

Removal of Phenol from Wastewater Using Micellar-Enhanced Ultrafiltration with Quaternary Ammonium Salt Cationic Gemini Surfactants

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ABSTRACT. Micellar-enhanced ultrafiltration (MEUF) as a surfactant-based separation process is promising for the treatment of low concentration organic wastewater. Gemini surfactant becomes the focus of the field of MEUF increasingly due to its excellent performance in MEUF. In order to investigate the effect of operational factors on the effectiveness of MEUF process, quaternary ammonium salt Gemini surfactants were used for the removal of phenol. The critical micelle concentration (CMC) values of cationic gemini surfactants (CG12, CG14, and CG16) were analyzed. Then the influence of various factors, include CG concentration, phenol concentration, electrolyte concentration and operating pressure, on the removal performance was investigated. Finally, BBD experimental design was carried out to identify optimal operational conditions. The results showed that hydrophobic chain length of Gemini surfactants have significant effect on its CMC value and the MEUF process. The longer the hydrophobic chain length, the higher phenol rejection. However, it can result in membrane fouling. CG concentration has significant influence on phenol rejection and permeation flux. And phenol concentration has a direct impact on its removal. In addition, the optimal operational condition is that CG12 concentration at 10 CMC, phenol concentration at 1 mM, and NaCl concentration at 25 mM. The corresponding optimal phenol rejection is 91.16%, the optimal permeate flux is 29.33 L/m²h.

Keywords: Micellar-enhanced ultrafiltration (MEUF), gemini surfactant, critical micelle concentration (CMC), phenol, flux, response surface analysis

1. Introduction

Phenol-containing wastewater is one of the most polluted types of industrial wastewater. It contains phenolic compounds including phenol, cresol, xylenol, naphthol aminophenol, nitrophenol and phenoxy acid (Qiu et al., 2009; Daramola et al., 2019). Phenolic compounds are used to manufacture polymer materials, synthetic fibers, explosives, ion exchange resins, phenolic resins, etc. The wastewater is mainly discharged from resin plants, plastic plants, oil refineries, coking plants, synthetic fiber plants and wood preservation plants (Wei et al., 2017; Chen et al., 2019; Yao et al., 2020) Drinking water is one of the pathways for phenolic compounds into human body. Phenol can directly enter the blood without detoxification through the liver, causing cell and protein degeneration, chronic poisoning, anemia, dizziness, memory loss, and various neurological diseases (Alshabib and Onaizi, 2019). In addition, irrigation of farmland with phenol-containing wastewater will cause crops failure, and fish in polluted water bodies will be poisoned (Wang et al., 2017; Yao et al., 2017).

Micellar Enhanced Ultrafiltration (MEUF) is a coupling technology that uses surfactants in the ultrafiltration process to improve the removal rate of a number of pollutants (Zhang et al., 2012). Due to the simple process, convenient operation, low energy consumption, high removal rate, and the ability to concentrate and recover high-value organics and heavy metals of MEUF (An et al., 2011), this technology has been applied for the removal of various pollutants (An et al., 2017; Chen et al., 2020). As the research on MEUF has become more extensive and in-depth, various membrane materials and new surfactants with superior performance and targeted properties have emerged (Wei et al., 2013; Chen et al., 2018). As the cost of membrane materials dropped, and membrane performance being continuously improved, MEUF technology possess has a broad application prospect and has become a competitive wastewater treatment technology.

Cationic Gemini surfactant (CG) is a series of dialkyl diquaternary ammonium salt surfactants with methylene chain as the linking group (Wei et al., 2012). The linking group of gemini surfactants has little effect on the critical micelle concentration, but the length of the linking group affects the curvature and aggregation number of micelles. The length of the hydrophobic chain has a great influence on the critical micelle concentration. The longer the hydrophobic chain, the smaller

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Table 1. Specifications of Surfactants

Surfactant	Molecular Formula	MW (g/mol)	CMC (mM)
CG ₁₂	C ₁₂ H ₂₅ N+(CH ₃) ₂ (CH ₂) ₂ N+(CH ₃) ₂ C ₁₂ H ₂₅ .2Br -	615	0.9
CG ₁₄	C ₁₄ H ₂₉ N+(CH ₃) ₂ (CH ₂) ₂ N+(CH ₃) ₂ C ₁₄ H ₂₉ .2Br -	671	0.125
CG ₁₆	C ₁₆ H ₃₃ N+(CH ₃) ₂ (CH ₂) ₂ N+(CH ₃) ₂ C ₁₆ H ₃₃ .2Br -	727	0.026
SDS	C ₁₂ H ₂₅ SO ₄ Na	288.38	7.8

