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Application of taguchi method in the optimization of wastewater treatment using spiral-wound reverse osmosis element

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Abstract

A pilot study for wastewater treatment in Exir pharmaceutical Co. (Borojerd, Iran) was conducted using a RO system with the capacity of 14.38 m^3/d. A Filmtec TW30HP-4641 RO element (polyamide, thin-film composite) was used in this study. The pilot plant consists of two spiral-wound RO elements. The RO train was configured in series. Trial runs were conducted at different operating conditions including pressures, temperature and concentration.

The pilot results showed that flux of water containing nitrate, nitrite, phosphate and sulfite was about 58 l/m^2 h. Taguchi method was employed for flux optimization. Analysis of the experiments indicated that the temperature of feed solution and transmembrane pressure have the most contribution in water flux. The flux was improved to 69 l/m^2 h by setting the control factors according to the Taguchi method. The technique showed that concentration of feed solution has the highest contribution in rejection of a solution containing nitrate, nitrite, sulfite and phosphate. After setting the control factor according to the Taguchi method rejection was enhanced to 99.9% for this case study.

Keywords: Reverse osmosis; Nitrate; Nitrite; Sulfite; Phosphate; Taguchi method

1. Introduction

Industrial wastewater components show different degrees of contamination hazard due to their chemical characteristics as well as excessive concentration. Therefore, the treatment of wastewater, which is particularly hazardous to the environment, requires a number of complementary techniques that sufficiently remove pollutants and enable the wastewater to be discharged into the receiving water or be reused for industrial purposes. Membrane processes can eliminate shortcomings, which are characteristic of the traditional methods of wastewater treatment. Due to their selectivity and high effectiveness, they can replace traditional techniques or may operate together in combinations as hybrid systems [1].

Lacks of fresh water and improved membrane performance have together resulted in the increasing use of reverse osmosis (RO) for the production of potable water and the reuse of wastewater. The majority of commercial RO plants are used for the desalination of seawater and brackish water, while the number of RO plants treating municipal and industrial wastewater for reuse is still limited [2].

Nitrogenous compounds (ammonia, nitrite, and nitrate) are considered of major contaminants in wastewater. Reverse osmosis membranes are capable to remove these pollutants. Polyamide RO membrane is capable of removing 90–97% of nitrogenous compounds [3,4]. The nitrate rejection depends on the membrane type. A rejection between 40 and 97% has been obtained by different researchers using various membranes [5–11] or membrane-based hybrid processes [12]. Microfiltration has been used as a pretreatment to reverse osmosis for production of high-quality water from secondary effluent [13].

Balannec et al. [14] studied phosphate rejections of three RO membrane: Desal 3 SF (polyamide/polysulfone), TFC HR (composite polyamide) and BW30 (composite polyamide). Phosphate removals of 99.6%, 100% and 99.8% were obtained, respectively. Vourch et al. [15] used nanofiltration and reverse osmosis and reported phosphate rejection higher than 95%.

The technique of defining and investigating all possible conditions in an experiment involving multiple factors is known as the design of experiments [16]. In robust parameter design, the primary goal is to find factor settings that minimize response variation, while adjusting (or keeping) the process on target.

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After determination which factors affect variation, you can find settings for controllable factors that will either reduce the variation, make the product insensitive to changes in uncontrollable (noise) factors, or both. A process designed with this goal will produce more consistent output. Robust design is an engineering methodology for low costs [17].

Taguchi's parameter design is an important tool for robust design. It offers a simple and systematic approach to optimize design for performance, quality and cost [17,18]. When a critical quality characteristic deviates from the target value, it causes a loss. Continuously pursuing variability reduction from the target value is the key to achieve high-quality and reduce cost.

The successful applications of Taguchi methods by both engineers and statisticians within British industry have lead to the formation of UK Taguchi Club [19]. Taguchi's approach is totally based on statistical design of experiments [17]. This can economically satisfy the needs of problem solving and product/process design optimization [20]. By applying this technique one can significantly reduce the time required for experimental investigation. This is important in investigating the effects of multiple factors on performance as well as to study the influence of individual factors to determine which factor has more influence, which less [17,20].

