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Construction and Performance Evaluation of a High Efficient Mixed Foaming System

Caili Dai, Qinfang Ding, Mingwei Zhao*, Jianhui Zhao, Jichao Fang, Hui Li

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To develop a high efficient foaming agent, the fluorocarbon surfactant (4-hydroxyethylether) (pentadecafluoroalkyl) amide is designed and synthesized, surface activity and foaming performance are investigated through the surface tension, surface viscosity and foaming performance measurements. The results show that the fluorocarbon surfactant has superior surface activity and can reduce surface tension of water to 15.3 mN/m. When used as the foaming agent, it also shows great advantage over conventional hydrocarbon surfactants. For the sake of cost reduction, sodium dodecyl sulfate (SDS) is added to construct high efficient foaming agents. The results show that the mixture has super surface activity and great foaming ability. Surface viscosity measurements show that surface viscosity of the mixture is much higher than that of single SDS. The foam integrated value of the mixture is enhanced by about 67% than SDS. The optimized formula of foaming system is 0.03~0.08% HPA4 + 0.20~0.40% SDS.

Introduction

Foam fluids are widely used in select flotation [1], dust suppression [2], food processing [3], building industry [4], pharmacy [5] and oil & gas field development [6]. These applications depends on the foaming property, while foaming performance of a foaming system is closely related with foaming agent, thus the foaming agent is always the research emphasis.

Improving the foaming property of a foaming system is always a challenge. Scientists have been devoting themselves to searching for better foaming system theses years and have made some progress. Harold A. Green developed a series of dimethyl-amine oxide and investigated their foaming performance. The results showed that surfactants with carbon chains of 8-10 in their molecule matrix have high salt-resistance and oil-resistance ability, while the surfactants with branched chains have relatively better foaming property [7]. David J. Dino's group synthesized imidazoline based amphoacetate surfactants as foa ming agents. They have excellent foaming property and foam stability in pure water, highly mineralized formation brines or oil-water mixture containing light hydrocarbons [8]. Lai designed and synthesized comb-like polymer surfactants from maleic anhydride, polyethylene glycol and ethylenically unsaturated carboxylic acid. The foamability was tested by ordinary methods and the results show that the results exhibits pretty good foamability, foam stability and high water-expelling ability in oil-containing saline water [9]. Besides creating new surfactant as foaming agents, scientists also tend to make pre-existing surfactants mixed with each other to produce foaming agents system with high efficiency. In Wang's study, sodium dodecyl, sulfate ($C_{12}H_{25}OSO_3Na$, SDS) , sodium dodecyl benzene sulfonate ($C_{18}H_{29}SO_3Na$), sodium dodecyl polyoxyethylene ether sulfate ($C_{12}H_{25}O(CH_2CH_2O)nSO_3Na$) and tert-dodecanethiol ($C_{12}H_{26}S$) were mixed with each other to build long –lasting foaming system. The foaming property tests show that the mixtures exhibit much stronger foamabiliy and longer stabilization periods [10]. In Yang's study, several anionic surfactants such as lauryl sodium sulfate ($C_{12}H_{25}OSO_3Na$), sodium dodecyl polyoxyethylene ether sulfate ($C_{12}H_{25}O(CH_2CH_2O)nSO_3Na$) etc. were mixed with cationic surfactant dodecyl dimethyl benzyl ammonium chloride ($C_{21}H_{38}NCl$). The results showed that the compatibility of the mixture directly influences the foaming ability and a small amount of cationic surfactant can be added into anionic surfactant to improve foam stability [11]. Although there have been many reports about new foaming agents, improving foaming ability of foaming agents is still a challenge.

Fluorocarbon surfactants are special surfactants with hydrogen atoms replaced by fluorine atoms, which supports several new properties, such as high surface activity, high chemical and thermal stability [12]. These properties make fluorocarbon surfactants have wide applications in nanoreactors for organic synthesis [13~15], proton transporting materials for polymer electrolyte membrane fuel cells [16], oxygen transporting gels for surgery, and drug-delivery system [17]. The most attractive property of fluorocarbon surfactants is the superior surface activity. Such superior surface activity inspires us that the fluorocarbon surfactant may be used a high efficient foaming agent.