the CMC, and the greater the viscosity of the aqueous solution. The micelles are spherical at low concentrations and columnar at high concentrations (Wei et al., 2011). The covalent bond in the gemini surfactant molecule is very strong, so that the electrostatic repulsion between the two ion head groups of the same charge is greatly weakened, which promotes the gemini surfactant to the liquid-liquid interface. The advantages of CG include: (1) the activating properties to water bodies are much greater than single-chain ones; (2) the CMC value is lower than that of general surfactants, and it is easier to form micelles at low concentrations; (3) the ability of solubilizing organic matter with long hydrophobic chain is superior; (4) because it has two hydrophilic groups, it has a lower Krafft point and has good solubility under low temperature conditions; (5) usage with other surfactants can reduce the mixed CMC. In this research, the performance of several CG is investigated, (i.e., CG12-2-12, CG14-2-14, CG16-2-16). The linking groups of these three CGs are short-chain methylene groups with two carbon atoms, and the hydrophobic chain is the symmetrical structure of the alkane chain. This study aims at in-depth comparison of the difference between the quaternary ammonium salt series CG in the process of MEUF for the removal of phenol from wastewater.

2. Materials and Methods

Chemicals: Phenol (purity greater than 98%), solubility is 8.3 g/L (20 °C); CG12, CG14, CG16 (purity greater than 98%, Chinese Academy of Sciences Chengdu Organic Institute of Chemistry); SDS (analytical grade); Hydrochloric Acid (analytical grade), Sodium Chloride (analytical grade). The properties of the surfactants are shown in Table 1.

The membrane device used in this experiment is a flow-cell pilot test equipment (model SMRXM-4), which can concentrate and filter various liquids. The equipment is suitable for operating at 5 ~ 50 °C, the system pressure is lower than 0.6 Mpa. The components of the membrane device include: 25DNL11Z-V vortex pump, flow rate is 1.8 m³/h, maximum pressure is 0.6 Mpa; the pipeline is a stainless steel pipe with an outer diameter of Φ12 mm and an inner diameter of Φ10 mm; thermometer at the bottom of the tank; diaphragm pressure gauge; ultrafiltration membrane PES10, with a molecular weight cut-off of 10 KDa.

The phenol wastewater used in this experiment was prepared with ultra-pure water, and then CG was added to the wastewater, stirred, and mixed uniformly by a magnetic stirrer, then allowed to stand for 1 hour, then added to the tank of the membrane device. The liquid is fed into the membrane module and the water inlet and outlet valves are adjusted. To make the

membrane module system reach the required pressure, the retentate is returned to the tank through the pipeline. The treated water is collected in cylinder to record the volume and then poured back into the tank. The pressure gauges at the inlet and outlet of the membrane module show the inlet and outlet pressure, and the temperature gauge at the bottom of the tank shows the real-time temperature of the liquid.

Box-Behnken experimental design is used in this study. It is an experimental design method that can show the non-linear relationship between evaluation indicators and factors. Compared with the central composite design, and in the case of the same number of factors, the number of experiments is less. The Box-Behnken test design is often used in tests when it is necessary to study the nonlinear effects of factors, and meet the requirements of this study. Determining the experimental factors is to find out the key factors that affect the process of MEUF. The experiment shows that the proportional relationship between the phenol concentration and the surfactant concentration is a key factor affecting the rejection rate of phenol. The electrolyte concentration can reduce the CMC value of CG, and the ionized charged ions are also related to phenol ions. There is a competitive relationship, so the concentration of NaCl is also a key factor, and the operating pressure has a general effect on the rejection rate of phenol and CG in addition to the obvious effect on the permeation flux. Therefore, the three key experimental factors determined by the BBD optimization method in this experiment are CG concentration, phenol concentration, and NaCl concentration.

3. Results and Discussion

3.1. Performance of CG-MEUF for Phenol Removal

The CG concentration was used as a variable, and other factors were kept as fixed conditions: the phenol concentration was 1 mM, the NaCl concentration was 20 mM, and the operating pressure was 0.2 MPa. As shown in Figure 1, as the concentration of CG12, CG14, and CG16 increases, the number and size of micelles in the solution increases, and phenol removal rate also increases. When the CG concentration reaches 6 mM or more, the phenol rejection rate increases slowly and gradually tends to be stable. Among the three CGs, CG16 has the best solubilization and removal of phenol. When CG concentration is lower than CMC, micelles cannot be formed in the solution, and phenol cannot be solubilized. However, during the ultrafiltration process, more and more solutes on the surface of the membrane cause concentration polarization and the CG concentration on the membrane surface is greater than that in the influent. The CG concentration here could reach the CMC value, forming micelles and solubilizing

phenol, so that there is a certain effective rejection rate. When the CG concentration is 0 mM, the benzene is solubilized in CG micelles and the interception concentration percentage is 30.2%. This could be the result from the membrane pores adsorbing some phenol.

