

Synergistic Effect of Mixed Surfactant Systems on Foam Behavior and Surface Tension

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Abstract Foam and surface tension behaviors of different ionic/nonionic surfactant solutions along with their different combinations have been investigated. Among different surfactants, sodium dodecyl sulfate showed the highest foamability over other surfactants. Mixed surfactant systems were always found to have higher foamability than the individual surfactant. It was also noticeable that nonionic surfactants show good foamability when they combine with anionic and cationic surfactants. In the case of mixed surfactant systems, nonionic/cationic surfactant mixtures showed lower surface tension than nonionic/anionic surfactant mixture due to a synergistic effect.

Keywords Surfactant · Mixed surfactants · Surface tension · Foamability · Foam stability · Synergism

Abbreviations

ST	Surface tension
SDS	Sodium dodecyl sulfate
EOR	Enhanced oil recovery
IFT	Interfacial tension
CTAB	Cetyltrimethylammonium bromide
CMC	Critical micelle concentration
HLB	Hydrophilic-lipophilic balance

Introduction

The foam behavior and interfacial properties of mixed surfactant systems have been extensively investigated

because of their wide applications in industries such as detergent, fabric softening, analytical chemistry, pharmaceuticals and enhanced oil recovery (EOR) technique [1–4]. In the EOR technique, different types of surfactants have been used to alter the interfacial properties and foam behaviors. In some cases, the mixture of surfactants often has better ability to modify the interfacial properties and to generate high foam volume than those of the individual interfacially active compounds and also the mixtures are advantageous because the purification of a single compound may be very expensive and difficult [5–9]. The mixture of two different surfactant types exhibit synergism or cooperative interaction and they can also produce unique microstructures like vesicles and rod-like micelles which are useful in certain applications [10–14]. However, mixed surfactant systems have the limitation that they form crystalline precipitates in aqueous solution as a result of the coulombic interaction between oppositely charged species [15]. The low interfacial tension (IFT) and surface tension (ST) in aqueous media at lower surfactant concentration is desired so that the surfactant pair can be chosen to exhibit synergism. Therefore, in a mixed surfactant system, IFT and ST are lower than that of IFT and ST of either surfactant in the same medium. In the chemically based EOR technique, surfactants have been considered as important chemicals for getting better recovery of trapped oil from natural oil reservoirs because of their high efficiencies at reducing oil–water IFT [16–19]. The main purpose of tertiary oil recovery by surfactant flooding is to lower the IFT between oil–water systems. Surfactant individually can lower the IFT between oil and water systems but the degree of reduction is not significant sometimes for an oil recovery process. So some new mixed surfactant systems have been investigated to characterize their efficiencies of surface activities. For foam flooding in EOR process foam is

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widely used as injected fluid in oil and gas reservoir to improve the mobility ration. Therefore, different mixed surfactant systems are required to achieve the goal.

From a fundamental point of view, foams are complex, highly nonequilibrium dispersions of gas bubbles in a relatively small amount of liquid generally containing surfactants. Foams can be produced using a number of techniques, including shaking, bubbling, bubbling and shaking, bubbling and stirring and a sudden drop in pressure. Due to thermodynamic instability of foams they undergo a self-destruction process due to liquid drainage, bubble disproportionation and coalescence (Ostwald ripening) [20–26]. One of the best ways to increase the foam stability is the addition of solid particles, which can irreversibly adsorb at the liquid–gas interface and noticeably increase the interfacial elasticity needed to prevent the film rupture and bubble coalescence. Various surfactants are used to produce and stabilize the aqueous foams by preventing bubbles in the foams from coalescing. The Gibbs-Marangoni effect is one of the most important factors that control the foaming properties [21, 27–31]. Characterization of foam behaviors of surfactants and mixed surfactants generally involves the investigation of both foamability and foam stability [32, 33]. Surfactants are adsorbed at the liquid–gas interface and are responsible for both the foamability and foam stability of the resulting foams. Foam stability depends on both the surfactant concentration and the rate of diffusion at the air–water interface as well as particle hydrophobicity and size [34–36]. Foam stability refers to the intrinsic resistance of the lamella to a decrease in the interfacial area and does not imply its stability in a thermodynamic sense [37].

In the present research paper, foaming properties (foamability and foam stability) of five different surfactants (SDS, Cetyltrimethylammonium bromide (CTAB), and three ethoxylated alcohols) and mixed surfactants were investigated by standard shaking method for application of

surfactants requiring high foaming in the petroleum oil industry. Critical micelle concentration (CMC), surface density and molecular cross-sectional area of the surfactants were determined to correlate these parameters with foaming properties of the surfactants. Surface tensions of different surfactants (individual and mixed) in pure water, brine and synthetic brine were measured for verification of their surface activities in a different environment.

