

Article

Synergistic Effects of SDS and Non-Ionic Surfactants on Ceramic Membrane Cleaning Performance Under Acidic Conditions

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Abstract: To reinforce the cleaning agent's wetting and cleaning capabilities on ceramic microfiltration membranes in acidic environments, the wetting properties of sodium dodecyl sulfate (SDS) in combination with multiple nonionic surfactants were examined in a systematic manner. The research findings suggested that there was a potential synergistic effect among SDS, isooctyl alcohol polyoxyethylene ether (JFC), and fatty alcohol polyoxyethylene ether (AEO-7). Moreover, atomic force microscopy (AFM) and infrared spectroscopy were utilized to assess the pre- and post-cleaning contamination levels. The research findings also demonstrated that using a compound cleaning agent conspicuously regenerated the structure and elevated the hydrophilicity of the ceramic membrane surface. The synergistic mechanism between JFC and SDS can be explained by the fact that the inclusion of JFC can lessen the electrostatic repulsion between the ionic groups of SDS and heighten their hydrogen bonding effect, which in turn enhances the dispersion of contaminants and lowers the surface tension of composite solution.

Keywords: surfactant; ceramic membrane; synergistic effect; membrane cleaning; separation



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1. Introduction

Phosphorus is one of the most abundant elements on Earth and an indispensable nutrient for life. As one of primary macronutrients in plants, around 95% of phosphorus originates from phosphate rock, which has been extensively employed in agriculture, chemical industry, food, and other fields, and phosphate rock is a non-renewable resource [1]. China ranks second globally in phosphate rock reserves and is the largest producer and supplier of phosphorus resources. Even though China is abundant in phosphate rock resources, the resources are characterized by less rich and leaner ores with complex beneficiation, i.e., medium- and low-grade ores with an average P₂O₅ grade of only 16.95% [2,3]. In general, the phosphate rock market requires a P₂O₅ content of >30%. Nonetheless, phosphate rock with a low P₂O₅ content may not meet the requirements of the fertilizer industry, and its P₂O₅ content may need to be increased to reach the required level.

It is essential to increase the phosphorous content of phosphorous rock slurry to 34.8% high-grade phosphorus concentrate through a sequence of processes that include ore crushing, screening, ball milling, flotation, concentrate concentration, and tailing concentration. A crucial part of this sequence is product dehydration, necessitating ceramic membrane separation technology. A ceramic membrane is a kind of porous separation membrane made of inorganic materials (such as alumina, silicon carbide, etc.), which has

the characteristics of high temperature resistance, corrosion resistance, and mechanical strength [4,5]. Furthermore, the ceramic membrane separation technology is a method of separating, recovering, and purifying various substances using membranes. It can effectively separate materials with different apparent properties, sizes, weights, morphologies, and electrical properties by selecting different process parameters such as temperature, pressure, flow rate, concentration, and pH value in accordance with the actual application requirements [6].

In the filtration process using ceramic membranes, van der Waals forces, electrostatic interaction forces, and the Lewis acid–base effect separately exist between the non-colloidal particles, colloidal particles, and soluble compounds in the slurry and the membrane. After a long period of operation, the ceramic membrane gradually becomes contaminated, which reduces the membrane's separation efficiency and the membrane flux. The ceramic membrane is gradually clogged to the point that it cannot meet the separation requirements, and at this point, it is necessary to clean the ceramic membrane [7–10].

Noteworthy, there are numerous techniques for cleaning ceramic membranes, which can be divided into three categories on the basis of their cleaning mechanisms: physical, chemical, and biological [11]. Although biological cleaning is mild and safe, its scope of application is limited, and the cleaning efficacy is always less satisfactory than anticipated. The physical cleaning is the basic and preferred cleaning method. This technique involves the removal of impurities from the membrane surface by employing external pressures such as hydraulic pressure, ultrasonic waves, and mechanical force, which lessens the level of ceramic membrane contamination. Even though it cannot completely restore the membrane flux and is most effective in the early phases of membrane fouling, physical cleaning can successfully reduce membrane fouling. After this stage, chemical cleaning becomes imperative to remove irreversible fouling [12]. In detail, this method changes the structure of contaminants or the chemical characteristics of the fouling layer by triggering a chemical reaction between the fouling and the cleaning agent, as well as shear stress, to dislodge the fouling from the ceramic membrane's surface and into the solution [13].

Chemical cleaning efficiency largely depends on the cleaning agents used since fouling can be dissolved and dispersed into a solvent only if the bond between the fouling and the membrane is weakened. Gul et al. identified an optimal cleaning agent formulation by comparing the surface morphology and membrane flux of nanofiber membranes before and after cleaning. Their results indicated that adding Triton X-100 surfactant (SFT) to the alkaline cleaning solution improved the membrane flux by 2 to 5 times compared to that for cleaning without SFT [14].

As the contaminants in the phosphate rock ceramic membrane include stable substances like silica, fluorapatite, and dolomite, acid cleaning is the currently used to clean this membrane, which primarily utilizes the active groups of the mixed acids to dissolve the contaminants. Nonetheless, simple pickling is normally characterized by low cleaning efficiency and lowers the ceramic membrane's service life. Thus, this study is intended to add SFT to the initial cleaning solution. SFT is a chemical that can produce a polymer layer at the water–oil interface [15,16]. SFT utilized for ceramic membrane cleaning not only lessens the cleaning agent's surface tension, heightens its wettability, and allows quick penetration into the fouling, but also disperses and emulsifies the fouling to be cleaned [17,18]. Based on the observations of membrane structural changes before and after cleaning, Lv et al. used SDS, an anionic SFT, to enhance the cleaning effectiveness following NaClO washing [19].

