Journal of Hazardous Materials 192 (2011) 585–592

Contents lists available at ScienceDirect

Journal of Hazardous Materials

journal homepage: www.elsevier.com/locate/jhazmat

Fuzzy modeling and simulation for lead removal using micellar-enhanced ultrafiltration (MEUF)

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ARTICLE INFO

Article history:
Received 16 February 2011
Received in revised form 15 May 2011
Accepted 17 May 2011
Available online 2 June 2011

Keywords:
Modeling
Fuzzy logic
Box–Behnken design
MEUF
Lead

ABSTRACT

In the present paper, a three factor, three-level response surface design based on Box–Behnken design (BBD) was developed for maximizing lead removal from aqueous solution using micellar-enhanced ultrafiltration (MEUF). Due to extremely complexity and nonlinearity of membrane separation processes, fuzzy logic (FL) models have been driven to simulate MEUF process under a wide range of initial and hydrodynamic conditions. Instead of using mathematical model, fuzzy logic approach provides a simpler and easier approach to describe the relationships between the processing variables and the metal rejection and permeation flux. Statistical values, which quantify the degree of agreement between experimental observations and numerically calculated values, were found greater than 91% for all cases. The results show that predicted values obtained from the fuzzy model were in very good agreement with the reported experimental data.

1. Introduction

Widespread use of heavy metals in industrial applications as well as discharging this contaminated wastewaters into environment is a serious problem [1]. Lead is classified as a hazardous waste and is highly toxic to humans, plants and animals, which tend to be accumulated in the food chain and cause serious health problems such as anemia, brain damage, anorexia, vomiting and malaise [2,3]. Conventional methods of heavy metal-containing wastewater treatment include precipitation [4], electro-deposition [5], evaporation, ion exchange [6], and crystallization [7]. However, industries are looking for competing alternative technologies which may overcome some of the inherent disadvantages of these methods [8]. Membrane separation processes of different types of membranes show great promises for commercial application [9–13]. However, both RO and NF processes use relatively "dense" membranes. Permeability of these membranes is low and thus, to get the desired throughput, a high operating pressure is required [14]. Recently, a modified ultrafiltration membrane separation process 'micellar-enhanced ultrafiltration (MEUF)' has been used for the removal of various organic and/or inorganic pollutants from aqueous phase [15–20].

Traditionally, the study of MEUF has been conducted using the one variable at a time approach, i.e., a single factor is varied while all other factors are kept unchanged for a particular set of experiments. Such conventional or classical methods of experimentation usually involve many experimental runs, which are time consuming, ignore interactions effects between the considered parameters of the process and lead to a low efficiency in optimization issues [21,22]. The application of statistical experimental design for membrane systems seems to be the best methodology for process control and optimization.

This study reports data of a metal-polluted wastewater model in order to evaluate the efficiency of MEUF for the removal of Pb^{2+} from aqueous solutions. The factors that have been considered are operative pressure difference, membrane molecular weight cut-off, molar ratio of surfactants to the solute, kind of surfactant and additives, pH, ionic strength, etc. According to the previous published research on MEUF, three factors including the surfactant concentration, solution pH and the surfactant to metal concentration ratio (S/M) have most significant effect on response variables. In this research, MEUF process using Box–Behnken as an experimental design was performed and the ability of FIS was evaluated to

Abbreviations: ARE, average relative error; AARE, absolute average relative error; SD, standard deviation; MEUF, micellar-enhanced ultrafiltration; CMC, critical micellar concentration; FL, fuzzy logic; FIS, fuzzy inference system; RSM, response surface methodology; BBD, Box–Behnken design; MFs, Gaussian membership functions; TMP, transmembrane pressure (bar).

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doi:10.1016/j.jhazmat.2011.05.051

Nomenclature

μ_f	membership function
R	filtration efficiency
C_f	concentration of Pb^{2+} (mg/L) in the feed solution (mM)
C_p	concentration of Pb^{2+} in the permeate stream (mM)
P_i	inlet pressure (bar)
P_o	outlet pressure (bar)
P_p	permeate pressure (bar)
Q	permeate volume (m^3)
A	area of membrane (m^2)
J_p	permeation flux (L/m^2 min)
X Y and Z	linguistic variables

modeling and simulation. It is unnecessary to carry out extensive pilot plant testing for data collection, which can be interpolated with potentially great savings, both in time and in cost by using this procedure.

1.1. Box–Behnken designs (BBD)

Response surface methodology (RSM) is a collection of mathematical and statistical techniques that can be used for studying the effects of several factors at different level and their influence on each other. A further benefit of using the RSM design is the reduction of number of experiments needed rather than a full experimental design at the same level. Another advantage of RSM designs is that it allows the effects of a factor to be estimated at several levels of the other factors studied. In this study, Box–Behnken designs (BBD) were applied as experimental design strategies to investigate MEUF process performance. One of the main advantages of Box–Behnken design matrix is that it does not contain combinations for which all factors are simultaneously at their highest or lowest levels. So this design is useful to avoid experiments performed under extreme conditions [23]. The advantages of Box–Behnken designs include the fact that they are all spherical designs and require factors to be run at only three levels. The designs are also rotatable or nearly rotatable [24].