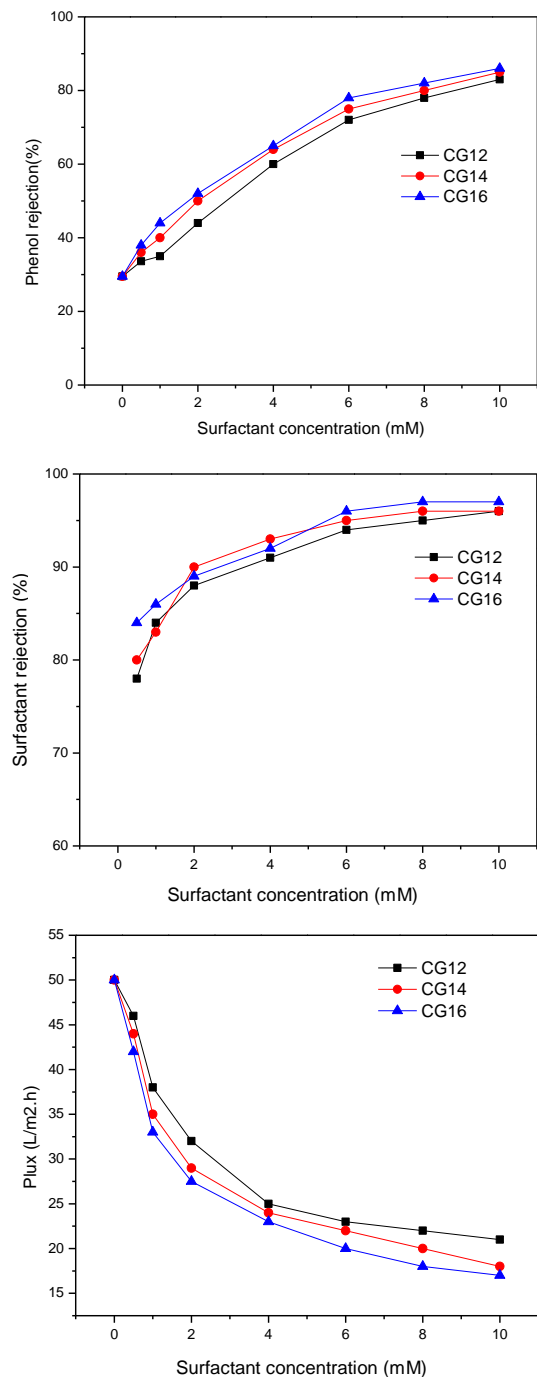


Figure 1. Effects of gemini surfactant concentration.

With the increase of CG, the interception rate of CG itself has also increased and finally reached the balance. The increase in the concentration of CG leads to an increase in the yield and

size of the micelles grown in the solution, which are more likely to be retained by the membrane. The three types of CG have different hydrocarbyl chain lengths. CG16 has the longest chain length, which forms the largest micelles and has the highest rejection rate, followed by CG14. At the same time, the increase of CG concentration also leads to the decrease of permeation flux, because there is more of micelles formed, thereby blocking the membrane pores, which leads to the decrease of permeation flux.

