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Original Article

Micellar Enhanced Ultrafiltration (MEUF) Using Cetylpyridinium Chloride (CPC) and Hexadecyltrimethylammonium Bromide (HDTAB) Surfactants for Fluoride Rejection: Modeling and Process Optimization

Sodabeh Heidarnejad[1,2,3], **Ali Jafari**[1,3*], **Seyyed Alireza Mousavi**[1,3], **Mohammad Javad Shokoohizadeh**[1,3]

[1]Department of Environmental Health Engineering, School of Health, Kermanshah University of Medical Sciences, Kermanshah, Iran
[2]Student Research Committee, Kermanshah University of Medical Sciences, Kermanshah, Iran
[3]Health, Safety and Environment Technologies Research Core, Health Technology Institute, Kermanshah University of Medical Sciences, Kermanshah, Iran

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***Corresponding author:**
Ali Jafari,
Email: Jafari_a99@yahoo.com

Abstract

Introduction: The current study aimed to optimize micellar-enhanced ultrafiltration (MEUF), a low-pressure membrane method, for removing fluoride from water, a contaminant linked to serious health risks such as fluorosis-through two cationic surfactants.

Methods: In this study, a dead-end ultrafiltration system was employed to remove fluoride using cetylpyridinium chloride (CPC) and hexadecyltrimethylammonium bromide (HDTAB). Important operational parameters such as pH (4-10), initial fluoride concentration (4-10 mg/L), and surfactant concentration (0.5-1.5 mM) were modeled and optimized via Response Surface Methodology (RSM) based on a Box-Behnken design.

Results: Analysis of variance (ANOVA) confirmed the high significance of the developed quadratic models, with regression coefficients (R^2) of 0.99 and 0.97 for CPC and HDTAB, respectively. The optimal removal efficiencies were 94.5% for CPC (at pH 8, 1.2 mM CPC, 8 mg/L F^-) and 89.6% for HDTAB (at pH 7, 1.4 mM HDTAB, 10 mg/L F^-). Under the optimum conditions, flux values of 268.1 L/m^2.h and 276.5 L/m^2.h were also obtained for CPC and HDTAB, respectively.

Conclusion: It was found that MEUF is an effective technique for fluoride rejection. And the RSM successfully modeled and optimized the process, with CPC demonstrating superior performance and model predictability compared to HDTAB. These observations suggest that the proposed system can be reliably applied to larger-scale fluoride removal from aqueous solutions.

Keywords: Fluoride removal, Micellar-enhanced ultrafiltration, Surfactant, Response surface methodology, Parameter optimization, Water treatment

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Introduction

The World Health Organization (WHO) recommends a fluoride concentration of 0.5 to 1.5 mg/L for safe drinking water.[1] Elevated fluoride levels in groundwater and the environment primarily stem from inorganic fertilizer use, untreated effluent discharges, and hydrological processes. While calcium-rich rocks like fluorspar help retain fluoride in soil, the application of inorganic fertilizers reduces the soil's capacity to bind it.[1] Exceeding the recommended limit can lead to dental and skeletal fluorosis, as well as other skeletal problems.[1] Fluoride intake from various water sources has been reported across different regions, with the level of exposure determining the specific health effects and risks.[2] To mitigate these risks, excess fluoride must be reduced to the WHO guideline value using appropriate treatment techniques.[1] Commonly used methods for fluoride removal from water include adsorption, membrane technology, and coagulation-flocculation.[3] In recent years, membrane processes have been widely studied and are considered effective for this purpose.[4]

Membrane technologies offer numerous benefits, such as leaving the aqueous phase unchanged, producing no intermediate products or sludge, allowing flexible scalability and continuous operation, enabling integration with other units, and removing various pollutants simultaneously.[5,6] However, these systems are frequently linked to substantial operating expenses and high energy demands. As a result, considerable research efforts have been directed toward the development of more efficient membrane technologies.[7]

While high-pressure membrane processes like reverse osmosis (RO) can sufficiently remove fluoride, their low permeability, high capital investment, and significant energy requirements often limit their feasibility.[8] In contrast, low-pressure ultrafiltration (UF) is widely applied in industry for separation, concentration, recovery of valuable materials, and effluent recycling[5] through the sieving of larger particles. However, conventional UF is inefficient for removing small ions like fluoride.

