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ORIGINAL ARTICLE

Synthesis and Properties of Organosilicon Quaternary Salts Surfactants

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Abstract Diethyl-benzyl-[3-methyldimethoxyl)]silpropyl ammonium chloride (DEBSAC) was synthesized from *N*,*N*-diethyl-aminopropyl-methyldimethoxysilane (DEAPMDES) and benzyl chloride. Diethyl-2,3-epoxypropyl-[3-methyldimethoxyl)] silpropyl ammonium chloride (DEEPSAC) was synthesized from DEAPMDES and epoxy chloropropane. Trimethyl-[3-methyldimethoxyl)] silpropyl ammonium chloride (TMSAC) was synthesized from trimethylamine and γ-chloropropylmethyldimethoxysilane (CPMDMS). The products were characterized by ^{1}H NMR, ^{13}C NMR, and IR. The surface tension measurements showed that the organosilicon quaternary salts exhibit a lower surface tension and a lower critical micelle concentration (CMC) than the hydrocarbon counterparts. The plate count method experiments illustrated that DEEPSAC has a better degree of antibacterial activity against *Escherichia coli* than DEBSAC. The solubilizing effects of the organosilicon quaternary salts on organosilicone were studied by pseudo-ternary phase diagrams of synthesized products/cosurfactant(*n*-butanol)/water/octamethylcyclotetrasiloxane. The solubilizing activities decreased in the order of TMSAC > DEBSAC > DEEPSAC.

Keywords Organosilicon quaternary salt · Surface tension · Antibacterial activity · Phase behavior diagram

Introduction

Organosilicon quaternary salts are a novel type of cationic surfactants with hydrophobic groups containing silicon. This type of surfactant has, in general, a lower critical micelle concentration (CMC) and provides lower surface tensions than surfactants whose hydrophobic portions are entirely hydrocarbon in nature. Organosilicon quaternary salts exhibit excellent properties due to the characteristics of organosilicon, such as thermal stability, low-temperature flexibility, anti-static properties, water resistance, biological and chemical inertness, and very low surface energy. Quaternary ammonium group structure also lends to novel antibacterial and surface activities. Thus, silicone-containing cationic surfactants have excellent surface activity, low toxicity and antimicrobial activity on Gram-positive and Gram-negative bacteria, yeast, fungi, and mold. Organosilicon quaternary salts have been used in the textile industry to provide fabric for antibacterial activity, as well as improved wicking, softness, elasticity, and anti-static behavior. The reagents prepared by organosilicon quaternary salts can also be used in pesticide formulation [1–7].

Dow Corning first synthesized an organosilicon quaternary salt $[(CH_3O)_3Si(CH_2)_3N^+(CH_3)_2C_{18}H_{37}]Cl^-$, named DC-5700. It is not only a novel cationic surfactant, but also a reactive silane that is able to react with many materials, such as glass, cotton, or polyester fibers by hydrolyzed silanol. The treated materials show an antibacterial property that is maintained after repeated washings [7]. The toxicity experiments with this product indicated that the median lethal dose is $LD_{50} = 12.27$ g/kg ± 0.116 g/kg,

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and that it cannot be absorbed into the body through finished fabric. This essentially shows that the organosilicon quaternary salt is probably a safe antibacterial agent.

Organosilicon quaternaries were prepared generally by an Si–H addition reaction to get tertiary amino silicon, which then reacted with haloalkane [8–10] or by the Menschutkin reaction of halogenoalkyl silicone unit with tertiary amines [2, 7, 11].

Hazziza-Laskar et al. [12, 13] prepared polysiloxane quaternaries by the following three steps: the first was the preparation of 1-(*N*,*N*-dimethylaminopropyl)1,1,3,3-tetramethyl disiloxane (M'_2A) by hydrosilylation with platinum catalysts. The second step was the grafting of M'_2A onto the 1,2-units of a hydroxytelechelic polybutadiene by hydrosilylation. The last step was the quaternization of the pendant tertiary amino groups in methanol with alkyl bromides from $C_8H_{17}Br$ to $C_{16}H_{33}Br$. The final products were characterized by ^{1}H-NMR and FT-IR. A series of concentrations of the compounds made by Hazziza-Laskar et al. were covered to form films. It was concluded that the water solution of 1 mmol/g had good germ inhibition to *Escherichia coli*.

