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Micellization and Antimicrobial Properties of N-alkyl-(2-hydroxypropyl)morpholinium Bromides

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Abstract

New ionic-liquid cationic surfactants have been synthesized on the basis of alkyl (C_9, C_{10}, C_{12}, C_{14}) bromides and 1-(4-morpholinyl)-2-propanol. Surface activity and specific electroconductivity of aqueous solutions of the obtained surfactants have been studied by tensiometric and conductometric methods. Depending on the length of the alkyl chain, the values of critical micellization concentration (CMC), maximum adsorption (Γ_{max}), area of minimal cross-sectional surface of polar group of the surfactant (A_{min}), adsorption efficiency (pC_{20}), surface pressure (π_{CMC}), binding degree of the counter-ion (β), changes of Gibbs free energies of micellization and adsorption processes (ΔG_{mic} and ΔG_{ad}) have been determined. Antimicrobial properties of the synthesized ionic-liquid surfactants have been studied on the example of different bacteria and fungus.

Key words: cationic surfactant, micellization, surface tension, adsorption, antimicrobial

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Introduction

Cationic surfactants have attracted an attention of chemists for many years because of general synthesis methods and wide application possibilities in various spheres [1-3]. These surfactants are used in emulsion polymerization processes [4], minerals flotation [5], textiles processing [6], as corrosion inhibitors. They also have a big importance in medicine being used as antimicrobial substances [7], drugs [8], gene delivery agents [9], and in DNA extraction methods [10]. Under the name of cationic surfactants, such compounds are meant which contain one or several hydrophobic groups linked to positively charged nitrogen atom. These compounds are called ammonium salts. In recent years, of obtainment and study of the surfactants containing imidazolium, pyridinium, pyrrolidinium and morpholinium fragments as cationic groups have induced a large interest [11-14]. One of the main reasons of such interest is an ionic-liquid nature of this type surfactants due to bulkiness of the cationic group [12-15]. Ionic liquids are substances consisting of only ions and melting of temperatures lower than 100 °C, sometimes, even [16] in room conditions. They have unique properties such as low melting temperatures, high thermal stability, low vapor pressure and others. Varying the nature of the cation and anion, it is possible to change the properties of these compounds in the needed direction. Among the above-mentioned classes of ammonium-type ionic liquids, the least poisonous and the cheapest are morpholinium-based salts [17]. They are used in design of ionic-liquid crystals [18, 19], as a solvent of cellulose [20], catalyst in organic syntheses [21], a component of electrochemical processes [22]. Zh. Qian and et al. [15] synthesized N-alkyl-N-methylmorpholinium bromide (C_nMMB, n = 12, 14, 16)-type ammonium salts and studied their surface activity properties. It was established that CMC of C_nMMB cationic surfactants is a little lower than these of N-alkyl-N-methylpiperidinium bromides (C_nPDB) and N-alkyl-N-methylpyrrolidinium bromides (C_nMPB) surfactants. A. B. Mirgorodskaya et al. [23] synthesized C_nMMB (n =10, 12, 14, 16, 18) cationic surfactants and studied their antimicrobial properties against different microorganisms. These surfactants were also investigated as corrosion inhibitors in H_2S and CO_2 media. It was revealed that the surfactants having the alkyl chain length n=14 and n=16 have stronger both corrosion-inhibiting and antimicrobial properties than the others. Varying the alkyl chain length and the anion nature, a number of properties of these compounds may be changed. Alkylation of nitrogen atom in morpholine by hydroxy group-containing agents seriously affects the nature of the hydrophilic part. When methyl group in the cationic surfactant N-cetyl-N-methylmorpholinium bromide is replaced by 2-hydroxyethyl group, CMC of the surfactant decreases [23]. Therefore, addition of hydroxy-group containing fragments to nitrogen atom will bring about a change in surfactivity properties of this class surfactants. Conduction of researches in this direction is of practical importance.

The present work is devoted to synthesis of new cationic surfactants based on 1-(4-morpholinyl)-2-propanol and alkyl bromides, a study of their aqueous solutions by tensiometric and conductometric methods as well as an investigation of antimicrobial properties of the obtained surfactants.

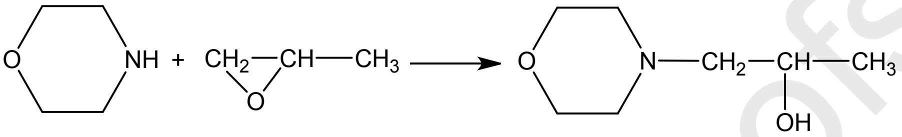
Experimental part

Reagents and instrumentation

Bruker TOP SPIN spectrometer (300.13 MHz and 75.46 MHz) was applied for registering ^1H NMR and ^{13}C NMR spectra. Values of chemical shift (δ) in ppm are registered downfield with regard to TMS. D_2O was taken as a solvent. IR spectra were registered (in KBr disks) on an ALPHA FT-IR (Bruker). The thermogravimetric (TG) analysis was carried out with a NETZSCH STA 449F3 equipment at a scanning rate of 10 °C per minute under N_2 atmosphere. 1-bromononane (Alfa Aesar GmbH & Co KG, Germany), 1-bromodecane (Alfa Aesar, England), 1-bromododecane (Alfa Aesar, England), 1-bromotetradecane (Sigma Aldrich, Japan), morpholine (98%, Alfa Aesar) and propylene oxide (98%, Alfa Aesar) of analytical grade were used.