Several researchers have undertaken substantial theoretical and experimental studies on the wetting and cleaning properties of SFT. Mousavi et al. observed that SDS has favorable wettability. It improves the wettability of dolomite by desorbing oil components from the rock surface, which is advantageous for facilitating the interaction of the cleaning agent

with obstructions [20]. Wang et al. discovered via molecular dynamics simulations that SDS molecules form a spherical micelle structure on the SiO₂ surface because of the electrostatic repulsion between SDS molecules and the SiO₂ surface head groups, as well as the hydrophobic interactions among alkyl chains. This micelle formation heightens the system's critical micelle concentration, which is beneficial for the elimination of contaminants [21]. Consequently, SDS was added to the cleaning solution to boost its cleaning efficacy.

However, the addition of SDS to the pickling process produces a flux-blocking effect, which may aggravate the contamination deposited on the surface of the membrane plate; thus, the addition of a single molecule of SDS to the cleaning agent reduces the effectiveness of the cleaning agent. Thus, the aim of this study was to combine different types of SFTs to enhance the cleaning performance. By studying the ternary system of nonionic SFT, anionic SFT, and β -cyclodextrin, Jiang et al. arrived at a pertinent conclusion that the solubility and hydrophobic interaction of this system is changed under the action of intermolecular hydrogen bonds and electrostatic forces after SFT is mixed. Furthermore, the secondary oil recovery rate can be improved by controlling the concentration of SFT [22]. Liao et al. discovered that utilizing a complex SFT blend of SDS/Tween80 hinders the demulsification of oil–water phase, thus elevating the stability of the phase interface and lowering the interfacial tension to 0.12 mN/m, thereby optimizing the membrane separation performance [23]. Shi et al. discovered that the synergy of certain anionic and nonionic SFTs can lower the system's surface tension by comparing the surface tension of solutions with non-cationic SFT mixtures, which produce a synergistic wetting effect [24]. It is reasonable to combine different kinds of SFTs, and the active groups of different SFTs can be utilized to improve the cleaning effect by enhancing the intermolecular hydrogen bonding and reducing the surface tension of the system.

With the aim of resolving the problem of insufficient cleaning performance of existing cleaning agents, this study considered the ceramic membranes of Guizhou Wengfu Group as the research object, and it explored the influence of the compound system of sodium dodecyl sulfate (SDS) and nonionic SFT on the cleaning performance of ceramic membranes under acidic conditions. By systematically analyzing the key parameters such as the surface tension of cleaning agent and the membrane flux recovery rate, a compound formulation system with efficient cleaning performance was constructed.

2. Experiment

2.1. Materials and Methods

2.1.1. Surfactants Used in the Study

The surfactants (SFTs) used in this study are divided into anionic and nonionic types, as detailed in Table 1. These SFTs were chosen for their significant surface activity and were used to evaluate their synergistic effects during the ceramic membrane cleaning process. All chemicals used were of analytical grade, and tap water was used to simulate real-world conditions during ceramic membrane cleaning.

Table 1. Surfactants (SFTs) used in the study.

Type	Reagent Name	Abbreviation	Molecular Formula
Nonionic SFT	Primary alcohol ethoxylate	Penetrating agent JFC	RO(CH ₂ CH ₂ O) ₅ H, R = C7-9
	Fatty alcohol polyoxyethylene ether-7	AEO-7	CH ₃ (CH ₂) _n (EO) ₇ , <i>n</i> = 11
	Fluorocarbon SFT	FSO-100	RfCH ₂ CH ₂ O(CH ₂ CH ₂ O) _n H, Rf = F(CF ₂ CF ₂) _m , <i>n</i> = 0–15 <i>m</i> = 1–7
	Polyethylene glycol 200	PEG-200	[CH ₂ -O-CH ₂] _n
Anionic SFT	Sodium dodecyl sulfate	SDS	C ₁₂ H ₂₅ SO ₄ Na

These SFTs were selected based on their ionization behavior in water and classified as anionic, cationic, and nonionic SFTs. Generally, cationic and anionic SFTs should not be mixed. Thus, in this study, we selected an anionic SFT (SDS) and several nonionic SFTs with significant surface activity to evaluate the wetting and cleaning performance of ceramic membranes.

2.1.2. Ceramic Membranes and Pre-Treatment

The ceramic membranes used in this study were plate-type membranes composed of Al_2O_3 , with a pore size of approximately 10 μm , provided by the Guizhou Wengfu Group (Guiyang, China). Before surface analysis, the membranes were pre-treated by soaking in a cleaning solution for 12 h, followed by thorough rinsing with distilled water. Then, the membranes were vacuum-dried in an oven at 40 °C for 24 h to ensure their proper drying before further analysis.

2.1.3. Instrumentation

The surface tension measurements of cleaning solutions were performed using a Surface Tensiometer (MC-1021, Minge Instrument Equipment Co., Ltd. (Shanghai, China)). Fourier-transform infrared (FTIR) spectroscopy was conducted using an IRSpirit spectrometer, scanning from 4000 cm^{-1} to 400 cm^{-1} with a resolution of 0.5 cm^{-1} . The software OMNIC was used for data processing. The surface roughness of the membranes was analyzed using an atomic force microscope (Bruker Dimension Icon, Karlsruhe, Germany) with a 10 $\mu\text{m} \times 10 \mu\text{m}$ scanning area. Samples were prepared, and membrane surface morphology was examined using a scanning electron microscope (ZEISS Sigma 300, Jena, Germany). The samples were coated with gold for 45 s, placed in vacuum for 20 min, and then imaged at different magnifications. Porosity analysis was also performed using an Autosorb IQ3 porosity analyzer, which provides insight into the porosity and surface area characteristics of the membranes [25].

2.2. Experimental Procedures

2.2.1. Methods of Analysis

The contaminant samples were scraped from the surface of the ceramic membrane, and the filtered slurry samples were collected for compositional analysis. The filtered slurry was first analyzed by XRF, and then the contaminant sample composition was analyzed using XRD. These clogging materials were analyzed through the integrated analysis of the peak positions, intensities, and peak shapes. Since ceramic membrane filtration is a physical process that does not involve chemical reactions, the contaminant compositional components of the ceramic membranes can be determined by comparison.