The most important stage in the design of an experiment lies in the selection of control factors. For this purpose, as many factors as possible should be included and nonsignificant variables must be identified at the earliest opportunity. Taguchi creates a standard orthogonal array to accommodate these requirements. Depending on the number of factors and levels needed, the choice is left to the user to select the standard orthogonal array.

This paper describes a case study investigating the parameters that influence wastewater treatment using reverse osmosis element. Flux and rejection are key factors for evaluating the performance of membranes. These two factors demonstrate the membrane's ion removal capability [21]. Factors such as transmembrane pressure, temperature and concentration affect the flux and rejection. The main objective was to find a combination of parameters to achieve high flux and high rejection for treatment of water containing nitrate, nitrite, sulfite and phosphate.

2. Materials and methods

2.1. Apparatus

A pilot plant reverse osmosis (Aqua-Cleer MFP/3–800-Culligan Company, Italy) with two elements (spiral-wound module) operating in series was used for all trails (Fig. 1). Permeate and concentrate were returned to the tank. The feed tank was stainless steel, with a capacity of 1000 l and two walls which allowed the feed temperature to be kept constant using the circulation of cooled water or steam. Volume of permeate was measured using a calibrated volume counter. For measuring the time a calibrated chronometer was used.

All the hydraulic components used in Aqua-Cleer MFP/3 plants consist of corrosion-resistance materials and are designed to withstand the operating condition as follows:

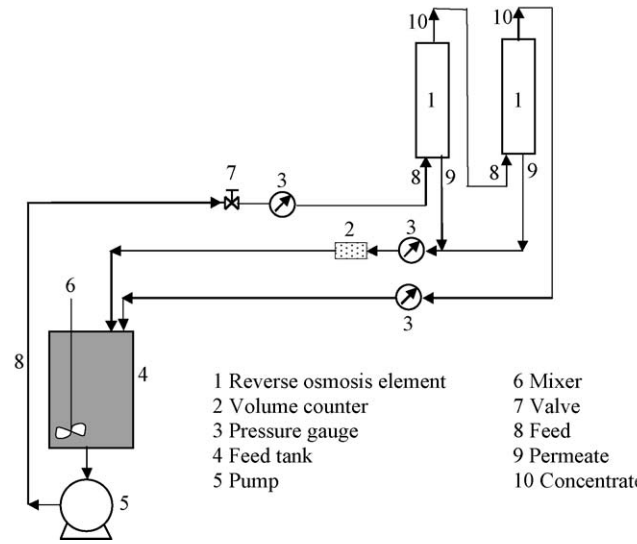

Fig. 1. Schematic of reverse osmosis system.

- The elements upstream of the high-pressure pump are resistant to a nominal pressure of 8 bar (114 psi).
- The elements of the high-pressure circuit (membrane inlet and reject lines) withstand a nominal pressure of 16 bar (228 psi).

Some parts of the line made of stainless steel, other parts were high-pressure hoses. The system consisted of a valve to control the applied pressure and flow.

2.2. Membrane

The thin-film composite polyamide membrane (Filmtec TW30HP-4641) was employed in the spiral-wound modules with the 128 ft^2 (11.89 m^2) active area.

2.3. Feed

For this work a solution containing nitrate, nitrite, sulfite and phosphate ions was prepared as the feed using deionized water at the pH between 5.5 and 6. The reagents used were: sodium nitrate, sodium nitrite, potassium dihydrogen phosphate and sodium sulfite all from Merck.

2.4. Ion rejection

Concentrations of ions were measured on the basis of standard methods. For mixture of ions, electrical conductivity (μs/cm) was employed as the basis for calculation of ion concentration. The rejection (R_Ω) based on the conductivity was calculated as

$$R_\Omega = \left[\frac{1 - \Omega_p}{\Omega_f}\right] \times 100 \tag{1}$$

where Ω_P and Ω_F are the conductivity of permeate and feed, respectively.