Experimental Section

Materials Used

Different categories of surfactants such as anionic, cationic and nonionic surfactants were used for generation of foam to study their foaming properties (foamability and foam stability) and to measure surface tensions of all the surfactants in water, different brines and synthetic brine. The anionic surfactant, SDS (with 0.98 % purity) was purchased from Fisher Scientific, India and the cationic surfactant, CTAB of 98 % pure was procured from Merck, India for use in the present study. The polyethoxylated nonionic surfactants Brij 30 (abbreviated C12E4), Brij S20 (abbreviated C18E20) and Brij 58 (abbreviated C16E20) all 99 % pure were purchased from Sigma-Aldrich, Germany. The properties of the surfactants are summarized in Table 1. Sodium Chloride (NaCl) was used for the preparation of different concentrations of the brines. The synthetic brine was prepared in distilled water using different salts (NaCl, 23.54 g/L; KCl, 0.675 g/L; CaCl₂, 0.115 g/L; MgCl₂, 5.840 g/L; Na₂SO₄, 3.840 g/L; SrCl₂, 0.024 g/L; KBr, 0.110 g/L; NaF, 0.090 g/L; NaHCO₃, 0.200 g/L; H₃BO₃, 0.030 g/L). All the chemicals used to prepare synthetic brine were supplied by Merck Specialties Pvt. Ltd., Mumbai, India. Reverse osmosis water from a Millipore water system (Millipore SA, 67120

Table 1 Physicochemical characteristics and adsorption parameters of the surfactants employed in this work at 300 K

Serial no.	Chemical names and category	Linear formula and molecular weight	Trade name	HLB value	CMC (mmol/L ¹)	$\Gamma \times 10^{10}$ (mol/cm ²)	A (Å ² /mol)
1.	Sodium dodecyl sulfate (anionic)	CH ₃ (CH ₂) ₁₁ OSO ₃ Na M.W. = 288.38	SDS	40.0	8.2	2.223	74.45
2.	Cetyltrimethylammonium bromide (cationic)	CH ₃ (CH ₂) ₁₅ N(Br) (CH ₃) ₃ M.W. = 364.48	CTAB	21.4	0.96	8.438	19.67
3.	Polyoxyethylene (4) lauryl ether (nonionic)	C ₁₂ H ₂₅ (OCH ₂ CH ₂) ₄ OH M. W. = 362.56	Brij30	9.7	0.002	4.156	39.95
4.	Polyoxyethylene (20) stearyl ether (nonionic)	C ₁₈ H ₃₇ (OCH ₂ CH ₂) ₂₀ OH M. W. = 1,152	BrijS20	15.3	0.0057	3.281	50.60
5.	Polyoxyethylene (20) cetyl ether (nonionic)	C ₁₆ H ₃₃ (OCH ₂ CH ₂) ₂₀ OH M. W. = 1,123.52	Brij58	16.0	0.0077	2.573	69.53

Molsheim, France) was used for preparation of the solutions.

Methods

Determination of Foamability and Foam Stability

For foamability and foam stability tests, 10 ml of different solutions of brine and synthetic brine were mixed with 1 ml of 0.5 wt% solution of different surfactants in test tubes. Then all the test tubes were shaken in a Rotospin rotary mixer (Tarsons Products Pvt. Ltd., Kolkata, India) at a fixed speed of 50 RPM for 6 h. Changes in foam volume and evolution of foam structure were followed by visual observation at different interval of time and the foam volume was plotted as a function of time to observe the foam stability. Initial foam volumes were taken for the determination of the foamability of the surfactants. All possible mechanical vibrations were avoided during foam stability measurements. Ensuring all the measurement conditions remained identical, brine concentration and synthetic brine effects on the foamability and foam stability for the surfactant systems were studied at 300 K. The same procedure was followed for mixed surfactants which were prepared by 1:1 (w/w) combinations of different surfactants (SDS + C12E4 = MS1; SDS + C18E20 = MS2; SDS + C16E20 = MS3; CTAB + C12E4 = MS4; CTAB + C18E20 = MS5; CTAB + C16E20 = MS6).

Surface Tension Measurement

Measurement of surface tension is a very useful supplementary test method for characterization of the surface activity of surfactants. In the present study, surface tensions of the surfactant solutions (0.5 wt%) were measured with the help of a programmable tensiometer (Kruss GmbH, Germany, Model: K20 EasyDyne) at 300 K by the Du Noüy ring method. To determine the CMC, surface tensions of different concentrated surfactant solutions were measured and the CMC was calculated from the point of inflexion. The platinum ring was thoroughly cleaned with acetone and flame-dried before each measurement. In all cases, the measurement range was set at 10 and the standard deviation did not exceed ± 0.1 mN/m.