2.2.2. Formulation and Optimization of Surfactant Combinations

This study explored various combinations of anionic (SDS) and nonionic SFTs under acidic conditions, where the acids were mainly citric acid (0.73 mol/L) and oxalic acid (1.17 mol/L). These combinations were prepared in mass ratios of 4:0, 3:1, 2:2, 1:3, and 0:4, with the concentration of the test solutions maintained at 0.15 wt%. The most suitable combination was determined based on surface tension measurements [26], and the SFT concentrations were adjusted to achieve optimal cleaning performance. Further experimentation focused on varying the mass ratios (10:0, 9:1, 8:2, ..., 0:10) to optimize the SFT blend.

2.2.3. Surface Tension and Membrane Flux Measurements

Surface tension is a key parameter for evaluating the wetting ability of the cleaning agent [27,28]. Each test solution's surface tension was measured three times, and the

average values were recorded. The membrane flux was measured under negative pressure (0.085 MPa) to evaluate the improvement in cleaning performance due to SFT solutions. The membrane flux refers to the volume of filtered liquid passing through the membrane area per unit time (with a filtration time of 5 min and an effective water permeation area of approximately 3.14 cm² for the flat ceramic membrane). The membrane flux was calculated using the following Equation (1):

$$J = \frac{V}{S \times t} \quad (1)$$

where

J is the membrane flux with units of L/(cm²·min) or LMH;

V is the volume of water permeating the ceramic membrane in liters (L);

S is the effective water permeation area of the membrane in cm²;

t is the filtration time in minutes [29].

The membrane flux recovery ratio R_0 was calculated to quantify the cleaning effect using the following Equation (2) [30]:

$$R_0 = \frac{J_w}{J_0} \times 100\% \quad (2)$$

J_0 is the pure water flux before membrane fouling;

J_w is the pure water flux after cleaning [31].

The contaminated ceramic membrane was placed into the cleaning tank with a cleaning agent and subsequently soaked in the cleaning agent for 30 min. Afterwards, ultrasonic cleaning was employed so that the cleaning agent fully reacts with contaminants. Finally, the membrane surface was washed with water to ensure the accuracy of cleaning effect for measuring the membrane flux.

3. Results and Discussion

3.1. Analysis of Ceramic Membrane Contaminants

The results of XRF analysis of the filtered slurry are shown in Table 2. As exhibited in the above table, the predominant components in the slurry are CaO and P₂O₅, as well as a small amount of fluorine, implying that the predominant constituent in the slurry is fluorapatite (Ca₅F(PO₄)₃), with minor impurities of magnesium, aluminum, and iron compounds.

Table 2. Composition analysis of mineral pulp.

Substances	P ₂ O ₅ (%)	MgO (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	MER Value (%)	SiO ₂ (%)	CaO (%)	F (%)
Slurry	34.32	1.08	0.98	0.83	8.71	6.72	47.74	8.33

Subsequently, the contaminant composition was analyzed using XRD, and the results obtained using the Jade auto-matching COD database are shown in Figure 1, where the clogging materials were mainly Ca₅F(PO₄)₃, SiO₂ and CaMg(CO₃)₂ (dolomite), etc. These clogging materials were analyzed through the integrated analysis of the peak positions, intensities, and peak shapes. The analytical results in Table 1 and Figure 1 made it possible to determine the composition of contaminants in ceramic membranes mainly as SiO₂, Ca₅F(PO₄)₃, CaMg(CO₃)₂, etc.

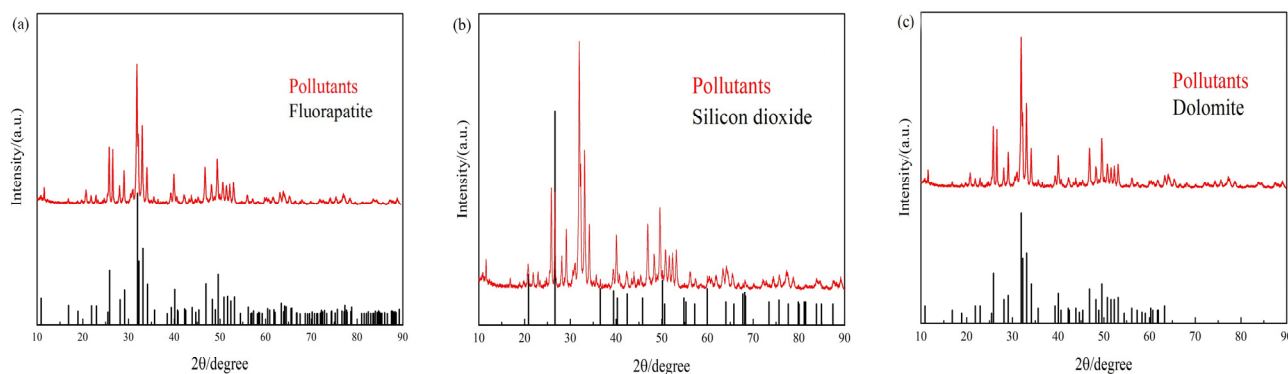


Figure 1. XRD patterns of contaminants on ceramic membrane: (a) fluorapatite ($\text{Ca}_5\text{F}(\text{PO}_4)_3$), (b) silicon dioxide (SiO_2), and (c) dolomite ($\text{CaMg}(\text{CO}_3)_2$).

3.2. Surface Tension of Composite Solution

The chemical cleaning of ceramic membranes predominantly employs chemicals and solvents to remove contaminants through dissolution, wetting, emulsification and dispersion, chelation, and other steps. Consequently, the lower the surface tension of the cleaning agent, the better the wetting cleaning effect on contaminants. Assuming that the surface tension of several SFT solutions is less than that of a single SFT solution, the mixed SFTs display a synergistic wetting effect, whereas antagonistic wetting suggests the opposite trend [32].