Table 1
Factors and their levels for design of experiments

Factor	Level		
	1	2	3
(A) Temperature (°C)	25 ± 2	30 ± 2	35 ± 2
(B) Pressure (bar)	5.75 ± 0.2	6.25 ± 0.2	6.75 ± 0.2
(C) Concentration (ppm)	50 ± 5	80 ± 5	110+5

2.5. Design of experiments

An operation limit of hydraulic components for pressure was in the range of 5–7 bars. We were able to control the pressure with ±0.2 bar accuracy. The feed temperatures were selected in the range of 20–40 °C according to operation limits of RO elements with accuracy of ±2 °C. Based on the concentrations of ions in the raw wastewater of the Exir pharmaceutical Co. (Borojerd, Iran), a range of 40–120 ppm with ±5 ppm accuracy was selected for feed concentration. Each operation limit of factors was divided into three levels.

For design of experiments with three factors (pressure, temperature and concentration) and three levels for each factor, the fractional factorial design, i.e. a standard L_9 orthogonal array [17] was employed. This orthogonal array was chosen due to no interaction among factors. Each row of the matrix represents one run. However, the sequence in which these runs are carried out is randomized. The three levels of each factor are represented by a “1” or a “2” or a “3” in the matrix.

The factors and their levels are assigned in Table 1. Table 2 shows the standard L_9 orthogonal array. Factors A, B, and C are arranged in column 1, 2, and 3, respectively, and column D is unused.

For analysis of the results and optimization of conditions for setting the control factors, QUALITEK-4 software was used. QUALITEK-4 (QT4) Version 4.75 is the windows version software for Automatic Design and Analysis of Taguchi Experiments.

3. Results & discussions

3.1. Primary experimental data

The results for flux of deionized water at different transmembrane pressures are shown in Fig. 2. The flux increases with increasing the transmembrane pressure according to Eq. (2).

Table 2
L_9 orthogonal arrays

Run#	Factor levels		
	A	B	C
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

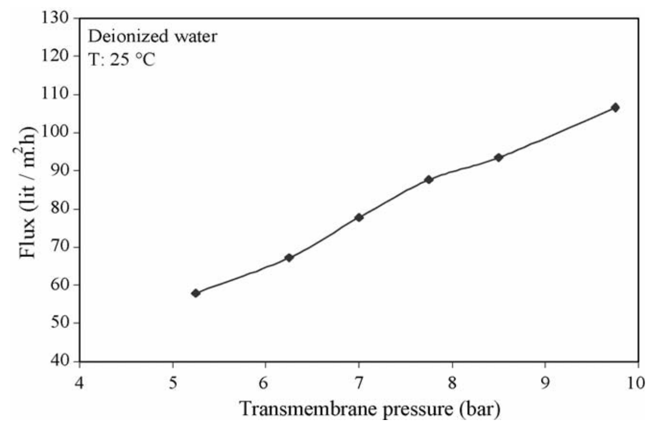

Fig. 2. Flux of deionized water for Filmtec TW30HP-4641 element vs. transmembrane pressure (25 °C).

$$J_w = \frac{P_w}{l}(\Delta P - \Delta\pi) \tag{2}$$

where J_w is the solvent flux; P_w the solvent permeability; l the thickness of membrane; ΔP the transmembrane pressure; $\Delta\pi$ is the osmotic pressure differential.

Filmtec TW30HP-4641 element showed instability in flux versus time at high transmembrane pressure (Fig. 3). For energy saving and flux stability the range of transmembrane pressure was selected between 5 and 6.5 bars.

The membrane permeability was calculated from Eq. (2). The average value of the water permeability (P_w) in the measured range was around 11 l/h m^2 bar (Fig. 4).

For investigation of the effects of transmembrane pressure, temperature and concentration on flux, four individual solutions containing phosphate (80 ppm), sulfite (50 ppm), nitrite (50 ppm) and nitrate (80 ppm) were prepared. In the next step a solution containing a mixture of all ions was prepared. The obtained fluxes are exhibited in Fig. 5.

These ranges of ion concentrations are three times of the ion concentrations in raw wastewater of Exir Pharmaceutical Co. that must be treated by reverse osmosis system.