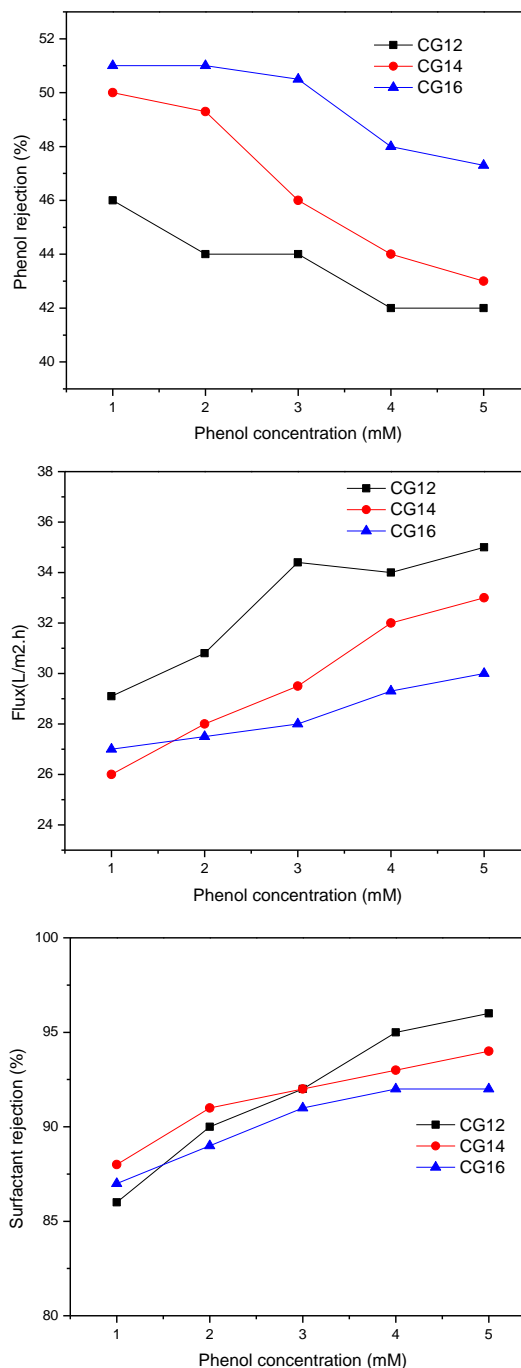


Figure 2. Effects of phenol concentration.

The phenol concentration is used as another variable, and the fixed conditions are: CG concentration is 2 mM, NaCl concentration is 20 mM, and operating pressure is 0.2 MPa. It can be seen from Figure 2, that when the concentration of CG is fixed, the removal rate of phenol is relatively stable when the concentration ratio of CG to phenol is large. This is because there are many micelles and less phenol, so only a part of the micelles is needed. It can completely adsorb phenol, so the rejection rate of phenol is relatively stable within a range CG concentration. With the increase of phenol concentration, more and more micelles reach adsorption saturation, and there is no more capacity to solubilize phenol. The excess phenol cannot be adsorbed by the micelles, it can only enter the filtrate as the solution passes through the ultrafiltration membrane, making the concentration of phenol in the filtrate increase, which leads to a decrease in the rejection rate of phenol.

Phenol is negatively charged in the aqueous solution and adsorbs to micelles with a large number of positive charges. As the concentration of phenol increases, the amount of phenol adsorbed by the micelles also increases. The micelles become more and more neutral and stable. The electrically neutral micelles accumulate on the surface of the membrane, and the repulsive force with the charged groups on the membrane surface is reduced, and it is easier to adsorb on the membrane surface. The concentration polarization phenomenon will become significant, and the gel layer is formed, which makes the CG micelles retention rate increases. Because CG16 has the longest chain length and the smallest number of micelles, it is easier to be adsorbed by negatively charged phenol to become neutral. Therefore, the curve of phenol concentration-CG rejection rate grows slowly. On the contrary, for CG12, the hydrophobic chain length is the shortest, and the number of micelles aggregated is larger than that of CG16. The micelles formed by it also carry more positive charges. Therefore, the negatively charged phenol molecules neutralize CG12 more slowly. Then the curve of phenol concentration-CG rejection rate grows fastest.

This part of the experiment takes NaCl concentration as the experimental variable, and keeps other factors as fixed conditions: CG concentration is 2 mM, phenol concentration is 1 mM, and operating pressure is 0.2 MPa. From Figure 3, it can be seen that with the increase of NaCl concentration, the rejection rate of phenol first increases and then decreases, and the rejection rate of CG is always increasing. This is because the NaCl ionization have a shielding effect between the head groups of the CG molecules in the solution due to the electrostatic repulsion, which reduced CMC value of the CG molecules. Besides, it increases the number of micelles and the micelle size is larger and more stable, resulting in an increase of phenol removal.

However, when the concentration of NaCl increases to a certain level, the rejection rate of phenol decreases. This is because Cl^- can also be adsorbed by micelles, which forms a competitive relationship with phenol molecules. As the concentration of NaCl increases, the rejection rate is reduced. In the NaCl concentration-phenol rejection rate graph, the turning point of the CG12 curve is at the NaCl concentration of 5 mM.

The peak value of the phenol rejection rate on the CG14 relationship curve corresponds to a NaCl concentration greater than 5 mM. For CG16 the turning point of the NaCl concentration is close to 10 mM. This phenomenon can be explained by the fact that the longer the hydrophobic chain of CG, the larger the micelle size, the stronger the ability to solubilize phenol, and the larger the amount of phenol molecules that can be adsorbed. Therefore, the peak of the NaCl concentration-phenol rejection rate curve of CG16 appears later. The increase in NaCl concentration has little effect on permeation flux.