In this work, a fluorocarbon surfactant (4-hydroxyethyl ether) (pentadecafluoro-alkyl) amide, abbreviated as HPA4, is designed and synthesized to be foaming agent. The surface property and foaming performance is investigated in detail. In addition, the surface property and foamability of HPA4 and SDS mixture are also systematically studied. Through this work, we expect to develop a foaming system with high efficiency.

Experimental Section

Chemicals

The nonionic fluorocarbon surfactant (4-hydroxyethyl ether) (pentadecafluoro-alkyl) amide was synthesized according to the following steps: under the protection of nitrogen atmosphere, 15.333g perfluoro caprylic acid and 4.667g diglycolamine were added into a three-mouth flask. The temperature was heated to 130 ℃ and keep mixing for 5 h. Then the catalyst sodium hydroxide was added into the flask and the temperature was kept at 90℃. After mixing for 5 h, the reaction was finished. The product was purified through chromatographic separation. Perfluoro caprylic acid and diglycolamine were purchased from Aladdin Industrial Corporation. SDS and sodium hydroxide were purchased from Sinopharm Chemicals Reagent Limited Corporation. The water used was deionized water.

Surface tension measurements

Surface tension measurements were conducted on a Model JYM-200B surface tension meter (Chengde Dahua Instrument Limited Corporation, accuracy ±0.1 mN·m^{-1}) using the ring method. Temperature was controlled by thermostatic cell holder. The surface tension was determined by a single measurement method and all measurements were repeated for at least three times.

Foaming performance evaluation

The foam performance was tested through Waring Blender method. First, 100 mL surfactant solution was poured into the stirring cup of Waring Blender (Waring Commercial Company, America) and the rotate speed was kept at 3000 r/min. After stirring continuously for 1 minute, the volume of the foam is measured in a graduated cylinder, marked as V_0, which is a parameter to reflect the difficulty level to generate foam. The time from the Waring Blender stops stirring to the volume of the liquid at the bottom of graduated cylinder reaches to 50 mL is called drainage half-life, marked as $t_{1/2}$. The drainage half-life reflects drainage rate of the bubble film and is a reasonable indicator to evaluate foam stability. Foam volume and drainage half-life are relatively independent parameters and none of them can evaluate the foaming ability of foaming agents separately. Usually foam integrated value F is utilized to measure the foaming performance, which is defined as the product of foam volume and drainage half-life, and can be calculated according to the following equation:

$$F = V_0 \times t_{1/2} \qquad (1)$$

Surface viscosity measurements

A KRUSS DSA100 interfacial dilatational rheometer was used to measure the surface viscosity of surfactant solutions. First, the illuminant, needle of injector and the camera were arranged in a line. Then the injector which was full of surfactant solutions was adjusted to create a hanging drop and the hanging drop's profile was converted to the computer through the camera. After that the hanging drop was set to sine-wave oscillation through the oscillation chamber and the oscillation frequency and amplitude was set as 0.1Hz and 0.3 μL, respectively. The surface area of the hanging drop was recorded synchronously. Surface viscosity can be obtained through whole drop outline analysis and Fourier analysis from experiment data.