Sauvet et al. [7, 14] and Huang and Huang [11, 15] synthesized organosilicon containing quaternary ammonium salt groups by reacting polysiloxane-bearing chlorohydrocarbon with tertiary amine. Huang and Huang found that the reaction is almost a second-order reaction when chloropropyl groups have low reaction yield, and the rate constant obviously increases when the reaction yield of chloropropyl groups exceeds 40%. They treated 100% cotton textiles with this compound and found that this kind of organosilicon quaternary salt had a novel antibactericidal activity on *E. coil* and *Staphylococcus aureus* when the mass concentration exceeds 1.5%. Sauvet et al. found that the compounds have high antibactericidal activity in a water solution toward *E. coli* and *S. aureus*. They also reported that there is no essential difference in the activity with different quaternary ammonium unit arrangement in the compounds.

In the present work, using organosilicon tertiary amine and chloroalkane, or γ-chloropropylmethyldimethoxysilane and tertiary amine as the raw materials, a series of organosilicon cationic surfactants have been successfully synthesized via the Menschutkin reaction, through a one-step reaction without catalyst. The surface activity, antibacterial property, and solubilization are reported.

Experimental Procedures

Materials

γ-Chloropropylmethyldimethoxysilane was purchased from Nanjing Yudeheng Coupling Reagent Corporation, redistilled under vacuum, *N*,*N*-diethyl aminopropyl methyldimethoxysilane (DEAPMDEOS) was provided by Shandong Provincial Key Laboratory of Fine Chemicals, epoxy chloropropane and benzyl chloride were purchased from Shanghai Chemical Reagent Corporation, and redistilled. 30% trimethylammonium alcohol solution was purchased from Shanghai Chemical Reagent Corporation. Octamethylcyclotetrasiloxane (D_4) was purchased from Dow Corning, and redistilled.

Instruments

Surface tensions were measured by the du Noüy ring method on a Chengde Dingsheng JYW-200 auto interfacial tensiometer. Fourier transform infrared (FT-IR) spectra were obtained on a Nicolet NEXUS 470 FT-IR spectrometer. ^{1}H-NMR and ^{13}C-NMR solution spectra were recorded on a Bruker Avance II 400 MHz NMR spectrometer.

Synthesis of Trimethyl-[3-methyldimethoxyl)]silpropyl Ammonium Chloride (TMSAC)

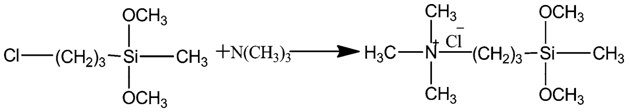
30% trimethylammonium ethanol solution and γ-chloropropylmethyldimethoxysilane were mixed in a clean, dry, and closed reactor, with a mole ratio of the respective substances of 1.3:1. The reaction temperature was adjusted to 90 °C and the mixture was stirred for 12 h. After the completion of the reaction, the solvent and excess reactants were evaporated. The remaining product was washed with ether several times and dried in a vacuum oven at 40 °C. The yield of the reaction shown in Scheme 1 was about 90%.

^{1}H-NMR (δ,ppm,D_2O): $\delta_{0.05}$(m,SiCH_3), $\delta_{3.0}$[m,$N^+(CH_3)_3$], $\delta_{3.3}$[m,Si$(OCH_3)_2$], $\delta_{0.95}$(m,Si$CH_2CH_2CH_2N^+$), $\delta_{1.65}$(m,Si$CH_2CH_2CH_2N^+$), $\delta_{2.7}$(m,Si$CH_2CH_2CH_2N^+$).

^{13}C-NMR [δ,ppm,$(CD_3)_2SO$]: $\delta_{-1.3}$(m,SiCH_3), δ_{67}[s,$N^+(CH_3)_3$], $\delta_{51.5}$[s,Si$(OCH_3)_2$], $\delta_{13.5}$(m,Si$CH_2CH_2CH_2N^+$), $\delta_{25.6}$(m,Si$CH_2CH_2CH_2N^+$), $\delta_{47.2}$(m,Si$CH_2CH_2CH_2N^+$).

