Optimization of operating conditions for permeation of low concentrated salts through nanofiltration membrane using response surface methodology

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Abstract— **Response surface methodology (RSM) was used to investigate the effects and mutual interactions of operating parameters (effective pressure, temperature and feed concentration) on simulated rejection and specific energy consumption responses for an ion permeating process through NF90 nanofiltration membrane. The analysis of variance demonstrates that the regression models were significant for both responses. The regression analysis assumption of normally distributed residuals was confirmed using the normal probability plot of the residuals and performing the Shapiro-Wilk test. It was found that the effective pressure has the most significant effect on both responses, followed by the feed concentration. The process temperature is more significant for rejection than for specific energy consumption. The optimum conditions which simultaneously maximize the rejection and minimize the specific energy consumption (SEC) were assessed using the desirability function approach and was found to be at effective pressure of 16 bar, temperature of 30° C and feed concentration of 25 mol.m^{-3}, with a resulted rejection value of 97.6% and a SEC value of 0.96 kWh.m^{-3}. When considering a higher feed concentration of 75 mol.m^{-3}, the optimum conditions are at effective pressure of 24 bar and temperature of 30° C, with a rejection value of 96.2% and a SEC value of 1.54 kWh.m^{-3}.**

Index Terms— **Nanofiltration; Modeling; Factorial design; Response surface methodology; Optimization.**

I. INTRODUCTION

Nanofiltration (NF) membrane separation processes have received significant attention and remains promising in the future due to characteristics as low energy requirement, ease of operation and high selectivity, which leads to low operational costs and low environmental impacts. NF is a pressure-driven membrane separation process with characteristics between those of reverse osmosis and ultrafiltration and is currently applied in many industrial processes such as desalination [1]. During the last two decades, the prediction of membrane performance has been a relevant area of research due to the complexity associated with modeling the ion transport at a nanoscale [1], [2].

The cost of permeate production in desalination plants using membrane processes generally consists of the cost of energy consumption, equipment, membranes, labor, maintenance and financial charges, the major part of the total cost attributed to energy consumption [3]. Considerable effort has been made to minimize the specific energy consumption of water desalination in membrane processes, including advances in areas of innovation such as membrane and element design, motor pump efficiency and energy recovery technology improvement [4]. Therefore, it is worthwhile using a systematic design combined to numerical simulation models to investigate the optimum conditions which lead to simultaneously minimal energy consumption and maximum salt rejection.

Response surface methodology (RSM) has been proven to be an effective statistical tool to find the conditions which optimizes one or several responses for complex systems, including membrane filtration processes [5, 6]. The efficient use of RSM as an optimizing tool in membrane processes was validated in many studies [5], [7], [8], [9], [10]. The use of RSM has gained importance in process design and optimization due to its simplicity in correlating the parameters and accuracy in predicting the measured responses [10].

The aim of this work is to investigate the effects and interactions of operating parameters such as effective pressure, temperature and feed concentration on rejection and specific energy consumption in the permeation process of NaCl solution through the commercial NF90 nanofiltration membrane and identify the conditions which optimize the performance responses using RSM with a three level factorial design.

II. METHODOLOGY

A. Membrane and Feed Solutions

The membrane considered in this work was NF90 (Dow FilmTech). The main characteristics of this membrane are shown in Table I.

Table I. Summary of NF90 membrane characteristics

	NF90
Supplier	Dow FilmTec
Material of skin layer[a]	Polyamide
Max. temperature, °C[a]	45
Pore radius, nm	0.55[b]
pH range[a]	3-9

[a]According to membranes supplier; [b] [11]

In a previous study [12], a predictive model for permeation of single salt solutions through the NF90 membrane, based on DSPM (Donnan-Steric Pore Model) equations and considering zeta potential measurements, was assessed and

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validated using experimental data of Nicolini et al. [13]. In the present work, an investigation of operating conditions for the permeation of NaCl through the NF90 membrane was conducted using this model. Three independent parameters (effective pressure, temperature and feed concentration) were varied between three values, generating 27 different combined simulations. The objective of simulations were to investigate the effect of these parameters on two performance responses (rejection and specific energy consumption) and to determine the optimum value of these parameters in order to optimize the response variables (i.e., maximize the rejection and minimize the specific energy consumption).

The effective pressure values considered were 10, 20 and 30 bar; the temperature values were 20, 25 and 30 º C and the feed concentration values were 25, 50 and 75 mol.m^{-3}. The response variables, rejection and specific energy consumption (SEC, kWh.m^{-3}), were calculated with (1) and (2), respectively:

$$\mathrm{Re}j = 1 - \frac{C_{i,p}}{C_{i,f}} \tag{1}$$

$$SEC = \frac{1}{Y} - \frac{\Delta P}{36\eta_{eff}} \tag{2}$$

where $C_{i,p}$ is the permeate concentration (mol.m^{-3}), $C_{i,f}$ is the feed concentration (mol.m^{-3}), ΔP is the applied pressure (bar), Y is the recovery rate (ratio between permeate and feed flow, considered to be 1 in this work) and η_{eff} is the global pump system efficiency (considered to be 50% in this work).