Results and Discussion

Surface properties of fluorocarbon surfactant

As is known, the foaming property of surfactant is closely related to the surface activity and self-assembly behavior in aqueous solution [18] [19], therefore it is necessary to study the basic properties of foaming system. Surface tension measurement was carried out to investigate surface properties of fluorocarbon surfactant HPA4 in aqueous solution. Figure 1 shows the surface tension of HPA4 at different concentrations. When the surfactant concentration is low, less than 1 mmol·L^{-1}, surface tension decrease sharply compared with the surface tension of pure water, which means fluorocarbon surfactant HPA4 molecules adsorb quickly on the air/water interface. With surfactant concentration keeps increasing, the surface tension decrease gradually and the decline rate of surface tension is getting low. Until the surface concentration reaches a relatively high level, the surface tension remains almost constant, indicating the surfactant molecules achieve saturated adsorption at the air/liquid interface. Fluorocarbon surfactant can reduce surface tension of aqueous solution to 15.3 mN/m. It is a much lower value compared with conventional hydrocarbon surfactants such as SDBS and sodium polyoxyethylene nonylphenyl ether sulphate, their minimum surface tension of surfactant solution is 37.10 mN/m and 29.20 mN/m, respectively [20] [21].This property contributes to its great potential as a foaming agent. The concentration of the breaking point of surface tension curve is called critical micelle concentration, abbreviated as cmc. The cmc value of fluorocarbon surfactant is about 3.41 mmol·L^{-1} at 25 ℃. Generally speaking, the cmc value is measure for surface activity for surfactant, the smaller the cmc value, the better the surface activity. Compared with convention hydrocarbon surfactants, including $C_8H_{17}SOCH_3$, $C_8H_{17}O(C_2H_4O)H$, $C_8H_{17}O(C_2H_4O)_2H$ and $C_8H_{17}O(C_2H_4O)_3H$, their cmc value is 27, 4.27, 5.62 and 7.62, respectively [22], fluorocarbon surfactant HPA4 has lower cmc value, indicating better surface activity than hydrocarbon surfactants. When comparing with fluorocarbon surfactants with the same fluorocarbon chains, including $C_7F_{15}COONa$, $C_8H_{17}SO_3NH_3C_2H_4OH$ [23], and $C_7F_{15}C(O)NH(CH_2CH_2O)_2H$ [24], (their cmc values are 36, 4.6, and 0.069 mM, respectively). The cmc value of fluorocarbon surfactant HPA4 is lower than two of the three fluorocarbon surfactants above, indicating its super surface activity.

The reduction of surface tension is ascribed to the adsorption of fluorocarbon surfactant molecules at the air/water interface. The surface excess Γ can be obtained from the Gibbs equation [25]:

$$\Gamma = -\frac{1}{nRT}\left(\frac{d\gamma}{d\ln c}\right)_T \qquad (2)$$

Where R is the gas constant, T is the Kelvin temperature, c is the bulk surfactant concentration and n=1 for HPA4 surfactant solution, dγ/d(ln c) is the slope of surface tension γ versus ln c curve. Thus, γ vs ln c curve is plotted and the fitting equation is obtained. Then derivative of the equation is taken and a derivative equation is obtained. The concentration of the surfactant is plugged into the derivative equation and surface excess *Γ* at different concentrations are calculated. The results are shown in Figure 2. The surface excess *Γ* increases sharply at lower surfactant concentration and then goes up gradually till reaching a plateau. This suggests that the adsorption of surfactant molecules reaches saturation, which is consistent with the surface tension curve. When surfactant concentration is low, a linear relationship can be seen in Figure 2. Because for a very dilute and single solution, there is no interaction among surfactant molecules at air/water interface, surface access *Γ* can be expressed by Henry equation as follows [26]:

$$\Gamma = K_h c \qquad (3)$$

Where K_h is a constant. For a higher concentration relatively, surface access Γ can be described by Langmuir adsorption isotherm equation, so the surface excess curve is L-shaped after the linear part. It's worth mentioning that the surface excess increases particularly fast when concentration is low. It's worth mentioning that the surface excess increases particularly fast when concentration is low. This phenomenon attributes to higher value of adsorption energy of $–CF_2$- group than $–CH_2$- group. So after hydrogen atoms in surfactant molecules are replaced by fluorine atoms, it is easier for surfactant molecules to move from the water to the air/water interface. This is an advantage for fluorocarbon surfactant used as foaming agent, since surfactant molecules move very quickly to the bubble film and form a compact monolayer to strengthen the bubble film, making the foam more stable. In addition, the maximum surface excess Γ_{max} of HPA4 (5.75 μmol·m^{-2}) is far more than that of typical fluorocarbon surfactants, such as $C_7F_{15}CH_2OP(O)[N(CH_2CH_2)_2O]_2$ and $C_6F_{13}CH_2CH_2NC_5H_5$ Cl, their maximum surface excess Γ_{max} are 2.77 and 2.70μmol·m^{-2} [27] [28], which means HPA4 molecules arrange more closely at the air/water interface and make the monolayer stronger.