Synthesis of Diethyl-benzyl-[3-methyldimethoxyl)] silpropyl Ammonium Chloride (DEBSAC)

DEAPMDEOS and anhydrous ethanol were mixed into a 100-mL, four-necked flask equipped with N_2 protection, stirring, thermometer, and reflux condenser. The mixture temperature was adjusted to reflux and benzyl chloride was



$$Cl-(CH_2)_3-Si(OCH_3)_2-CH_3 + N(CH_3)_3 \longrightarrow H_3C-\overset{+}{N}(CH_3)_2\,Cl^- -(CH_2)_3-Si(OCH_3)_2-CH_3$$

Scheme 1 Synthesis of trimethyl-[3-methyldimethoxyl)] silpropyl ammonium chloride (TMSAC) added dropwise. The mole ratio of DEAPMDEOS to benzyl chloride is 1:1.2. The mixture was refluxed at 80 °C for 22 h. After the completion of the reaction, the solvent and excess reactant were evaporated under high vacuum. The remaining product is light-yellow and water-soluble. The reaction equation is shown in Scheme 2.

IR (KBr smear): 1,629.12 and 3,196.76 $\mathrm{cm^{-1}}$ are characteristic of benzene, 2,955.90 $\mathrm{cm^{-1}}(\nu_{\mathrm{CH3}})$, 1,456.02 $\mathrm{cm^{-1}}$ (δ_{CH2}), 1,397.04 $\mathrm{cm^{-1}}(\delta_{\mathrm{CH3}})$, 2,904.95 $\mathrm{cm^{-1}}(\nu_{\mathrm{N\text{–}CH2}})$, 796.78 $\mathrm{cm^{-1}}(\delta_{\mathrm{N\text{–}CH2}})$, 1,198.87 $\mathrm{cm^{-1}}(\nu_{\mathrm{N\text{–}C}})$, 1,260.30 $\mathrm{cm^{-1}}$ (ν_{SiC}), 1,081.51 $\mathrm{cm^{-1}}(\nu_{\mathrm{SiO}})$.

^{1}H-NMR (δ,ppm,$\mathrm{CD_3Cl}$): $\delta_{1.38}$[m,$\mathrm{N^+(CH_2}\mathit{CH_3}\mathrm{)_2}$], $\delta_{3.22}$ [m,$\mathrm{N^+(}\mathit{CH_2}\mathrm{CH_3)_2}$], $\delta_{2.0}$(m,$\mathrm{N^+}\mathit{CH_2}\mathrm{CH_2CH_2Si}$), $\delta_{4.60}$(m,$\mathrm{N^+}\mathit{CH_2}\mathrm{C_6H_5}$), $\delta_{0.61}$(m,$\mathrm{N^+CH_2CH_2}\mathit{CH_2}\mathrm{Si}$), $\delta_{1.05}$(m,$\mathrm{N^+CH_2}\mathit{CH_2}$ $\mathrm{CH_2Si}$), $\delta_{0.19}$(m,$\mathrm{Si}\mathit{CH_3}$), $\delta_{7.3\text{–}7.4}$(s,s $\mathrm{N^+CH_2}\mathit{C_6H_5}$), $\delta_{3.51}$ (m,$\mathrm{SiO}\mathit{CH_3}$).