Micellar-enhanced ultrafiltration (MEUF) has been proposed as a substitute technology to overcome these limitations. This process uses surfactants to create conditions where pollutants are entrapped within micelles, which are then removed via low-pressure UF.[5]

MEUF is particularly effective for removing various metals and organic pollutants such as dyes from solution.[9] A micelle is an aggregate of surfactant molecules that forms in solution when the surfactant concentration exceeds the critical micellization concentration (CMC). Ionic surfactants electrostatically attract oppositely charged ions, allowing heavy metal ions, or similarly charged species, to bind to the micelle surface for subsequent UF separation.[5] The MEUF process is influenced by many parameters, including UF membrane characteristics, operating pressure, solution pH, and the properties of both the pollutant and the surfactant.[10] To improve control, analysis, prediction, and optimization, response surface methodology (RSM) has been widely applied.[11] Hence, this work aimed to study fluoride removal via MEUF using cetylpyridinium chloride (CPC) and hexadecyltrimethylammonium bromide (HDTAB) surfactants and to optimize the process using RSM based on a Box-Behnken design.

Martials and Methods

Materials

A polymeric ultrafiltration (UF) membrane, polyacrylonitrile (PAN-350) (Septera, USA), with molecular weight cut-off (MWCO) of 20 kDa was used for all the experiments. All the chemicals were of laboratory grade and the highest available purity. Also, Sodium fluoride (NaF, Sigma-Aldrich) was sourced from a local supplier to prepare synthetic fluoride solutions. The cationic surfactants CPC and HDTAB (both from Sigma-Aldrich) were also procured locally. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) (Merck, Germany) were used for pH adjustment. All solutions were prepared using distilled water.

MEUF set up

A dead-end membrane set up was configured and applied in this work (Figure 1). The system consisted of a 250 mL cell equipped with a magnetic stirrer, providing an effective membrane surface area of 20 cm^2. A constant operating pressure of 100 kPa was supplied by a nitrogen gas cylinder for the experiments.

All experiments were conducted at laboratory temperature (approximately 25 °C) with a constant filtration time of 10 min. Permeate was collected throughout this period and its mass was measured immediately using a digital balance to determine the flux. For each experimental run, a sample of the permeate was taken for fluoride concentration analysis. Fluoride concentration was analyzed via the SPADNS spectrophotometric method according to standard procedures.[12]

Design of experiments and Statistical Analysis

The experiments were designed using RSM based on a Box-Behnken design, which is commonly used to fit a second-order equation. Design-Expert® software (Version 7.1.6, trial) was used for this purpose.[13] In this work, a three-level Box-Behnken design was employed. The three independent variables were tested at coded values of -1, 0, and + 1, corresponding to the actual levels presented in Table 1: fluoride concentration (4, 7, and 10 mg/L), pH (4, 7, and 10), and surfactant concentration (0.5, 1, and 1.5 mmol/L) for both CPC and HDTAB. The central point was replicated three times. For each surfactant (CPC and HDTAB), a total of 15 experimental runs were generated and executed in random order. For the final validation, the optimized conditions predicted by the model for each surfactant were tested experimentally in triplicate. The average removal efficiency from these runs was then compared to the model's predicted value. The experimental procedure, as outlined in Table 2, was as follows: A specified amount of surfactant (CPC or HDTAB) was added to 200 mL of a fluoride solution. The pH was adjusted, and the solution was transferred to the membrane cell. Filtration was performed for 10 minutes at room temperature under 1 bar of N_2 gas pressure and with constant magnetic stirring at 100 rpm. The fluoride concentration and flux of the permeate were subsequently analyzed. Finally, the fluoride removal percentage was calculated using Eq. 1.

$$\%R=\left(1-\frac{\mathrm{CFp}}{\mathrm{CFf}}\right)\times 100 \tag{1}$$

where, R, CF_f and CF_p are fluoride removal (%), concentration of fluoride in feed solution (mg/L), and concentration of fluoride in the permeate (mg/L), respectively. Also, flux was calculated using Eq. 2.