The model parameters of NF90 membrane determined with the previously proposed model considering the NaCl salt were: effective membrane thickness equal to 0.49 µm and dielectric constant of ordered water layer equal to 30. These values were considered in all simulations which were carried on with the software Scilab using the model equations and numerical resolution described in [12].

B. Simulation design

A 3^k factorial design with two independent variables (rejection and specific energy consumptions) and three k-factors (effective pressure, temperature and feed concentration) was used to evaluate the effects of the factors on response variables in the NaCl permeation process through NF90 membrane. The simulation design with the two resulted responses are shown in Table II in terms of original and coded (1, 0, -1) variables.

A second order polynomial model was used for regression with the simulated data using the software Statistica (Trial version 13, Statsoft Inc.) according to (3):

$$Y_k = b_{k0} + \sum_{i=1}^{3} b_{ki} x_i + \sum_{i=1}^{3} b_{kii} x_i^2 + \sum_{i=1}^{3} \sum_{j=j+1}^{3} b_{kij} x_i x_j \tag{3}$$

where b_{k0}, b_{ki}, b_{kii} and b_{kij} are the regression coefficients, Y_k are the response variables and x_i are the independent variables. The coefficient of determination R^2 was considered to measure the fitness of the regression model. The regression analysis assumption of normally distributed residuals was also assessed using the normal probability plot of the residuals and performing the Shapiro-Wilk test.

In membrane permeation processes it is usually desirable to obtain the highest rejections with the lowest specific energy consumption. Thus, an optimization study was carried on to obtain the best operating conditions which lead to the optimum results. For this, the desirability function approach, proposed by Derringer and Suich [14], was used with Statistica.

The desirability approach consists in converting each response variable Y_k into an individual desirability function d_k in the scale $0 \le d_k \le 1$. If the response is fully desirable, $d_k = 1$ and if the response is outside the acceptable region, $d_k = 0$. The independent variables are chosen in order to maximize the global desirability function, D, defined by (4) [15].

$$D = (d_1 \times d_2 \times \ldots \times d_k)^{1/k} \tag{4}$$

where k is the number of response variables.

If the target value for response Y_k is a maximum value, the individual desirability d_k is defined by (5); if it is a minimum value, (6) is applied [15].

$$d_k = \begin{cases} 0 & Y_k \le Y_{k\min} \\ \left(\dfrac{Y_k - Y_{k\min}}{Y_{k\,\mathrm{arg}\,et} - Y_{k\min}}\right)^s & Y_{k\min} \le Y_k \le Y_{kt\,\mathrm{arg}\,et} \\ 1 & Y_k \ge Y_{kt\,\mathrm{arg}\,et} \end{cases} \tag{5}$$

$$d_k = \begin{cases} 1 & Y_{kt\,\mathrm{arg}\,et} \le Y_k \\ \left(\dfrac{Y_{k\max} - Y_k}{Y_{k\max} - Y_{kt\,\mathrm{arg}\,et}}\right)^t & Y_{kt\,\mathrm{arg}\,et} \le Y_k \le Y_{k\max} \\ 0 & Y_k \ge Y_{k\max} \end{cases} \tag{6}$$

where $Y_{kt\,\mathrm{arg}\,et}$ is the target value, Y_{kmin} is the minimum acceptable value for response and Y_{kmax} is the maximum acceptable value for response. The parameters s and t are the response weights. In this work, it was set s = t = 1, generating a linear desirability function.

III. Results and Discussion

A. Statistical Analysis

The effective pressure, temperature and concentration data were analyzed and related to the two responses considered (rejection and specific energy consumption) by second order polynomial regression models with the software Statistica (Trial version 13, Statsoft Inc.). The adequacy of the models was tested by the analysis of variance (ANOVA). The statistical significance of each term was indicated by p-value. It was found that the quadratic term of temperature and the interactions between quadratic terms of all variables (except that of ΔPe^2 x C_f for rejection and SEC responses and ΔPe x Cf^2 for SEC response) were not statistical significant (p-value > 0.05) and were removed form models. The resulting ANOVA with statistical significant terms for

Table II. Factorial design and independent response variables.