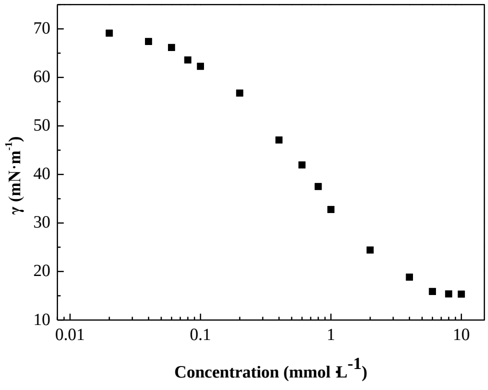


Figure 1 Relationship between surface tensions and concentrations for HPA4 solution at 25℃

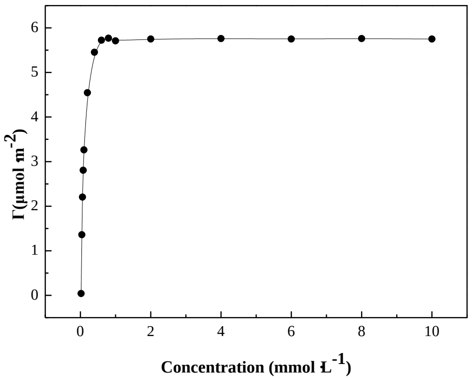


Figure 2 Relationship between surface excess and concentrations for HPA4 solution at 25℃

Foaming property of fluorocarbon surfactant HPA4

The Waring Blender method is used to determine the foaming performance of fluorocarbon surfactant, which is generally estimated by foam volume, drainage half-life and foam integrated value. The foam integrated value is the product of the foam volume and drainage half-life, which is a comprehensive index to evaluate the foaming property of a foaming agent. The larger the foam integrated value, the better the foaming performance.

The foaming performance of fluorocarbon surfactant HPA4 was investigated at 25 ℃and the results are shown in Figure 3. The foam volume, drainage half-life and foam integrated value all increase over surfactant concentration till a plateau. The foam volume almost reach up to about 690mL at concentration of 0.20%, this is better than conventional hydrocarbon surfactants such as SDS and SDBS. Their foam volume are 665mL and 410mL respectively, even their concentrations are up to 0.80%. The drainage half-life of HPA4 is 685s, much longer than that of SDS and SDBS, their drainage half-life are 432s and 426s, respectively [29]. Foam integrated value of fluorocarbon surfactant HPA4 (472650 mL s) is even much higher than that of SDS (287280 mL s), SDBS (174660 mL s). This is an amazing improvement. When foam is generated, surface area and surface energy of the system increases sharply. However the surface tension of HPA4 solution is much lower than hydrocarbon surfactants, thus the foam is easier to be produced.

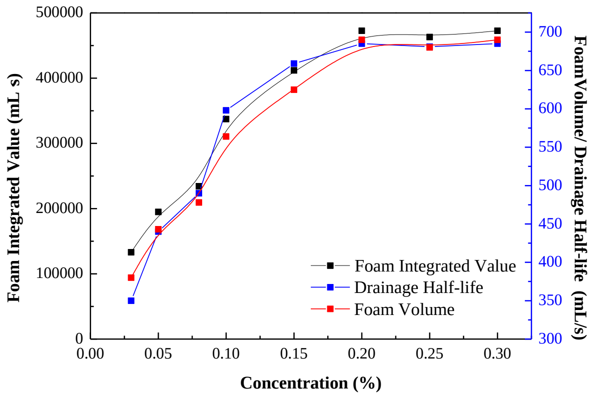


Figure 3 Parameters of foam ability for HPA4 at different concentrations

Obviously, increment of drainage half-life is bigger than that of foam volume, because low surface tension contributes to two aspects of stabilizing the foam. On the one hand, based on energy theory, low surface tension of foaming agent gives low surface energy to foam system, which is beneficial for stabilizing the foam. On the other hand, it is generally accepted that foam's decay attributes to two processes: liquid drainage of the bubble film and gas diffusion through the bubble film [30]. For drainage mechanism, it is reported that liquid drainage is the result of extrusion among bubbles, which is caused by curved face pressure. The interface among three bubbles is shown in Figure 4, which is the so-called Plateau Border. It can be derived from Laplace's equation.

