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Using response surface methodology and Box-Behnken design in the study of affecting factors on the dairy wastewater treatment by MEUF

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Abstract. Micelle-Enhanced Ultrafiltration (MEUF) is a membrane separation processes that improving ultrafiltration process with the formation of micelles of the surface active agents. Surface active agents are widely used to improve membrane processes due to the ability to trap organic compounds and metals in the treatment of industrial waste water. In this study, surface active agents are used to improve micelle-enhanced ultrafiltration (MEUF) to reduce chemical oxygen demand (COD), total dissolved solid (TDS), turbidity and clogging the membrane in dairy wastewater treatment. Three important operational factors (anionic surface active agent concentration, pressure and pH) and these interactions were investigated by using response surface methodology (RSM) and Box-Behnken design. Results show that due to the concentration polarization layer and increase the number of Micelles; the anionic surface active agent concentration has a negative effect on the flux and has a positive effect on the elimination of contamination indices. pH, and the pressure have the greatest effect on flux. On the other hand, it could be stated that these percentages of separation are in the percentages range of Nano-filtration (NF). While MEUF process has higher flux than NF process. The results have been achieved at lower pressure while NF process needs high pressure, thus making MEUF is the replacement for the NF process.

Keywords: MEUF, surface active agents, Box-Behnken, dairy waste water

1. Introduction

Membrane ultrafiltration (UF) is mainly used in separation of solutions that are a mixture of favorable and unfavorable materials. The driving force required for transference through the membrane is a pressure difference of about 2-10 bars. UF membranes are able to separate genera with a molecular weight of about 300-500000 Dalton and within the pore size of 10-1000 Å. This method is usually used in separation of macromolecules including proteins from low molecular weight solvents. Micellar enhanced ultrafiltration (MEUF) is a membrane separation process which improves the performance of ultrafiltration process by forming a micelle from surfactants (Landaburu-Aguirre *et al*. 2010).

Surfactants are widely used in industries including glue, foaming and wetting agents, emulsifiers and the like thanks to their ability in dissolving particles. In addition to this capacity, owing to entrapping toxic organic compounds and metals, these materials have attracted a great deal of attention for environmental uses including treatment of water and soil contaminants and are widely used for improving MEUF separation technologies. It was first used by Scamehorn *et al.* (1980) to control wastewater contamination and separate soluble organic compounds including 4-tert butyl-phenol as well as polyvalent metal ions such as cadmium and zinc from aqueous solutions. The main rule of separation in this process is that surfactants (anion, cation or nonionic) are added to the aqueous contaminant flow (wastewater). The monomers of the superficial active agent accumulate and form a complex at critical micelle concentration (CMC). Formation of the complex causes increased size of the contaminant molecules (the hydrodynamic diameter of micelle is larger than the diameter of pores of ultrafiltration membrane) in the wastewater. Therefore, MEUF process is used for separation of different organic or inorganic contaminants from aqueous phase. Non-bonding particles in the monomers of the official active agent easily move through the membrane along with water and go towards the permeate flow. In this process, the contaminant's separation efficiency and the flux of permeate are dependent on the characteristics of the membrane and the contaminant as well as different operational conditions. The unique feature of MEUF processes that it enjoys both the high efficiency of reverse osmosis process and high permeate flux of ultrafiltration process. In addition, the performance of rejection of materials for MEUF process regarding low molecular weight materials is almost similar to that of the reverse osmosis and Nano filtration process. Therefore, MEUF process may be used as a gradual process to overcome the intrinsic limitations of reverse osmosis process (Puasa *et al.* 2011).

Surfactants have a hybrid property (hydrophilicity and hydrophobicity) in their molecular structure. They also have a molecular part with a poor affinity for solvent (lyophobic group) and a molecular part with a strong affinity for solvent (lyophilic group). The hydrophobic tail is usually a long hydrocarbon chain or cyclohexane ring. On the other

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hand, the hydrophilic head is a very powerful polar or charged group. Given the nature and properties of the hydrophilic group, surfactants are categorized into anionic (negatively charged), cationic (positively charged), Zwitterion (positively and negatively charged) and nonionic (without a clear ionic charge) groups (Landaburu-Aguirre 2012).