It was decided to first mix SDS with a nonionic SFT and subsequently determine the surface tension of the composite solution. Figure 2 depicts the experimental procedure. Figure 2a exhibits the surface tension of composite solutions of the anionic SFT SDS and several nonionic SFTs at different mass ratios. Irrespective of its low surface tension, the surface tension of the mixed solution of SDS and FSO-100 is between the surface tensions of the monomers SDS and FSO-100. Simultaneously, the surface tension values of the SDS and AEO-7 composite solution and the SDS and PEG-200 composite solution lie in between the monomeric SFTs' surface tension values. This suggests that SDS and FSO-100, AEO-7, and PEG-200 do not display any synergistic or antagonistic interactions.

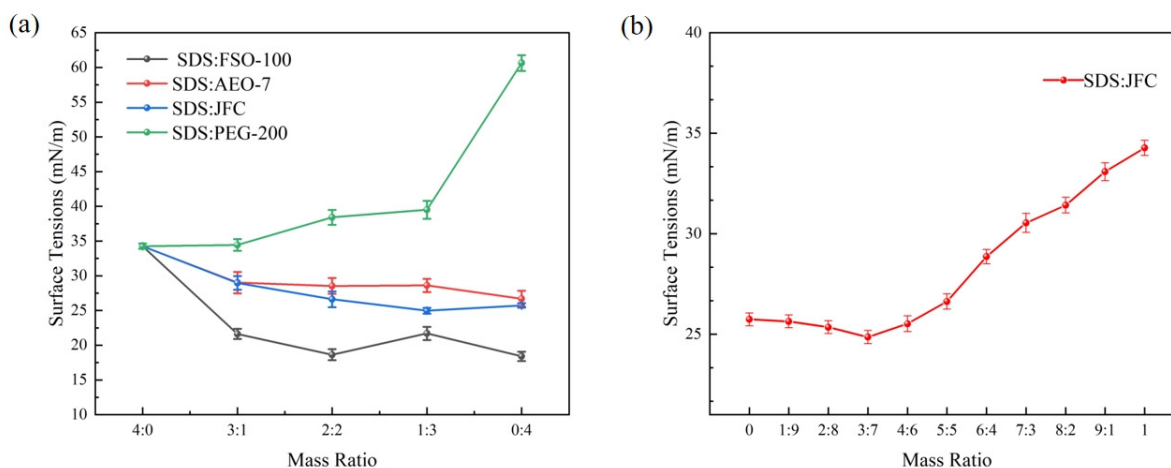


Figure 2. Surface tension measurements of SDS and nonionic SFT composite solutions: (a) Surface Tension Measurement of Composite Solutions of SDS Mixed with Nonionic SFT; (b) Determination of surface tension of composite solutions of SDS and JFC at different mass ratios.

Nonetheless, a synergistic effect is observed for the composite solution when the mass ratio of SDS and JFC is about 1: 3. The surface tension of the composite solution (24.97 ± 0.42 mN/m) is lower than that of the SDS monomer solution (34.27 ± 0.38 mN/m)

and the JFC monomer solution (25.74 ± 0.32 mN/m). Figure 2b is the surface tension of the composite solution of SDS and JFC under different mass ratios. By employing the stepwise dilution approach, it is possible to identify the ideal mass ratio of SDS to JFC in a solution to be 3:7 and the surface tension to be 24.86 ± 0.33 mN/m, which is 2.75% and 3.42% lower than that of the JFC monomer solution and the SDS monomer solution with the same concentration [33]. This suggests that SDS mixed with JFC has some synergistic wetting effect under appropriate conditions.

Figure 2a reveals that there is no antagonistic correlation between SDS and any of the selected nonionic SFTs. Moreover, each nonionic SFT has its own unique advantages. Thus, JFC was combined with various nonionic SFTs to determine the surface tension of the combination, as displayed in Figure 3.

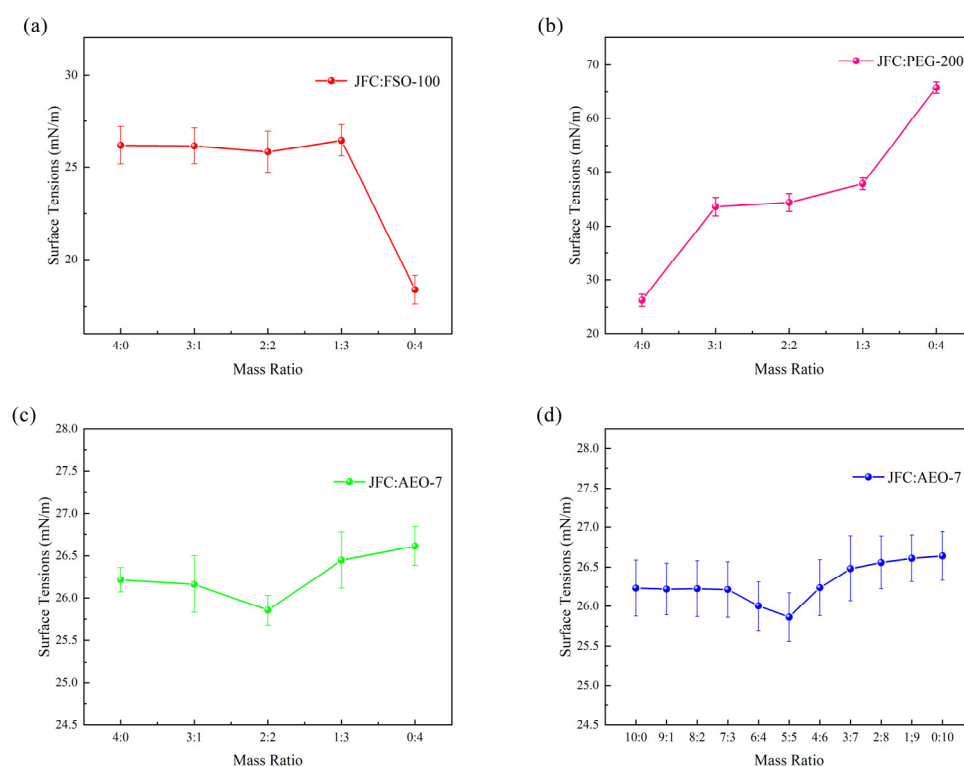


Figure 3. Surface tension of composite solutions of JFC with (a) FSO-100, (b) PEG-200, and (c,d) AEO-7 at various mass ratios.