$$P_B - P_A = \frac{\gamma}{R} \tag{4}$$

Where P_A is fluid pressure in A, P_B is fluid pressure in B, R is the radius of the bubble (assuming that each bubble is the same size), γ is the surface tension of the surfactant solution. Pressure in B is higher than that in A, the liquid will flow from B to A under pressure differentials. The higher the pressure differential, the more quickly the water will flow. For fluorocarbon surfactants used as foaming agents, their surface tension γ is much lower than hydrocarbon surfactants. As a result, liquid drainage is slowed down and the foam stability is greatly enhanced. Furthermore, for gas diffusion mechanism, as mentioned above, the fluorocarbon surfactant molecules adsorb densely at the air/water interface and form a tight monolayer to keep the gas from diffusing through the bubble film, resulting in more stability of the foams.

In a word, when used as foaming agents, HPA4 acts outstandingly. Its foam volume, drainage half-life and foam integrated value are much higher than conventional hydrocarbon surfactants SDS and SDBS. However, it is so expensive that it is not suitable to apply on a large scale. Taking this factor and excellent compatibility of fluorocarbon surfactants into consideration [31], we decide to make fluorocarbon surfactant HPA4 compound with commonly used hydrocarbon surfactants. Thus we can take all advantages of superior surface activity of HPA4 and synergistic effect of the mixture, while significantly reduce the application cost. The most commonly used hydrocarbon foaming agents is SDS [32]. It is very cheap also with characteristic of wide range of sources. Fluorocarbon surfactant HPA4 is mixed with SDS, aiming at creating a foaming agent system with high efficiency under the condition of relatively low cost. In order to reduce the cost, the concentration of fluorocarbon surfactant is set less than 0.10%.

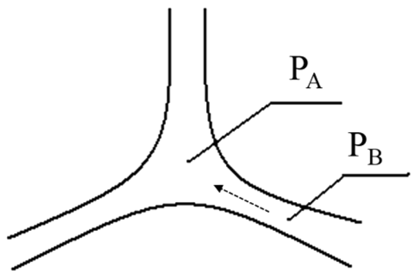


Figure 4 The Plateau Border

Surface property of fluorocarbon-hydrocarbon surfactant mixture

Surface tension measurement was carried out to investigate surface properties of foaming agent system: HPA4 + SDS. The concentration of fluorocarbon surfactant is set as 0.03%, 0.05% and 0.08%. Figure 5 shows the surface tension of the fluorocarbon-hydrocarbon surfactant mixture at room temperature 25 ℃.

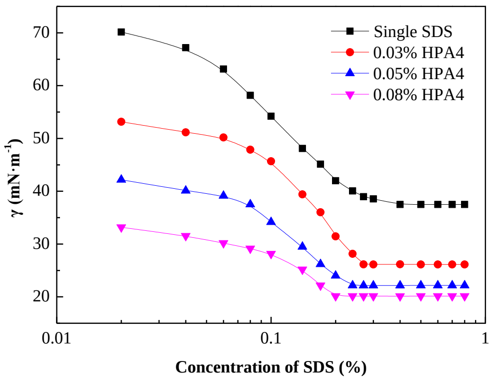


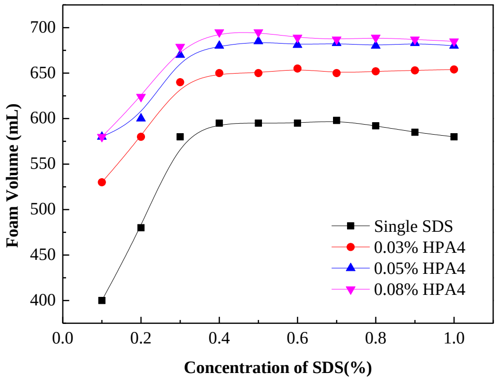
Figure 5 Surface tension of fluorocarbon-hydrocarbon surfactant mixture at 25℃

Surface tension of fluorocarbon-hydrocarbon surfactant mixture is significantly low than single SDS solution, owning to superior surface activity of HPA4. More fluorocarbon surfactant is added in the mixture, the lower surface tension can be reached. Obviously, surface tension of fluorocarbon-hydrocarbon surfactant mixture falls faster than single SDS solution. In addition, cmc value of the mixture solution is lower than single SDS solution. More HPA4 is added, the lower the cmc value is. As mention above, cmc value is measure of surface activity. Comparing with single SDS, the fluorocarbon-hydrocarbon surfactant mixture gets much better surface activity.