Method of 1-(4-morpholinyl)-2-propanol synthesis

1-(4-morpholinyl)-2-propanol was synthesized by interaction of morpholine and propylene oxide at 1:1 molar ratio according to the following scheme (Scheme 1):



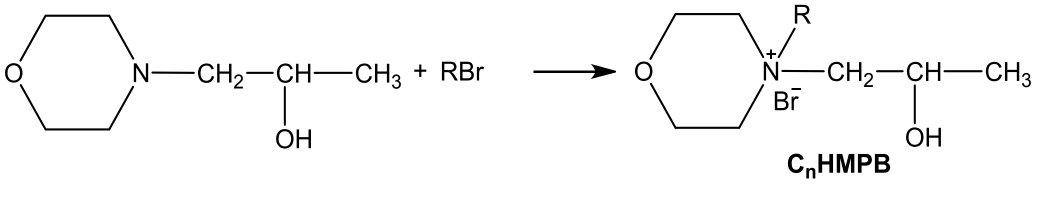
Scheme 1. Reaction scheme of the synthesis of 1-(4-morpholinyl)-2-propanol

To synthesize 1-(4-morpholinyl)-2-propanol, 0.1 mol of morpholine was introduced to a flat-bottom flask and 0.1 mol of propylene oxide was added. The reaction was conducted at room temperature, stirring with a magnetic mixer under the nitrogen atmosphere for 48 hours. As the reaction is exothermal, the flask was placed into a water bath. Thus, appearance of high temperature was prevented. By vacuum distillation of the final mixture, the reaction product was purified from the admixtures. The yield of the reaction product was 98%. It is a yellowish transparent liquid. It dissolves well in water, ethanol, acetone, partially-in hexane. The structure of the synthesized 1-(4-morpholinyl)-2-propanol was confirmed by NMR- and IR-spectroscopy methods. IR ν, cm^{-1}: 3434 ν (OH), 2931, 2854 and 2809 ν (C–H), 1454 and 1373 δ (C–H), 1271 and 1208 ν (C–N), 1139 and 1113 ν (C–O). (Fig. S1). ^1H NMR, (300.13 MHz, D_2O), δ (ppm): 1.03-1.05 (CH–C$\underline{H}_3$), 2.27-2.31 (N-C$\underline{H}_2$-CH), 2.45-2.46 (C$\underline{H}_2$–N), 3.61-3.64 (C$\underline{H}_2$–O–C$\underline{H}_2$), 3.93 (C$\underline{H}$–OH) (Fig. S2). ^{13}C NMR, (75.47 MHz, D_2O), δ (ppm): 20.9, 44.5, 53.1, 64.0, 65.3, 66.0, 66.7. (Fig. S3) (see Figs. S1-S3 in Supporting Information).

Synthesis of cationic ionic-liquid surfactants based on 1-(4-morpholinyl)-2-propanol

0.1 mol of 1-(4-morpholinyl)-2-propanol and 30 mL acetonitrile were introduced into a flat-bottom two-neck flask. After homogenizing the mixture 0.1 mol of 1-bromononane (1-bromodecane, 1-bromododecane or 1-bromotetradecane) was added to it. The reaction was conducted using magnetic mixer, reflux condenser and heater for 18-20 hours at the temperature of boiling of the mixture.

The scheme of synthesis of ionic-liquid surfactants may be illustrated as following (Scheme 2):



$R=C_9H_{19}, C_{10}H_{21}, C_{12}H_{25}, C_{14}H_{29}$.

Scheme 2. Synthesis of cationic ionic-liquid surfactants by interaction of 1-(4-morpholinyl)-2-propanol with 1-bromoalkanes

