
PHYSICOCHEMICAL STUDIES
OF SYSTEMS AND PROCESSES

Dependence of the Oil-Collecting and Oil-Dispersing Ability of Surfactants on the Kind of Alkyl Radicals in Their Head Group

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Abstract—Ionic liquid (IL) surfactants were prepared from 1-bromododecane and ethanolamines (2-dimethyl- and 2-diethylethanolamines) in an equimolar ratio to reveal how their oil-collecting and dispersing ability is influenced by the structure of the alkyl fragment. The surface activity of the substances synthesized was studied tensiometrically, and the electrical conductivity, conductometrically. The effect exerted on the colloid-chemical parameters of the surfactants by the replacement of the methyl fragment in the head group by the ethyl fragment is described. The relative oil-collecting and oil-dispersing ability of these substances was evaluated under laboratory conditions by the example of a thin oil film on the surface of waters with different levels of mineralization.

Keywords: dodecyldimethylethylolammonium bromide, dodecyldiethylethylolammonium bromide, surface activity, specific conductivity, oil collection, oil dispersion

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Ionic liquids (ILs) have recently made a revolution in research centers and chemical industry. The interest in them is due to many valuable properties of ILs, such as low volatility, good solubility, high catalytic activity, low corrosion activity, low toxicity, very high electrical and thermal conductivity, thermal stability, etc. [1–4]. The diversity of properties makes ILs promising for various fields of economy and opens the way to green chemistry [5–13].

The majority of ILs are amphiphilic; i.e., they contain hydrophilic and hydrophobic fragments. This determines their surface activity and tendency to self-organization and aggregation both in the neat form and in solution [14]. The studies being performed are mainly focused on IL micellization in solutions [15]. Numerous studies have been made to obtain IL-type surfactants. The results of previous studies show that the surface activity and applied properties of surfactants can be improved by appropriate modification of their hydrophilic and hydrophobic groups [16–20]. Harkot and Janczuk [21]

determined the isotherms and thermodynamic parameters of the adsorption for aqueous solutions of two cationic surfactants, dodecylethyldimethylammonium bromide and benzyldimethyldodecylammonium bromide, from the surface tension data. It follows from the experimental and calculated data that the difference in the structure of two cationic surfactants due to replacement of the methyl group bonded to the polar group by the aryl group improves the economic parameter (pC_{20}) and decreases the efficiency of the adsorption (Γ) at the aqueous solution–air interface. According to [21], the standard free energy of adsorption can be predicted from the surface tension of the surfactant solution assuming that the aryl group is equivalent to 2.5 methylene groups. Xing et al. [22] studied the micellization of dodecyltrimethyl/ethyl/propyl/butylammonium bromide ($C_{12}NM$, $C_{12}NE$, $C_{12}NP$, and $C_{12}NB$) by measuring the electrical conductivity at various temperatures. In the temperature range examined (15–45°C), the critical micelle concentration (CMC) and the degree of association of

counterions (β) decreased with an increase in the size of the head polar group. The decrease in CMC with an increase in the size of the surfactant head group is due to the fact that an increase in the length of the alkyl chain in head groups makes the surfactant more hydrophobic and thus favors aggregation of its molecules.

The environment protection is one of the most important problems of the modern world. Contamination of the surface of water bodies by petroleum products, associated with the active development of oil-extracting, oil-refining, and petrochemical industry and with an increase in the volume of oil and petroleum products transported by ships, can disturb the ecological balance [23–25], and the removal of such contaminations from water bodies is a pressing problem. Thick oil films are removed mechanically, but thin (less than 1 mm thick) oil films cannot be localized mechanically, and physicochemical means are efficient in this case. Reagents exhibiting oil-collecting and oil-dispersing properties occupy a particular place among such means [26, 27]. Thus, studies on the synthesis of IL surfactants exhibiting good oil-collecting and oil-dispersing properties are important for maintaining the ecological balance in the hydrosphere and are also of scientific and practical interest.

In this work, we prepared new IL surfactants based on 1-bromododecane and ethanolamines and studied their surface activity and applied properties.