Run	ΔP_e (bar)		T (° C)		C_f (mol.m^{-3})		Rej	SEC (kWh.m^{-3})
	Original	Coded	Original	Coded	Original	Coded		
1	10	-1	20	-1	25	-1	0.9597	0.62
2	10	-1	20	-1	50	0	0.9290	0.68
3	10	-1	20	-1	75	1	0.9011	0.74
4	10	-1	25	0	25	-1	0.9625	0.62
5	10	-1	25	0	50	0	0.9342	0.68
6	10	-1	25	0	75	1	0.9085	0.74
7	10	-1	30	1	25	-1	0.9650	0.62
8	10	-1	30	1	50	0	0.9388	0.69
9	10	-1	30	1	75	1	0.9150	0.75
10	20	0	20	-1	25	-1	0.9783	1.18
11	20	0	20	-1	50	0	0.9617	1.24
12	20	0	20	-1	75	1	0.9465	1.30
13	20	0	25	0	25	-1	0.9795	1.18
14	20	0	25	0	50	0	0.9642	1.24
15	20	0	25	0	75	1	0.9501	1.31
16	20	0	30	1	25	-1	0.9808	1.18
17	20	0	30	1	50	0	0.9664	1.25
18	20	0	30	1	75	1	0.9533	1.31
19	30	1	20	-1	25	-1	0.9845	1.73
20	30	1	20	-1	50	0	0.9726	1.80
21	30	1	20	-1	75	1	0.9617	1.86
22	30	1	25	0	25	-1	0.9853	1.73
23	30	1	25	0	50	0	0.9742	1.80
24	30	1	25	0	75	1	0.9640	1.87
25	30	1	30	1	25	-1	0.9860	1.74
26	30	1	30	1	50	0	0.9755	1.80
27	30	1	30	1	75	1	0.9660	1.87

Table III. Analysis of variance (ANOVA) for the polynomial models – rejection.

	SS[a]	df[b]	MS[c]	F-value	P-value
ΔP_e	0.0070	1	0.0070	14214.37	< 0.0001
ΔP_e^2	0.0006	1	0.0006	1186.49	< 0.0001
T	0.0001	1	0.0001	297.20	< 0.0001
C_f	0.0055	1	0.0055	11150.82	< 0.0001
C_f^2	0.0000	1	0.0000	7.98	0.0117
ΔP_e x T	0.0000	1	0.0000	67.81	< 0.0001
ΔP_e x C_f	0.0008	1	0.0008	1628.88	< 0.0001
ΔP_e^2 x C_f	0.0001	1	0.0001	136.55	< 0.0001
T x C_f	0.0000	1	0.0000	40.95	< 0.0001
Error	0.0000	17	0.0000		
Total SS	0.0142	26			
R-squared: 0.9994					
Adj R-squared: 0.9991					

[a] SS – sum of squares
[b] df – degrees of freedom
[c] MS - mean sum of squares

Table IV. Analysis of variance (ANOVA) for the polynomial models – SEC.

	SS[a]	df[b]	MS[c]	F-value	P-value
ΔP_e	5.6177	1	5.6177	218931375	< 0.0001
ΔP_e^2	0.0000	1	0.00007	562	< 0.0001
T	0.0001	1	0.00017	4936	< 0.0001
C_f	0.0728	1	0.0727	2834850	< 0.0001
C_f^2	0.0000	1	0.0000	280	< 0.0001
ΔP_e x T	0.0000	1	0.0000	21	0.0003
ΔP_e x C_f	0.0001	1	0.0001	2875	< 0.0001
ΔP_e x C_f^2	0.0000	1	0.0000	40	< 0.0001
ΔP_e^2 x C_f	0.0000	1	0.0000	240	< 0.0001
T x C_f	0.0000	1	0.0000	971	< 0.0001
Error	0.0000	16	0.0000		
Total SS	5.6907	26			
R-squared: 1.0					
Adj R-squared: 1.0					

[a] SS – sum of squares
[b] df – degrees of freedom
[c] MS - mean sum of squares

rejection and SEC responses are shown in Tables III and IV, respectively.

The regression coefficients in terms of original variables are shown in Table V for both responses.

Table V. Regression coefficients of polynomial models of rejection and SEC.

Variables	Regression coefficients	
	Rejection	SEC
Intercept	0.965334	0.002811
ΔP_e	0.001844	0.055272
ΔP_e^2	-0.000017	0.000009
T	0.000721	0.000039
C_f	-0.002296	0.002228
C_f^2	0.000001	-0.000003
ΔP_e x T	-0.000033	-0.000004
ΔP_e x C_f	0.000099	0.000022
ΔP_e x C_f^2	-	0.000000
ΔP_e^2 x C_f	-0.000002	-0.000000
T x C_f	0.000010	0.000012

The reduced forms of polynomial models are as follows:

$$\begin{aligned} Rej &= 0.965334 + 0.001844\Delta P_e - 0.000017\Delta P_e^2 \\ &+ 0.000721T - 0.002296C_f + 0.000001C_f^2 \\ &- 0.000033\Delta P_e \times T + 0.000099\Delta P_e \times C_f \\ &- 0.000002\Delta P_e^2 \times C_f + 0.000010T \times C_f \end{aligned} \quad (7)$$

$$\begin{aligned} SEC &= 0.002811 + 0.055272\Delta P_e + 0.000009\Delta P_e^2 \\ &+ 0.000039T - 0.002228C_f - 0.000003C_f^2 \\ &- 0.000004\Delta P_e \times T + 0.000022\Delta P_e \times C_f \\ &+ 0.000012T \times C_f \end{aligned} \quad (8)$$

B. Residual Analysis

The residual distribution was analyzed in order to evaluate the assumption of normal distribution. If this assumption is correct, a straight line should be obtained on a normal probability plot. The normal probability plots for both rejection and SEC are shown in Fig. 1.