Figure 3a suggests that the surface tension of the composite solution is higher than that of the monomer SFT solution after mixing JFC and FSO-100 at a mass ratio of 1:3, indicating that the composite solution of JFC and FSO-100 has an antagonistic effect.

Figure 3b demonstrates that, after mixing JFC and PEG-200, the surface tension value of the composite solution gradually increased as the mass ratio of PEG200 increased. Nonetheless, the surface tension value of the composite solution was found to lie between the surface tension values of the individual SFT solutions, namely, the JFC solution and the PEG-200 solution, indicating that there was neither a synergistic nor an antagonistic effect between JFC and PEG-200.

Figure 3c suggests that, at a mass ratio of JFC:AEO-7 of 1:1, the composite solution shows a synergistic effect, and the surface tension of the composite solution (25.78 ± 0.31 mN/m) is lower than that of the AEO-7 monomer solution (26.63 ± 0.33 mN/m) and that of the JFC monomer solution (26.36 ± 0.21 mN/m). As depicted in Figure 3d, the optimum mass of the compound solution of JFC and AEO was determined as 1:1 by adopting the stepwise dilution method, and the surface tension was 25.73 ± 0.27 mN/m, which was 1.48% and 2.82% lower

than that of the same concentration of the monomer solution of JFC (26.21 ± 0.29 mM/m) and AEO-7 (26.46 ± 0.34 mM/m).

As Figures 2 and 3 collectively illustrate, SDS, JFC, and AEO-7 synergistically wet the ceramic membrane when the above three SFTs were added simultaneously under acidic conditions, and the composite ratio is 3:7:7. On this basis, the composite solution of SDS, JFC, and AEO-7 was chosen as the cleaning formulation.

3.3. R_0 of Composite Solution

To further evaluate the feasibility of using the cleaning agent formulation, this study added SFTs under acidic conditions and performed cleaning trials. We recognized that variations in sample processing protocols among experimental groups (e.g., cleaning temperature, etc.) may introduce confounding factors, particularly given that this experiment was conducted primarily under ambient conditions. While the potential impact of treatment differences cannot be entirely dismissed, the robustness of our conclusions was bolstered by the rigorous harmonization of data obtained from multiple trials. The cleaning tests primarily demonstrated the cleaning impact of utilizing compound SFTs directly via the R_0 . The larger the R_0 , the lower the blockage of the ceramic membrane and the better the cleaning effect. In general, the larger the R_0 , the less blocked the ceramic membrane and the more effective the cleaning. For this reason, the different combinations of composite solutions were employed to clean the clogged ceramic membranes under acidic conditions. Furthermore, the measured membrane fluxes were converted to R_0 through calculations, yielding Figure 4.

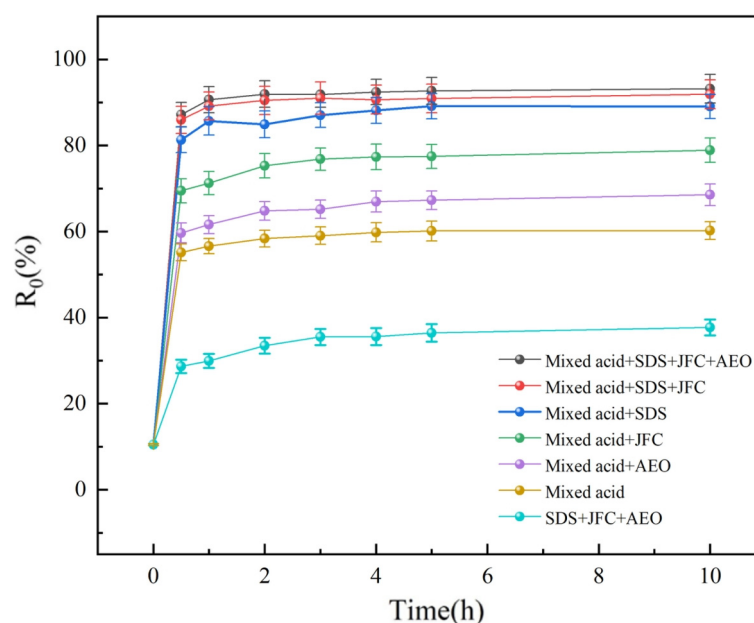


Figure 4. Effect of cleaning time on membrane recovery coefficient for different composite solutions.

Figure 4 illustrates the effect of cleaning time on the R_0 for different composite solutions [34]. As revealed by Figure 4, with the extension of cleaning time, the R_0 rises rapidly at 0–2 h and basically reaches a stable level after 2 h. After the cleaning reaches a stable level, the R_0 of mixed acid cleaning is about 60%, while the R_0 of cleaning after adding AEO-7 alone in mixed acid is about 68%. Additionally, the R_0 of cleaning after adding JFC alone in mixed acid is about 75.5%, the R_0 of cleaning after adding SDS alone in mixed acid is about 88%, and the R_0 of cleaning after adding SDS and JFC at the same time in mixed acid is about 90%. Moreover, the R_0 of cleaning after adding SDS, JFC, and JFC at the same

time in mixed acid is about, and the R_0 of cleaning after adding SDS, JFC, and AEO-7 to the acid combination is 93%.