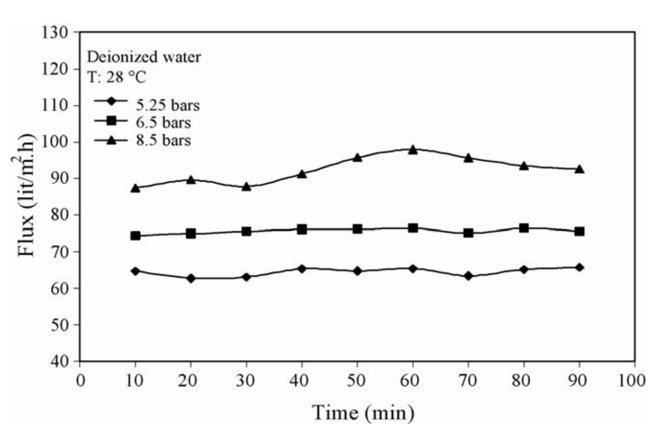

Fig. 3. Flux of deionized water for Filmtec TW30HP-4641 element vs. time as a function of transmembrane pressure (28 °C).

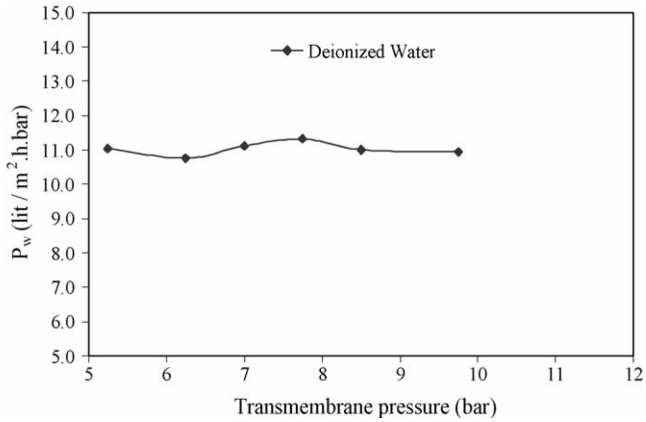

Fig. 4. Deionized water permeability for Filmtec TW30HP-4641 element vs. transmembrane pressure (25 °C).

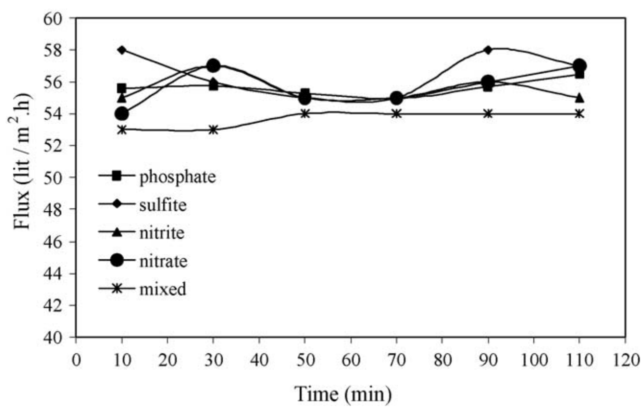

Fig. 5. Flux of water containing phosphate (80 ppm), sulfite (50 ppm), nitrite (50 ppm), nitrate (80 ppm) and mixed ions for Filmtec TW30HP-4641 element vs. time (6 bars, 25 °C).

Recycling the permeate during the experiments, results in an increase in feed temperature. The temperature of the feed was maintained constant with the accuracy of ±2 °C. Temperature fluctuation (±2 °C) may results in a deviation (about ±3 l/m^2 h) in flux (Fig. 5) or permeability (Fig. 6).

Temperature increment enhances the flux (Fig. 7). The empirical equation for the increment in our case study (at 6 bars

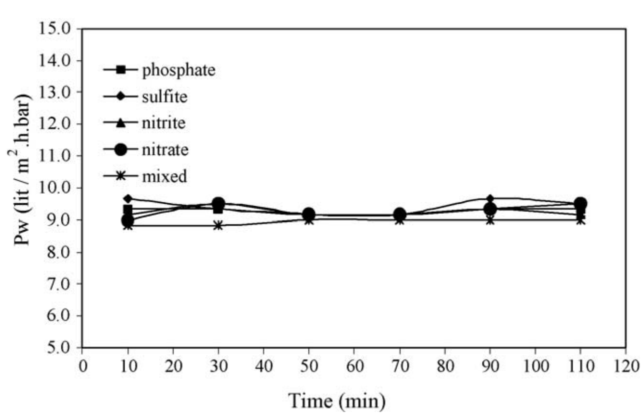

Fig. 6. Water permeability for Filmtec TW30HP-4641 element vs. time (6 bars, 25 °C).