It can be noted that for both rejection and SEC responses an approximately straight line is obtained, indicating that the regression models are of good fit and it is adequate. This also reflects the high values of adjusted R^2 for both cases.

To complement this investigation, the Shapiro-Wilk test was performed in order to obtain a quantitative analysis of the normality test. Fig. 3 shows the frequency histogram of residues for both responses.

It should be noted that the p-value > 0.05 for both cases (p-value = 0.64067 for rejection and p-value = 0.38572 for SEC), confirming that the assumption of normality is correct. The null hypothesis of normally distributed residues is then accepted.

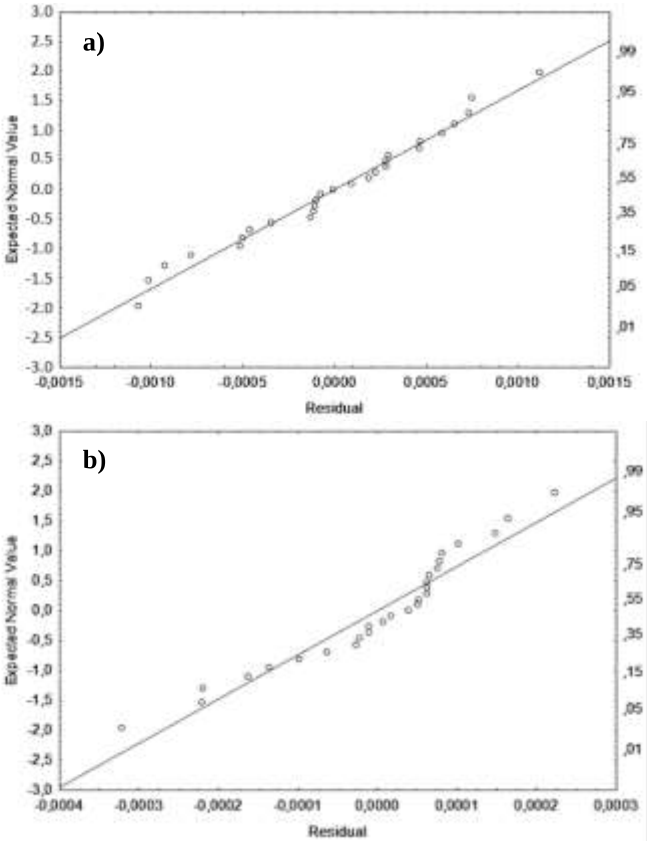

Fig. 1. Normal probability plot of residuals of observed values compared to predicted values: (a) for rejection and (b) for SEC.

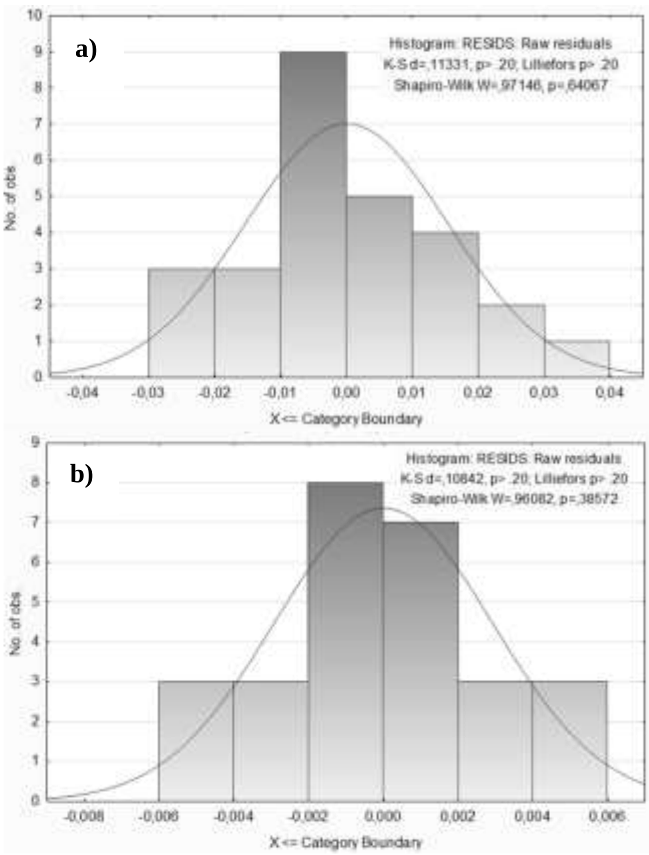

Fig. 2. Frequency histogram of residues: (a) for rejection and (b) for SEC.

C. Effect of variables

The degree of influence of each factor on the rejection and SEC responses is indicated in the Pareto charts as shown in Fig. 3. The bar lengths indicate the absolute values of the effects.