EXPERIMENTAL

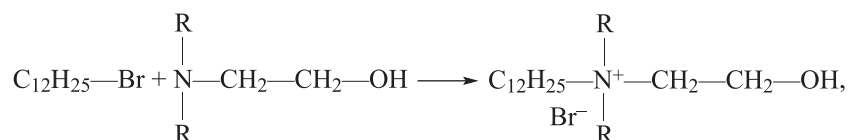
2-Dimethylaminoethanol (DMAE) and 2-diethylaminoethanol (DEAE) (reagent grade, >98%) were purchased from Merck. 1-Bromododecane (BDD) (reagent grade, >98%) was purchased from Sigma–Aldrich. The ^1H and ^{13}C NMR spectra were recorded with a Bruker Avance II+ 300 spectrometer (UltraShield™ Magnet) at the operating frequency of 300.18 and 75.46 MHz, respectively, using D_2O as a solvent. The IR spectra were measured with a Spectrum BX FTIR spectrometer using KBr pellets. The surface tension of the products was determined with a Du Nouy

KSV Sigma 702 tensiometer at the water–air interface by the ring detachment method. The specific electrical conductivity (κ) was measured with an ANION 4100 conductometer. The distilled water that we used had $\kappa = 3\text{--}4 \mu\text{S cm}^{-1}$. The dynamic light scattering (DLS) studies were carried out at CMC for each surfactant. The scanning was performed at 25°C with a HORIBA LB-550 particle size analyzer equipped with a 650 nm laser diode capable of generating a 5 mW light beam. Particles with the diameter from 1 to 6000 nm were scanned.

The oil-collecting and oil-dispersing performance of each surfactant was studied in three Petri dishes filled with seawater (from Caspian Sea), freshwater, and distilled water (40–45 mL). Oil films were prepared using crude oil from the Pirallahi oil field (Absheron Peninsula, Azerbaijan). The crude oil (density $\rho^{20} = 924.4 \text{ kg m}^{-3}$, kinematic viscosity $\nu^{30} = 105 \text{ cSt}$) was added into the Petri dishes in an amount of 2.5 vol % relative to the total water amount, and the surfactants were added in an amount of 0.02 g. On adding 5 wt % aqueous solutions of the surfactants, the spilled thin (thickness $\sim 0.17 \text{ mm}$) oil film is instantaneously localized to form a spot. The surfactant performance was judged from the change in the oil spot size on the water surface under the action of the surfactant solution. The oil-collecting performance was evaluated by the collection factor K , which was calculated as the ratio of the initial surface area of the spilled oil film to the surface area of the oil localized under the action of the surfactant. The surfactant performance in the dispersion of the oil film was characterized by the degree of decontamination of the water surface, K_D (%). The time of retention of the collected oil film is denoted as τ .

RESULTS AND DISCUSSION

The new IL surfactants were prepared by the reaction of DMAE and DEAE with BDD (reactant molar ratio 1 : 1) at $80\text{--}100^\circ\text{C}$ with stirring. In the first case, the reaction occurs within 1 h, and in the second case, it is complete in 2 h. The reaction equation can be written as follows:



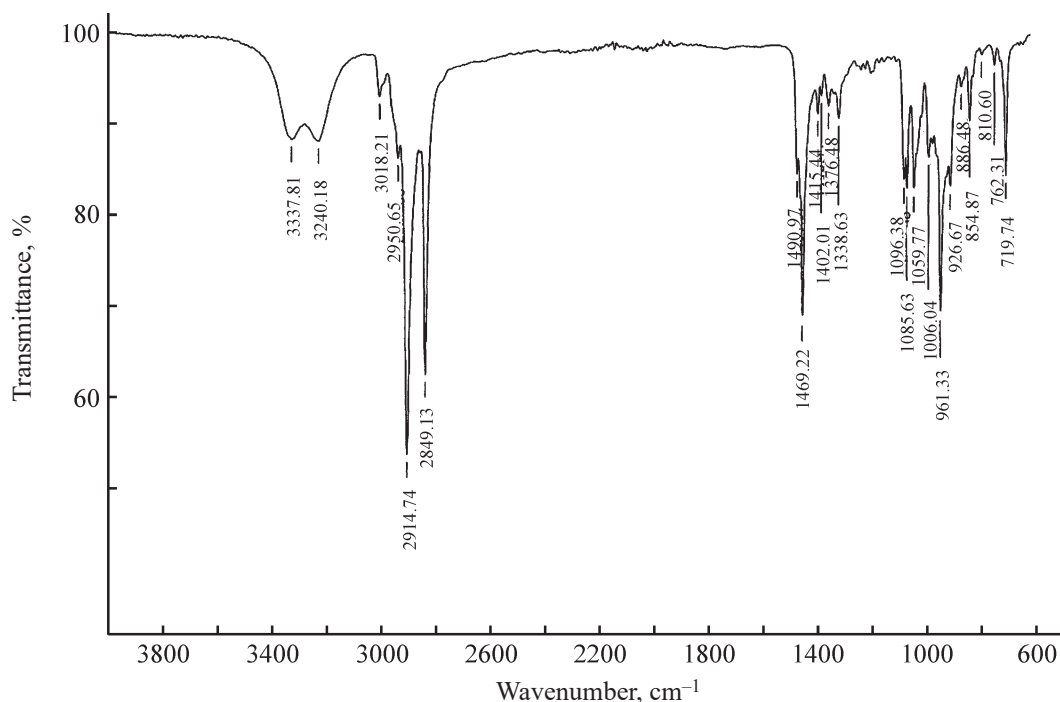


Fig. 1. IR spectrum of C₁₂DMEAB.

where R = CH₃ (dodecyldimethylethylammonium bromide, C₁₂DMEAB) or C₂H₅ (dodecyldiethylethylammonium bromide, C₁₂DEEAB).

The quaternary ammonium salt C₁₂DMEAB is a milky white solid with the melting point of 74.8°C. The salt is very readily soluble in acetone, ethanol, and ethyl acetate and undergoes strong foaming in water on shaking.

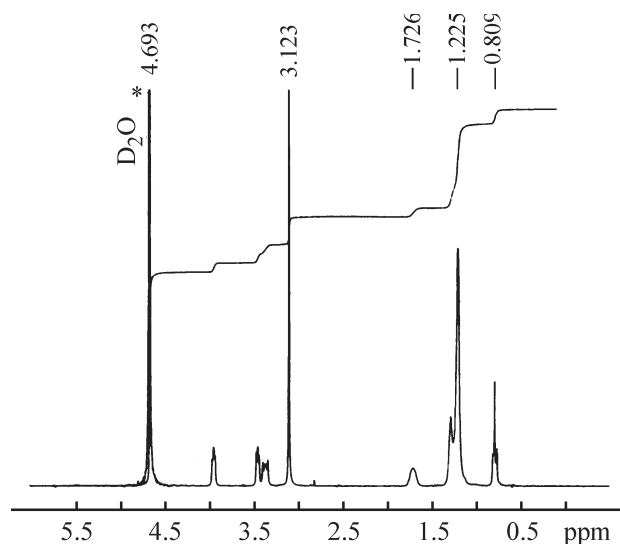


Fig. 2. ¹H NMR spectrum of C₁₂DMEAB.

The salts were identified by IR and ¹H, ¹³C NMR spectroscopy.