Results and Discussions

Foamability

Foamability is the foam generating power of surfactant solutions and is favored by the ability of the surfactant to

attain low surface tension in short time when a new interface is created. The foamability of different surfactants (0.5 wt%) in distilled water, 2 wt% NaCl, 4 wt% NaCl and synthetic brine were studied and the results are summarized in Fig. 1. From the Fig. 1, it is clear that in all the solutions SDS has produced higher foam volumes i.e., higher foamability than the other surfactants employed in this work. The result shows similarity with other research works [38, 39]. Generally, foamability is highly influenced by the accumulation of the foaming agents at the air–water interface to produce foam [40, 41]. It is important to reach the minimal concentration of a surfactant that is required for the formation of a saturated monolayer at the bubble surface. The equilibrium adsorption of surfactant at the air–water interface may be calculated with help of measured surface tension isotherm using Gibbs surface adsorption equation as follows:

$$\Gamma = -\frac{1}{RT} \left(\frac{d\gamma}{d \ln C} \right) \quad (1)$$

where Γ is the surface density (mmol/cm^2); R is the universal gas constant ($8.314 \text{ J}/\text{mol}^1 \text{ K}^1$), T is the thermodynamic temperature (K), γ is the surface tension (mN/m^1), C is the surfactant concentration (mmol/L^1) at the CMC. The term, $\left(\frac{d\gamma}{d \ln C} \right)$ used in Eq. 1 can be calculated from the slope of the plot of the logarithm of the surfactant concentration with the surface tension shown in Fig. 2. From Fig. 2, it is clear that, as the surfactant concentration increases, the surface tension decreases rapidly and each curve has a level off point at the concentration corresponding to the CMC, then remains at an almost constant value at higher concentrations of surfactant in solution.

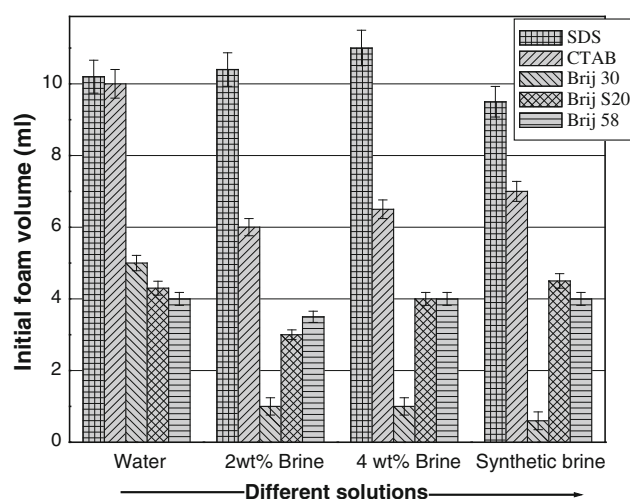


Fig. 1 Initial foam volume of different surfactant solutions in distilled water, 2 wt% NaCl, 4 wt% NaCl, and synthetic brine at 300 K

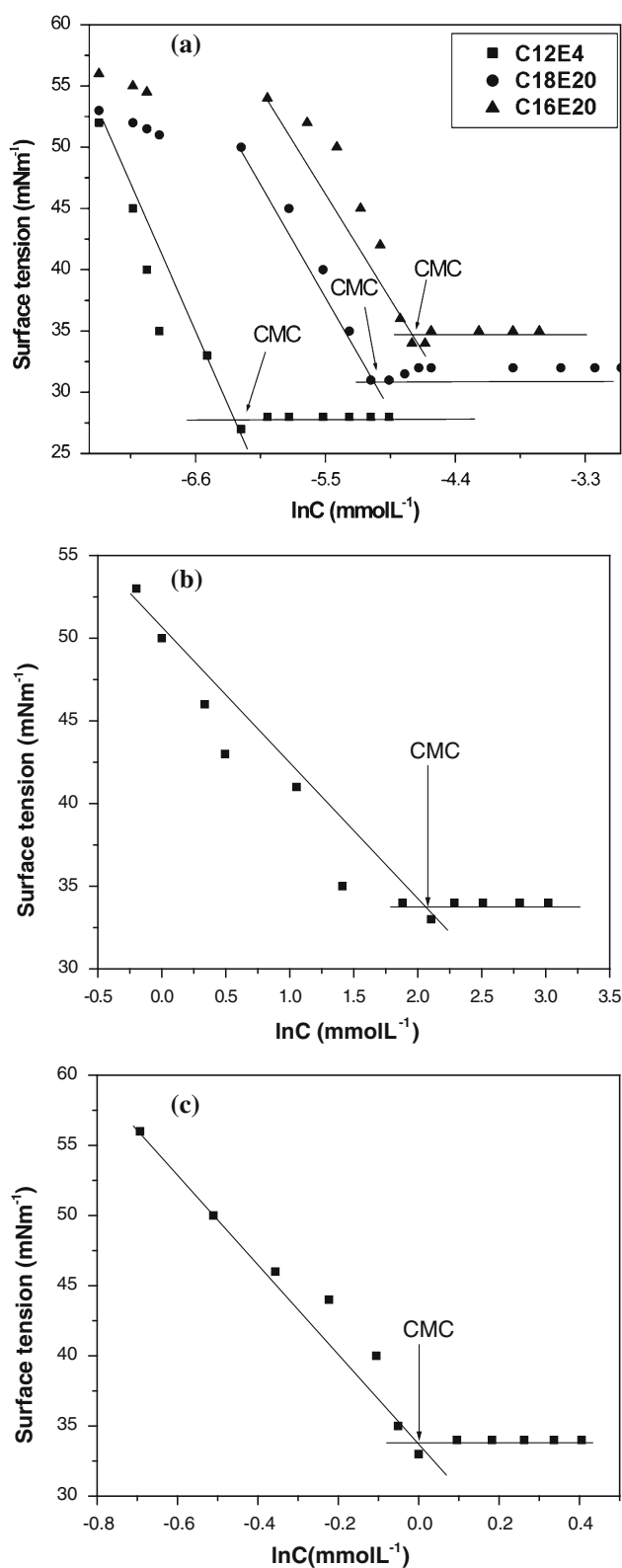


Fig. 2 The surface tension versus concentration plot for the determination of the CMC of the surfactants at 300 K: **a** polyethoxylated alcohol surfactants; **b** SDS and **c** CTAB