1.2. Fuzzy model

Fuzzy modeling is one of the most powerful techniques to estimate input–output relation in complex nonlinear systems. Zadeh [25] introduced this concept, in which fuzzy numbers are assigned to variables to represent uncertainties. A fuzzy number describes the relationship between an uncertain quantity x and a membership function μ, which ranges between 0 and 1 [26]. There are two types of fuzzy inference systems: Takagi–Sugeno models [27], and Mamdani models [28]. The output membership functions of Takagi–Sugeno models are either constant or linear functions of the input variables, whereas the output membership functions of Mamdani models are fuzzy sets, which can incorporate linguistic information into the model. Mamdani models are more suitable for modeling qualitative information is used in this work [29]. Fuzzy logic starts with and builds on a set of user-supplied human language rules. The fuzzy systems convert these rules to their mathematical equivalents. This simplifies the job of the system designer and the computer, and results in much more accurate representations of the way systems behave in the real world. Additional benefits of fuzzy logic include its simplicity and its flexibility. Fuzzy logic can handle problems with imprecise and incomplete data, and it can model nonlinear functions of arbitrary complexity [30,31].

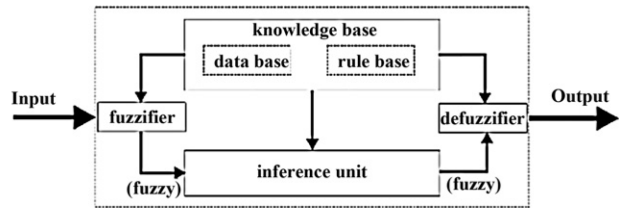

Fig. 1. Fuzzy inference system.

Fuzzy logic provides advantages over ANNs in that it can better handle noisy and distorted multivariate data. Another advantage of fuzzy modeling lies in the training phase where it is possible to establish rules even in the absence of data relying instead on expert knowledge to constitute the rule base. Importantly, the resultant fuzzy model and its rules can be more easily interpreted compared with ANNs [32]. Fuzzy models describe input–output relationships by fuzzy if-then rules (fuzzy propositions). They make use of fuzzy sets and approximate reasoning to find an overall 'good enough' solution to a particular problem domain without using detailed first-principle knowledge of that domain. Fuzzy rules may be formulated on the basis of expert knowledge of the system [33]. The Mamdani scheme is a type of fuzzy relational model where each rule is represented by an IF–THEN relationship. It is also called a linguistic model because of both the antecedent and the consequent are fuzzy propositions [34]. The model structure is manually developed and the final model is neither trained nor optimized. The output from a Mamdani model is a fuzzy membership function based on the created rules. Since this approach is not exclusively reliant on a data set, with sufficient expertise on the system involved, a generalized model for effective future predictions can be obtained [35].

Mamdani models structure following rule base (where X, Y and Z are linguistic variables).

R_i: if X is A_i and Y is B_i,. . . then Z is $C_{i,}$. . ., $i = 1\ldots n$

Given the input fact (x_0, y_0), the goal is to determine the output "Z is C".

Each fuzzy expert system model is developed through three stages, i.e., fuzzification, inference engine and defuzzification as depicted in Fig. 1.

2. Experimental

2.1. Chemicals

All reagents used were of analytical grade. Lead (II) acetate ($Pb(CH_3COO)_2$, 99% purity), with a molecular weight of 379.34 and sodium dodecylsulfate (SDS) as a anionic surfactant (>99% purity), with a molecular weight of 288.38 were obtained from Merck company. Experimentally, the (critical micelle concentration) CMC is usually was found by plotting a graph of electric conductivity versus concentration. An abrupt change of slope marks the CMC. Its plot against the concentration of surfactant yields two straight lines from whose intersection the CMC is obtained [3]. The CMC of SDS was determined 2.34 g/L using conductivity meter method. Distilled water was used in all of the experiments. HCl and NaOH, were used for pH adjustment, both having concentration of 1N.

2.2. Membrane

The experimental spiral-wound ultrafiltration regenerated cellulose membrane with an effective area of 1.83 m^2 was supplied by Amicon (PL series, Millipore) and used without further treatment. The membrane cut-off was 10 kDa.