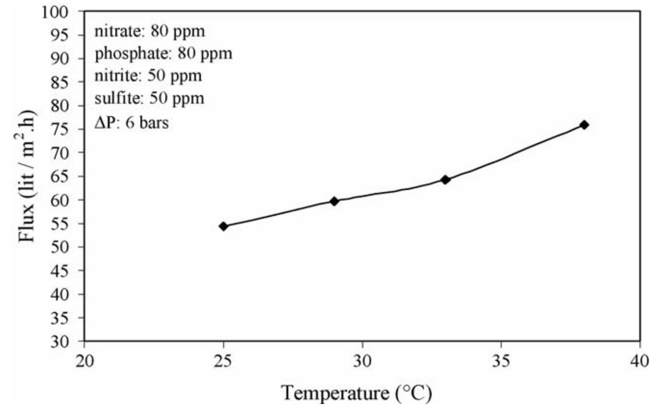

Fig. 7. Flux of water containing nitrate, nitrite, phosphate and sulfite ions for Filmtec TW30HP-4641 element vs. temperature (6 bars).

transmembrane pressure) is

$$J_{\mathrm{w}}(\text{water flux}) = 0.0119T^3 - 1.0624T^2 + 32.591T - 282.76 \quad (3)$$

where T is the temperature of feed solution.

The membrane permeability was plotted as a function of time. The five curves nearly superimposed. The average values of the water permeability (independent of pressure) was 9.2 l/h m^2 bar (Fig. 6).

In general, the flux declines due to the increase in the concentration polarization or fouling [22]. Recycling of permeate increases the concentration of the nitrate, nitrite, sulfite and phosphate ions close to the membrane-solution interface leading to an increase in osmotic pressure [23]. This decreases the effective driving force and results in flux decline. However, in our case, these two phenomena (i.e. concentration polarization and reduction of driving force) did not affect the flux. This can be explained due to the dilute nature of the feed as well as short period of experiment.

3.2. Flux optimization

According to the design of experiments based on Taguchi method (Table 2), Runs 1–9 were performed with a solution containing nitrate, nitrite, sulfite and phosphate during 120 min (Fig. 8).

The analysis of the results (Table 3) carried out using QUALITEK-4 software. In Taguchi method the main effect of control factors indicates the trend of influence of a factor. The main effects were calculated using average flux. The results (Fig. 9) indicate the effects of pressure, temperature and concentration on flux.

Another technique for optimization of the results suggested by Taguchi method is analysis of variance (ANOVA). This is information displays relative influence of factor and interaction to the variation of results.

Analysis of variance (ANOVA) is similar to regression which is used to investigate and model the relationship between a response variable and one or more independent variables. However, analysis of variance differs from regression in two ways:

Table 3
Experimental results for flux (l/m^2 h)

	Flux 1 (0 min)	Flux 2 (20 min)	Flux 3 (40 min)	Flux 4 (60 min)	Flux 5 (80 min)	Flux 6 (100 min)	Flux 7 (120 min)	Average flux
Run 1	49	50	52	50	49	48	48	49.428
Run 2	52	52	52	51	50	50	50	51
Run 3	70	55	56	54	55	53	54	56.714
Run 4	55	57	57	56	55	58	58	56.571
Run 5	55	56	56	55	56	55	55	55.428
Run 6	63	64	66	62	67	66	63	64.428
Run 7	58	58	58	58	58	57	58	57.857
Run 8	63	65	66	66	66	66	65	65.285
Run 9	73	66	65	65	66	67	68	67.143

Table 4
Analysis of variance (ANOVA) for flux measurement

Factors	DOF	Sum of squares	Variance	F-ratio	Pure sum	Percent
Temperature (°C)	2	1292.974	646.487	96.792	1279.616	51.383
Pressure (bar)	2	725.755	362.877	54.330	712.397	28.606
Concentration (ppm)	2	97.555	48.777	7.303	84.197	3.380
Other/error	56	374.027	6.679			16.631
Total	62	2490.313				100.00%