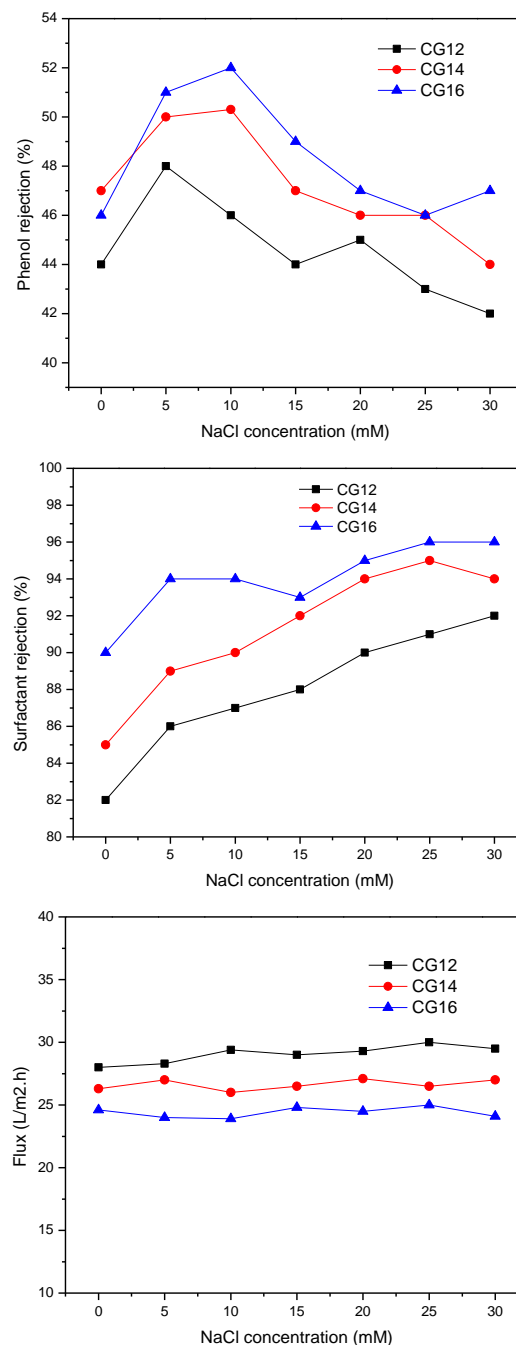


Figure 3. Effects of NaCl concentration.

The membrane used in this experiment is a PES10 membrane. For hydrophilic membranes, the greater the operating pressure, the higher the retention rate of micelles. As shown in Figure 4, the retention of three CGs, the rate increases with the increase of operating pressure. Transmembrane pressure is the most intuitive experimental factor for permeation flux interference. The operating pressure-permeation flux curve in the figure shows that permeation flux is positively correlated with pressure, but this trend is not stable. The pressure change range adopted in this experiment is not large, therefore the phenomenon that can be seen is not obvious.

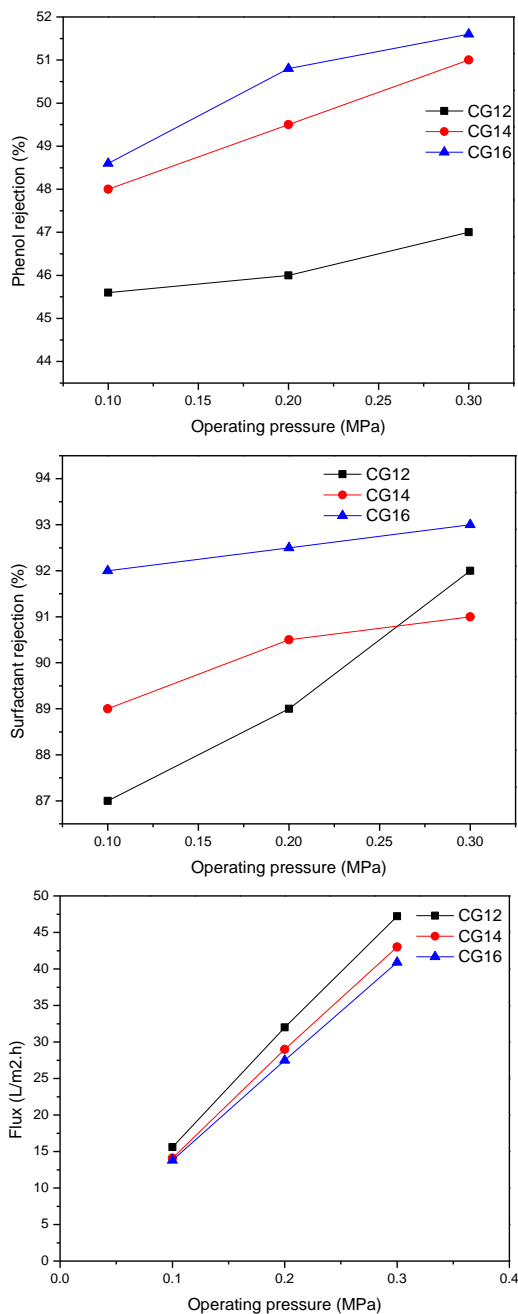


Figure 4. Effects of operation pressure.

