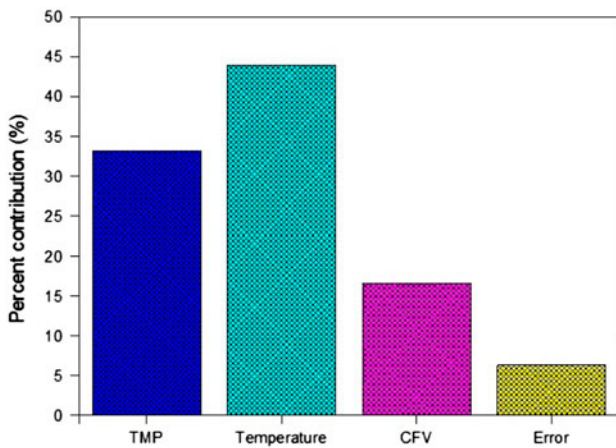


Fig. 6. Percent contribution of each factor on the performance statistics.

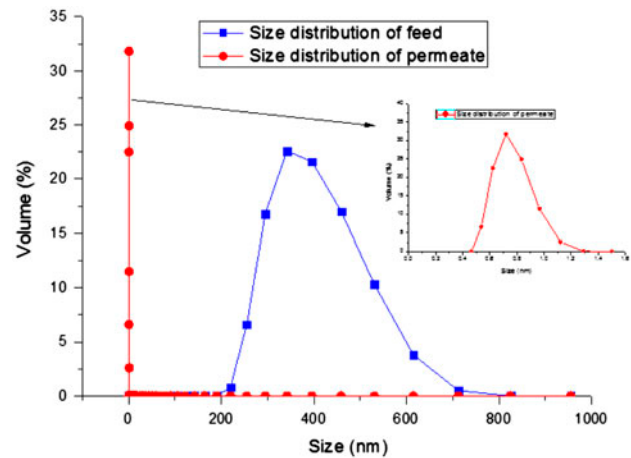


Fig. 8. Size distribution of feed and permeate particles at the end of 8-h long-time experiment.

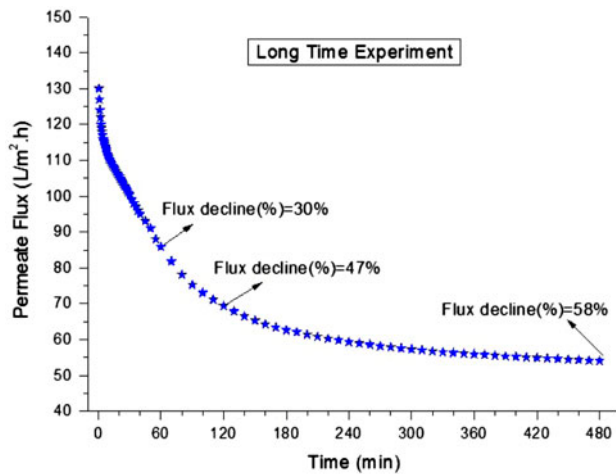


Fig. 7. The changes in permeate flux as a function of time at optimum conditions for PAN350 ($T=40^{\circ}\text{C}$, $\text{TMP}=1.5$ bar, $\text{CFV}=1$ m/s) membrane.

Table 7
Process performance of PAN350 membrane at optimum conditions

Parameter	Produced water quality	Permeate quality	Percent removal
Oil and grease	42	<1.5 ppm	99
TSS	108	Trace	100
Turbidity	90	0.5	99
TOC	109	35	68

system. It was demonstrated that the final permeation flux after 8 h was completely free of oil and TSS. However, TOC could not be entirely removed.

Moreover, to show the performance of the PAN350 membrane, particles size distribution of permeate and

feed is illustrated in Fig. 8. As it is shown, the size of particles in feed decreased from the range of 200–800 nm to 1.5–3 nm. As a result, PAN350 membrane is a good choice for UF of produced water treatment, because it successfully passed the requirement of desalination unit (size distribution < 100 nm, oil and grease < 5 ppm, TSS = nil and turbidity < 5).

4. Conclusion

In this paper, a two-stage study in an attempt to meet the demand of a desalination unit in Iran was conducted. In the first stage, Taguchi based designed experiments were used in order to optimize the effects of significant factors on flux decline caused by membrane fouling. To analyze the results of experiments, S/N ratio (smaller-the-better) was employed. In the second stage, the performance of PAN350 membrane under optimum conditions was studied. The results of these stages are: (1) The optimum conditions were chosen according to the highest value of S/N ratio as the first level of transmembrane pressure (1.5 bar), second level of the temperature (40°C), and third level of cross-flow velocity (1 m/s). In terms of percentage of each factor shown in the ANOVA table, the most effective parameter for minimizing flux decline was found temperature, while CFV was the least effective one. (2) At optimum conditions, under an 8-h long-time experiment a result of 99% oil and grease 100% TSS, 99% turbidity and 68% TOC rejection was obtained. (3) The size of particles in feed decreased from the range of 200–800 nm to 1.5–3 nm.

According to the obtained results, the PAN350 membrane successfully passed the request of desalination unit for achieving size distribution < 100 nm, oil and grease < 5 ppm, TSS = nil, and turbidity < 5.

Symbols

A_i	—	sum of all observations of factor A at level i
DOF	—	degrees of freedom
F	—	F-ratio value
F_A	—	F-ratio value of factor A
J_i	—	initial permeate flux ($\text{Lm}^{-2}\text{h}^{-1}$)
J_f	—	final permeate flux ($\text{Lm}^{-2}\text{h}^{-1}$) after filtration
k_A	—	number of the levels of factor A
MS	—	mean of square (variance)
MS_A	—	variance of A
MS_e	—	variance of error
n	—	number of repetition performed for an experimental combination
n_{A_i}	—	number of all observations at level i of factor A
N	—	the number of all observation
P	—	percentage of the product obtained experimentally
SS	—	sum of squares
SS_A	—	sum of squares of A
SS_e	—	sum of squares of error
SS_T	—	total sum of squares
S/N	—	signal-to-noise ratio
T	—	sum of all observations
Y_i	—	is the observation of i

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