Anionic surfactants are a group of surfactants where the polar part of the molecule consists of an anion. This means that the head group in them has a negative charge. Therefore, they are able to interact with (absorb) contaminants with a positive charge such as metal ions from wastewater. Alkaline or alkaline earth salts of carboxylic acid, ester salts of sulfuric acid and phosphoric acid belong to this group and Sodium n-Dodecyl Sulphate (SDS) and Linear Alkyl benzene Sulfonate (LAS) are two important examples of them.

The efficiency of MEUF process in separation of organic and inorganic contaminants is dependent on various operational factors including pressure difference, type and concentration of the surfactant, pH of the solution, presence of additives, type of membrane and the size of its pores and eventually presence or absence of nonionic surfactants and salt. Considering the significance of treatment of industrial wastewaters and the progressive application of membrane processes for treatment of water and wastewater, many researchers have been trying to present better methods for treatment and improve the properties of membranes (Samper *et al.* 2009, Baek *et al.* 2004, Monem *et al.* 2011).

Luo *et al.* investigated different conditions and compared their results with Nano filtration (Luo *et al.* 2012, 2011a). They found that the best feed pH for treatment by Nano filtration and spinning disk module is 7-8 (Luo *et al.* 2011a). These researchers also treated synthetic milk wastewater by a two-stage process of ultrafiltration and Nano filtration and found that this method enjoys a greater efficiency and lower membrane fouling in comparison with the Nano filtration method (Luo *et al.* 2011b). Similar works have been studied by Carta *et al.* in treating a synthetic dairies wastewater by aerobic reactors (Carta *et al.* 2004), Arbeli *et al.* for dairies wastewater by an anaerobic tank (Arbeli *et al.* 2006) and Yavuz *et al.* for treating the real wastewater of a dairies factory by electrocoagulation (EC) and electrofenton (EF) methods (Yavuz *et al.* 2011). In this paper, Box-Behnken's method has been used to investigate the effect of SDS, pH and pressure on MEUF function in dairy wastewater treatment.

2. Material and methods

In order to extract the model and find the effect of different parameters, surface response method was used. This method a series of statistical techniques used in the optimization of processes where the response of interest is affected by a number of variables (Talebpour *et al.* 2009). It is also a valuable instrument for investigation of the role of factors and their effect on the reaction, as eventually the effect of variables on the response is plotted as a graph, on which maximum and minimum points are clear (Bezerra *et al.* 2008). This method is even able to evaluate the extent of effect of parameters and their effect on each other (Switzar *et al.* 2011). In this method, a model is defined for every dependent variable and it expresses the main and interactive effects of the influential variables on it separately. The defined model is mostly in the form of Eq. 1.

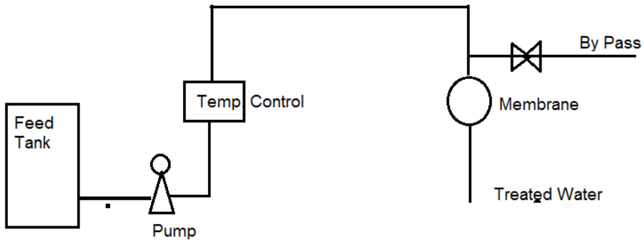

Fig. 1 Membrane pilot schematic

Table 1 Levels and factors for Box-Behnken design

	Factor	Unit	level		
			-	0	+
A	SDS	mM	2	5	8
B	pressure	bar	2	3	4
C	pH		2	7	12

Table 2 Membrane pilot specifications

tank volume	Feed flow rate	Type of heat exchanger	Pump type
43 liters	Lit/min 2.5	plates	Piston

$$Y_n=D_0+\sum D_i x_i+\sum\sum D_{ij}x_i x_j+\sum D_{ii}x_i^2+\varepsilon \quad (1)$$

where, Y_n is one of the responses, D_0 represents a constant coefficient, D_i is linear effects, D_{ii} denotes square effects and D_{ij} shows interactive effects.