^{13}C-NMR [δ,ppm,$\mathrm{(CD_3)_2SO}$]: $\delta_{58.85}$[d,$\mathrm{N^+(}\mathit{CH_2}\mathrm{CH_3)_2}$], $\delta_{12.68}$[m,$\mathrm{N^+(CH_2}\mathit{CH_3}\mathrm{)_2}$], $\delta_{60.15}$(m,$\mathrm{N^+}\mathit{CH_2}\mathrm{CH_2CH_2Si}$), $\delta_{45.43}$(d,$\mathrm{N^+}\mathit{CH_2}\mathrm{C_6H_5}$), $\delta_{7.49}$(m,$\mathrm{N^+CH_2CH_2}\mathit{CH_2}\mathrm{Si}$), $\delta_{-1.17}$ (m,$\mathrm{Si}\mathit{CH_3}$), $\delta_{15.29}$(m,$\mathrm{N^+CH_2}\mathit{CH_2}\mathrm{CH_2Si}$), $\delta_{132.31}$[s,$\mathrm{N^+CH_2}$ $\mathit{C}\mathrm{(CH)_2(CH)_2CH}$], $\delta_{52.35}$(s,$\mathrm{SiO}\mathit{CH_3}$), $\delta_{129.92}$[s,$\mathrm{N^+CH_2}$ $\mathrm{C(}\mathit{CH}\mathrm{)_2(CH)_2CH}$], $\delta_{128.26}$[s,$\mathrm{N^+CH_2C(CH)_2(}\mathit{CH}\mathrm{)_2CH}$], $\delta_{127.75}$ [m,$\mathrm{N^+CH_2C(CH)_2(CH)_2}\mathit{CH}$].

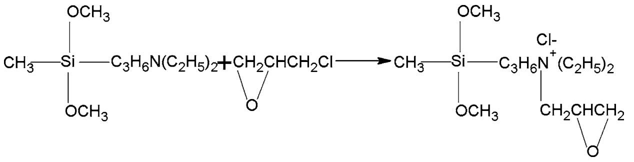
Synthesis of Diethyl-2,3-epoxypropyl-[3-methyldimethoxyl)] silpropyl Ammonium Chloride (DEEPSAC)

DEAPMDEOS and anhydrous methanol were added into a 100-mL, four-necked flask equipped with $\mathrm{N_2}$ protection, stirring, thermometer, and reflux condenser. The temperature was adjusted to 60 °C and a mixture of epoxy chloropropane and anhydrous methanol was added dropwise (the mole ratio of DEAPMDEOS and epoxy chloropropane is 1:1.2). The reaction was maintained at 60 °C for 6 h. After the completion of the reaction, the solvent methanol was evaporated. The left product was washed four times with ether and dried in a vacuum oven for 5 h. The reaction equation is shown in Scheme 3.

IR (KBr smear): 920 and 775 $\mathrm{cm^{-1}}$ are characteristic of epoxy, 2,955.67 $\mathrm{cm^{-1}}(\nu_{\mathrm{CH3}})$, 2,988.01 $\mathrm{cm^{-1}}(\nu_{\mathrm{CH2}})$, 1,482.55 $\mathrm{cm^{-1}}(\delta_{\mathrm{CH3}})$, 2,904.95 $\mathrm{cm^{-1}}(\nu_{\mathrm{N\text{–}CH2}})$, 799.83 $\mathrm{cm^{-1}}(\delta_{\mathrm{N\text{–}CH2}})$, 1,201.46 $\mathrm{cm^{-1}}(\nu_{\mathrm{N\text{–}C}})$, 1,282.47 $\mathrm{cm^{-1}}(\nu_{\mathrm{SiC}})$, 1,085.47 $\mathrm{cm^{-1}}$ (ν_{SiO}).

^{1}H-NMR (δ,ppm,$\mathrm{CD_3OD}$), $\delta_{0.025}$(m,$\mathrm{Si}\mathit{CH_3}$), $\delta_{0.45}$ (m,$\mathrm{Si}\mathit{CH_2}$), $\delta_{0.97}$(m,$\mathrm{N^+CH_2}\mathit{CH_2}\mathrm{CH_2Si}$), $\delta_{3.29}$(m,$\mathrm{SiO}\mathit{CH_3}$), $\delta_{3.1}$(m,$\underset{\diagdown\,\mathrm{O}\,\diagup}{\mathit{CH_2}\mathrm{CH}}$——), $\delta_{1.65}$(m,$\mathrm{N^+}\mathit{CH_2}$—), $\delta_{1.15}$[m,$\mathrm{N^+(CH_2}$ $\mathit{CH_3}\mathrm{)_2}$], $\delta_{3.14}$(m,$\underset{\diagdown\,\mathrm{O}\,\diagup}{\mathrm{CH_2}\mathit{CH}}$——).