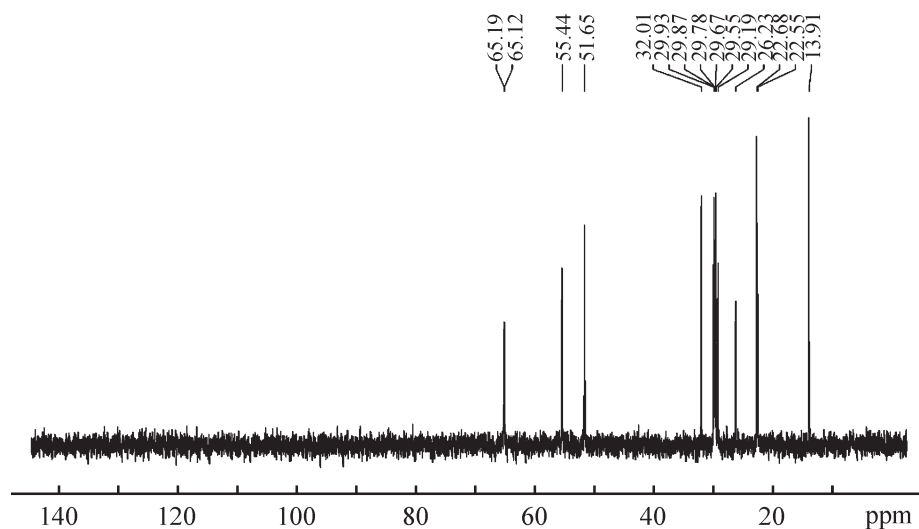
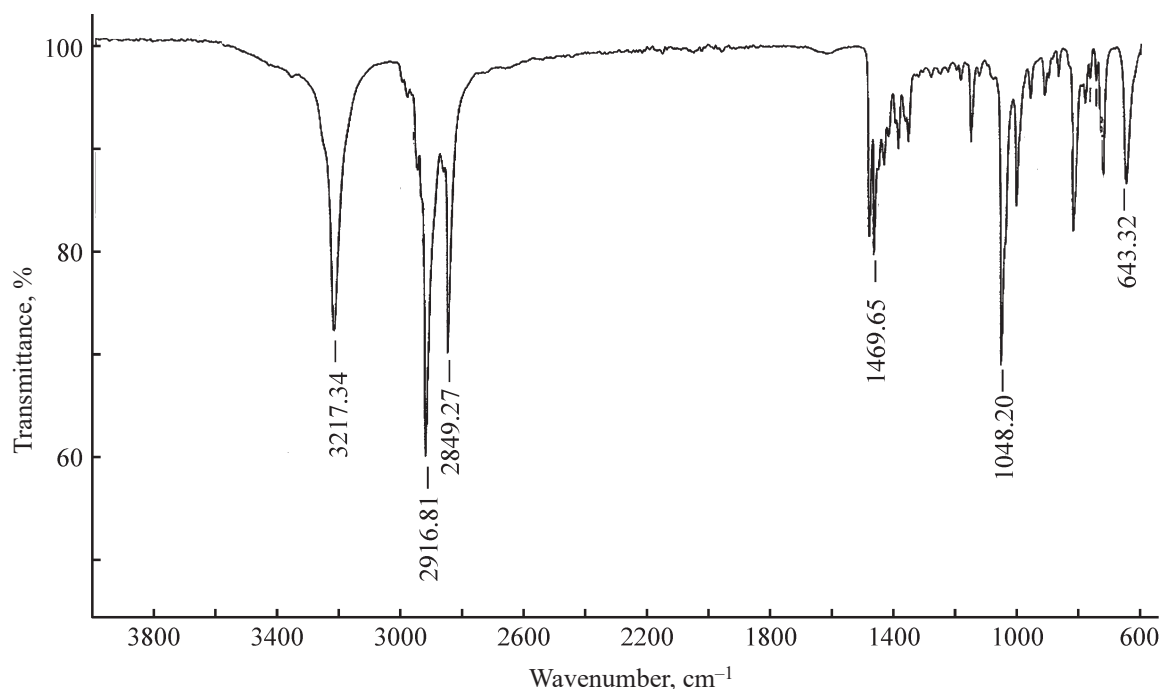
In the IR spectrum of C₁₂DMEAB (Fig. 1), there are the following absorption bands ν , cm⁻¹: 3338 and 3240, ν (OH); 2950, 2914, and 2849, ν (C–H); 1490, 1469, and 1376, δ (C–H); 1085, ν (C–N); 1059, ν (C–O); 719, δ (CH₂)_n.

¹H NMR spectrum of C₁₂DMEAB (Fig. 2) (300.18 MHz, D₂O), δ , ppm: 0.809 (CH₂–CH₃), 1.225 (CH₂ in chain), 1.726 (CH₂–CH₂–CH₂–N⁺), 3.123 (CH₂–CH₂–CH₂–N⁺), 3.361–3.377 (N⁺–CH₃), 3.400–3.480 (N⁺–CH₂–CH₂–OH), 3.953–3.984 (N⁺–CH₂–CH₂–OH).

¹³C NMR spectrum of C₁₂DMEAB (Fig. 3), δ , ppm: 13.91 (CH₃), 22.55–32.01 (CH₂ in alkyl group), 51.65 (N⁺–CH₃), 55.44 (N⁺–CH₂–CH₂–OH), 65.12–65.19 (CH₂–CH₂–CH₂–N⁺), (N⁺–CH₂–CH₂–OH).

The quaternary ammonium salt C₁₂DEEAB is a solid of coffee–milk color with the melting point of 63.7°C. The product is readily soluble in water, ethanol, acetone, and ethyl acetate. The aqueous solution undergoes strong foaming in shaking.