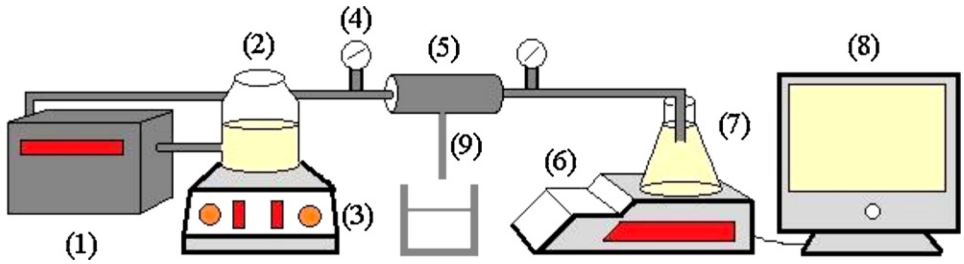

Fig. 2. Schematic of MEUF process: (1) peristaltic pump, (2) feed reservoir, (3) magnetic stirrer, (4) monometer, (5) spiral-wound ultrafiltration module, (6) balance, (7) permeate stream reservoir, (8) computer in which data of permeate weight are registered, (9) retentate stream.

2.3. Experimental setup and micellar-enhanced ultrafiltration experiments

All UF experiments were carried out in a batch stirred cell (Amicon 8200, Millipore), with an initial volume of 100 mL. The cell was stirred with a magnetic stirrer at 300 rpm. In the preparation of feed solutions, lead acetate and surfactant (SDS) were mixed at the required concentrations according run number. The applied pressure was adjusted by pressurized air at 60 kPa, and the operating temperature was $25 \pm 2\,^{\circ}$C. A peristaltic pump Model XXX80 (Millipore Co.) with constant flow and 50 kPa inlet pressure was used to provide cross-flow ultrafiltration. The TMP of cross-flow UF was evaluated by two pressure gauges before inlet and at the outlet of system, respectively.

The used membrane was immediately flushed at room temperature for 15 min at 60 kPa using distilled water, 0.01 M HCl, 0.1 M NaOH, 1% NaCl. After each step in the cleaning procedure, distilled water was circulated at 60 kPa and room temperature, until the pH of permeate became neutral. When maintained as described above, the membrane exhibited a constant initial permeate flux after use. Fig. 2 shows a schematic diagram of the experimental spiral-wound membrane system set-up assembled.

The spiral-wound UF device was operated in continuous and cross-flow mode which has a much higher flux and much more effective membrane area than the conventional batch cell system. In this study, the permeation flux for the cross flow UF operation is defined as

$$J_p = \frac{Q}{A\ \Delta t} \tag{1}$$

where J_p is the permeation flux (mL/(m^2 s)), Q is the feed volume (mL), A is the area of membrane (m^2) and Δt is time interval of MEUF process.

The effectiveness of MEUF process was represented by percentage rejection of Pb^{2+} ions and surfactants as following:

$$R = 1 - \frac{C_p}{C_f} \tag{2}$$

where C_p and C_f denote the concentrations of solutes in permeate and feed streams, respectively. TMP is the transmembrane pressure which can be calculated by the following equation:

$$\mathrm{TMP} = \frac{P_i + P_o}{2} - P_p \tag{3}$$

where P_i and P_o are inlet and outlet pressures, respectively, and P_p is permeate pressure.

2.4. Measurement and analysis

The permeate flux was measured continuously and gravimetrically using a digital balance laboratory scale that was connected to a computer and monitored via one by flow program. Conductivity and pH were measured with a Crison microCM 2200 conductivity meter and a Shimadzu 2000 pH meter analyzer, respectively. The Pb^{2+} ion concentration in permeate was analyzed by atomic absorption spectrometry (GBC, 908AA Model) at 228.8 nm. The idealized calibration or standard curve of atomic absorption spectrometry is stated by Beer's law that the absorbance of an absorbing analyte is proportional to its concentration; air–50% oxygen–acetylene flame source was used for Pb^{2+} concentration measurement. The rule of thumb is that a minimum of five standards and a blank should be prepared in order to have sufficient information to fit the standard curve appropriately. If the sample concentration is too high to permit accurate analysis in linearity response range, there are three alternatives that may help bring the absorbance into the optimum working range: (1) sample dilution, (2) using an alternative wavelength having a lower absorptivity, (3) reducing the path length by rotating the burner hand.

3. Experimental design

The number of experiments (N) that is required for development of BBD is defined as:

$$N = 2k(k-1) + C_o \tag{4}$$

where k is the number of factors and C_0 is the number of central points.

The significant variables like SDS feed concentration, surfactant to metal molar ratio (S/M ratio) and solution pH were chosen as the critical variables and designated as X_1, X_2 and X_3, respectively. The low, middle, and high levels of each variable were designated as −, 0, and +, respectively, and given in Table 1.