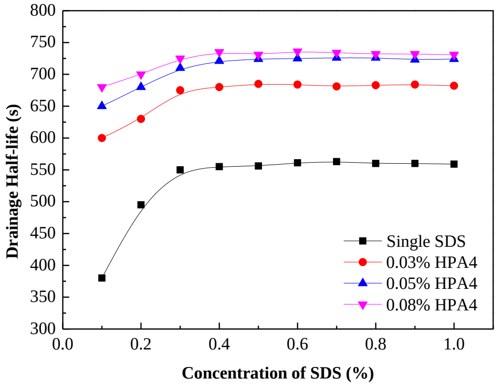
There is only one turning point in surface tension curve of the mixture shown in Figure 5, indicating mixed micelles are formed. In the mixed micelle, nonionic fluorocarbon surfactant HPA4 molecules adsorb among SDS molecules and weaken electrostatic repulsion of anionic link in SDS molecules. In addition, the hydrophilic group of fluorocarbon surfactant is polarized, in other words, its centre of negative charge and positive charge is not the same point. Its dipole can attract anion group of SDS, strengthening interaction force between fluorocarbon surfactant HPA4 and SDS [33]. These properties make micelle easier to form, showing lower cmc values. In addition, since the adsorbed layer is tighter, indicating when used as foaming agent, the mixture will packed densely at the air/water interface to make bubble film more stable.

Foaming property of fluorocarbon-hydrocarbon surfactant mixture

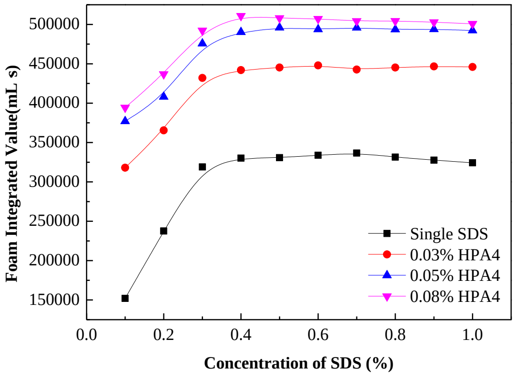
The Waring Blender method is used to investigate the foaming performance of the fluorocarbon-hydrocarbon surfactant mixture. In this part, foam volume, drainage half-life and foam integrated value are still adopted to evaluate the comprehensive foaming performance of the mixture. The results are shown in Figure 6.



(a)

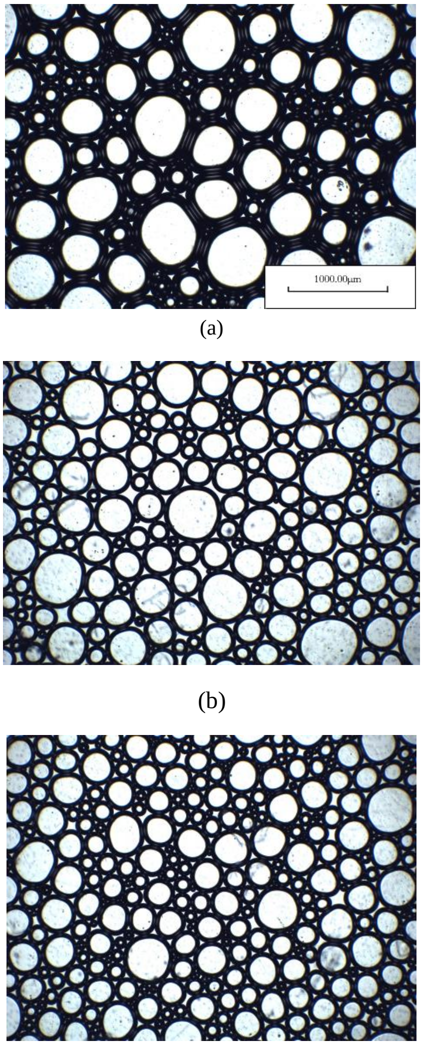


(b)



(c)

Figure 6 Foam volume (a), drainage half-life (b) and foam integrated value (c) for fluorocarbon-hydrocarbon surfactant mixture