3.2. Response Surface Analysis for CG-MEUF Treatment

When optimizing the experimental conditions for the solubilization and retention of phenol by CG12 MEUF, A, B, and C represent the CG12 concentration, phenol concentration and NaCl concentration, respectively. +1, 0, -1 represent the three levels of each factor from high to low, and the phenol rejection rate and permeation flux are the response values (shown in Tables 2 to 3).

Table 2. BBD Experimental Design

Factor	Level		
	-1	0	+1
A: Phenol Conc.	1	2	3
B: CG Conc.	2	6	10
C: NaCl Conc.	5	15	25

Figure 5(a) shows the phenol rejection rate as affected by the interaction of CG12 concentration and phenol concentration. In the selected level range, keep A (CG12 concentration) unchanged, and the response value R (phenol rejection rate) decreases with the increase of B (phenol concentration); keep B (phenol concentration) unchanged, and respond the value R (phenol retention rate) increases rapidly with the increase of A (CG12 concentration), then slowly flattens out, and finally approaches a gradual value. This shows that the interaction between CG12 concentration and phenol concentration is very strong.

As shown in Figure 5(b), when C (electrolyte concentration) is fixed, the response value R (phenol rejection rate) increases as A (CG concentration) increases. On the contrary, when CG concentration is fixed, the response value R (phenol rejection rate) slightly decreases with C (electrolyte concentration). A (CG concentration) has a significant effect on the response value, while C (electrolyte concentration) has an insignificant effect on the response value. Although the electrolyte NaCl has a less significant impact on the water quality, it has a significant impact on the formation of CG micelles and the adsorption of phenol on the micelles. At the same time, NaCl also affects the retention rate of CG micelles.

As shown in Figure 5(c), when the concentration of NaCl does not change, the response value decreases with the increase of B. When B is fixed, the response value first increases and then decreases with the increase of C. The reason why the phenol removal rate has such a trend of first increasing and then decreasing is because the higher NaCl concentration increases the number of CG micelles and increases the CG retention rate. The phenol encapsulated in the micelles is intercepted, thereby increasing the rejection rate of phenol. However, with the further increase of NaCl concentration, the negatively charged ions have a competitive adsorption relationship with phenol. Then rejection rate of phenol decreases. The experimental data analysis report shows that the NaCl concentration corresponding to this turning point is 8.97 mM. That is to say, when the NaCl concentration is 8.97 mM, the NaCl concentration has a maximum effect on the increase of the phenol rejection rate. According to the data analysis in Figure 5, the best operation

conditions of the three factors are: CG12 concentration 10 CMC, phenol concentration 1 mM, electrolyte NaCl concentration 8.97 Mm. In this combination, the response value R phenol rejection rate reaches the best point at 92.55%.

change of A and then approaches a gradual value. Therefore, A has a significant impact on the response value, and B has an impact on the response value, but it is not significant. The interaction between the CG concentration and the phenol concentration on the response value is not obvious.