The CMC of a surfactant is a good measure of its efficiency as a foaming agent; the lower the CMC, the more efficient the surfactant as a foamer. Therefore, a low CMC helps to form a greater amount of foam than a surfactant with a higher CMC of same type. The CMC of a surfactant also influences the foam stability. If the CMC of the surfactant solution is low then foam stability is high.

The molecular cross-sectional area can be determined using the following equation:

$$A = \frac{1}{\Gamma N_A} \quad (2)$$

where A is the molecular cross-section of the polar head group (\AA^2) and N_A is the Avogadro constant ($6.023 \times 10^{23} \text{ mol}^{-1}$). The surface excess concentration and molecular cross-sectional area were calculated using Eqs. 1 and 2 and are depicted in Table 1. A noticeable result was found in the case of nonionic surfactant systems where adsorption decreases with the increase in CMC of the surfactants. In the case of C12E4, the surfactant has a lower CMC and the Γ value is greater than the other surfactants (Brij C18E20 and C16E20). The surface activity of a surfactant decreases with an increasing hydrophilic-lipophilic balance (HLB) value (given in Table 1) implying that the surfactant with a lower HLB value tends to concentrate at the air/liquid interface instead of remaining in the bulk. As the HLB value increases, the CMC and the surface tension at CMC is also increased. The hydrocarbon chain length of the surfactant also influences the CMC. Generally, with increasing the hydrocarbon chain of the surfactant leads to the surfactant molecules being more hydrophobic. Therefore, it is also experimentally found that surfactants with longer hydrocarbon chains have a driving force for aggregation, and thus dramatically reduce the solution CMC. As the CMC values of the surfactants decrease the Γ values of the surfactants also increase. However this effect is not always applicable as the surfactants are sometimes of technical grade. It can also be deduced from Table 1 that as the Γ value increases, the molecular cross-section of the polar head group (A) decreases for the nonionic surfactants. As SDS and CTAB are different types of surfactants, such a comparison is therefore not valid. When the test solution shifted from water to brines and synthetic brine, the initial foam volume of SDS increases in brine and decreases in synthetic brine but in the case of CTAB it decreases in both brines and synthetic brine. In the case of nonionic surfactants, the initial foam volume decreases in all cases when it is shifted from water to brines and synthetic brine. The variation of NaCl concentration affects the foamability of the SDS solution. Since SDS is an anionic surfactant, in presence of

NaCl, the SDS is therefore adsorbed on the liquid film array tightly and the foaming power of SDS increased with increasing NaCl concentration as shown in the Fig. 1. However, in the case of synthetic brine, foamability of SDS decreases due to the presence of Ca^{2+} and Mg^{2+} cations, which cause the SDS to precipitate in synthetic brine. On the other hand, CTAB is a cationic surfactant in which a positive charge resides on the nitrogen atom therefore the monovalent Na^+ cations of brines (and other cations of synthetic brine) experience a repulsive force and the foaming power of CTAB is reduced by decreasing the adsorbing power of the CTAB surfactant on the air–water interface. Since the nonionic surfactants are neutral, the adsorption of surfactants on the liquid film is very low in the presence of NaCl; therefore, foamability of the surfactants decreases in all cases.

The foamability of mixed surfactant systems is depicted in Fig. 3. More or less all the mixed surfactant systems show higher foamability than the individual system. In synthetic brine, the initial foam volume of the MS1 system decreases drastically compared to water. In the case of mixed surfactant systems, a salt effect on foamability has been also found. A probable explanation can be made on the basis of the effect of salt on the CMC of the surfactant. With an increase in salt concentration, the CMC of the surfactant decreases and as a result the foamability increases [42]. It is also noticeable that nonionic surfactants show good foamability when they combine with anionic and cationic surfactants. Due to synergism, mixed surfactant systems exhibit better foaming power than that of single one. From the Fig. 3, it is clear that the synergism is very much more prominent in the case of a cationic/

nonionic surfactant mixture than an anionic/nonionic surfactant mixture.