$$J=\frac{V}{A.\Delta t} \tag{2}$$

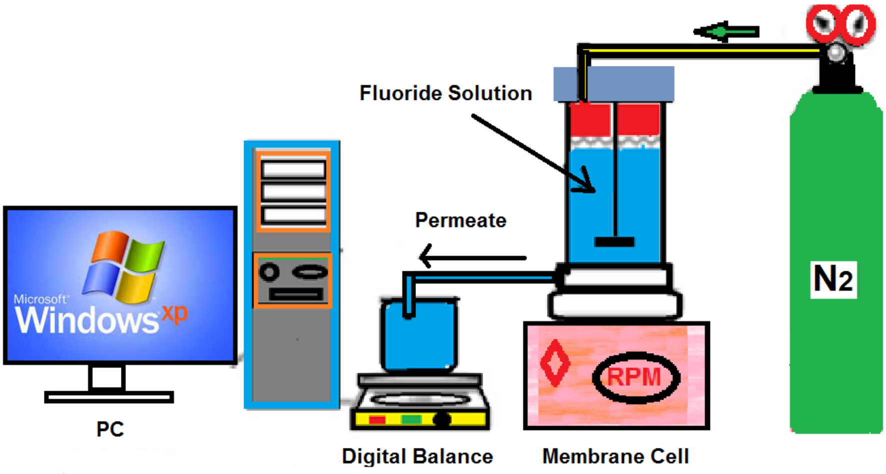

Figure 1. Dead-End MEUF set up employed in this study

Table 1. Independent variables used in the study and levels using Box-Behnken

Variable	Factors	Unit	Levels		
			Low (-1)	Middle (0)	High (+1)
pH	A	-	4	7	10
Surfactant concentration(CPC & HDTAB)	B	mM	0.5	1	1.5
F concentration	C	mg/L	4	7	10

Table 2. Experimental runs and result according to the Box–Behnken design

Run order	CPC					HDTAB				
	pH	CPC	F	Flux (L/m^2.h)	Removal (%)	pH	HDTAB	F	Flux (L/m^2.h)	Removal (%)
1	10	1.0	4	280	91.0	10	1.0	10	274	83.0
2	7	1.0	7	270	92.0	10	0.5	7	260	80.0
3	7	1.0	7	271	92.5	7	1.5	4	273	93.0
4	4	1.0	10	260	60.0	7	0.5	4	268	83.0
5	4	1.5	7	274	68.0	7	1.5	10	274	91.0
6	7	0.5	4	280	93.0	4	0.5	7	261	70.0
7	4	0.5	7	268	58.0	7	1.0	7	277	90.0
8	7	1.0	7	271	91.5	10	1.5	7	268	89.0
9	7	1.5	4	274	95.8	7	0.5	10	260	68.5
10	10	1.5	7	270	91.0	4	1.0	10	275	70.0
11	10	1.0	10	260	86.5	7	1.0	7	278	91.0
12	4	1.0	4	280	85.3	4	1.0	4	274	82.0
13	7	1.5	10	260	89.2	10	1.0	4	276	81.0
14	10	0.5	7	270	73.5	7	1.0	7	275	89.0
15	7	0.5	10	255	67.6	4	1.5	7	275	83.0

where, J is permeate flux (L/m^{2}h), A represents membrane effective area (m^2), and V shows the collected volume of permeate (L) within filtration period Δt (h).

Development of model and MEUF optimization

RSM was used to model the effect of the individual coded variables: A (fluoride), B (pH), and C (surfactant concentration)—on the response function, fluoride removal percentage (Y or R%), as defined in Table 1. Based on RSM, a second-order (quadratic) model was developed for the response (Y) by accounting for the linear, quadratic, and interactive effects of the variables. The general form of the model equation is given in Eq. 3.[13-15]

$$Y = \beta_{\circ} + \beta 1A + \beta 2B + \beta 3C + \beta 11A2 + \beta 22B2 + \beta 33C2 + \beta 12AB + \beta 13AC + \beta 23BC \quad (3)$$

Where, Y represents the predicted response. The coefficients are defined as follows: β_0 is the constant term; β_1, β_2, and β_3 are the linear coefficients for the individual variables; β_{11}, β_{22}, and β_{33} are the second-order (quadratic) coefficients; and β_{12}, β_{13}, and β_{23} are the coefficients for the interaction effects. The terms A, B, and C represent the independent variables. Also, the significance of the model, along with the individual and interaction effects, was investigated using Analysis of Variance (ANOVA) conducted with the Design-Expert (DX) software. The sufficiency of the developed model and the validity of the experimental data were also evaluated using the software's diagnostic outputs.