(a)

(b)

(c)

Figure 7 Two dimension microstructure of foam (a):SDS, (b):HPA4, (c):hyrocarbon-fluorocarbon surfactant mixture

As shown in Figure 6, the foam volume, drainage half-life and foam integrated value of the mixture all obviously improve, comparing with single SDS. The more fluorocarbon surfactant is added, the more the increment value. The foam integrated value is enhanced by about 67% at most when fluorocarbon surfactant HPA4 is compounded with SDS. In addition, the foam volume and drainage half-life of the mixture can reach the top level of single SDS even when concentration of SDS is 0.20%. This improvement attributes to the synergistic effect between fluorocarbon surfactant HPA4 and SDS. Two-dimension microstructures of the foams generated by 0.40% SDS, 0.20% HPA4 and the mixture 0.40%SDS + 0.08% HPA4 are shown in Figure 7. Their surface tensions are 37.5, 18.8 and 20.1 mN/m, respectively. It is seen that the gas bubbles of the HPA4 and the mixture are smaller and more uniform than that of SDS, which is another evidence that HPA4 and the mixture perform better than SDS as foaming agents. According to the foaming property of fluorocarbon-hydrocarbon surfactant mixture, the foaming agent formula is 0.03-0.08% HPA4+ 0.20-0.40% SDS.

As discussed above, the fluorocarbon-hydrocarbon surfactant mixture has superior surface activity and lower surface tension. Therefore it can generate foam with larger volume as the pure fluorocarbon surfactant HPA4 does. The lower the surface tension of the mixture, the more easily the foam be produced [34]. As mentioned in the foam property of fluorocarbon surfactant, liquid drainage of the bubble film is one of the main reason for foam decay. For pure fluorocarbon surfactant, low surface tension slows down the liquid drainage of bubble film and stabilizes the foam for a longer time. However, for mixed foaming agents, there are other aspects in favor of foam stability. In the first place, SDS in system makes both sides of the bubble film similarly charged and mutual exclusive, which can protect the bubble film from thinning. In the second place, foam decay is a process of bubble coalescence and foam rupture. When one gas bubble breaks up or two gas bubbles coalesce, it gives an external interference to the other gas bubbles. However, there is self-restoration ability for bubble films, which is called Marangoni effect or Gibbs elasticity. It is one of the key elements for stabilizing the foam. According to previous report, the more the surface tension changes after foaming agents is added, the stronger the self-restoration ability [35] [36]. So the fluorocarbon-hydrocarbon surfactant mixture has much long drainage half-life than single sodium dodecyl sulfate. In addition, as mentioned in the micellization behavior of the mixture that the fluorocarbon and hydrocarbon surfactant molecules pack more densely because of their interaction, similarly the molecules of HPA4 and SDS will adsorb at air/water interface and arranges tighter than single HPA4 and SDS as shown in Figure 8.This synergistic effect improves the strength and surface viscosity of the adsorbed film. The surface viscosity of foaming agents versus concentration are shown in Figure 9.