Based on the model presented in Eq. 1, to investigate the effect of SDS concentration, pressure and pH on the extent of COD rejection, TDS, turbidity and flow rate, a Quadratic equation has been considered as Eq. 2, in which the variables A, B and C represent the SDS concentration, pressure and pH, respectively.

$$Y = D_0+ D_1.A+ D_2.B+ D_3.C+ D_{11}.A^2+ D_{22}.B^2+ D_{33}.C^2+ D_{12}.A.B+ D_{13}.A.C+ D_{23}.B.C \quad (2)$$

This equation has 10 constant coefficients. For calculation of these coefficients, at least 10 experiments under different conditions are required. Box-Behnken design is one of the methods to design experiments and obtain the coefficients in Eq. 2 (Khajeh *et al.* 2010).

The Box-Behnken design is an independent quadratic design in that does not contain an embedded factorial or fractional factorial design. In this design the treatment combinations are at midpoints of edge of the process space and at the center. These designs are required 3 levels of each factor.

The experimental designs were performed across 3 levels of pressure difference, 3 levels of SDS concentration, 3 levels of pH by Design Expert 7.

First the polymer membrane was prepared with Dimethylformamide (DMF) solvent, 1-Methylpyrrolidine

Table 3 Properties of waste water

pH	Turbidity(NTU)	TDS(mg/l)	COD(mg/l)
9.3-10.4	7000-7500	1800-2100	2800-3200

(NMP), polyethersoulfone (PES) polymer and polyaniline (Pani) on a film dragger device and installed on multipurpose membrane ultrafiltration device made by Pishtaz Nanofanavar Toos Iranian Company (Fig. 1, Table 2).

The utilized dairies wastewater was sampled and the properties of the wastewater were measured (Table 3). The utilized surfactant was sodium dodecyl sulfate (SDS) from a series of anionic agents with the molecular weight of 288.372 and chemical formula of $NaC1_2H_{25}SO_4$ with a purity of 99%, which was purchased from Merck Co., Germany.

To perform each experiment, the weighed surfactant was poured into 100 cc distilled water and stirred by a magnetic stirrer for 15 min at 300 rpm. Next, this solution was added to 8 L of wastewater and the final solution was stirred for 15 min and after brining the pH to the intended value, it was poured into the feed container of the device using acid and base. Then, the pressure was brought to the desired value using the pressure adjustment valve and once the flux reached the steady state, the output samples were collected at certain time intervals.

After each treatment experiment, the wastewater sample was analyzed in order to evaluate its potential in rejecting each of the contamination indices. The investigated indices are COD, TDS and turbidity. To determine the turbidity, turbidity meter was used and for determining the dissolved solid particles, TDS meter was employed. Finally, to determine the chemical oxygen demand (COD), spectrophotometry method was used.

3. Results and discussion

Following calculation of the extent of rejection, the obtained results were recorded in Table 4. To calculate the rejection percentage of materials by the membrane under different operational conditions, Relation 3 is used (Huang *et al.* 2007).

$$R(\%)=(1-C_p/C_f)\times 100 \quad (3)$$

In this equation: R: Rejection percentage, C_p: Product property, C_f: Feed property.

The accuracy of the presented model was tested by analysis of variance. Analysis of variance shows that the selected model is significant and P-value below 0.05 suggests that the factors and the fitted model are very significant and are not random or due to noise and disturbance. The insignificance of the lack-of-fit data is also the last confirmation for the model. The R value for the equations is above 99%. This suggests that over 99% of the changes in the response can be accounted for by the model (Rahmanian *et al.* 2011a, 2011b). Based on the coefficients obtained from the design, Relation 2 is converted to Relations 5-8 for each response.

Table 4 Result of experiments

NO	SDS	P (bar)	pH	Rate (LMH)	COD	TDS	Tur. NTU
1	5	3	7	38.58	63.8	44.11	98.95
2	2	3	2	45.31	50.5	33.53	98.49
3	8	2	7	35.08	71.1	50.82	99.24
4	5	4	12	33.79	72.8	50.88	99.29
5	2	2	7	43.08	56.4	38.00	98.66
6	5	4	2	42.36	54.9	36.74	98.62
7	5	2	2	42.34	57.2	39.75	98.75
8	2	3	12	37.76	65.9	45.25	99.01
9	2	4	7	40.00	60.1	40.78	98.84
10	8	3	12	31.84	77.1	54.69	99.42
11	5	3	7	38.48	63.8	44.19	98.90
12	5	3	7	38.58	63.8	44.11	98.95
13	8	4	7	36.15	67.7	46.83	99.07
14	5	3	7	38.18	63.7	44.21	98.89
15	5	2	12	35.81	70.2	49.07	99.14
16	5	3	7	38.50	63.8	44.10	98.97
17	8	3	2	39.39	61.6	42.96	98.89