To check the model adequacy, the sum of squares of the model is usually analyzed.[16] Significance of quadratic conditions confirms the model fitness. To assess the model sufficiency, coefficient of determination (R^2) and lack of fit (LOF) values are mainly considered.[17,18] The R^2 is defined as the ratio of the changes described by the model to the total changes. Therefore, the higher value of R^2, closer to one, reveals more power of model fitness. An acceptable fitted model, should have an R^2 value of at least 0.8.[19] The significance of LOF showing that the data are not well positioned near the model and the model cannot predict the experimental data. In contrast, a non-significance LOF value reveals that the model can sufficiently fit with the experimental data.[15,17,18]

Results and Discussion

Model selection and sufficiency assessment

The experimental and predicted response values (Y) have been shown in Table 2, with the corresponding ANOVA results in Table 3. From this analysis, the final quadratic models for fluoride removal in terms of coded factors are given by Eqs. 4 and 5:

$$R_{CPC}(\%) = +92.00 + 8.84 \times A + 6.49 \times B - 7.73 \times C + 1.88 \times A \times B + 5.20 \times A \times C + 4.70 \times B \times C - 12.54 \times A^2 - 6.84 \times B^2 + 1.24 \times C^2 \quad (4)$$

Table 3. ANOVA results for the developed fluoride removal model

	CPC		HDTAB	
Source	**F Value**	***P*-value Prob > F**	**F Value**	***P*-value Prob > F**
Model	182.22	<0.0001	22.35	0.0016
A	429.87	<0.0001	22.55	0.0051
B	231.65	<0.0001	85.43	0.0002
C	328.45	<0.0001	20.20	0.0064
A B	9.67	0.0265	0.92	0.3814
A C	74.41	0.0003	11.28	0.0202
B C	60.79	0.0006	8.99	0.0302
A^2	399.30	<0.0001	41.88	0.0013
B^2	118.76	0.0001	3.91	0.1049
C^2	3.89	0.1056	11.29	0.0201
Lack of Fit	9.02	0.1014	21.06	0.0457
R-Squared	0.99		0.97	
Adj R-Squared	0.99		0.93	
Pred R-Squared	0.95		0.62	

$$R_{HDTAB}\ (\%) = +89.67 + 3.50 \times A + 6.81 \times B - 3.31 \times C - 1.00 \times A \times B + 3.50 \times A \times C + 3.13 \times B \times C - 7.02 \times A2 - 2.15 \times B2 - 3.65 \times C2 \quad (5)$$

Statistical analysis confirmed that the quadratic models developed for CPC and HDTAB were both significant ($P < 0.0002$; Table 3). The higher F-value for the CPC model indicates it possesses greater explanatory power in the ANOVA. This aligns with the principle that a higher F-value and a lower p-value collectively denote superior model fit. All main effects (variables A, B, and C) were significant for both surfactants. Regarding interaction effects, all terms were significant except for the AB interaction in the HDTAB model.

Table 3 shows the LOF test was not significant for the CPC model ($P > 0.05$) but was significant for the HDTAB model ($P < 0.05$). This indicates that the quadratic model is well-fitted to the experimental data for fluoride removal using CPC. The LOF test is a critical diagnostic tool that assesses whether the model fails to account for variation in the data not captured by the experimental error. A non-significant LOF suggests good model fitness[5,16-18], which was confirmed for CPC but not for HDTAB.