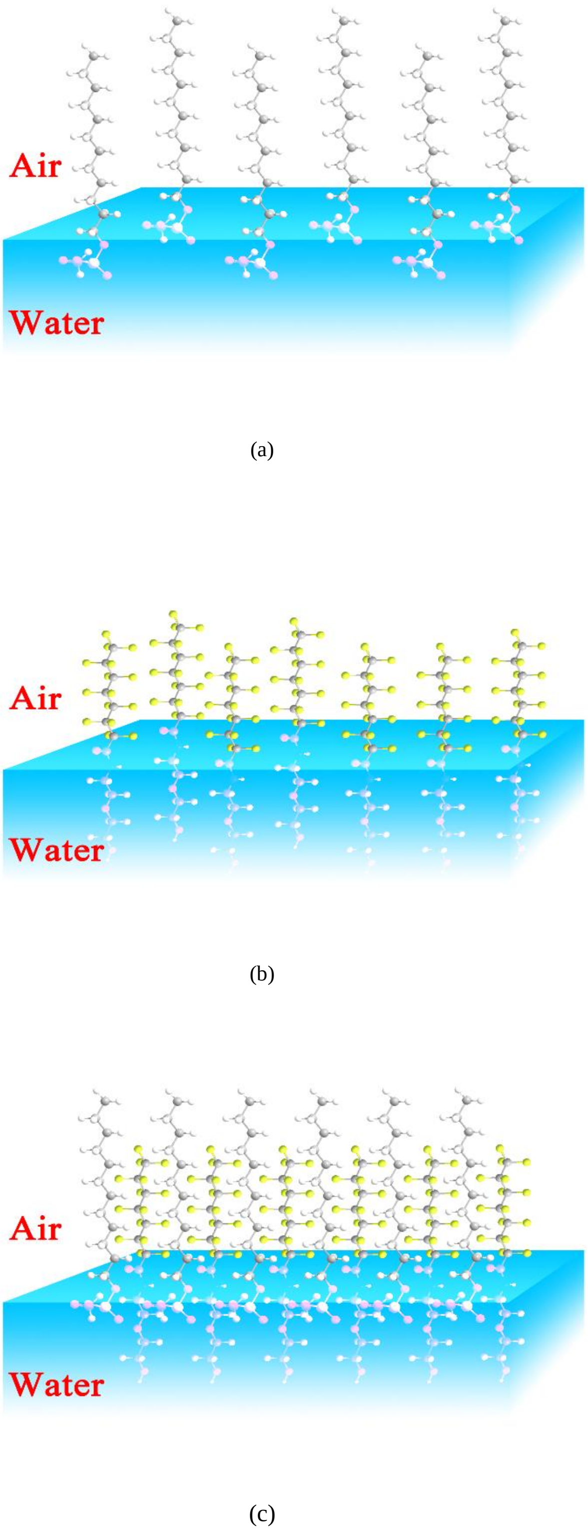


Figure 8 Schematic diagram of how surfactant molecules arrange at air/water interface (a):SDS, (b):HPA4, (c):hyrocarbon-fluorocarbon surfactant mixture

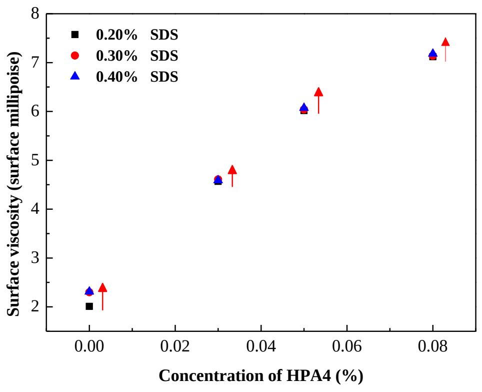


Figure 9 Surface viscosity for fluorocarbon-hydrocarbon surfactant mixture at 25℃

The surface viscosity of the mixture is much higher than that of pure sodium dodecyl sulfate. Surface viscosity is a key element to stabilize the foam and the drainage half-life will increase over the surface viscosity [37]. Besides when strength of the adsorbed film is improved, its air permeability is getting lower. Based on gas diffusion theory the foam is more stable [38].

Conclusion

In this work, fluorocarbon surfactant (4-hydroxyethyl ether) (pentadecafluoro-alkyl) amide is synthesized, its surface property and foaming performance are investigated. The results show that the fluorocarbon surfactant has superior surface activity. Its foaming performance is evaluated and it shows great advantage over hydrocarbon surfactants. To reduce applied cost, SDS is mixed with the fluorocarbon surfactant. Surface property and foaming performance of the mixture are studied and the results show that addition of fluorocarbon surfactant greatly reduce the surface tension and cmc value of the mixture. Surface viscosity measurements show that surface viscosity of the mixture is much higher than that of single SDS. And after fluorocarbon surfactant is added, the foam integrated value of the mixture increases by about 67% than single SDS. The optimized formula of foaming system is 0.03~0.08% HPA4 + 0.20~0.40% SDS and it will have a broad application prospect with high efficiency.

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Notes and references

School of Petroleum Engineering, China University of Petroleum (East China), Qingdao, Shandong, 266580, Shandong, P. R. China.
Corresponding Author:
Mingwei Zhao: Email: zhaomingwei@upc.edu.cn
Tel: +86-532-86981183

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Fluorocarbon surfactant is synthesized and mixed with SDS to create a highly efficient foaming system.