$$COD = +39.7612+3.6066A +1.3031B +0.81C-0.5821A.B-0.2455B.C$$

$$TDS = 25.5387-3.265A+0.833383B-0.4495C-0.56417A.B-0.241B.C$$

$$Flow\ Rate = 53.325-2.025A-1.511B-0.4475C+0.345837A.B-0.1025B.C$$

$$Turbidity = +98.0847 +0.15542 A +0.0111B -0.0535 C -0.029375A.B +0.01387 B.C$$

To examine the role of each factor on the expected results, three-dimensional plotting of Relations 5-8 has been provided in Figs. 2-5. Basically, wastewater turbidity reduced by using membrane processes. As can be seen in Fig. 2, all three factors have linear influence on turbidity. But this effect is not high (about 1%).

According to Fig. 3 can be seen with increasing pressure difference, pH and concentration of surface active agent, rejection of TDS increased.

The effects of changes in the concentration of the variables on the extent of COD removal are shown in Fig. 4. The results indicate that the extent of COD rejection increases with elevation of the surfactant's concentration at any pressure difference. This behavior is due the fact that the probability of micelle formation in the layer close to the membrane surface increases due to concentration polarization phenomenon. This means that a very large number of the surfactant monomers has begun creating micelle in the concentration polarization layer, as the surfactant concentration in this layer is larger than its concentration in the wastewater solution bulk (Rahmanian *et al.* 2010, El Abasi *et al.* 2011). It is predicted that at high concentrations, the effect of this improvement diminishes, since the number of linking points or the sites of solution of organic compounds does not increase effectively with elevation of concentration (Zeng *et al.* 2011).