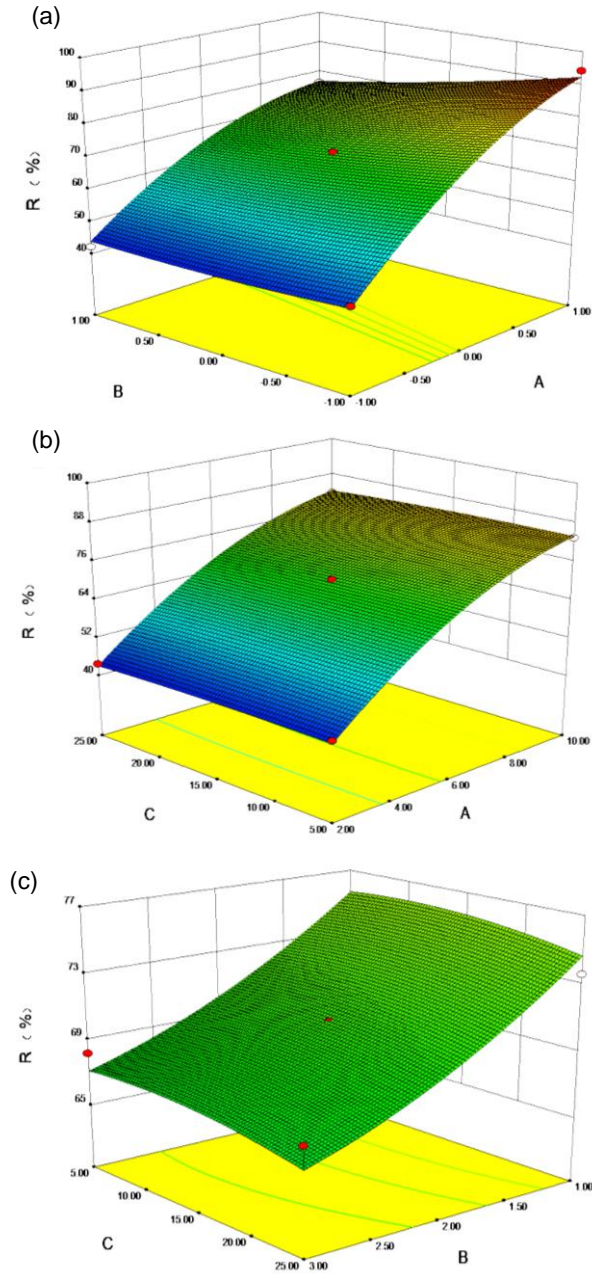


Figure 5. Surface analysis of phenol rejection rate as response value.

It can be seen from Figure 6(a) that when the concentration of CG is fixed, as the concentration of phenol changes from large to small, the response value of the permeation flux first decreases and then slightly increases, with a minimum value in the middle. When the concentration of phenol is fixed, the permeation flux of response value increases slowly with the

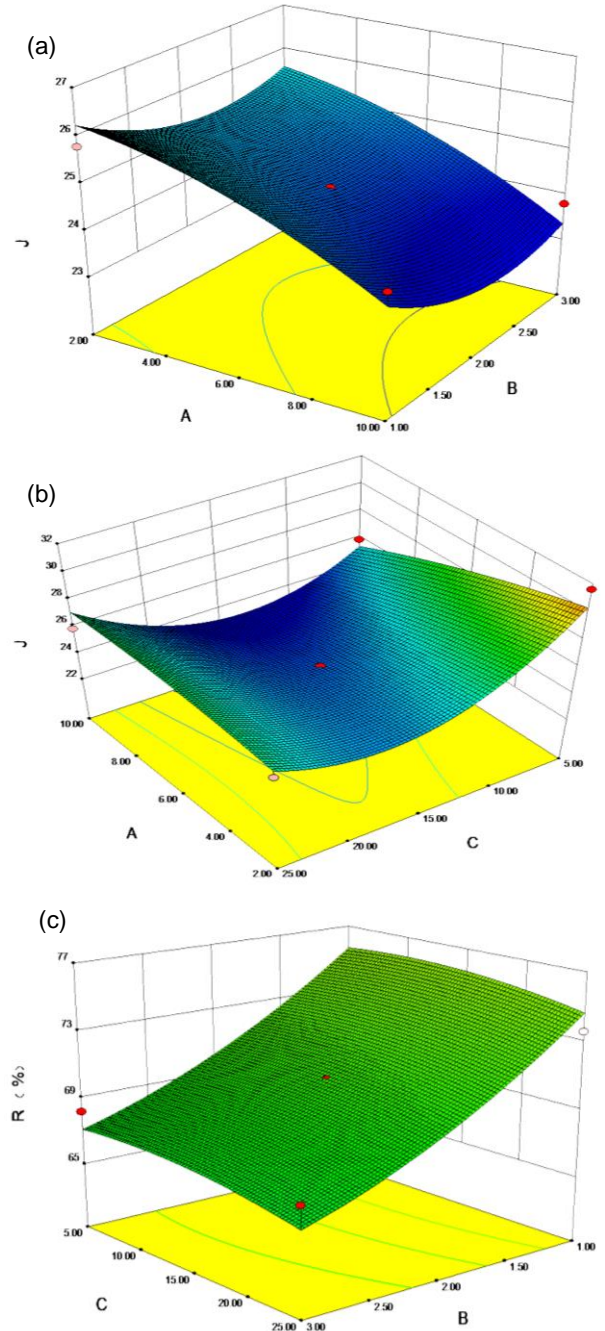


Figure 6. Surface analysis of flux as response value.