^{13}CNMR [δ,ppm,$\mathrm{(CD_3)_2SO}$]: $\delta_{-1.43}$(m,$\mathrm{Si}\mathit{CH_3}$), $\delta_{6.77}$(m,$\mathrm{Si}\mathit{CH_2}$), $\delta_{53.2}$(s,$\mathrm{SiO}\mathit{CH_3}$), $\delta_{14.76}$(m,$\mathrm{N^+CH_2}\mathit{CH_2}$ $\mathrm{CH_2Si}$), $\delta_{64.98}$(s, $\underset{\diagdown\,\mathrm{O}\,\diagup}{\mathit{CH_2}\mathrm{CH}}$——), $\delta_{12.46}$[m,$\mathrm{N^+(CH_2}\mathit{CH_3}\mathrm{)_2}$], $\delta_{59.55}$(m,$\mathrm{N^+}\mathit{CH_2}\mathrm{CH_2CH_2Si}$), $\delta_{58.19}$[m,$\mathrm{N^+(}\mathit{CH_2}\mathrm{CH_3)_2}$], $\delta_{63.17}$(s, $\mathrm{N^+}\mathit{CH_2}\underset{\diagdown\,\mathrm{O}\,\diagup}{\mathrm{CH\,CH_2}}$), $\delta_{73.84}$(s,$\underset{\diagdown\,\mathrm{O}\,\diagup}{\mathrm{CH_2}\mathit{CH}}$——).


Scheme 3 Synthesis of diethyl-2,3-epoxypropyl-[3-methyldimethoxyl)] silpropyl ammonium chloride (DEEPSAC)

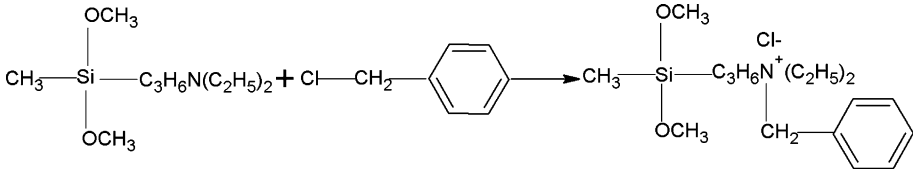
Measurements

Surface tensions of aqueous solutions of the surfactants were measured with the ring tensiometer technique.

The antibacterial activity of the water-soluble DEBSAC, DEEPSAC, and TMSAC was determined by testing different concentrations of the compounds against *E. coli* and *Aspergillus flavus* with broth-dilution and spread-plate methods. Surfactants solutions at a range of concentrations were prepared with sterile, double-deionized water in multiwell microtiter plates. The test organisms were added. The microtiter plates were incubated at 37 °C for 24 h in a shaker. At the end of this period, a small amount of the mixture from each sample was withdrawn and spread on agar plates with a swap, and the plates were incubated at 37 °C for 48 h. The growth of bacterial cells on agar plates was observed in order to determine the corresponding activity.

The phase diagram of the pseudo-ternary systems water-octamethyl cyclotetrasiloxane($\mathrm{D_4}$)-surfactant-cosurfactant were obtained with the cloud point technique. Active mixture (AM) and $\mathrm{D_4}$ were added into a bottle with a water jacket and kept under electromagnetic stirring for 30 min;

Scheme 2 Synthesis of diethyl-benzyl-[3-methyldimethoxyl)] silpropyl ammonium chloride (DEBSAC)



then, water was added dropwise in the AM/D_4 mixture until the solution visually turned cloudy.

Results and Discussion

Surface Tension

Organosilicons are very hydrophobic, but their water solubility is greatly increased by the quaternization. Figure 1 shows that DEBSAC, TMSAC, and DEEPSAC have typical surface activity. The concentration at which the surface tension stops dropping is the CMC. Figure 1 shows that the CMC is about 0.0166 mol/L, 0.0182 mol/L, and 0.0275 mol/L for DEBSAC, DEEPSAC, and TMSAC, respectively. The minimum of the surface tension is about 30, 36, and 38 mN/m for DEEPSAC, TMSAC, and DEBSAC, respectively, which indicates that the ability of the reduction of the surface tension is in the order of DEEPSAC > TMSAC > DEBSAC.