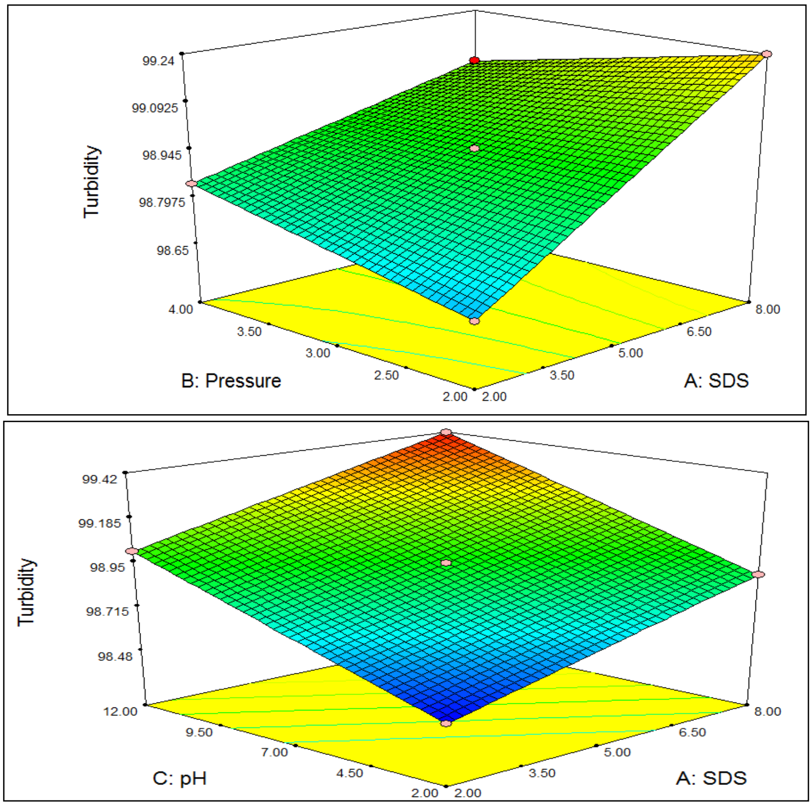

Fig. 2 Influence of pH, SDS and pressure on turbidity

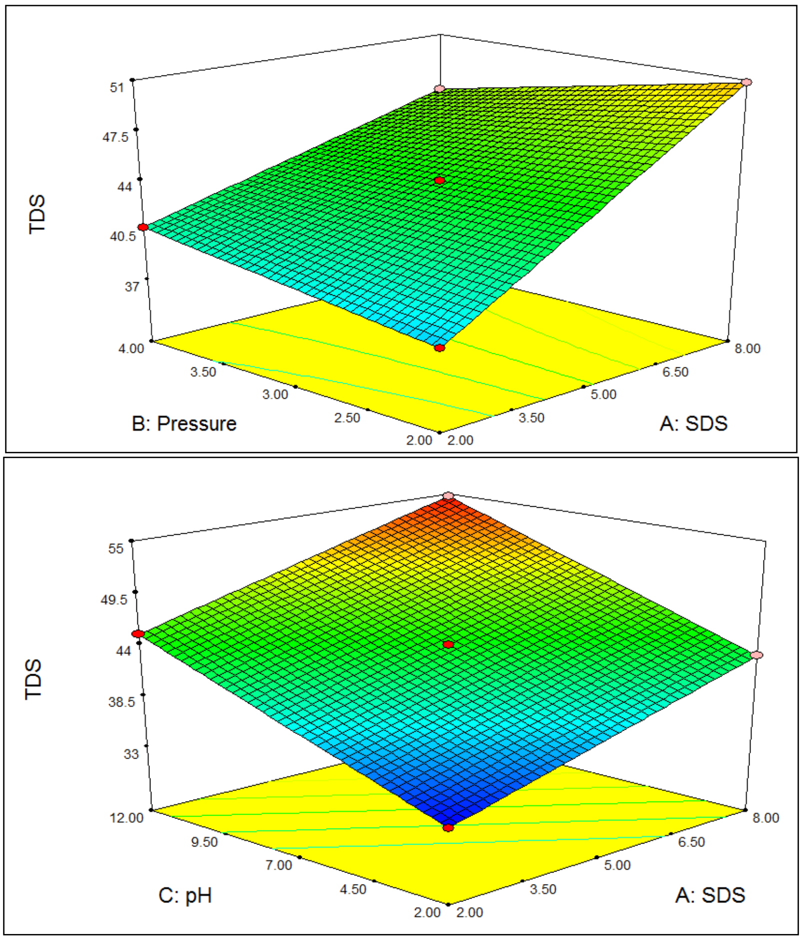

Fig. 3 Influence of pH, SDS and pressure on TDS

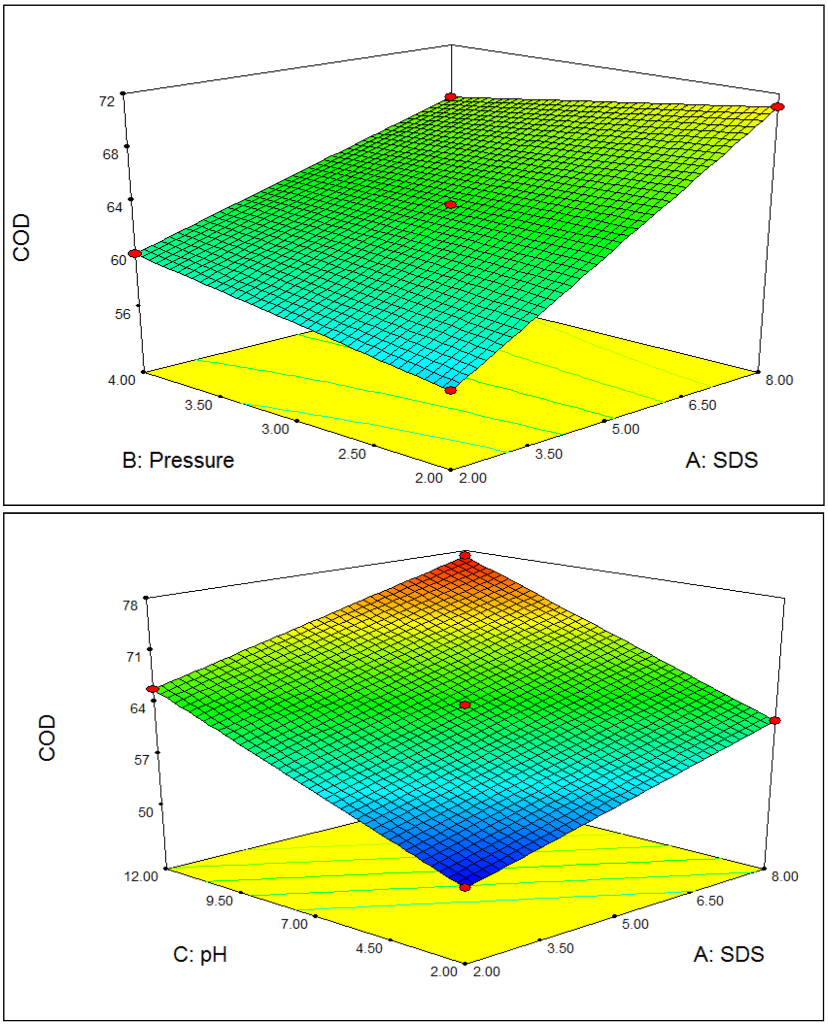

Fig. 4 Influence of pH, SDS and pressure on COD

Furthermore, according to Fig. 4, at high surfactant concentrations, the slope of elevation of COD rejection percentage diminishes with elevation of pressure difference. This is because at high pressure differences, micelles become compressed and the solubility capacity for contaminants in them declines (Purkait *et al.* 2004, Lue *et al.