The coefficients of determination (R^2) were 0.99 and 0.97 for fluoride removal via CPC and HDTAB, respectively. Values closer to 1 indicate a stronger model fit. Based on this metric, both models appear robust. Furthermore, the adjusted R^2 values were 0.99 and 0.93 for CPC and HDTAB, respectively, also suggesting a good fit. However, the predicted R^2 values—0.95 for CPC and 0.62 for HDTAB—reveal a critical distinction. The low predicted R^2 for HDTAB (below the common threshold of 0.9) suggests the model lacks sufficient predictive capability for new data. It is important to note that a high R^2 value does not alone confirm model adequacy.[16] Thus, it is recommended that adjusted R^2 be checked for the model evaluation. From statistical point, R^2 value increases by adding a variable, regardless regardless of whether the variable is significant or not, but adjusted R^2, increases only if the new term improves the model more than what would be expected by chance.[17,18,20] In this work, high-adjusted R^2 for the response (fluoride removal by CPC) revealed good state of the model sufficiency. Normal distribution of residuals was evaluated using normal probability plot as applicable visual tool in the experiment[16,21] as illustrated in Figure 2 a and b for CPC and HDTAB. As seen, the data are positioned near to the line visualizing the model adequacy for F removal by both CPC and HDTAB. Predicted and experimental values for fluoride removal by MEUF process for CPC and HDTAB have been presented in Figure 3 a and b). It was found that adequacy of the model is obvious as a good correlation appears between the actual data and predicted values for CPC compared to HDTAB. In addition, Visual evaluation of residuals versus runs (Figure 4 a and b) indicated that the residual values are placed at both sides of the zero line of residuals indicating no systematic

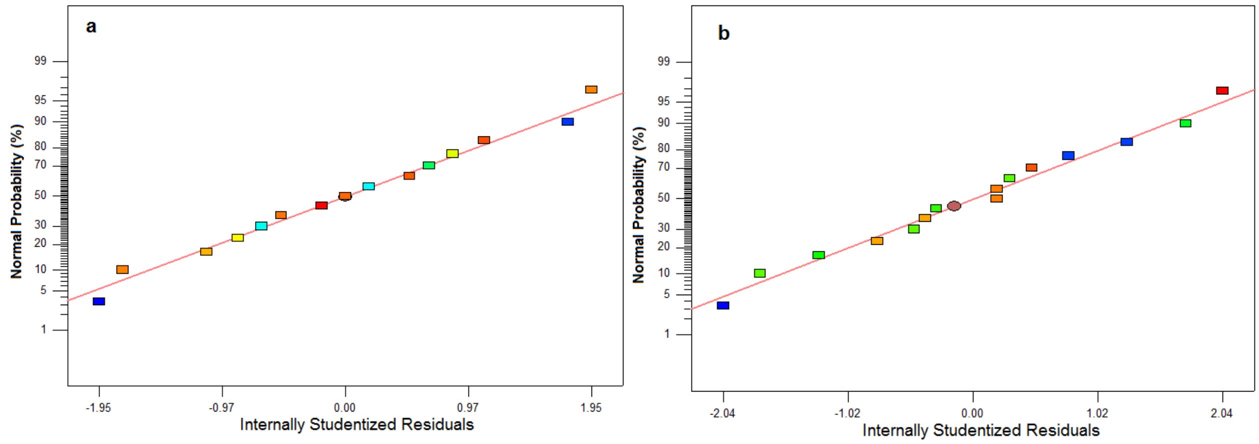

Figure 2. Normal probability plot of residuals for fluoride removal process by MEUF- a CPC and b-HDTAB

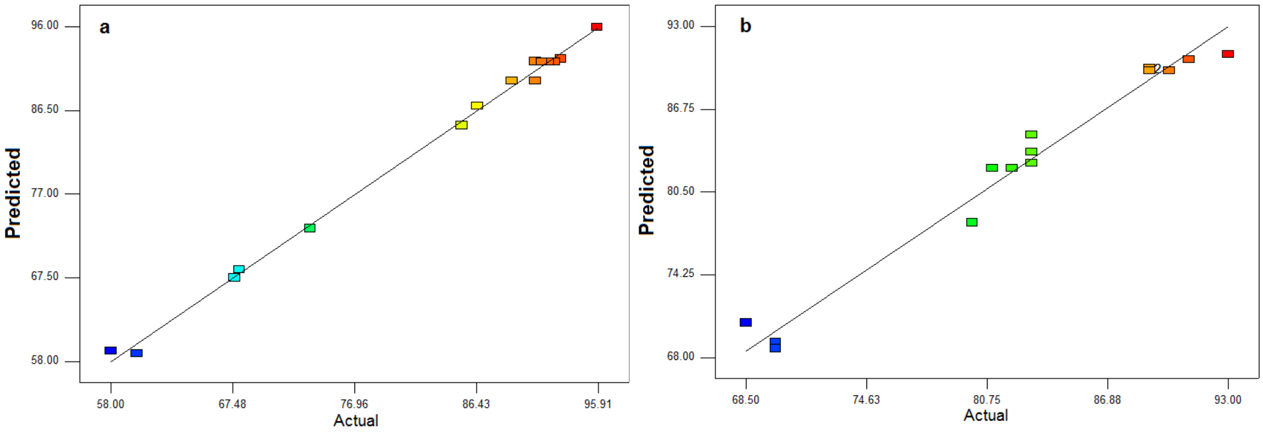

Figure 3. Predicted and experimental values for fluoride removal by MEUF process a CPC and b-HDTAB