Foam Stability

The foam stability of a surfactant solution is defined as the change in foam volume, i.e. the volume of liquid drained from the foam per unit time. Foams are thermodynamically unstable due to their high interfacial free energy, and their relative stability depends on several factors such as drainage, disproportionation and coalescence, viscosity and surface shear viscosity of the liquid phase. Foam stability is the property of the two air/water interfaces of the thin films which influences the formation and destruction of foam. Stabilization of foam is caused by van der Waals forces between the molecules in the foam, electrical double layers created by dipolar surfactants, and the Marangoni effect, which acts as a restoring force to the lamellae. Several destabilizing effects can break foam down. The influencing factors are (1) Gravitation causes drainage of liquid to the foam base, (2) osmotic pressure causes drainage from the lamellae to the Plateau borders due to internal concentration differences in the foam, while (3) Laplace pressure causes diffusion of gas from small to large bubbles due to pressure difference. Films can break under disjoining pressure. These effects can lead to rearrangement of the foam structure at scales larger than the bubbles, which may be individual or collective. The changes in foam volume as a function of time are shown in Figs. 4, 5, 6 and 7 for all the surfactants in distilled water, 2 wt% NaCl, 4 wt% NaCl and synthetic brine respectively. In all cases, the time evolution of the foam structure provides natural

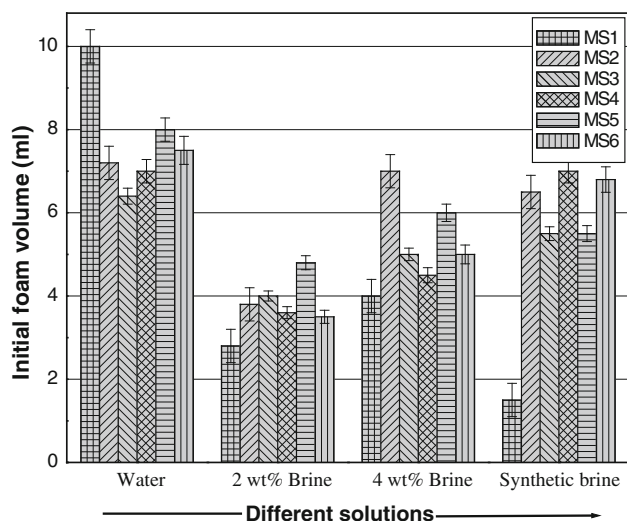


Fig. 3 Initial foam volume of different mixed surfactant solutions in distilled water, 2 wt% NaCl, 4 wt% NaCl, and synthetic brine

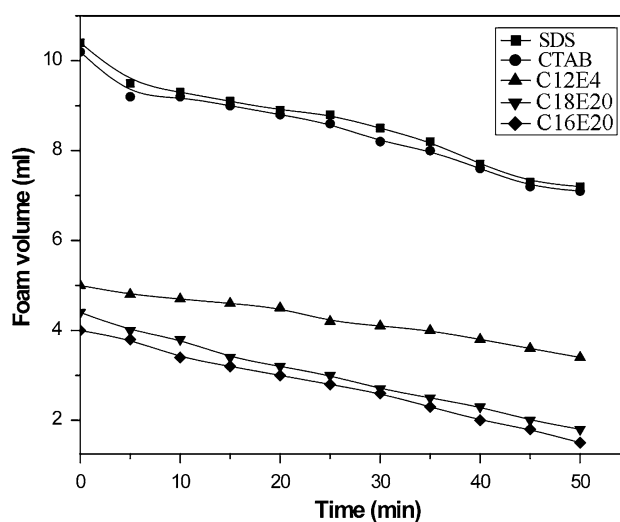


Fig. 4 Plot of foam volumes of different surfactants versus time in distilled water

quantifying foam stability [43]. The presence of dispersed particles of colloid in the continuous phase is one of the major factors responsible for the foam stability. They minimize the liquid drainage rate due to increased surface viscosity of the continuous phase. It was also reported that the characteristics of the colloid dispersion highly influence the stability and thinning behavior of foams [9, 44]. In the case of synthetic brine, the foam stability is quite low compared to other solutions. Due to the presence of different salts in the synthetic brine, the foam stability is affected noticeably. It is clear from the figures that the nonionic surfactants show good foam stability in some cases compared to other surfactants. Some researchers suggested that dry or metastable foams seem to show two different regimes of foam decay, one during the initial stage, immediately after foam formation, followed by a second one of comparatively slow drainage [33, 44]. The decrease in foam volume with time for the mixed surfactant systems is depicted in Figs. 8, 9, 10 and 11 in distilled water, 2 wt% NaCl, 4 wt% NaCl and synthetic brine respectively. In water, the mixed surfactant systems show better results regarding the foam stability than that of the individual surfactant systems. The foam stability of the mixed surfactant systems in water is also higher than the other solutions (2 wt% NaCl, 4 wt% NaCl and synthetic brine).

Surface Tension of Surfactant Solutions in Saline Water

For measurement of the surface tension of surfactant solutions, all the surfactants were used to prepare solutions at 0.5 wt% concentration in water, 2 wt% NaCl, 4 wt% NaCl and synthetic brine. In Fig. 12, surface tensions of different anionic, cationic and nonionic surfactants in water have been plotted for comparison of their surface activities.