As seen in Table 2, the design had only 16 experimental runs, instead of having 27 experimental points if the run was done in 3^3 complete factorial design, as similarly reported by others. The response surface design developed is based on The Box–Behnken design (BBD) with all combinations of the factors at two levels (high, +1 and low, −1 levels) and the center points (coded level 0), which are the midpoints between the high and low levels, is repeated forth. The actual design of experiments is given in Table 2. Center points used to an estimate of pure error between experimental data and fuzzy model predicted data. Another important reason for adding the replicate runs at the design center is that the center points do not influence the usual effect estimates in the design.

Table 1
The levels of variables chosen for the trials.

SDS feed concentration (mM) (X_1)	Molar ratio (SDS/Pb^{2+}) (X_2)	pH (X_3)
2(−)	5(−)	2(−)
4(0)	10(0)	7(0)
6(+)	15(+)	12(+)

Table 2
The Box–Behnken design (BBD) for lead removal by MEUF process.

Std	Run	A:C_{SDS}	B:S/M	C:pH	R_1: rejection (R%)	R_2: permeate flux (mL/(m^2 s))
1	13	2	5	7	77.78	8.21
2	10	6	5	7	99.41	7.45
3	8	2	15	7	77.35	7.71
4	12	6	15	7	90.89	5.56
5	7	2	10	2	55.96	9.02
6	9	6	10	2	56.64	8
7	15	2	10	12	49.12	8.48
8	16	6	10	12	73.61	7.3
9	4	4	5	2	37.27	7.35
10	11	4	15	2	57.38	7.38
11	3	4	5	12	61.93	7.91
12	6	4	15	12	52.86	5.89
13	1	4	10	7	91.2	8.24
14	5	4	10	7	86.11	8.56
15	14	4	10	7	94.34	7.51
16	2	4	10	7	89.19	8.27

4. Results and discussion

4.1. Effect of process variables on rejection rate and permeation flux

The rejection factor increased with increase of the feed SDS concentration while keeping the molar ratio and solution pH at the middle level. No significant enhancements of rejection factor can be seen at a higher SDS concentration because of efficient binding sites do not increase anymore. Furthermore, the permeation flux decreases with the increase of the SDS concentration in mentioned operating conditions.

The Pb^{2+} rejection factor increases gradually with an increase in solution pH at a fixed SDS concentration and molar ratio. This is due to the competition of H^+ trapped on the micelle surface with metal ions. At low pH (acidic condition), there are more H^+ compared with basic condition (pH > 10). Consequently, the effective binding sites are occupied by hydrogen ions. But in solutions with high pH there are less ions with the same charge, as a result Pb^{2+} rejection increased. In addition, the results show that shows that the increasing the solution pH has very little effect on permeation fluxes. For SDS which has CMC of 8.2 mM, the critical S/M ratio is 5 for obtaining metal removal efficiency of greater than 99% for most of the metals investigated. The gained results show adequate consistency with literature [36].

The lead ions presented a high retention at experienced levels that SDS concentrations were changed, even at those surfactant concentrations far below the CMC. This is a consequence of the formation of a SDS–Pb^{2+} precipitate: after the addition of lead ions, the SDS solution immediately turns a brown-black colour, indicating an association of Pb^{2+} with SDS [24]. Theoretically, there are no micelles formed at surfactant concentrations below the CMC and therefore, there is not any metal ion rejection, but according to our results, the Pb^{2+} rejection was observed when the SDS concentrations are below the CMC. This behavior can be demonstrated due to the concentration polarization effect which hinders the ions permeation through the membrane pores [17].

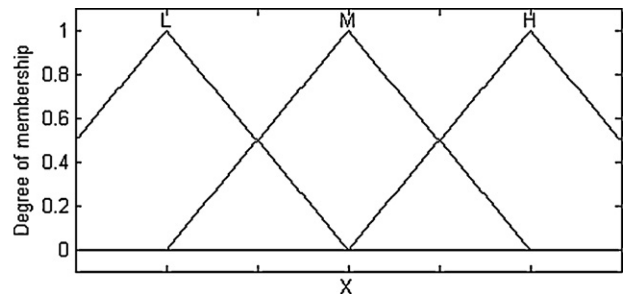

Fig. 3. Membership function of input variables (X: $C_{SDS} \in [1,7]$, S/M $\in [4,16]$, pH $\in [1,13]$) used in this study.

4.2. Development of fuzzy model

From Fig. 3, three inputs to the fuzzy models including: C_{SDS}, S/M and pH. The ranges of these inputs are from [1, 7], [4, 16] and [1, 13] respectively. For the input variables (C_{SDS}, S/M, pH) three membership functions L, M and H are used. They are L, low, M, medium and H, high.