In the IR spectrum of C₁₂DEEAB (Fig. 4), there are the following absorption bands, ν , cm⁻¹: 3171, ν (OH); 2988, 2953, 2916, and 2849, ν (C–H); 1469 and

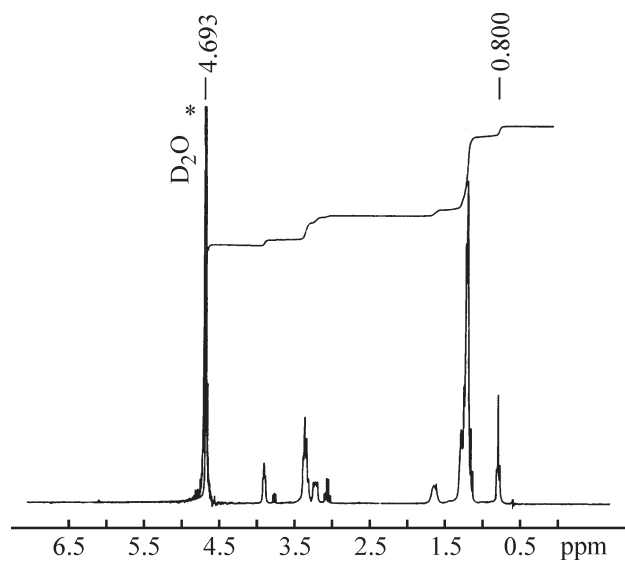
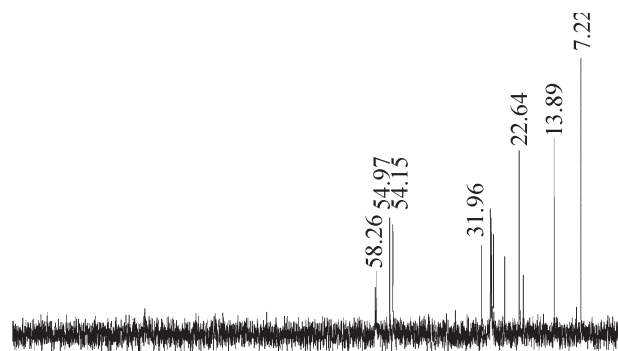
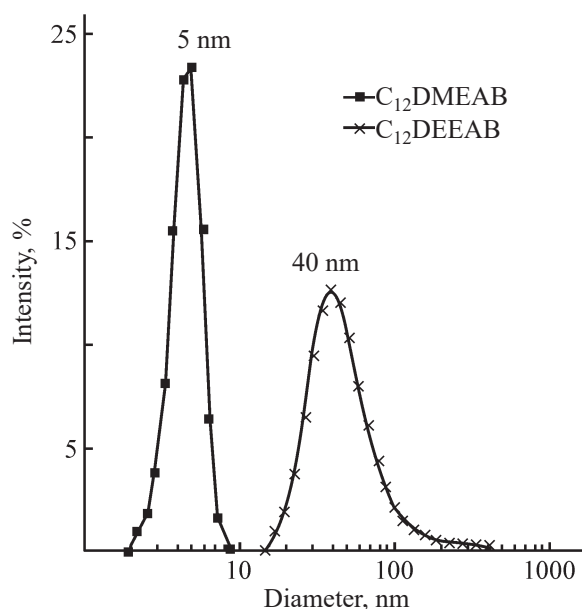
Fig. 3. ^{13}C NMR spectrum of $\text{C}_{12}\text{DMEAB}$.Fig. 4. IR spectrum of $\text{C}_{12}\text{DEEAB}$.

1360, $\delta(\text{C-H})$; 1154; $\nu(\text{C-N})$; 1048, $\nu(\text{C-O})$; and 720, $\delta(\text{CH}_2)_x$.

^1H NMR spectrum of $\text{C}_{12}\text{DEEAB}$ (Fig. 5) (300.18 MHz, D_2O), δ , ppm: 0.800 ($\text{CH}_2\text{-CH}_3$), 1.209–1.232 (CH_2 in chain), 1.622–1.651 ($\text{CH}_2\text{-CH}_2\text{-CH}_2\text{-N}^+$), 3.054–3.079 ($\text{CH}_2\text{-CH}_2\text{-CH}_2\text{-N}^+$), 3.201–3.256 ($\text{CH}_3\text{-CH}_2\text{-N}^+$), 3.345–3.392 ($\text{N}^+\text{-CH}_2\text{-CH}_2\text{-OH}$), 3.895–3.926 ($\text{N}^+\text{-CH}_2\text{-CH}_2\text{-OH}$).