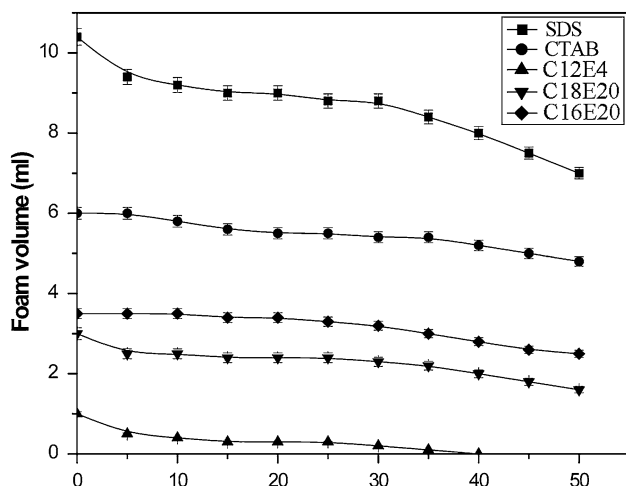


Fig. 5 Plot of foam volumes of different surfactants versus time in 2 wt% NaCl

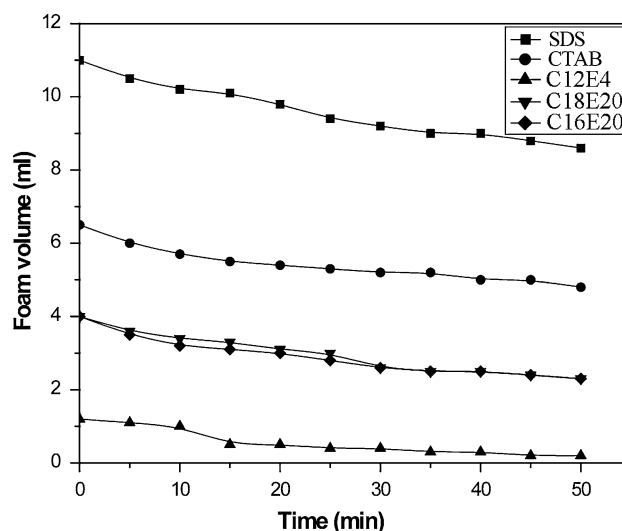


Fig. 6 Plot of foam volumes of different surfactants versus time in 4 wt% NaCl

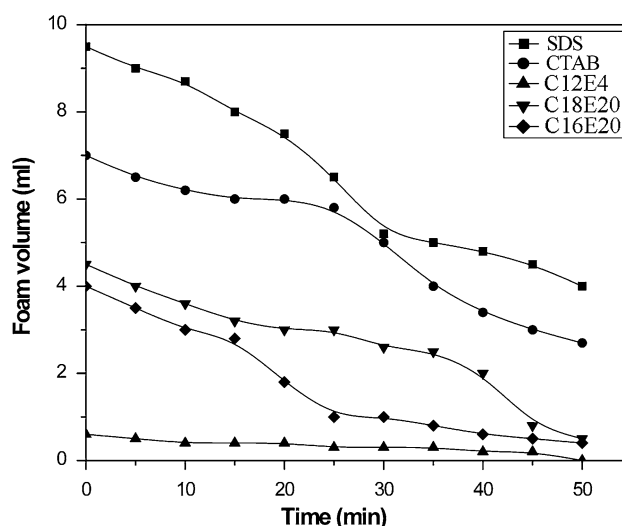


Fig. 7 Plot of foam volumes of different surfactants versus time in synthetic brine

Surface tensions of water, 2 and 4 wt% NaCl solutions, and synthetic brine have been also shown in the same graph for general comparison. Among the surfactants, C2E4 shows the lowest surface tension (26.3 mN/m) in water. Surface tensions of 2 wt% NaCl, 4 wt% NaCl, water and synthetic brine have been reported as 60, 61, 58 and 71 mN/m, respectively. The ST value of synthetic brine was evaluated for comparison purposes and a lower value when compared with water or brine i.e., 58 mN/m, was measured. ST values for all the anionic, cationic and nonionic surfactant solutions prepared in water, brines, and synthetic brine are shown in Fig. 13 together. ST values for surfactant

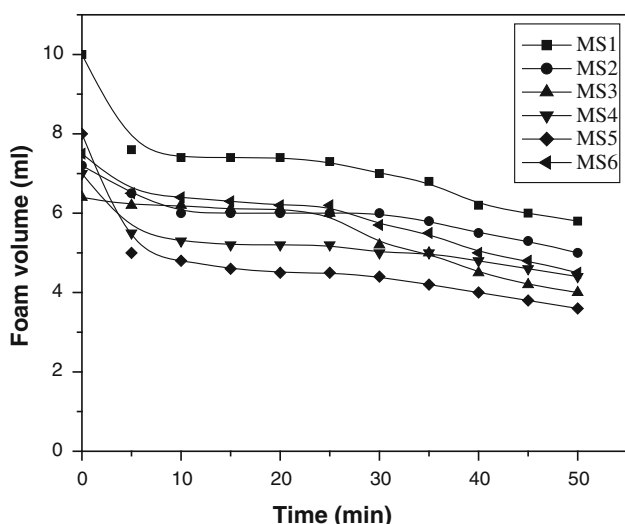


Fig. 8 Plot of foam volumes of different mixed surfactants versus time in distilled water