For each input variables, triangle membership functions (MFs) are requested to use. Because all of the MFs are triangle shapes, so we can express these MFs as follows: The triangular curve is a function of vector x, and depends on three scalar parameters a, b, and c, as given by

$$f(x, a, b, c) = \left\{ \begin{array}{cc} 0 & x \le a \\ & \text{or} \\ & x \ge c \\ \dfrac{x-a}{b-a} & a \le x \le b \\ \dfrac{c-x}{c-b} & b \le x \le c \end{array} \right\} \tag{5}$$

or, more compactly, as

$$f(x, a, b, c) = \max(\min\left(\frac{x-a}{b-a}, \frac{c-x}{c-b}\right), 0) \tag{6}$$

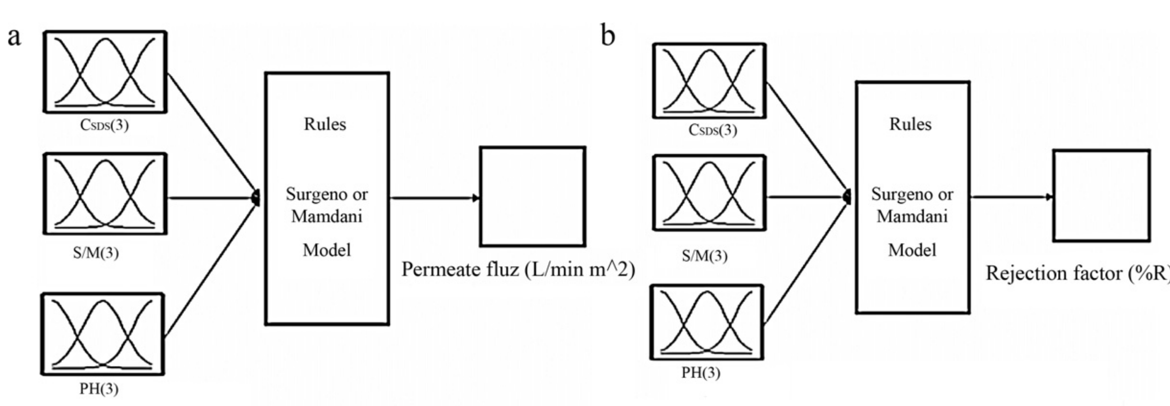

Fig. 4. Graphical representation of the fuzzy model structure (a) permeate flux output (b) rejection factor output.

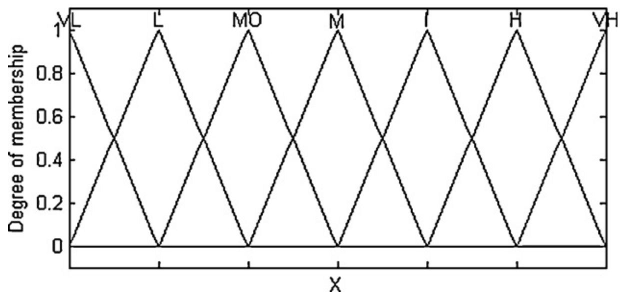

Fig. 5. Membership function of output variables (permeate output, rejection output) used in this study.

The parameters a, c locate the "feet" of the triangle and the parameter b locates the "peak" [37].

In practice, fuzzy modeling is applied using local inferences. That means each rule would be inferred and the results of the inferences of individual rules then would be aggregated. The most common inference methods including: the max-min method, the max-product method and the sum-product method, where the aggregation operator is denoted by either max or sum, and the fuzzy implication operator is denoted by either min or prod. Especially the max–min calculus of fuzzy relations offers a computationally nice and expressive setting for constraint propagation [34,38]. Finally, a defuzzification method is needed to obtain a crisp output from the aggregated fuzzy result.

Popular defuzzification methods include maximum matching and centroid defuzzification. Hence in this study, the fuzzy reasoning results of outputs are gained by aggregation operation of fuzzy sets of inputs and are designed fuzzy rules, where max-min aggregation method and centroid defuzzification method are used. Where fuzzy inference system for permeate flux and rejection factor output is shown in Fig. 4.

Table 3
ARE, AARE and SD for permeate flux and rejection which modeled by fuzzy.

Response variable	Method	% ARE	%AARE	%SD
Permeate flux	Fuzzy	0.371335	2.554286	2.102598
Rejection (R%)	Fuzzy	0.9349388	3.757003	1.998143

From Fig. 5 outputs fuzzy inference system including: permeate flux and rejection factor. The ranges of these outputs are from [5.5, 9.5] and [35,100] respectively. For the output variables seven membership functions VL, L, MO, M, I, H and VH are used. They are VL, very low, L, low, MO, moderate, M, medium, I, increase, H, high and VH, very high as shown in Fig. 5.

The fuzzy model rule surfaces show the relationship between S/M, pH, C_{SDS}, permeate flux; rejection is given in Figs. 6 and 7.