In Figure 6(b), when the CG concentration is fixed at a low value, the response value first decreases rapidly and then slowly increases with the increase of the electrolyte concentration. When the CG concentration is kept at a high value, the response

Table 3. Experimental Conditions and Results

No	CG Conc. (CMC)		Phenol Conc. (mM)		NaCl Conc. (mM)		Removal (%)		Flux (L/m ² h)	
	X ₁		X ₂		X ₃		Y ₁		Y ₂	
	Level	Value	Level	Value	Level	Value	Tested	Predicted	Tested	Predicted
1	-1	2.00	-1	1.00	0	15.00	46.30	45.75	25.80	26.24
2	+1	10.00	-1	1.00	0	15.00	94.20	92.35	24.40	24.11
3	-1	2.00	+1	3.00	0	15.00	42.10	43.95	25.32	25.62
4	+1	10.00	+1	3.00	0	15.00	76.50	77.05	23.80	23.36
5	-1	2.00	0	2.00	-1	5.00	44.50	43.72	31.60	30.36
6	+1	10.00	0	2.00	-1	5.00	83.40	83.93	25.92	25.42
7	-1	2.00	0	2.00	+1	25.00	43.60	43.08	26.12	26.63
8	+1	10.00	0	2.00	+1	25.00	81.80	82.58	24.92	27.17
9	0	6.00	-1	1.00	-1	5.00	74.30	75.63	27.20	28.00
10	0	6.00	+1	3.00	-1	5.00	68.20	67.13	28.72	29.67
11	0	6.00	-1	1.00	+1	25.00	73.60	74.67	30.32	29.37
12	0	6.00	+1	3.00	+1	25.00	67.40	66.08	27.12	26.32
13	0	6.00	0	2.00	0	15.00	70.80	70.50	24.56	24.56
14	0	6.00	0	2.00	0	15.00	70.67	70.50	24.14	24.56
15	0	6.00	0	2.00	0	15.00	70.10	70.50	24.70	24.56
16	0	6.00	0	2.00	0	15.00	70.20	70.50	24.98	24.56
17	0	6.00	0	2.00	0	15.00	70.73	70.50	24.84	24.56

value first increases with the increase of C. Decrease, and then increase rapidly, there is a minimum value. When electrolyte C is fixed at the lowest value, the response value permeation flux decreases rapidly with the increase of A. When C is fixed at the maximum value, the response value increases slightly with the increase of A, until A reaches 6 CMC. After that, the response value no longer increases and is almost a stable value. This indicates the CG concentration and electrolyte concentration have a strong mutual influence. This is because the addition of C electrolyte reduces the critical micelle concentration of CG12 and the number of micelles in the solution. The increase contributes to membrane pollution and affects the permeate flux.

In Figure 6(c), when the concentration of phenol is at high level, the response value decreases sharply with the increase of electrolyte C and then increases slightly. When the concentration of phenol is at a low value, the response value decreases slightly with the increase of C. Conversely, when C is at a low value, the response value increases slightly with the increase of B phenol concentration. When C is at a high value, the response value increases with the increase of phenol concentration. This shows that the phenol concentration and C electrolyte concentration have a certain effect on the response value of permeation flux, but it is not significant, while the interaction of phenol concentration and electrolyte concentration has a significant effect on permeation flux.

According to the data analysis in Figure 6, the best operating conditions of three factors to permeation flux can be obtained: CG12 concentration at 6.21 CMC, phenol concentration is 3 mM, electrolyte NaCl concentration is 5 mM. This optimal combination can achieve the largest permeation flux is 29.54 L/m² h. In practical applications, considering the two target values of phenol rejection rate and permeation flux at the same time, the optimal conditions should be: CG concentration at 10 CMC, phenol concentration at 1 mM, and NaCl concen-

tration at 25 mM. Under such condition, the rejection rate of phenol is 91.16%, and the permeation flux is 29.33 L/m²h.

4. Conclusions

This study investigated the effect of operational factors on the effectiveness of MEUF process. Quaternary ammonium salt Gemini surfactants were used for the removal of phenol. The CG concentration has a significant influence on the experimental effect. Increasing the concentration of NaCl can promote the reduction of the critical micelle concentration of CG, increase the amount of micelles, and increase the rejection rate of CG. By comparing the performance of three quaternary ammonium salt cationic surfactants with different hydrophobic chain lengths, it was found that the longer the hydrophobic chain of gemini surfactants, the lower the CMC value, the stronger the ability to solubilize phenol, the higher the rejection rate at the same concentration. But this could lead to serious membrane pollution. Through the analysis of the response surface experiment of CG12 applied to MEUF to remove phenol, it was found that the interaction between CG12 and NaCl has little effect on the rejection rate of phenol; while the effect on the permeation flux is significant. The interaction between CG12 and phenol has a great influence on the rejection rate of phenol. The next step will be to study the two experimental factors of temperature and pH. In addition, this study introduced the BBD response surface experimental design method in MEUF. However, the range of experimental factors of this model is not wide enough, and the parameter range should be expanded for further improvement.

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