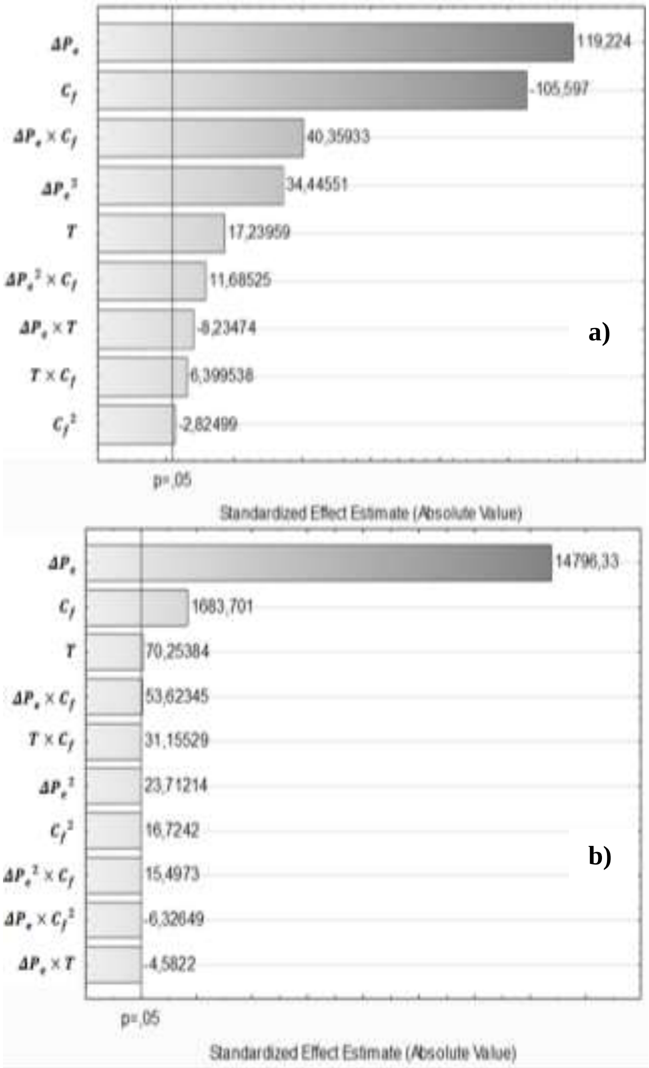

Fig. 3. Pareto charts of effects for the responses: (a) rejection and (b) SEC.

From Fig. 3a it should be noted that the linear effective pressure has the most significant effect on rejection, as expected for a pressure-driven separation process. The second significant factor is the feed concentration, which shows an effect almost as high as the effective pressure. However, these effects are inverses: while rejection increases with increasing effective pressure, it decreases with increasing concentration. As a consequence, for very high concentrated solutions the effective pressure can be also very high to attend the same rejection. It can also be noted in Fig. 3a that the interaction terms of linear effective pressure with feed concentration and the quadratic term of effective pressure are more significant than the linear temperature.

In Fig. 3b it can be noted that the specific energy consumption, like the rejection, is most affected by the effective pressure in first place, followed by the feed concentration. However, unlike the rejection, the effect of effective pressure is much more significant than the effect of feed concentration. This is because the energy consumption is directly related to the applied pressure that should be delivered by the pump to feed the system. The feed concentration also affects the energy consumption because high concentrations lead to higher osmotic pressures, requiring a higher applied pressure delivered by the pump to drive the process. The third significant effect is the temperature, followed by the interaction and quadratic terms which have similar significance.

D. Response Surface

The predicted models and the effects of variables are presented in Figs. 4 and 5 as the 3-D response surface and contour plots for rejection and SEC, respectively. These plots are obtained by representing two variables within simulation range and keeping the third variable constant at level zero (mean value).

As previously discussed, the effective pressure is the most significant factor for both rejection and SEC. As showed in Fig. 4a, a high rejection (above 96%) can be obtained when ΔP_e > 16-20 bar at temperatures between 20 and 30 and considering a constant feed concentration equal to 50 mol.m^{-3}. However, for higher concentrations, rejection values above 96% are only obtained with higher effective pressures, as showed in Fig. 4b. For feed concentration values above 75 mol.m^{-3} and considering a constant temperature equal to 25 º C, an effective pressure above 25 bar is required to attaint higher rejections. For a constant pressure equal to 20 bar, the higher the feed concentration and the lower the temperature, the lower the rejection, as showed in Fig. 4c. This can be explained by the increase of the diffusive transport caused by the increase of the concentration, causing the transport of salts through the membrane to be higher. On the other hand, the increase of the temperature makes the solvent viscosity to decrease, causing an increase in solvent transport that is not accompanied by the transport of salts, causing the rejection to increase. Referring to Fig. 4c, rejections above 96% can be obtained with C_f < 50 mol.m^{-3} at all temperatures within simulation range (20 º C < T < 30º C); however, a maximum feed concentration about 65 mol.m-3 is permissible at 30º C to attaint higher rejections at 20 bar.