^{13}C NMR spectrum of $\text{C}_{12}\text{DEEAB}$ (Fig. 6), δ , ppm: 7.22–8.32 ($\text{CH}_3\text{-CH}_2\text{-N}^+$), 13.89 ($\text{CH}_3\text{-CH}_2$), 21.65–31.96 (CH_2 in alkyl group), 54.15 ($\text{CH}_3\text{-CH}_2\text{-N}^+$), 54.97 ($\text{CH}_2\text{-CH}_2\text{-CH}_2\text{-N}^+$), 58.26 ($\text{N}^+\text{-CH}_2\text{-CH}_2\text{-OH}$), 58.57 ($\text{N}^+\text{-CH}_2\text{-CH}_2\text{-OH}$).

The diameters of aggregates formed by the surfactants in aqueous solutions were evaluated by dynamic light scattering. Figure 7 shows the size distribution

Fig. 5. ^1H NMR spectrum of $\text{C}_{12}\text{DEEAB}$.Fig. 6. ^{13}C NMR spectrum of $\text{C}_{12}\text{DEEAB}$.Fig. 7. Size distribution of aggregates formed by $\text{C}_{12}\text{DMEAB}$ and $\text{C}_{12}\text{DEEAB}$ in an aqueous medium at CMC (25°C).

of aggregates formed by the IL surfactants in an aqueous solution. As can be seen, the aggregate size varies with a change in the surfactant structure. The mean hydrodynamic diameter of the aggregates formed by $\text{C}_{12}\text{DMEAB}$ at CMC is 5 nm, and that of the aggregates formed by $\text{C}_{12}\text{DEEAB}$ at CMC is 40 nm, i.e., 8 times larger.

The hydrophilic-lipophilic balance (HLB) of the synthesized surfactants was calculated by the Davies method [28]; the results are given in Table 1. These products are very readily soluble in water at 20°C even at concentrations exceeding the critical micelle concentration. Therefore, there was no need to determine the Krafft temperature for the IL surfactants prepared.

The surface tension isotherms for $\text{C}_{12}\text{DMEAB}$ and $\text{C}_{12}\text{DEEAB}$ (Fig. 8) have the shape typical of micelle-forming surfactants. The CMC values determined for these surfactants by the tensiometric and conductometric methods are given in Table 1.

The maximal adsorption of aqueous surfactant solutions at the interface with air Γ (mol cm^{-2}) and the minimal cross section area of the polar group $A_{\min}$ (nm^2) were calculated by the following formulas [29, 30]:

$$\Gamma_{\max} = -\frac{1}{nRT} \frac{d\gamma}{d\ln C},$$

$$A_{\min} = 10^{16}/N_A \Gamma_{\max},$$

where T is the absolute temperature; R , universal gas constant; N_A , Avogadro constant; and n , number of particles adsorbed at the interface (Table 1).

The quantity pC_{20} determining the economic feasibility of using surfactants and characterizing the concentration at which the surface tension of surfactant solutions decreases by 20 mN m^{-1} was calculated by the formula [29, 30]

$$pC_{20} \equiv -\log C_{(-\Delta\gamma = 20)}.$$

The higher is pC_{20} , the more efficient is the surfactant adsorption at the interface and the more pronounced is the decrease in the surface tension. Thus, pC_{20} increases on replacing CH_3 by C_2H_5 .

The surface pressure (π_{CMC}) of aqueous solutions of the synthesized surfactants at the water–air interface was calculated by the formula [29, 30]

$$\pi_{\text{CMC}} = \gamma_0 - \gamma_{\text{CMC}},$$

where γ_0 is the surface tension at the water–air interface

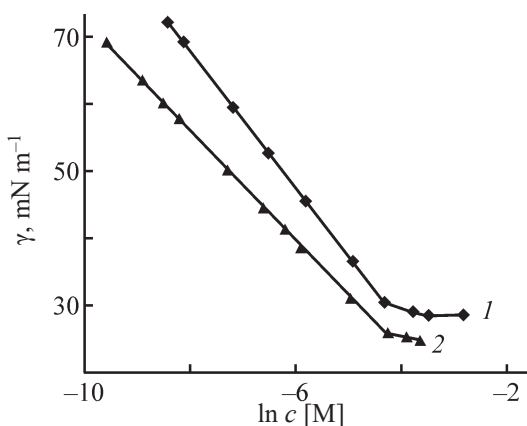


Fig. 8. Semilog plot of the surface tension vs. concentration of aqueous solution of (1) $C_{12}DMEAB$ and (2) $C_{12}DEEAB$.