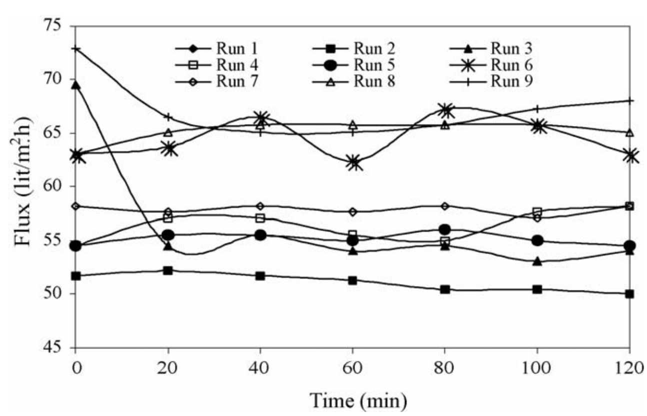

Fig. 8. Flux of water containing mixed ions for Filmtec TW30HP-4641 element vs. time (runs 1–9).

the independent variables are qualitative and no assumption is made about the nature of the relationship.

The last column of ANOVA indicates the influences of factors and interactions assigned to the column to the variations of the results. The row labeled other/error, on the other hand, contains information about the sources of variability of the results. This line indicates information about the influence from three sources.

1. Uncontrollable (noise) factors.
2. Factors which are not included in the experiment.
3. Experimental error.

The analysis of variance by QUALITEK-4 for this work is listed in Table 4. The numbers on the right hand side of the table indicate the "contribution" that a factor or interaction makes to the improvement of the expected performance.

After ANOVA, the optimum conditions for the experiment can be reported. The QUALITEK-4 software calculates the performance at the optimum condition. Optimum conditions and best performance for our case study are listed in Table 5. According to the Taguchi method, solution temperature has the highest contribution in flux of water containing nitrate, nitrite, sulfite and phosphate. The best setting for control factors is

- Transmembrane pressure = 6.75 bar.
- Concentration of feed solution = 50 ppm.
- Temperature of feed solution = 35 °C.

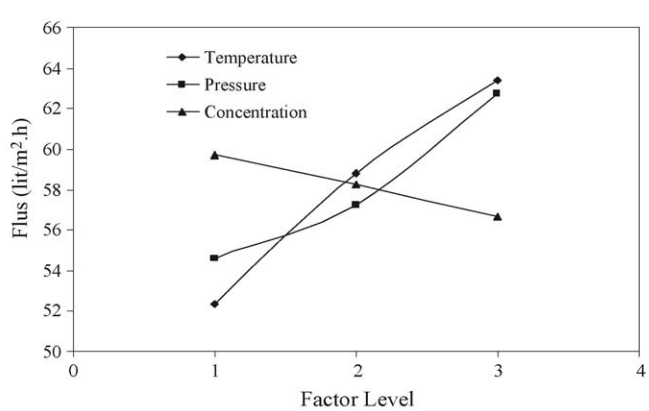

Fig. 9. Main effect plot for means of flux measurement.

Table 5
Optimum condition and performance of flux

Factor	Level description	Level	Contribution
Temperature (°C)	35	3	5.222
Pressure (bar)	6.75	3	4.555
Concentration (ppm)	50	1	1.507
Total contribution from all factors. . .			11.284
Current grand average of performance. . .			58.206 l/m^2 h
Expected result at optimum condition. . .			69.490 l/m^2 h

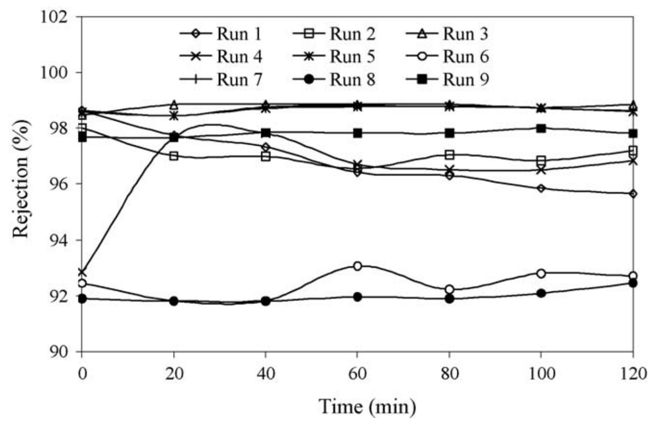

Fig. 10. Rejection of mixed ions using Filmtec TW30HP-4641 element vs. time (runs 1–9).