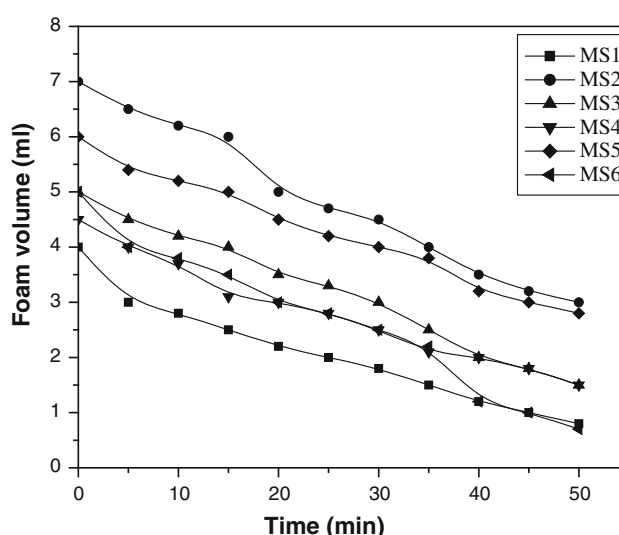


Fig. 10 Plot of foam volumes of different mixed surfactants versus time in 4 wt% NaCl

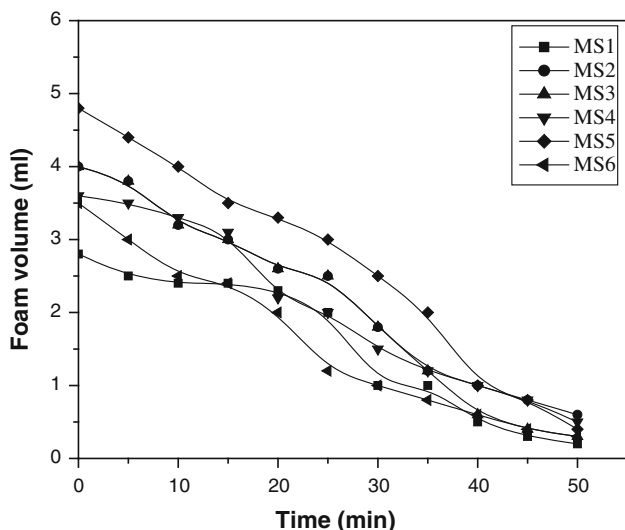


Fig. 9 Plot of foam volumes of different mixed surfactants versus time in 2 wt% NaCl

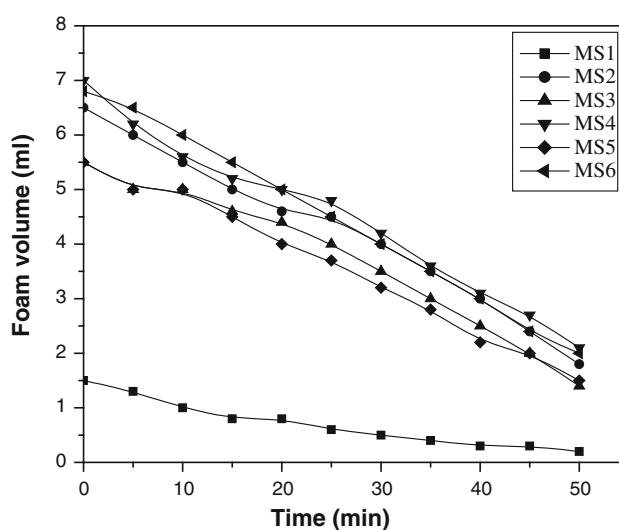


Fig. 11 Plot of foam volumes of different mixed surfactants versus time in synthetic brine

solutions in brine are found to be smaller than those of the corresponding water solution. It is important to report that in the case of synthetic brine due to the presence of a large amount of salts, including Mg and Ca, SDS was precipitated. Due to precipitation, ST values of the surfactant solutions in synthetic brine show high values compared to other solutions. To prevent precipitation of the SDS surfactant, different alternatives may be used like nonionic/anionic surfactants mixtures, and addition of sodium etc. In case of synthetic brine, all the nonionic surfactants show higher ST values than those of the other solutions. This is due to the presence of different salts in the synthetic brine, which reduces the activity of the surfactants.

Surface Tensions of Mixed Surfactants

Surface tension of a particular surfactant solution can be modified by mixing of any two types of surfactants (anionic/nonionic, cationic/nonionic, and anionic/cationic). In this study, a mixture of anionic/nonionic and cationic/nonionic surfactants was used for characterization. A noticeable change in surface tension of mixed surfactant systems was observed from Fig. 14. In water, the MS4 surfactant system showed a surface tension of 23.2 mN/m, which is lower than that of any surfactant solution alone which have been used to prepare the MS4 system. On the other hand, the MS2 surfactant system showed the highest