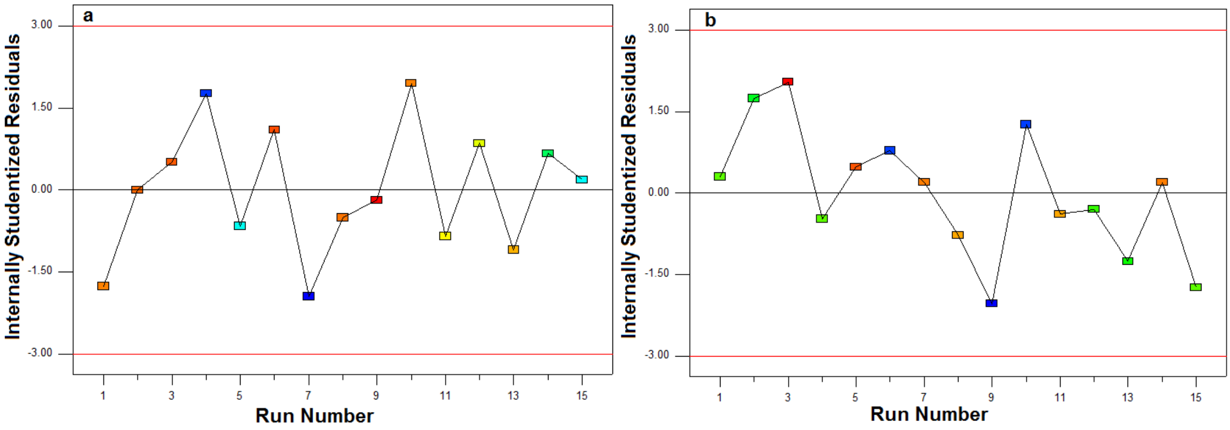

Figure 4. Predicted and experimental values for fluoride removal by MEUF process a-CPC and b-HDTAB

errors in experimental runs for CPC and HDTAB. This visual evaluation (Figure 4 a and b) verifies the validity of experimental performance for the models created for CPC and HDTAB. More precisely, in comparison, the visual results from the figure (Figure 4 a and b) indicate more strength for CPC data.

Flux analysis

The flux values for each experimental run using CPC and HDTAB surfactants have been presented in Table 2. The maximum, minimum, and average flux values for CPC were 280, 255, and 270 L/m^2.h, respectively. For HDTAB, the corresponding values were 278, 260, and 271 L/m^2.h. Throughout operation, the membrane flux decreased significantly from the pure water flux (1000 L/m^2.h) due to fouling caused by surfactants and micelles.[5] Membrane fouling and the consequent flux decline are influenced by numerous factors, including membrane properties, pollutant characteristics, and operational parameters.[5,22]

Table 4, reveals the optimal selected criteria for maximum fluoride removal via MEUF for both surfactants (CPC and HDTA). Accordingly, the optimal condition for the surfactants (CPC and HDTA) are presented in Table 5. From Table 4, it was attempted to set pH near the natural waters value (6-8)[1] and fluoride concentration, flux and removal at maximum value. As shown in Table 5, at optimum condition for CPC the removal and flux values are 94.4% and 268.1 L/m^2.h, respectively. Furthermore, for CPC, the removal and flux values are 89.6% and 276.5 L/m^2.h, respectively. From the results, (Table 5), CPC revealed higher rejection of fluoride and higher removal at optimal condition compare to HDTAB.