The R_0 of mixed acid cleaning was lower than that of the composite solution with the addition of SFT; the addition of SDS, JFC, AEO-7, and the composite solution of these three SFTs can enhance the cleaning effect of the mixed acid, whereas the R_0 of SDS, JFC, and AEO-7 alone was lower than that of the composite SFT. The mixture of acids with the simultaneous addition of SDS, JFC, and AEO had the highest R_0 , and the membrane recoveries were approximately $36.0 \pm 1.2\%$, $23.2 \pm 0.6\%$, and $3.1 \pm 0.8\%$ higher than the solutions with AEO, JFC, and SDS alone, respectively. The findings of the cleaning experiment coincide with those of the surface tension tests in Figures 1 and 2, which illustrate that the solution containing SDS, JFC, and AEO in a 3:7:7 ratio has a better wetting and cleaning performance under acidic conditions.

3.4. Effect of Cleaning Agent Treatment on Surface Groups and Structure of Membrane

In order to study the cleaning effect of compound SFTs on the surface of ceramic membranes, the comparative analysis of infrared spectra was performed for the original ceramic membranes without cleaning and the ceramic membrane samples treated with the cleaning agents containing the three SFTs. Afterwards, the infrared properties of the original ceramic membrane and the cleaned ceramic membrane are illustrated in Figure 5. The absorption peak at 1009 cm^{-1} is the Si-O-Si stretching vibration absorption peak, which shifts to a high wavenumber attributed to the change in crystal structure during the ceramic firing process; thus, it presents a broad peak. The overlapping peaks at 591 cm^{-1} are principally derived from the absorption peaks of Al-O stretching vibration and Al-O tetrahedral bending vibration in the Al_2O_3 spinel structure. The absorption peak at 443 cm^{-1} belongs to Al-O and Si-O bending modes.

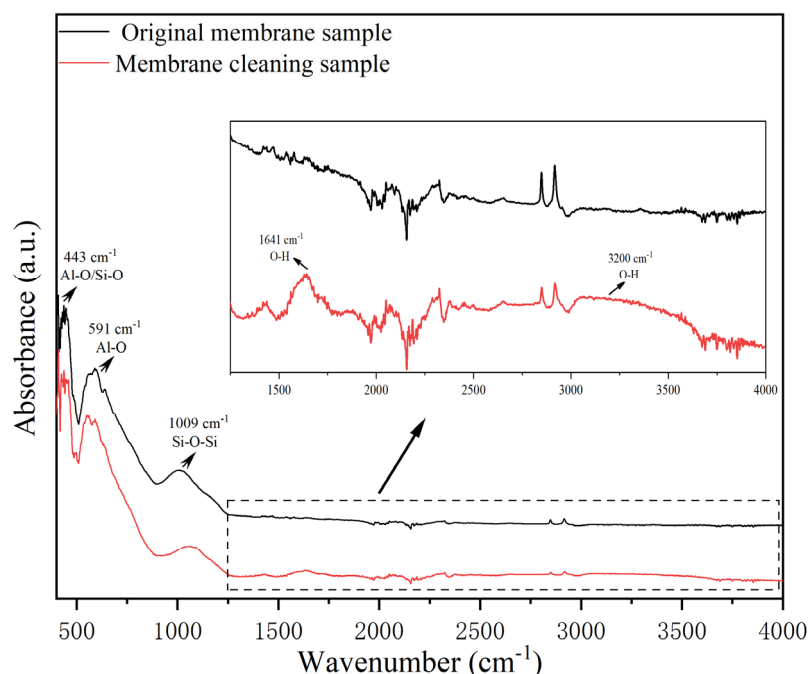


Figure 5. Infrared spectra of original membranes and cleaned membranes.

After cleaning, no conspicuous variations were found in the characteristics of ceramic membrane samples, and the characteristic absorption peaks such as Si-O-Si and Al-O remained apparent. Nevertheless, the absorption peak at 591 cm^{-1} shifted strikingly, illustrating that the Al-O crystal form of the ceramic membrane samples changed after cleaning, which may stem from the residual SFT on the surface of the ceramic membrane or

even partially inserted into the Al-O tetrahedral interlayer structure. The absorption peaks of O-H stretching and bending vibration at 3200 cm^{-1} and 1641 cm^{-1} were observed after the infrared spectra of $1250\text{ cm}^{-1}\sim 4000\text{ cm}^{-1}$ were further treated, which further confirmed that there were trace SFT components on the surface of ceramic membranes. Moreover, the introduction of hydrophilic groups such as O-H may have significantly augmented the hydrophilicity of ceramic membranes [35].

The surface structure change in ceramic membrane (such as surface roughness and membrane morphology) can also reflect the cleaning effect of the cleaning agent on the ceramic membrane. Ceramic membranes are fouled gradually due to electrostatic interactions and concentration polarization. The contaminants not only plug the pore of the membranes, but also gradually decrease the surface roughness of the membranes, reducing the separation efficiency and shortening the life of the membranes. The AFM plots in Figure 6 depict a new ceramic membrane (a), a clogged, contaminated ceramic membrane (b), and a ceramic membrane after cleaning with a composite of the three SFTs (c). The results indicate that the average surface roughness (R_a) of the new, fouled, and cleaned ceramic membranes are 2890 nm, 1550 nm, and 2337 nm, respectively, which reveals that the surface roughness of the ceramic membrane cleaned with SDS and JFC mixed acid solution has been effectively restored.