Fig. 5a shows that the higher the effective pressure, the higher the specific energy consumption, causing the process to be less energy efficient. At a fixed feed concentration equal to 50 mol.m^{-3}, it can be noted in Fig. 5a that the influence of the temperature is very small comparing to that of the effective pressure. As previously discussed, this is because the energy consumption is directly related to the applied pressure that should be delivered by the pump to feed the system. When the temperature is kept constant at 25 º C, the energy consumption decreases with decreasing feed concentration, as showed in Fig. 5b. This is because at lower concentrations the osmotic pressure is lower. When the effective pressure is kept constant at 20 bar, the influence of temperature is a little more significant: for each concentration considered, the lower temperature, the lower the SEC, as showed in Fig. 5c.

E. Optimization

An optimization process was carried out to find the operating conditions which lead to a maximum rejection and a minimum specific energy consumption simultaneously for the transport of NaCl through NF90 membrane within simulation conditions. For this, the desirability function of

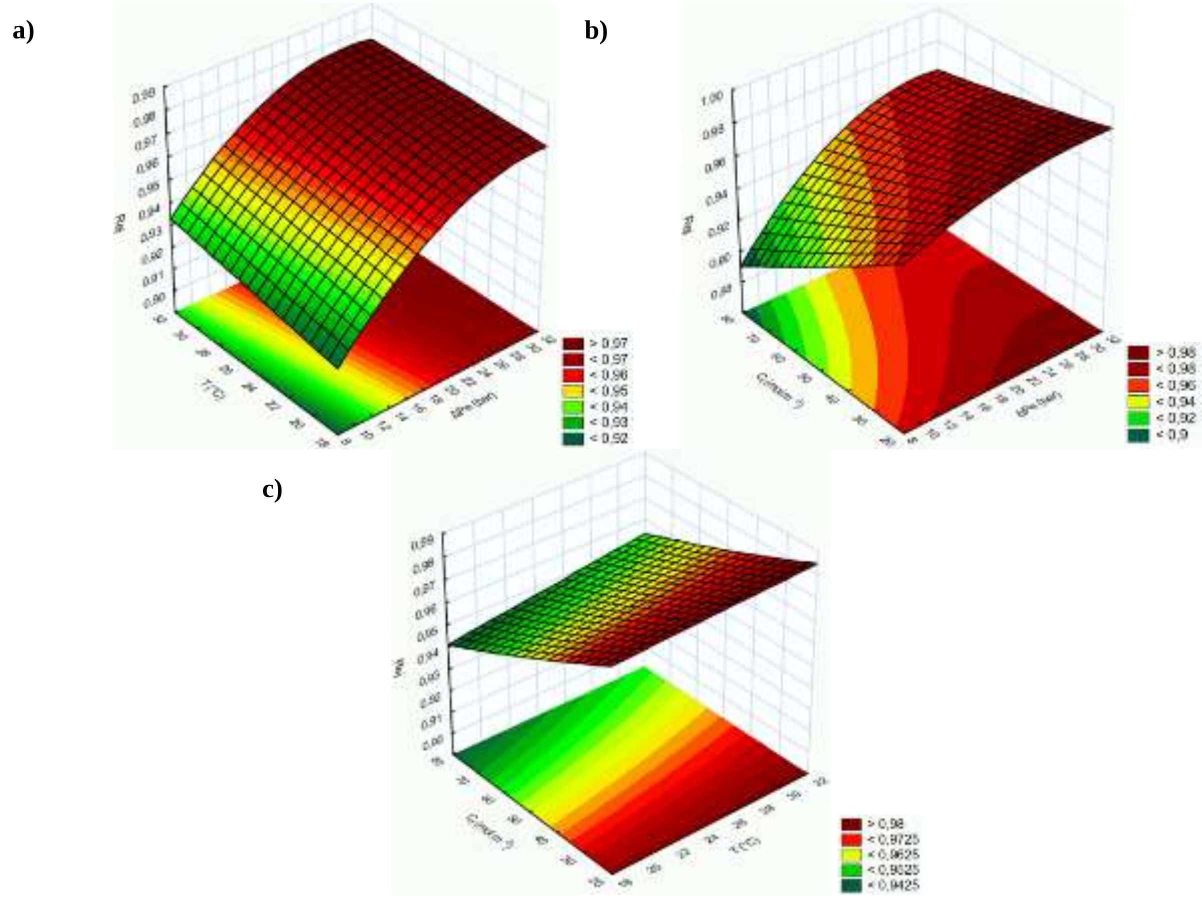

Fig. 4. Response surfaces of rejection as a function of: (a) temperature and effective pressure; (b) feed concentration and effective pressure and (c) feed concentration and temperature.