Impact of independent variables on the MEUF process

The ANOVA results (Table 3) indicate that all individual variables—solution pH (A), surfactant concentration (B), and fluoride concentration (C), significantly affected fluoride removal for both surfactants ($P < 0.05$). However, their relative influence differed between CPC and HDTAB. For CPC, the most influential factors were, in order, pH ($P < 0.0001$), fluoride concentration ($P < 0.0001$), and CPC concentration ($P < 0.0001$). For HDTAB, the order of influence was HDTAB concentration itself ($P < 0.0002$), followed by pH ($P = 0.0051$), and fluoride concentration ($P = 0.0064$). This highlights that the specific surfactant concentration had the strongest individual effect on the removal efficiency for HDTAB. The effects of these variables on the response are visualized in the three-dimensional (3D) response surface plots shown in Figure 5 (a, b, c).

From Figure 5a, at constant value of fluoride (7 mg/L), an increase in pH and CPC resulted in fluoride removal efficiency. However, the increase was limited to maximum 95.8%. The same phenomenon was seen for HDTAB, as illustrated in Figure 6a, but with lower removal efficiency (93%). Moreover, pH effect on the process can be seen in Figure 5b and Figure 6b. As indicated in Figure 5b and Figure 6b, an increase in pH and a decline in the initial fluoride concentration enhanced the removal efficiency, although this enhancement did not continue linearly. At a constant concentration of CPC and HDTAB, an increase in fluoride concentration limits the attraction of larger quantities of fluoride due to the swift occupation of adsorption sites. Consequently, as the initial fluoride concentration in the environment rises, the removal efficiency diminishes. Similar findings have also been well documented.[23,24] Furthermore, pH is an important factor in many processes as it can change the solution properties and affect the removal efficiency. Cationic surfactants are highly sensitive to pH and solution properties.[5] pH may change electrostatic interactions between the micelles and the charge of the pollutants.[25] Also, an increase in pH leads to a rise in the zeta potential of the membrane surface, resulting in a more negative surface charge, which in turn improves the efficiency of removal.

The data presented in Figure 5c and Figure 6c indicates that an increase in CPC and HDTAB concentration beyond the critical micelle concentration (CMC) resulted in enhanced fluoride removal. However, when the concentration exceeds removal efficiency declined, potentially attributed to the aggregation of micelles and their associated net changes.[26] The surfactant concentration initially promotes an increase in rejection rates up to the CMC, then a slight decline.[15] This decrease in removal efficiency may be linked to an inadequate ratio of fluoride ions to micelles, which is crucial for facilitating access to adsorption sites. Optimal conditions for fluoride removal are typically achieved at lower fluoride concentrations combined with higher surfactant level.[27]

MEUF optimization

The optimal operating conditions were determined using numerical optimization, prioritizing the maximization of fluoride removal while keeping other parameters within their specified experimental ranges. The resulting optimal variable levels and their corresponding performance metrics are presented in Table 4. Under these optimized conditions, the predicted fluoride removal rate was 96.8%. Experimental verification through supplementary tests yielded an average removal efficiency of 95.8% ± 0.32%. The close agreement between the predicted and

Table 4. Criteria selected to determine the optimal conditions

Name	Goal	Limit Lower	Limit Upper	Weight Lower	Weight Upper	Importance
pH	is in range	6	8	1	1	3
Surfactants (CPC, HDTAB) (mM/L))	is in range	0.5	1.5	1	1	3
F^-(mg/L)	maximize	-	max	1	1	3
Flux (L/M^2.h)	maximize	-	max	1	1	3
Removal (%)	maximize		max	1	1	3

Table 5. Optimal conditions for maximum removal efficiency and flux prediction using CPC and HDTAB

Surfactant Type	F^- (mg/L)	pH	Surfactant (mM)	Predicted Response	
				F removal (%)	**Flux** (L/m^2.h)
CPC	8	8	1.2	94.5	268.1
HDTAB	10	7	1.4	89.6	276.5