The results of the fuzzy modeling for permeate flux; rejection percentages are shown in Figs. 8 and 9 for each trials of Box–Behnken design respectively. The result reveals that there is a high degree of agreement between the experimental data and the dynamic simulation of this work.

Fig. 10 shows the prediction of extraction percentage of the present model as compared with the experimental data for Pb^{2+} rejection and permeation flux of MEUF process and deviation is found within ±10%.

The accuracy and ability of fuzzy model for predicting MEUF performance was checked with experimental data in Table 3 which

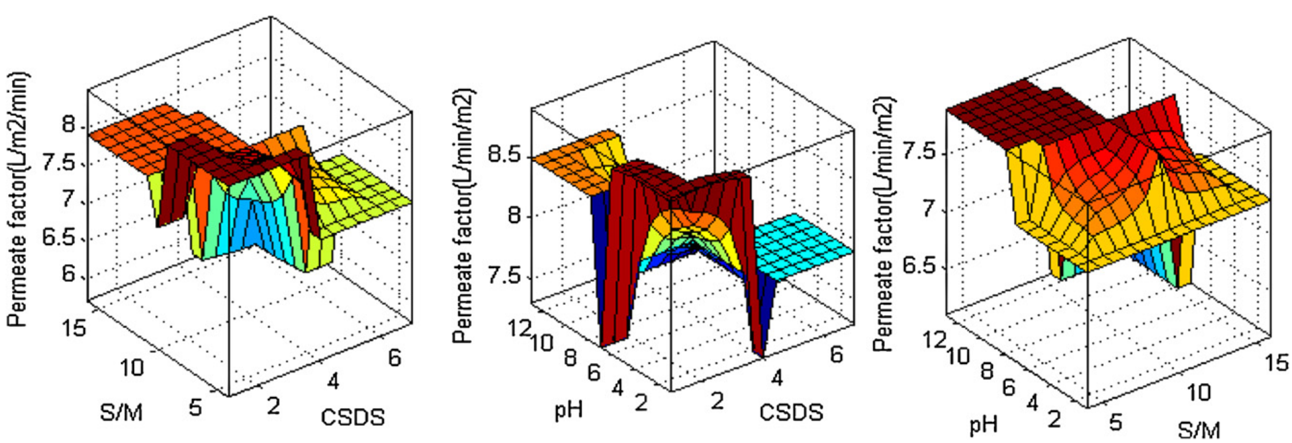

Fig. 6. Fuzzy model rules for permeate flux.

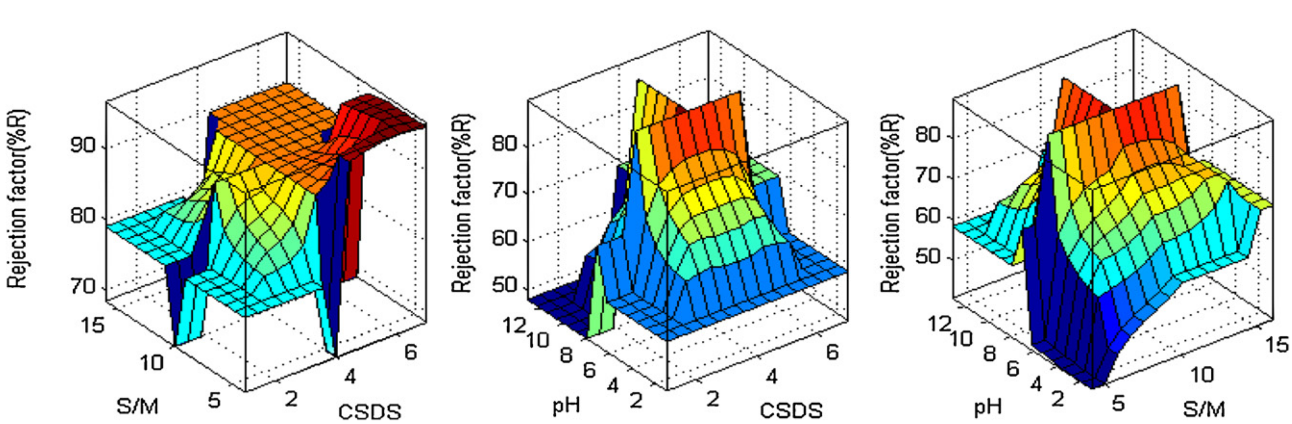

Fig. 7. Fuzzy model rules for rejection factor (R%).