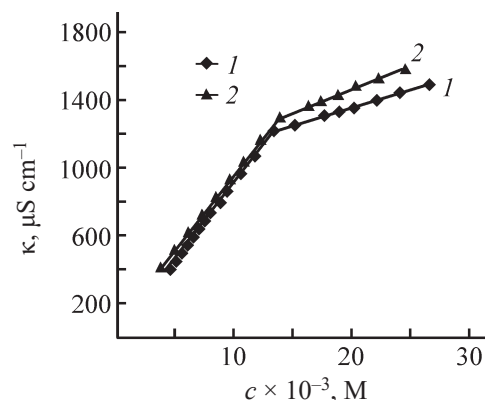


Fig. 9. Specific electrical conductivity of aqueous solutions of (1) $C_{12}DMEAB$ and (2) $C_{12}DEEAB$ as a function of their concentration.

and γ_{CMC} is the surface tension at the interface of the surfactant solution with air at CMC.

The main condition of using surfactants as oil collectors is that the surfactant spill pressure should exceed the oil spill pressure (20 mN m^{-1}). The surfactants should have the spill pressure in the range $30\text{--}40 \text{ mN m}^{-1}$ [31]. As seen from the surface pressures of the surfactants synthesized (Table 1), the π_{CMC} value calculated for $C_{12}DEEAB$ is higher than that for $C_{12}DMEAB$. To compare the surface properties of the surfactants with those of the known analogs, we present in Table 1 the published data for dodecyltrimethylammonium bromide ($C_{12}TAB$) [32]. Comparison of the colloid-chemical parameters shows that, for the surfactants synthesized, π_{CMC} is higher and CMC and γ_{CMC} are lower than those of the classical surfactant. It should also be noted

that the IL surfactants prepared are more favorable environmentally.

Figure 9 shows the dependences of the specific electrical conductivity on the concentration of aqueous solutions of the surfactants synthesized. As can be seen, the concentration dependence of the specific electrical conductivity consists of straight lines with two different slopes. The surfactant concentration corresponding to the change in the slope is equal to CMC. The degree of binding of counterions with micelles was calculated by the formula [29, 30]

$$\beta = 1 - \alpha,$$

where α is the degree of dissociation of the micelle, $\alpha = S_2/S_1$; S_1 and S_2 are the straight line slopes before and after CMC, respectively.

Table 1. Surface properties of aqueous solutions of ionic liquid surfactants at 298 K

β	$\text{CMC} \times 10^3, \text{mol dm}^{-3}$		$\Gamma_{\text{max}} \times 10^{10}, \text{mol cm}^{-2}$	$A_{\text{min}} \times 10^2, \text{nm}^2$	$\pi_{\text{CMC}}, \text{mN m}^{-1}$	$\gamma_{\text{CMC}}, \text{mN m}^{-1}$	pC ₂₀	$\Delta G_{\text{min}}, \text{kJ mol}^{-1}$	$\Delta G_{\text{ad}}, \text{kJ mol}^{-1}$	HLB
C ₁₂ DMEAB										
0.77	13.3 ^a	13.3 ^b	2.04	81.3	41.6	30.4	2.95	−36.56	−38.59	10.7
C ₁₂ DEEAB										
0.70	13.9 ^a	14.0 ^b	1.65	100.6	46.3	25.7	3.27	−34.90	−37.70	9.8
C ₁₂ TAB ^c										
0.79	14.6 ^a	14.5 ^b	2.85	58.15	34.43	37.57	—	−36.58	−48.63	9.3

β , degree of counterion binding; CMC, critical micelle concentration; γ_{CMC} , surface tension of the solution at CMC; $\Gamma_{\max}$, maximal adsorption; $A_{\min}$, minimal cross section area of the polar group; π_{CMC} , surface pressure; pC $_{20}$, economic parameter; ΔG_{mic} , Gibbs free energy of micellization; ΔG_{ad} , Gibbs free energy of adsorption; ^a CMC determined by the tensiometric method; ^b CMC determined by the conductometric method; ^c taken from [32].