Antibacterial Activity

The bactericidal activity of the aqueous solution of DEEPSAC and DEBSAC against *E. coli* was determined with plate count methods (Figs. 2 and 3). The antibactericidal activity exceeds 5 orders of magnitude (indicated as a log reduction) when the weight concentration of DEEPSAC in solution is only 0.1%; in comparison with DEBSAC, its bactericidal activity is a 1.1 log reduction when the concentration is 0.12%. TMSAC has no antibacterial activity against *E. coli*. Consequently, the structure of the alkyl chain bonded to N^+ has a considerable influence on the antibacterial activity. When the alkyl chain bonded to N^+ is an epoxypropyl, it has a very strong antibacterial activity against *E. coli*, whereas benzyl takes the second place. When the alkyl chain bonded to N^+ is a simple methyl, there is no antibacterial activity against *E. coli*.

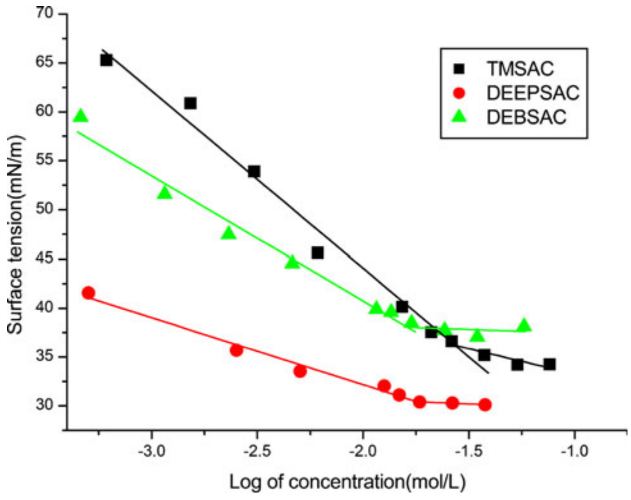


Fig. 1 Surface tension of the organosilicon quaternary salts

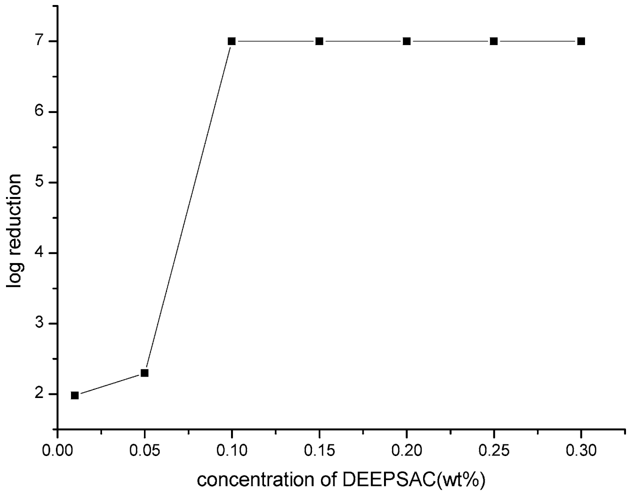


Fig. 2 Bactericidal activity of DEEPSAC against *E. coli*

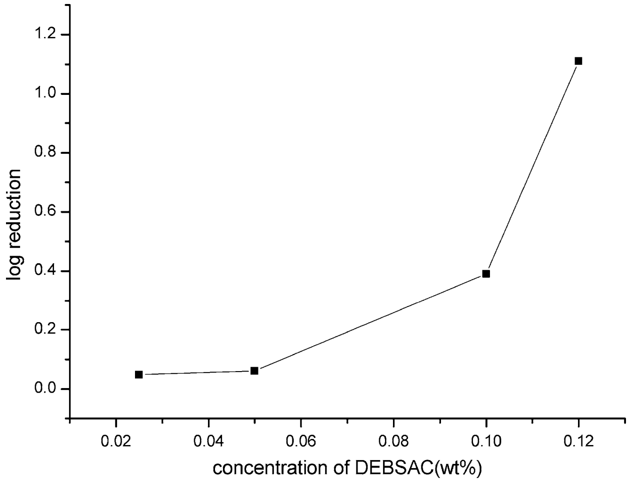


Fig. 3 Bactericidal activity of DEBSAC against *E. coli*

Regarding the antibacterial mechanism of DEEPSAC and DEBSAC, it is generally agreed that the compounds adsorb onto the bacterial cells surface, diffuse through the cell wall, bind to the cytoplasmic membrane, and disrupt the cytoplasmic membrane [16]. DEEPSAC can tightly bond to the bacterial cell surface by the epoxy group reacting with the hydroxyl and amino of cells, hence, resulting in an excellent antibacterial activity against *E. coli* compared to DEBSAC.