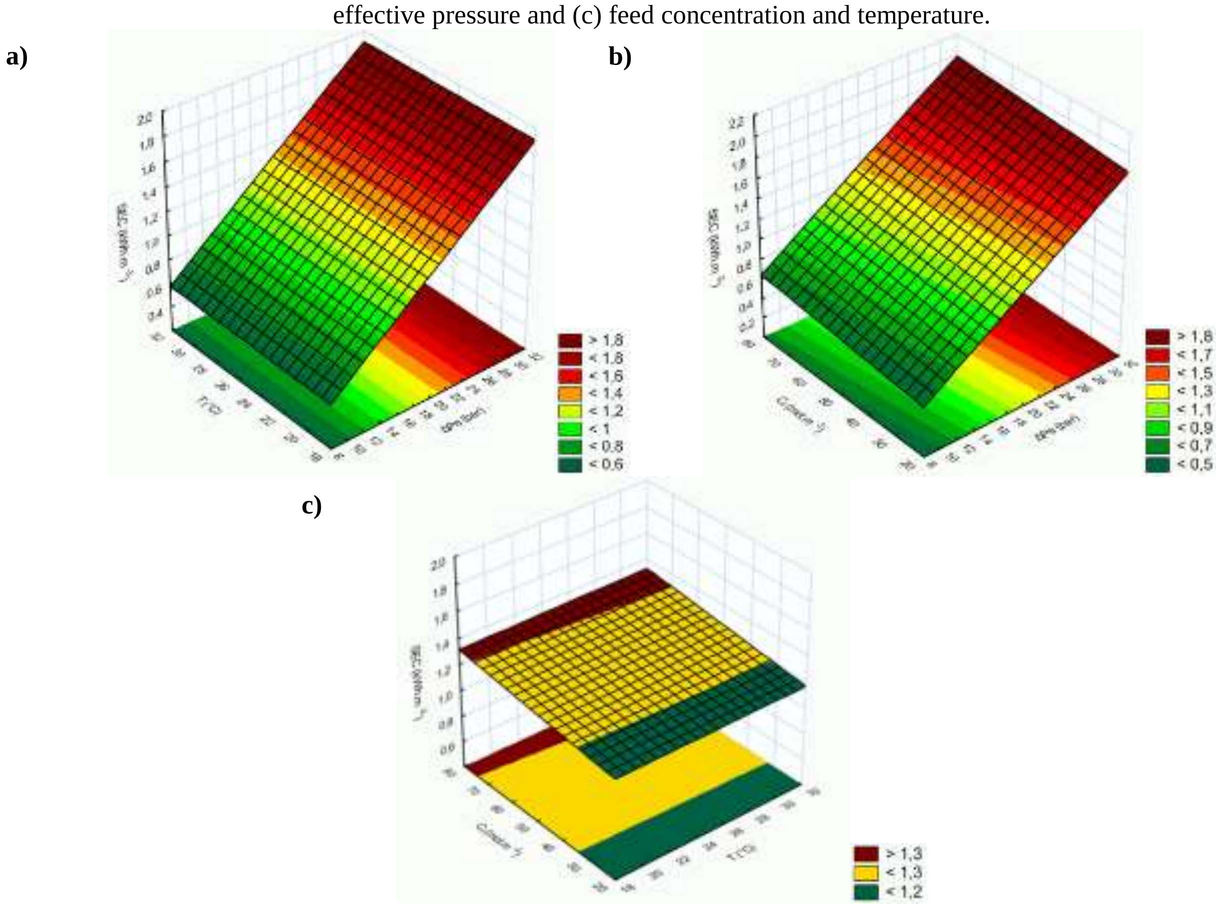

Fig. 5. Response surfaces of SEC as a function of: (a) temperature and effective pressure; (b) feed concentration and effective pressure and (c) feed concentration and temperature.

Statistica software was used. It was selected a target rejection value equal to *Rej* ≥ 98% (desirability level of 1 in a scale of 0 – 1) and a minimum acceptable rejection value equal to 95% (desirability level of 0 for rejection below this value). In the case of SEC response, the target value was SEC ≤ 0.6 kWh.m^{-3} (desirability level was equal to 1) and the maximum acceptable value set was 2.0 kWh.m^{-3} (desirability level equal to 0 above this value). The resulting desirability profiles are showed in Fig. 6a-c for minimum feed concentration equal to 25, 50 and 75 mol.m^{-3}, respectively.