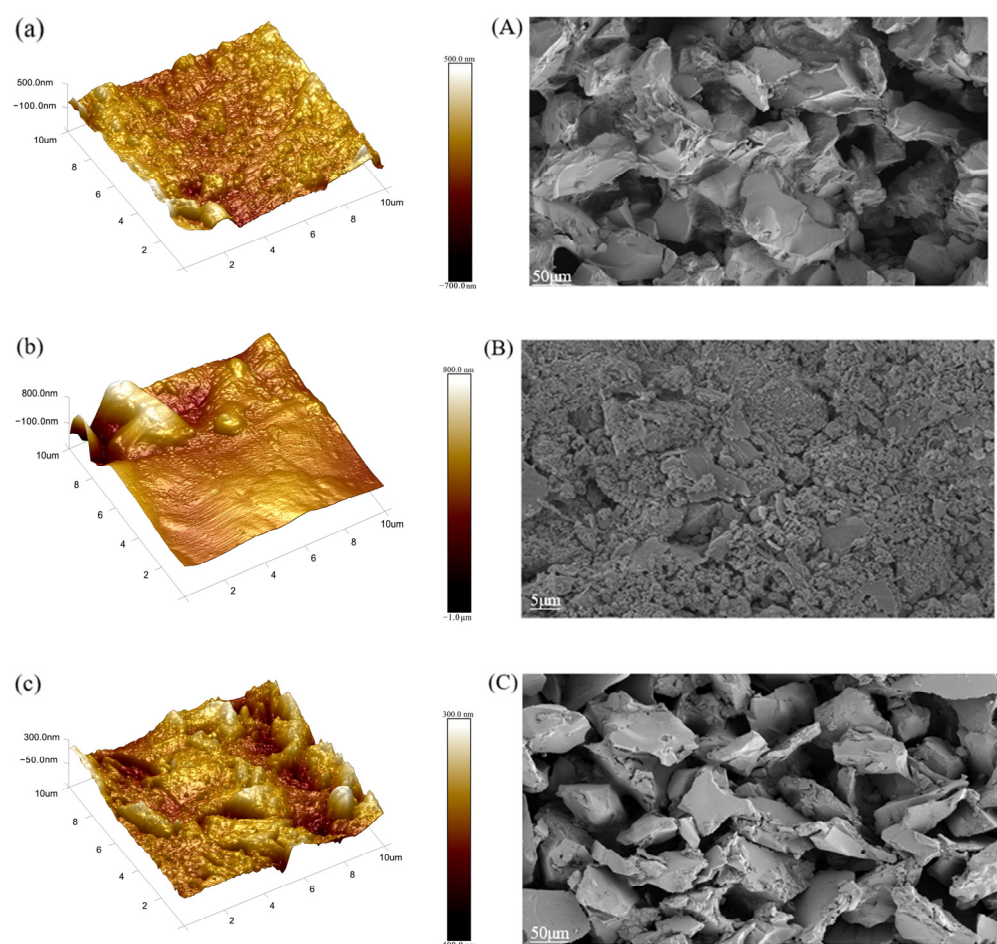


Figure 6. SEM and AFM images of three membranes: (a,A) are new membranes; (b,B) are contaminated membranes; (c,C) are cleaned membranes.

Notably, the SEM images in Figure 6 are of a new ceramic membrane (A), a clogged, contaminated ceramic membrane (B), and a ceramic membrane after cleaning with a cleaning agent containing the three SFTs in a proportional manner (C). The membrane

pores on the surface of the contaminated membrane were almost completely blocked compared to the new ceramic membrane, while the pores of the ceramic membrane after cleaning with the composite cleaning solution were greatly restored. Finally, mercury intrusion tests were performed on the newly prepared ceramic membranes, the blocked contaminated membranes, and the ceramic membranes after composite detergent cleaning. The porosities of these membranes were $40.72 \pm 0.94\%$, $36.46 \pm 0.91\%$, and $39.30 \pm 0.79\%$, respectively. The restoration of porosity indicated that the permeability of the ceramic membranes was also restored after cleaning. All these results reflect the better wetting and cleaning ability of the cleaning solution containing a combination of the three SFTs.

To study the change in hydrophilicity of ceramic membranes after cleaning with compound SFTs, we compared three types of membranes: the original uncleaned membrane (a), the clogged membrane (b), and the membrane cleaned with a detergent made from a blend of three SFTs (c). The contact angles measured are shown in Figure 7. The contact angle of the ceramic membrane recovered after cleaning, which is consistent with the surface tension measurements, indicating an improvement in hydrophilicity before and after cleaning.

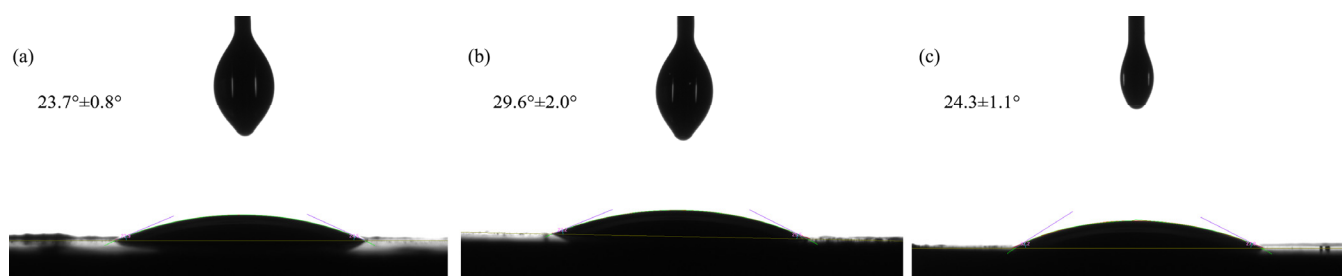


Figure 7. Contact angle test plots of three membranes: pristine ceramic membrane (a), clogged ceramic membrane (b), and cleaned ceramic membrane (c).