* 2010). The results indicate that the greatest COD rejection is obtained at the pressure difference of 12 bar and surfactant concentration of 8 mM. With the increase in the surfactant concentration, the probability of formation of the number of micelles increases. Also, the probability of compression of micelles increases at higher pressure differences. Therefore, their solubility capacity for the contaminant particles decreases. On the other hand, with the increase in the effective driving force (pressure difference), due to the increase in the transference of contaminant particles from the membrane, the rejection percentage declines (Huang *et al.* 2010).

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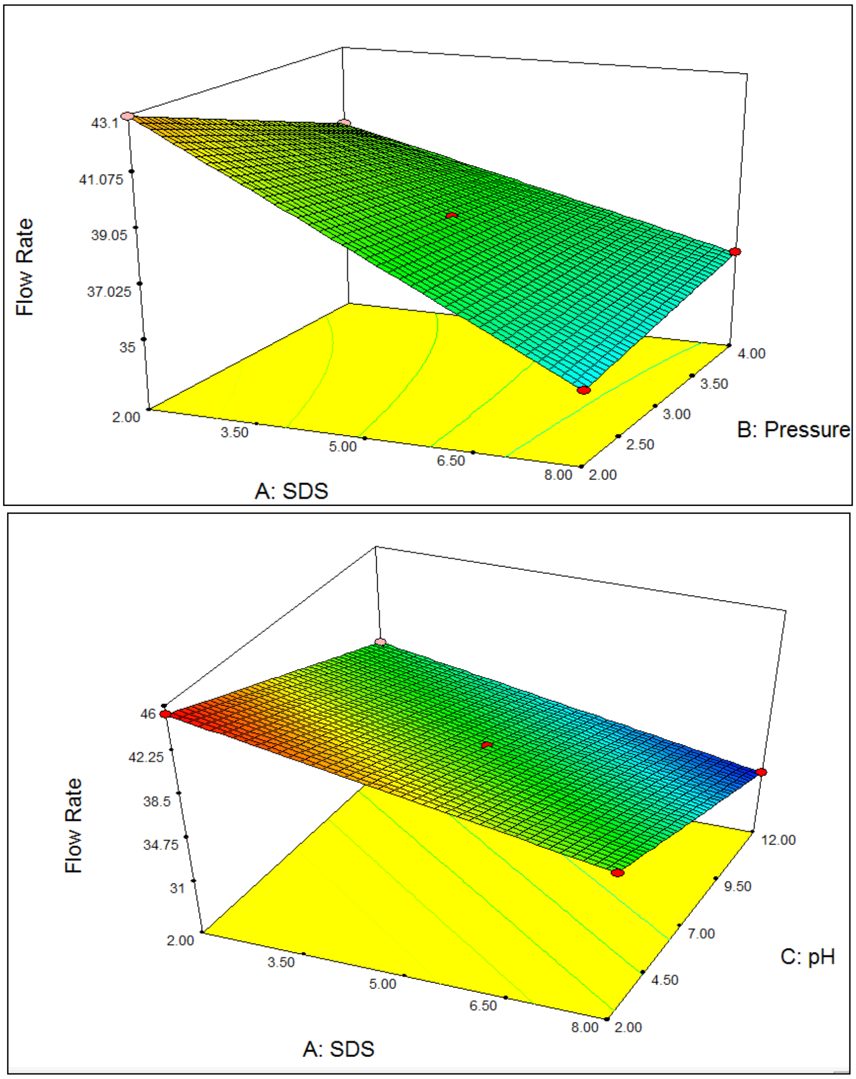