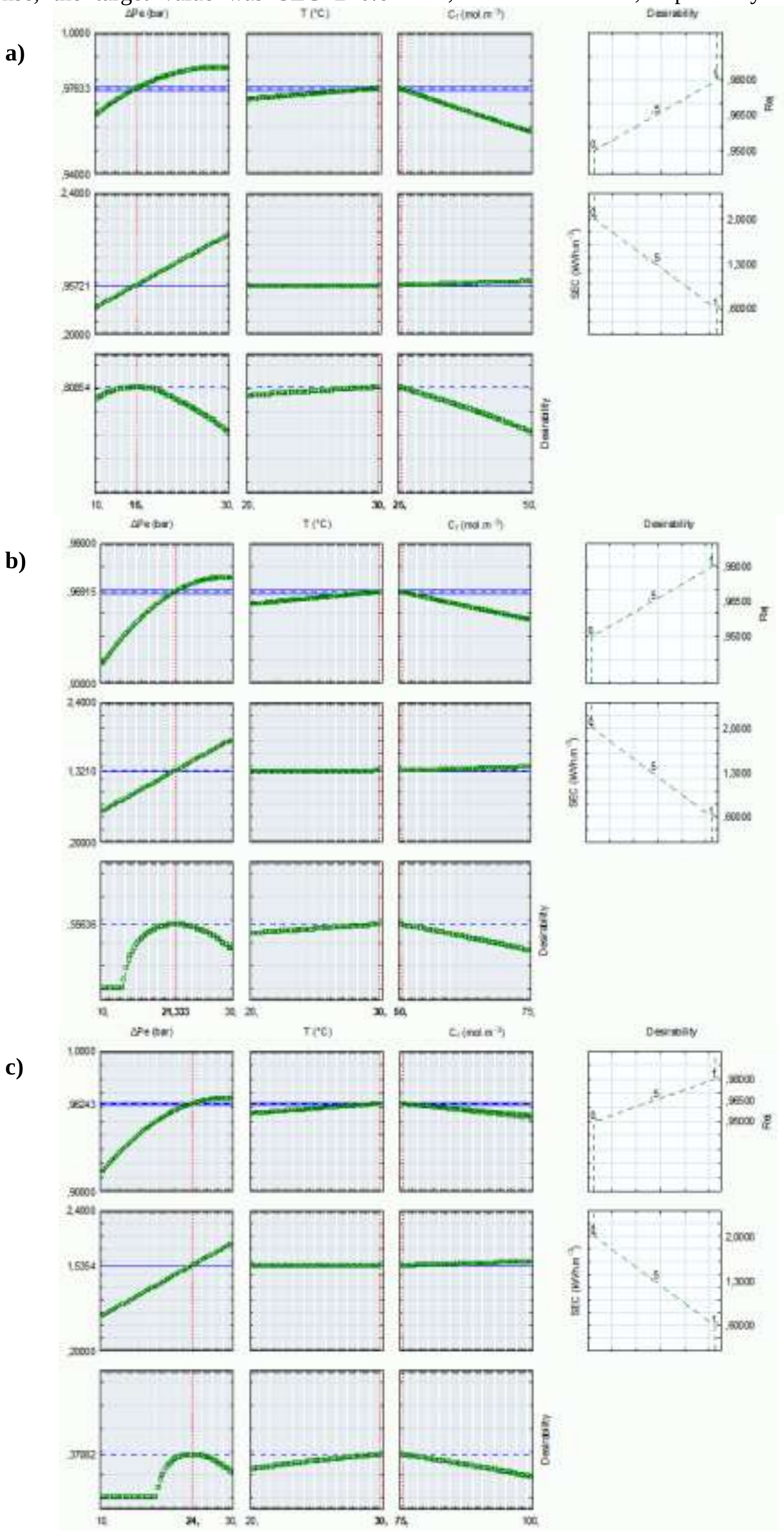

Fig. 6. Desirability profiles for optimum rejection and SEC values considering a minimum feed concentration as: (a) 25 mol.m^{-3}; (b) 50 mol.m^{-3} and (c) 75 mol.m^{-3}.

Figure 6a shows that the optimum operating parameters values within simulation range that optimizes the rejection and specific energy consumption for NaCl permeation through NF90 membrane were: ΔP_e = 16 bar, T = 30° C and C_f = 25 mol.m^{-3}. These operating values lead to a rejection value equal to 97.6% and a SEC value equal to 0.96 kWh.m^{-3}. Since lower concentrations lead to higher values of rejection and lower values of SEC, it was expected that the concentration value which leads to the optimum values of both responses would be the minimum concentration within the simulation range. To evaluate the optimum conditions considering fixed higher concentrations, the desirability approach was repeated considering higher minimum concentrations. Fig. 6b shows that, for a minimum feed concentration equal to 50 mol.m-3, the optimum effective pressure and temperature values are ΔP_e = 21.3 bar and T = 30° C, with a rejection value equal to 96.9% and a SEC value equal to 1.32 kWh.m^{-3}. For a minimum feed concentration equal to 75 mol.m^{-3}, the optimum values are ΔP_e = 24 bar and T = 30° C, with a rejection value equal to 96.2% and a SEC value equal to 1.54 kWh.m^{-3}. This indicates that, for more complex and concentrated solutions, higher pressures are required. In these cases, the use of energy recovery devices can be an alternative to reduce the energy consumption and achieve higher rejections.

IV. Conclusions

In the present work, the effects of operating parameters such as effective pressure, temperature and feed concentration on rejection and specific energy consumption of the NaCl permeation process through NF90 nanofiltration membrane were investigated. A second order polynomial model was obtained to predict the performance variables with changing process parameters. It was found that that the effective pressure has the most significant effect on both rejection and SEC responses, followed by the feed concentration. The temperature has little effect comparing to other parameters and it is more significant for rejection. The optimum process parameters were determined for different feed concentrations and was found to be ΔP_e = 16 bar, T = 30° C with a rejection value equal to 97.6% and a SEC value equal to 0.96 kWh.m^{-3} considering the minimum feed concentration (25 mol.m^{-3}) and ΔP_e = 24 bar and T = 30° C, with a rejection value equal to 96.2% and a SEC value equal to 1.54 kWh.m^{-3} for maximum feed concentration (75 mol.m^{-3}).

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