3.5. Study on Synergistic Cleaning Mechanism of Composite SFT Under Acidic Conditions

After the long-term operation of the ceramic membrane, it becomes contaminated by the numerous hydrophobic substances (such as flotation-modified apatite, Pyrite, etc.) deposited on the surface of the membrane. Thus, there are a myriad of hydrophobic water points on the surface of the contaminated membrane. In addition, simple pickling could not effectively wet and clean the blocking substances in the pores of the ceramic membrane. Nonetheless, the molecular structure of SFT is amphoteric: with one end having a hydrophilic group and the other end having a hydrophobic group.

As illustrated in Figure 8a, when SFTs are added to the pickling solution, their hydrophobic groups adsorb onto the hydrophobic sites on the membrane's surface, making the surface more hydrophilic. However, in some cases, the hydrophilic groups of SFTs may also adsorb onto the hydrophilic water points on the membrane surface, which could lead to a reduction in the wetting and cleaning efficacy.

In Figure 8b, the composite solution of anionic and nonionic SFTs addresses this issue. Through hydrophobic interactions, the hydrophobic tails of nonionic SFT molecules cluster around the hydrophobic tails of the anionic SFT molecules. This interaction enhances the hydrophilicity of the membrane surface. Additionally, the nonionic SFT molecules bond with the hydrophobic tails of the anionic SFT at hydrophilic sites, reducing the loss of wetting efficacy typically associated with anionic SFTs alone.

As shown in Figure 8c, anionic SFTs, such as SDS, dissociate in water, and their active groups carry a negative charge. Due to electrostatic repulsion, the distance between the SDS molecules increases, lowering the surface tension of the low-density adsorption layer. On the other hand, as shown in Figure 8d, nonionic SFTs do not dissociate in water

and remain electrically neutral. When nonionic SFTs are combined with SDS, they help reduce the electrostatic repulsion between the SDS molecules, leading to the more effective wetting of contaminants and the stabilization of the colloidal suspension. This prevents the recontamination of the ceramic membrane by ensuring that the cleaned contaminants remain evenly dispersed in the solvent.

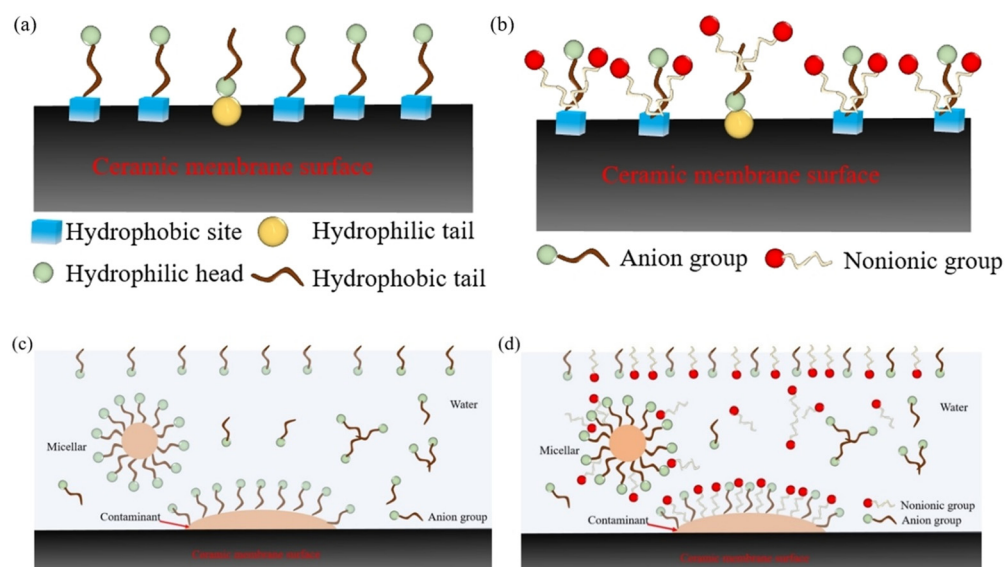


Figure 8. (a,b) Synergistic effect of anionic–nonionic SFTs on membrane surface; (c,d) Synergistic effect of anionic–nonionic SFTs in solution.

The selected nonionic SFT molecules fill the gaps in the adsorption layer of the SDS ionic groups through hydrogen bonding, further lowering the surface tension of the composite solution [36,37]. This synergistic effect enhances the overall wetting and cleaning performance of the solution, particularly in complex, hydrophobic fouling scenarios.

4. Conclusions

The SFT compounding tests suggest that no synergistic or antagonistic interactions are observed between the anionic SFT SDS and the nonionic SFTs FSO-100, AEO-7, and PEG-200. However, there are some synergistic wetting and cleaning effects observed between the anionic SFT SDS and the nonionic SFTs JFC and AEO-7 at a mass ratio of 3:7:7. The wetting and cleaning effects of SDS, JFC, AEO-7, and their composite solutions on ceramic membranes were assessed using the R0 for these solutions. The cleaning experiments demonstrate that the R0 of the SDS, JFC, and AEO-7 composite solution is approximately 36%, 23%, and 3% higher than the same concentration of AEO, JFC, and SDS as a monomeric solution, respectively. This suggests that SDS and the nonionic SFT JFC exhibit a synergistic wetting and cleaning effect.

New, clogged, and detergent-cleaned ceramic membranes were analyzed for surface changes. The results demonstrate that the detergent effectively unblocks membrane pores, removes surface impurities, and restores original roughness through synergistic mechanisms. The composite solution's wetting action combined with electrostatic/hydrophobic properties enables effective cleaning. The nonionic SFTs reduce surface tension by forming hydrogen bonds with water, while stabilizing colloidal suspensions that prevents secondary contamination by forming a uniform contaminant dispersion.

The cleaning efficiency of a ceramic membrane is influenced by multiple factors, including the cleaning agent interactions and external environmental conditions. Future

studies should systematically evaluate the impact of variables like temperature and pressure for optimizing the practical industrial applications of SFTs in cleaning solutions.

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