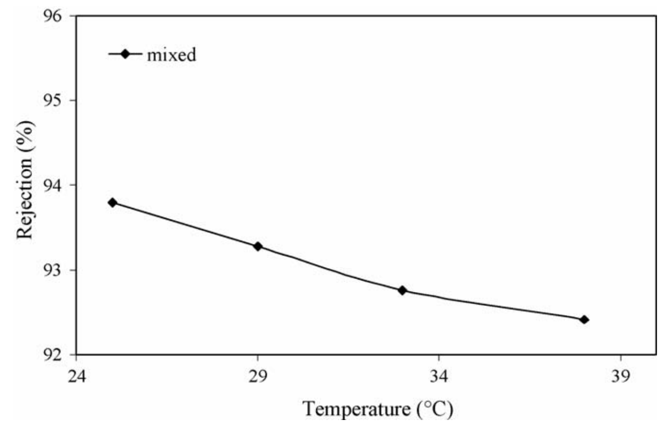

Fig. 11. Rejection of mixed ions using Filmtec TW30HP-4641 element vs. temperature (6 bars).

Current grand average (i.e. arithmetic average for all trials) for flux is around 58 l/m^2 h. However at optimum conditions, the flux is improved to around 69 l/m^2 h. The average value for water permeability is 11 l/h m^2 bar (for deionized water) and 9.2 l/h m^2 bar (for water containing nitrate, nitrite, sulfite and phosphate before optimization). At the optimum conditions, water permeability is increased to 10.2 l/h m^2 bar that is very close to P_w of deionized water.

3.3. Rejection optimization

For studying the effects of transmembrane pressure, temperature and concentration on rejection using Filmtec TW30HP-4641 element, a solution containing phosphate (80 ppm), sulfite (50 ppm), nitrite (50 ppm) and nitrate (80 ppm) was prepared and rejections of the ions were measured (Fig. 10). The temperature variation (±2 °C) causes a slight fluctuation in ion rejection.

Rejection is a function of solute molar flux which depends on solute permeability (Eq. (4)).

$$J_s = \frac{P_s}{l}(C_m - C_p) \quad (4)$$

where J_s is the solute molar flux; P_s the solute permeability; l the thickness of membrane; C_m the solute concentration; C_p is the solute concentration in permeate.

Solute permeability depends on the physico-chemical properties of ions and membrane material. Since membrane separation performance is based on molecular level, solute rejection is related to the macromolecular chain mobility. When solution temperature rises, polymeric molecular chains become more flexible and deform more easily. The higher sensitivity of the molecular chains to the temperature results in more possibility of expansion of membrane chains under hydraulic pressure when solution temperature is elevated. For pressure-driven membrane processes, solute flux through the membrane can be described as the sum of a convective and a diffusive transport. A membrane with high solute rejection indicates that the solute transport mainly caused by diffusive flux and the convective transport is mostly hindered. When solution temperature is elevated, the expansion of membrane chains results in convective transport leading to rejection decline. The degree of membrane expansion can be observed from solute rejection changes with varied solute temperatures in separation tests [24].

Fig. 11 exhibits a slight decline in ion rejection versus temperature increment for our case study. The empirical equation for the decrement (at 6 bars transmembrane pressure) is

$$R(\%) = 0.0038T^2 - 0.3463T + 100.1 \quad (5)$$

where T is the temperature of feed solution.

Eq. (5) indicates when the precision of temperature control is ±2 °C a fluctuation in rejection around ±0.3% is anticipated.

After running nine tests the rejections were measured and listed in Table 6 for different time intervals (0, 20, 40, 60, 80, 100 and 120 min).