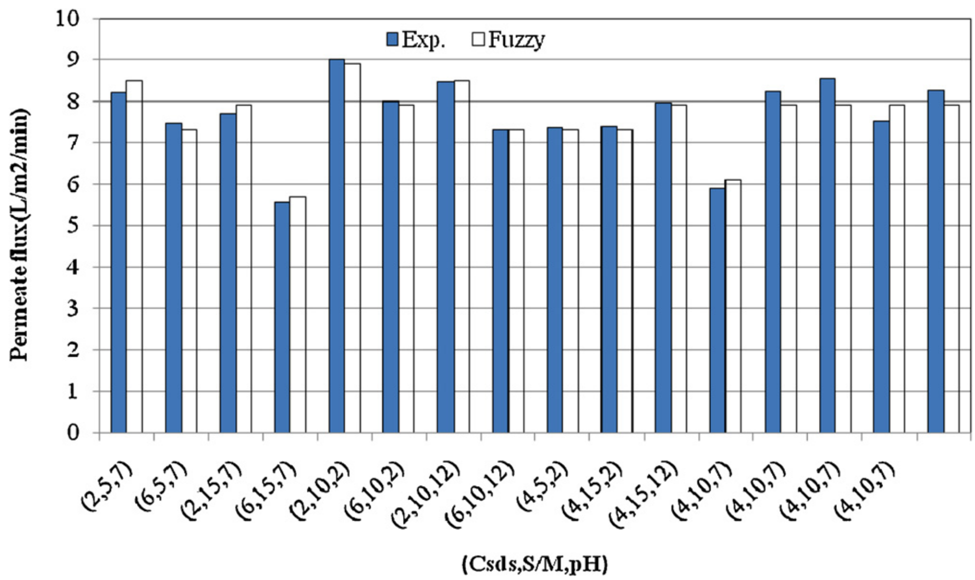

Fig. 8. Comparison between the fuzzy modeling of this work and experimental data for the permeate flux (J_p).

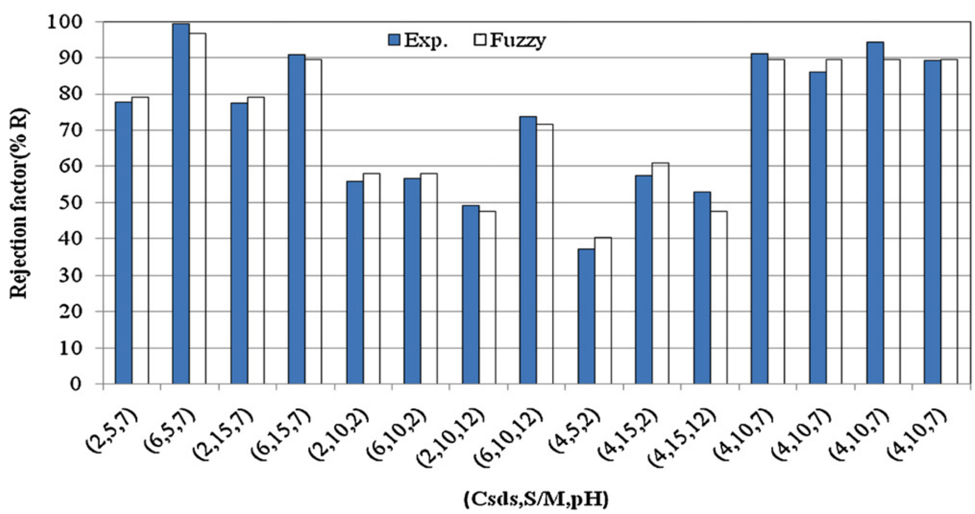

Fig. 9. Comparison between the fuzzy modeling of this work and experimental data for the rejection factor.

indicated that the results from proposed fuzzy model are in better agreement with experimental data. Table 3 reveals average relative error (ARE), absolute average relative error (AARE) and standard deviation (SD) for Table 3 gives ARE, AARE and SD for results of proposed fuzzy model. ARE, AARE and SD are defined as below:

$$\mathrm{ARE} = \frac{1}{N}\sum_{i=1}^{N}\left(\frac{X_{\mathrm{experimental}(i)} - X_{\mathrm{calculated}(i)}}{X_{\mathrm{experimental}(i)}}\right) \quad (7)$$

$$\mathrm{AARE} = \frac{1}{N}\sum_{i=1}^{N}\left(\left|\frac{X_{\mathrm{experimental}(i)} - X_{\mathrm{calculated}(i)}}{X_{\mathrm{experimental}(i)}}\right|\right) \quad (8)$$

$$\mathrm{SD} = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}\left(\left|\frac{X_{\mathrm{experimental}(i)} - X_{\mathrm{calculated}(i)}}{X_{\mathrm{experimental}(i)}}\right| - \mathrm{AARE}\right)^2} \quad (9)$$

Table 4 summarizes a comparison between the present work and the other methods given in the published earlier articles for evaluation membrane performance. However, in most of them, it can be said that the empirical models developed were reasonably accurate. In the present work, a fuzzy logic (FL) models has been used, since it was effective in finding complex non-linear relationships for this membrane separation process.

Table 4
Comparison between the current work and another simulation method.