Fig. 5 Influence of pH, SDS and pressure on Flow rate

declines (Huang *et al.* 2010).

Flow rate is one of the most important responses in evaluation of performance of MEUF systems. Fig. 5 represents the influence of pH and SDS concentration on flow rate. Flow rate decrease by pH and SDS concentration increasing.

As can be seen, a decrease in the permeate flux is observed in the very beginning of the operation (with the increase in the surfactant concentration across all pressure differences). This suggests that a severe fouling is developed with the increase in the surfactant concentration. Reduction of the permeate flux for the surfactant concentrations below the critical concentration can be due to several reasons. First, presence and sedimentation of the surfactant in the wastewater, which accumulates on the membrane surface, causing pore fouling and formation of a cake layer on the membrane surface. Second is development of concentration polarization phenomenon, which is due to accumulation of surfactant on the membrane surface. In other words, the surfactant's monomers precipitate like a layer at a distance very close to the membrane surface, thereby accumulating on each other. Therefore, the concentration of these materials increases in this thin layer and reaches the values above the critical concentration of micelle formation. This elevation causes formation of micelle in the layer close to the membrane surface and will result in a significant decrease in the permeate flux. The rate and amount of formation of this layer are influenced by different factors including pressure difference, operational temperature, surfactant concentration, etc.

Membrane eclipse results from the bonding of suspended solids and soluble materials to the membrane surface. In fact, the main constituents of silt water are soluble microbial products (SMP) produced by microorganisms. The study by Nuengiamnong *et al.* showed that the condensed sludge cake layer on the membrane surface contains cells and bacterial-derived materials such as extracellular polymeric substance (EPS) polymeric material (EPS) and soluble microbial production (Naengiamong *et al.* 2005, Yamato *et al.* 2006). Therefore, the use of surfactants before the wastewater enters the membrane system form bacterial material, such as EPS and SMP and thereby reduce the membrane clogging due to the bonding of suspended solids and soluble solids on the membrane surface (Huang *et al.* 2016, Shi *et al.* 2017).

4. Conclusion

Using surface response methodology and principles of Bon-Behnken experimental design, the degree of significance of the factors influencing dairies wastewater treatment (pressure, SDS and pH) was analyzed on four responses of extent of TDS rejection, extent of COD rejection, extent of turbidity rejection and the permeate flux in MEUF process in the form of a biquadratic equation. Then, following validation of the equations in the form of three-dimensional plots, their role was investigated. The results indicate although the effect of surfactant on the turbidity increases with elevation of concentration, it is below 1% and all of the three factors have a linear ascending effect. The surfactant has an enhancing effect on the extent of COD rejection, but the slope of this effect declines with elevation of pressure. TDS rejection has also a similar behavior. The concentration of surfactant and pH on the permeate flux has a descending effect, while elevation of pressure has an increasing effect. It can be concluded that in the range of experiment data, best condition obtain in higher pressure, pH and SDS. Eventually, considering the results obtained in COD and TDS separation from dairies wastewater, the advantage of MEUF process in relation with UF process is clear. On the other hand, it can be stated that these separation percentages are within the range of separation percentages of nanofiltration membranes (NF). However, the flux of MEUF process is far larger than that of the NE process. Furthermore, these results have been obtained at low pressures, while NF process works at high pressures, thus MEUF process is a very suitable substitute for NF process.

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