Table 6
Experimental results for rejection (%)

	Run 1 (%) (0 min)	Run 2 (%) (20 min)	Run 3 (%) (40 min)	Run 4 (%) (60 min)	Run 5 (%) (80 min)	Run 6 (%) (100 min)	Run 7 (%) (120 min)	Average rejection (%)
Run 1	99	98	97	96	96	96	96	96.857
Run 2	98	97	97	97	97	97	97	97.142
Run 3	98	99	99	99	99	99	99	98.857
Run 4	93	98	98	97	97	97	97	96.714
Run 5	99	98	99	99	99	99	99	98.857
Run 6	92	92	92	93	92	93	93	92.428
Run 7	99	98	99	99	99	99	99	98.857
Run 8	92	92	92	92	92	92	92	92
Run 9	98	98	98	98	98	98	98	98

Table 7
Analysis of variance (ANOVA) for rejection measurements

Factors	DOF	Sums of squares	Variance	F-ratio	Pure sum	Percent
Temperature (°C)	2	31.375	15.687	10.569	28.406	6.690
Pressure (bar)	2	24.201	12.100	8.152	21.233	5.000
Concentration (ppm)	2	285.915	142.957	96.315	282.947	66.636
Other/error	56	83.119	1.484			21.674
Total	62	424.612				100.00%

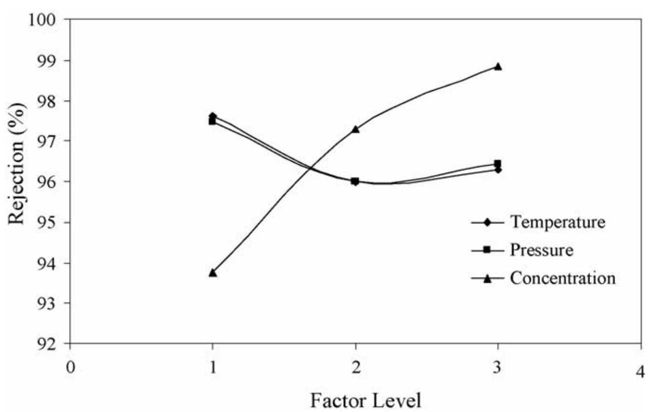

Fig. 12. Main effect plot for means of rejection measurement.

Table 8
Optimum condition and performance of rejection

Factor	Level description	Level	Contribution
Temperature (°C)	25	1	0.984
Pressure (bar)	5.75	1	0.841
Concentration (ppm)	110	3	2.222
Total contribution from all factors...			4.046
Current grand average of performance...			96.634%
Expected result at optimum condition...			100.681%

The main effects plot (Fig. 12) showed mat the concentration of feed solution has the highest contribution in rejection of ions in a solution containing nitrate, nitrite, sulfite and phosphate. The QUALITEK-4 software was used for ANOVA calculations (Table 7) and optimum conditions (Table 8) for setting the control factors.

The best setting for control factor is

- Transmembrane pressure = 5.75 bar.
- Concentration of feed solution = 110 ppm.
- Temperature of feed solution = 25 °C.

Current grand average of rejection is around 96% but at optimum condition the rejection improves to 100%.

4. Conclusions

Reverse osmosis treatment of water containing nitrate, nitrite, sulfite and phosphate using Filmtec TW30HP-4641 element evidenced that temperature of feed solution and transmembrane pressure have the highest contribution in flux. The feed concentration exhibited the lowest contribution. According to Taguchi method temperature of feed solution is more effective than transmembrane pressure and feed concentration. Raising the temperature and transmembrane pressure up to 35 °C and 6.75 bars and decreasing the feed concentration to 50 ppm results in highest flux. At this conditions, water permeability is about 10.2 l/h m^2 bar that is very close to water permeability of deionized water (about 11 l/h m^2 bar).

According to the Taguchi method, concentration of feed solution has the highest contribution in ion rejection of a solution containing nitrate, nitrite, sulfite and phosphate. Transmembrane pressure and temperature of feed solution have minor contributions. Increasing the feed concentration up to 110 ppm and decreasing the temperature and transmembrane pressure down to 25 °C and 5.75 bars results in a complete rejection in this case study.

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