Phase Diagrams of Pseudo-Ternary Systems

TMSAC, DEBSAC, and DEEPSAC are organosilicon cationic surfactants. A microemulsion can be obtained by

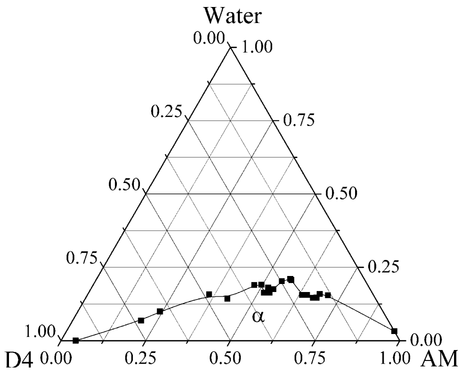


Fig. 4 Pseudo-ternary phase diagram of AM (10% TMSAC + 90% *n*-butanol)/D_4/H_2O

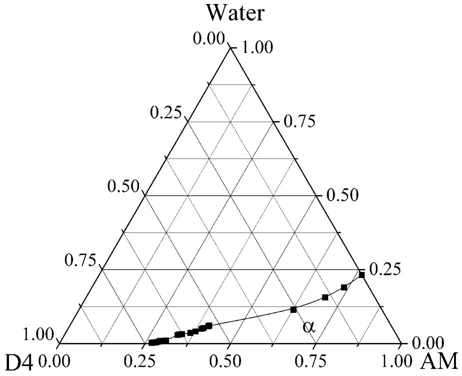


Fig. 5 Pseudo-ternary phase diagram of AM (10% DEBSAC + 90% *n*-butanol)/D_4/H_2O

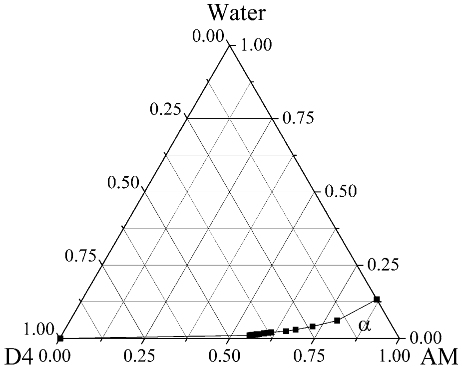


Fig. 6 Pseudo-ternary phase diagram of AM (10% DEEPSAC + 90% *n*-butanol)/D_4/H_2O

mixing these surfactants, cosurfactants, water, and oil in an appropriate composition. The studied system contains four components: water, octamethylcyclotetrasiloxane (D_4), organosilicon surfactant, and cosurfactant. One of the ways of reducing this system to a pseudo-ternary system is keeping the ratio of the surfactant and cosurfactant constant and calling this the active mixture (AM) [17]. Choosing D_4 as the oil phase, the solubilizing effects of the surfactants on organosilicon were studied. Phase diagrams involving D_4, water, and the AM are shown in Figs. 4, 5, and 6. The concentration of every organosilicon cationic surfactant is 10 wt% in AM. The temperature is 45 °C. In Figs. 4, 5, and 6, the region α is the microemulsion phase zone where the system remains monophasic. The area of region α is in the order of TMSAC > DEBSAC > DEEPSAC and, thus, indicates that TMSAC has the strongest ability to form a microemulsion. The three compounds have the same silane group, but different cationic groups. TMSAC has the best water solubilization power due to its more hydrophilic cationic group.

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