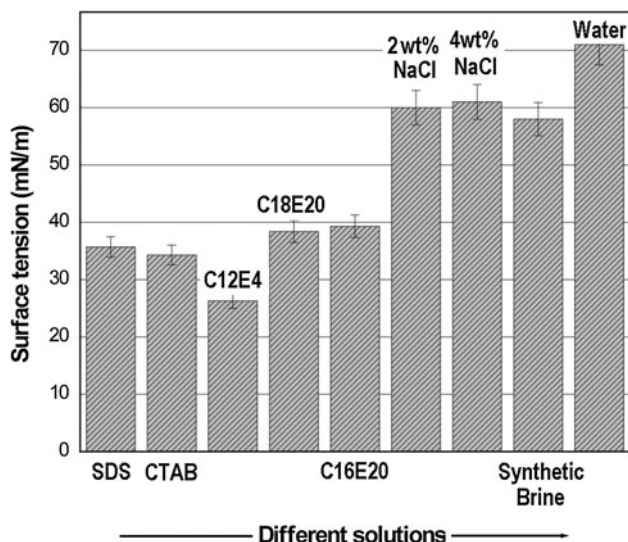


Fig. 12 Surface tensions for different surfactants (0.5 wt%) in water at 300 K

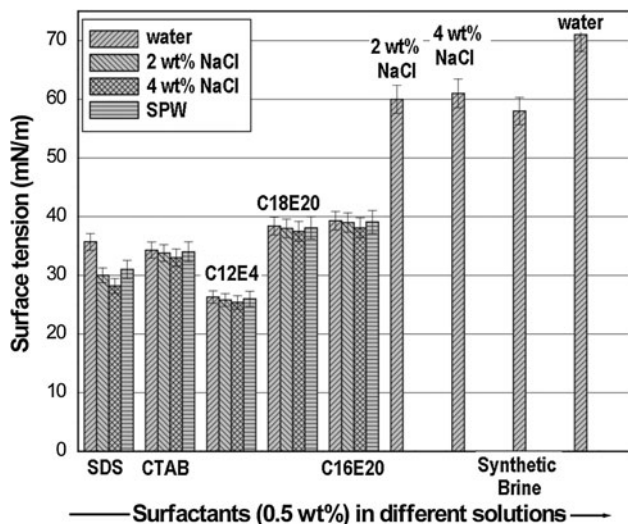


Fig. 13 Surface tensions for different surfactants (0.5 wt%) in water, brines, and synthetic brine at 300 K

ST value in water. Therefore, it is clear from the surface tension values of mixed surfactant systems that a cationic/nonionic system can modify surface properties more efficiently than an anionic/nonionic system. The reduction in surface tension of a mixed surfactant solution can be explained based on the synergism. In the case of a cationic/nonionic surfactant mixture, synergism is very much pronounced than an anionic/nonionic surfactant system. This phenomenon can be explained by the Γ value of the cationic surfactant. Since the Γ value is greater than other surfactants, therefore the combination with a nonionic surfactant gives better results than that of the others. In synthetic brine, both low and high synergism takes place in

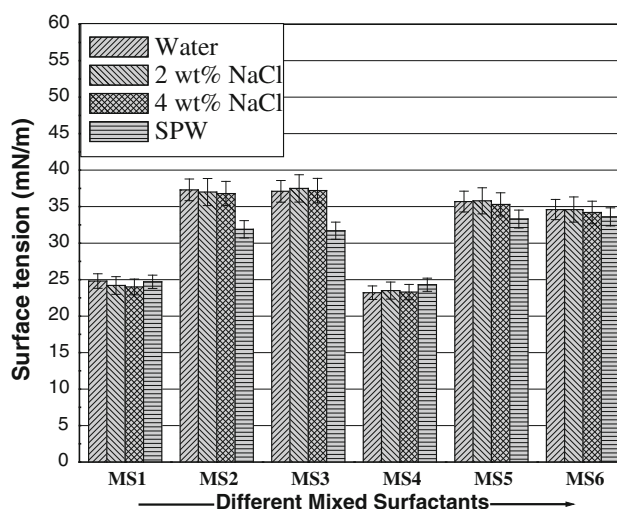


Fig. 14 Surface tensions for different mixed surfactant systems (0.5 wt%) in water, brines, and synthetic brine at 300 K

the case of a cationic/nonionic surfactant system due to presence of different salts.

Conclusions

Foamability and foam stability of different surfactants and mixed surfactant systems were studied for their application in enhanced oil recovery. SDS shows a maximum foamability in a 4 wt% NaCl solution. Foamability of nonionic surfactants depends on the CMC value. Adsorption of nonionic surfactants at an air–water interface decreases as the CMC values of the surfactants increase. In water, foamability of mixed surfactant systems increases for all the solutions. In a 4 wt% NaCl solution, all the surfactants shows better foam stability than the other solutions. Among the surfactants, C12E4 shows the lowest surface tension (26.3 mN/m) in water. For the mixed surfactant systems, the MS4 surfactant system shows a surface tension of 23.2 mN/m, which is lower than that of the individual surfactants. In the case of a cationic/nonionic surfactant mixture, synergism is very much more pronounced than in an anionic/nonionic surfactant system. For the synthetic brine, high synergism takes place in the case of a cationic/nonionic surfactant system due to the presence of different salts. The present study provides useful information for selecting suitable mixed surfactant systems for enhanced oil recovery.

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