Table 2. Oil-collecting and oil-dispersing power of C₁₂DMEAB and C₁₂DEEAB in the form of 5% aqueous solutions

Seawater		Freshwater		Distilled water	
τ , h	K (K_D , %)	τ , h	K (K_D , %)	τ , h	K (K_D , %)
C ₁₂ DMEAB					
0–29	36	0–31	30	0–61	21
45–61	42	49–62	41	63–135	25
79–109	35	73–113	36		
114–135	24	117–135	25		
C ₁₂ DEEAB					
0–113	96%	0–1	31	0	41
		1.17–114	97%	1	36
				1.17–114	96%

The calculated degrees of binding of the surfactant counterion are also given in Table 1. As can be seen, the degree of binding with the counterion β decreases in going from the methyl to ethyl fragment; hence, the degree of the ionic dissociation of C₁₂DEEAB is higher.

It is known from the literature [29, 30] that, for ionic surfactants, the Gibbs free energy of micellization (ΔG_{mic}) is calculated by the equation

$$\Delta G_{mic} = (2 - \alpha)RT \ln X_{CMC},$$

where X_{CMC} is the surfactant mole fraction.

The Gibbs free energy of the adsorption (ΔG_{ad}) at the water–air interface is calculated as follows:

$$\Delta G_{ad} = (2 - \alpha)RT \ln X_{CMC} - 0.6023\pi_{CMC}A_{CMC},$$

where $A_{CMC} \cong A_{min}$.

As can be seen, both ΔG_{mic} and ΔG_{ad} (Table 1) are negative. This means that the adsorption and micellization of the IL surfactants synthesized occur spontaneously, with ΔG_{ad} being more negative. That is, the adsorption occurs more spontaneously than the micellization. On replacing the methyl groups in the surfactant head by the ethyl groups, ΔG_{mic} and ΔG_{ad} increase.

Thus, elongation of the alkyl radical negatively affects the adsorption and micellization. In the case of the IL surfactants synthesized, the replacement of the methyl groups by the ethyl groups makes these processes less spontaneous. This may be due to the fact that this replacement makes the surfactant more hydrophobic.

The results of studying the oil-collecting and oil-dispersing ability of the IL surfactants synthesized are given in Table 2.

The experiments have shown that the reagents exhibit oil-collecting and oil-dispersing power. C₁₂DMEAB exhibits the oil-collecting power in all the three kinds of water. The maximal oil collection factor is 25 in distilled water, 41 in freshwater, and 42 in seawater. The collected oil spot is retained for more than 5 days. As seen from Table 2, the oil collection factor in freshwater and seawater is higher than in distilled water. That is, the water mineralization positively influences the oil collection. The replacement of methyl groups in the reagent by ethyl groups considerably alters the oil-collecting properties. C₁₂DEEAB in distilled water and freshwater exhibits a mixed effect; i.e., first the oil collection occurs for a short time, and then it is followed by the oil dispersion. The oil collection is observed for 1 h ($K_{max} = 41$ and 31, respectively) and is followed by prolonged (>4 days) oil dispersion ($K_D = 96$ and 97%, respectively). In seawater, C₁₂DEEAB exhibits exclusively the oil-dispersing effect ($K_D = 96\%$). Thus, the replacement of the methyl groups in the surfactant molecule by the ethyl groups favors the transition from the oil-collecting performance to the oil-dispersing performance. The reagents act for ~4–6 days.

CONCLUSIONS

IL surfactants containing ethyl and methyl fragments were prepared by the reaction of 1-bromododecane with 2-dimethylaminoethanol and 2-diethylaminoethanol. The colloid-chemical parameters of the surfactants prepared were determined. The influence of the kind of

substituent in the head group in these parameters was analyzed. On replacing methyl groups by ethyl groups, CMC, $A_{\min}$, π_{CMC} , pC_{20} , ΔG_{mic} , and ΔG_{ad} increase, whereas β , Γ_{max} , and γ_{CMC} decrease. With an increase in the alkyl chain length, the size of the aggregates formed by the surfactants in an aqueous medium at CMC increases. On replacing the methyl groups in the surfactant molecule by the ethyl groups, the oil-collecting ability gives way to the oil-dispersing ability.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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