Type of process	Method	Responses variables	Statistical index	Ref.
Milk ultrafltration	ANN	Permeate flux and the solutes rejection	Average errors <1%	[39]
Copper removal by MEUF	RSM	Rejection coefficient	R^2 = 0.79	[40]
Cross flow milk ultrafiltration	Fuzzy	Permeate flux and the components rejection	–	[41]
Zinc removal by MEUF	ANN	Permeate flux and the rejection rate	R^2 > 0.92	[42]
Current work	Fuzzy	Permeate flux and the rejection rate	R^2 > 0.91	

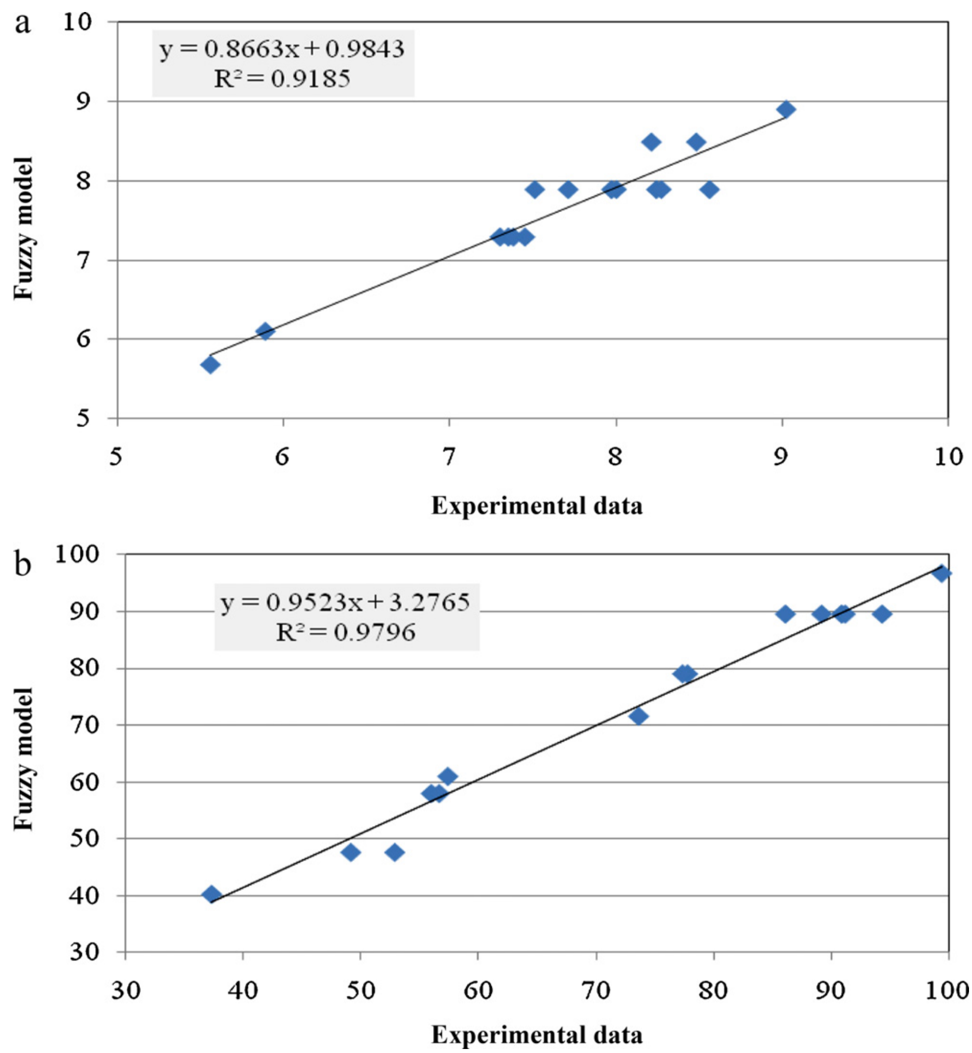

Fig. 10. Comparison of measured and predicted data for model formulation (a) permeate flux, (b) rejection output.

5. Conclusions

In the present investigation, a fuzzy logic based model was developed to predict and modeling of lead removal and permeation flux in separation processes using micellar-enhanced ultrafiltration. The Box–Behnken experimental design method investigates the effect of parameters such as surfactant concentration, solution pH and surfactant to metal concentration ratio (S/M) on MEUF performance. The simulation results reveal that the output variables of MEUF performance could be predicted with a high degree of accuracy. Predicting the performance of a MEUF separation process, prior to its ultrafiltration experiment, was introduced as the most prominent feature of this study. This can offer an intelligent method for evaluation of MEUF presentation instead of try and error method, which is both time and cost consuming.

Acknowledgments

The authors are indebted to Department of Khorasan Research Institute for Food Science and Technology (KRIFST), Mashhad, Iran for their financial assistance and support.

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