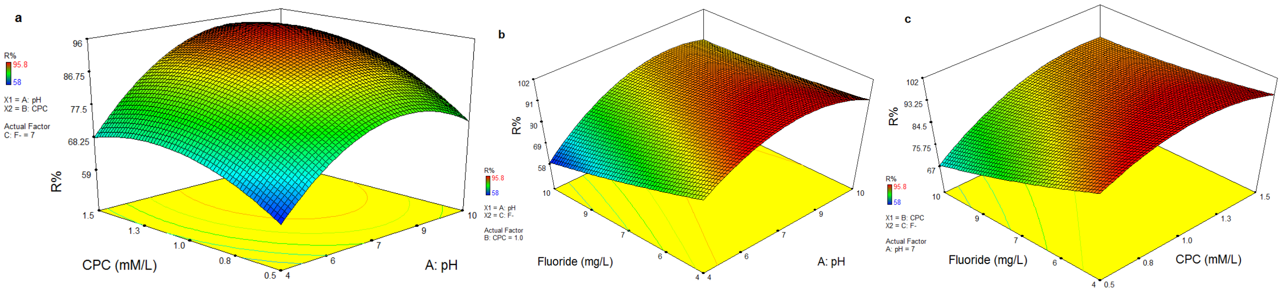

Figure 5. 3D response surface plots illustrating the interaction effects of variables on fluoride removal by CPC: (a) pH and CPC concentration; (b) pH and fluoride concentration; (c) fluoride concentration and CPC concentration

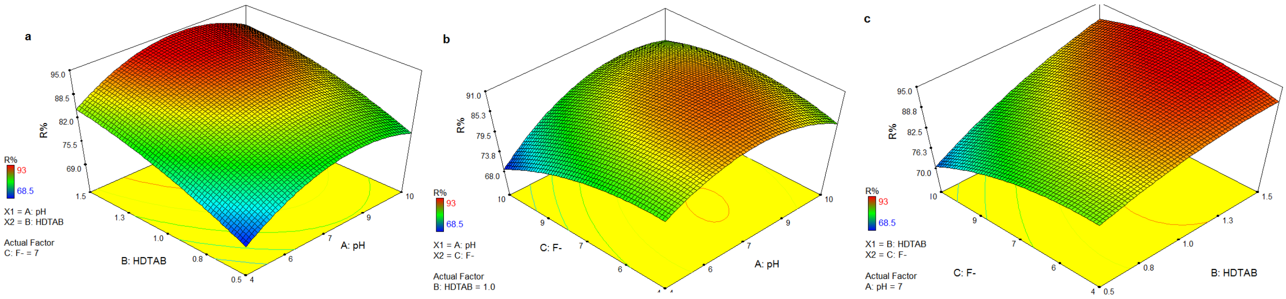

Figure 6. 3D response surface plots illustrating the interaction effects of variables on fluoride removal by HDTAB: (a) pH and HDTAB concentration; (b) pH and fluoride concentration; (c) fluoride concentration and HDTAB concentration

experimental results strongly validates both the identified optimum and the predictive accuracy of the model. Consequently, this study demonstrates that RSM is an effective and reliable tool for optimizing process variables and predicting system performance in this context.

Conclusion

The results of the present study demonstrated that the application and optimization of the MEUF process for fluoride removal using CPC and HDTAB surfactants was successful. The quadratic models generated via RSM aligned closely with experimental results, capturing the combined influence of pH, fluoride level, and surfactant dose on removal efficiency. CPC showed slightly better performance than HDTAB, reaching a peak fluoride removal (94.5%) under the optimized conditions. Statistically, the CPC model proved reliable with strong predictive ability, while the HDTAB model, though significant, was less consistent in forecasting outcomes. These results reinforce MEUF's potential as a practical and efficient low-pressure option for defluoridation. Further work should test the process with actual groundwater or industrial effluents and include a cost analysis to assess scalability.

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Authors' Contribution

Conceptualization: Ali Jafari.
Data Curation: Ali Jafari, Mohammad Javad Shokoohizadeh.
Formal Analysis: Ali Jafari, Mohammad Javad Shokoohizadeh.
Funding Acquisition: Ali Jafari.
Investigation: Sodabeh Heidarnejad.
Methodology: Ali Jafari, Sodabeh Heidarnejad, Seyyed Alireza Mousavi.
Project Administration: Sodabeh Heidarnejad.
Resources: Ali Jafari, Seyyed Alireza Mousavi.
Software: Ali Jafari, Mohammad Javad Shokoohizadeh.
Supervision: Ali Jafari, Mohammad Javad Shokoohizadeh.
Validation: Seyyed Alireza Mousavi.
Visualization: Sodabeh Heidarnejad, Mohammad Javad Shokoohizadeh.
Writing–Original Draft: Sodabeh Heidarnejad.
Writing–Review & Editing: Seyyed Alireza Mousavi.

Competing Interests

The authors declare that they have no conflict of interest.

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