For isolation of the synthesized products from the reaction mixture, vacuum distillation was used. Herewith, the obtained ionic-liquid surfactant was purified from the solvent and unreacted 1-bromoalkane. To achieve high purity of the surfactants, they were recrystallized in acetone three times. The yield of the obtained ionic-liquid surfactants was 90-94%. They dissolve well in ethanol, acetone, ethyl acetate, partially-in water. Purity of the obtained cationic ionic-liquid surfactants was determined by NMR- and IR-spectroscopy methods. IR ν, cm^{-1}: 3281 ν (OH), 2919 and 2851 ν (C–H), 1466 and 1374 δ (C–H), 1255 ν (C–N), 1118 and 1051 ν (C–O), 720 δ -$(CH_2)_x$- (Fig. 1). ^{1}H NMR, (300.13 MHz, D_2O), δ (ppm): 0.81 ($CH_2{-}C\underline{H}_3$), 1.12 ($CH{-}C\underline{H}_3$), 1.22 (CH_2 chain), 1.75 ($C\underline{H}_2{-}CH_2{-}N^+$), 2.98-3.16 ($CH_2{-}C\underline{H}_2{-}N^+$), 3.54 ($C\underline{H}_2{-}N^+{-}C\underline{H}_2$), 3.86 ($N^+{-}C\underline{H}_2{-}CH(OH){-}CH_3$), 4.01 ($C\underline{H}_2{-}N^+{-}C\underline{H}_2$), 4.32 (CH-O$H$) (Fig. 2). ^{13}C NMR, (75.47 MHz, $CDCl_3$), δ (ppm): 13.8, 14.0, 20.1, 21.6, 21.8, 22.6, 22.7, 26.2, 29.2, 29.4, 29.6, 29.8, 29.9, 30.0, 31.9, 32.1, 32.6, 52.1, 58.8, 59.4, 60.4, 61.5, 61.6, 63.3, 64.4. (Fig. 3). Figs. S4-S5 (see in Supporting Information) shows the results of thermogravimetric analysis of C_{12}HMPB and C_{14}HMPB. As can be seen in the figures, C_{12}HMPB begins to decompose at 212.5 °C and C_{14}HMPB at 227.8 °C. Most part of the surfactants (>50%) are completely decomposed up to 300 °C.

Determination of surface activity of the synthesized ionic-liquid surfactants

Surface tension of the synthesized ionic-liquid surfactants was determined using Du Nouy tensiometer KSV Sigma 702 tensiometer (Attension Biolin Scientific, Finland). 0.00025-0.2% wt. aqueous solutions of the ionic-liquid surfactants were prepared and, after 24 hours, their surface tension were measured at 25 °C [24]. For preparation of the aqueous solutions, distilled water was used. The surface tension of this water at the border with air at 25 °C was equal to 71.9 mN/m. Those results of the measurements were taken into consideration where an error did not exceed ±0.2 mN/m.

Determination of specific electrical conductivity of the synthesized ionic-liquid surfactants

Specific electrical conductivities (κ) of aqueous solution of the obtained ionic-liquid surfactants were determined by means of "ANION-4120" conductometer (Russian Federation). For this purpose, first, 0.0001%-0.4% wt. aqueous solutions of the surfactants were prepared and, after 24 hours, their specific electrical conductivities at 25 °C were determined [25]. Specific electrical conductivity of the distilled water for preparing aqueous solutions of the surfactants was 1.8-2.2 μS/cm at 25 °C. In the studies, the results of the measurements with an error loss than ±0.3 μS/cm were used.

Dynamic Light Scattering (DLS)

The size distribution of the aggregates, which formed by the synthesized cationic surfactants in water was determined at 25 °C by using the Particle Size Analyzer (HORIBA LB-550, Japan). As a light source in the device is used a laser diode with the wave length of 650 nm and the power of 5 mW. The measuring range of the device was in the range from 1 nm to 6 μm. The density of the samples, taken for the study are CMC and 3×CMC. Each sample was measured by repetition at least three times. The distribution of solvent diffusion coefficients (D) was obtained by analyzing the correlation function of data scattering in the CONTIN process. Then was determined hydrodynamic diameter (Dh) of the aggregates by using the Stokes-Einstein equation Dh=kT/3πηD, where k is the Boltzmann constant, T is the absolute temperature, and η is the solvent at that temperature.

Testing of antimicrobial properties of the obtained ionic-liquid surfactants

Two Gram-positive (*Staphylococcus aureus, Bacillus anthracoides*) bacteria, three Gram-negative (*Pseudomonas aeuroginosa, Escherichia coli, Klebsiella pneumoniae*) bacteria and fungi (*Candida albicans*) were included in the test cultures to study the antimicrobial effect of cationic surfactants. Suspensions prepared at 10^6 mic./ml were evenly distributed on the surface of the appropriate nutrient media using a spatula. The studied cationic surfactant and/or its solutions of different concentrations were soaked in sterile discs made of filter paper with a diameter of 6 mm and placed on the surface of the nutrient medium inoculated with microorganisms. The samples were incubated at 37 °C for one day. The effect of cationic surfactants is determined by the diameter of the sterile zone (expressed in mm) which the microorganism does not grow around the disc. The diameter of the sterile zone in which the microorganism does not develop is inversely proportional to the minimum inhibitory concentration of the chemical in the microorganism and indicates the degree of sensitivity.

Results and Discussion

Surface properties of the synthesized ionic-liquid surfactants in aqueous solution

The surface tension at the border of aqueous solutions of the 1-(4-morpholinyl)-2-propanol-based ionic-liquid surfactants with air was determined using tensiometric method. The obtained results are presented in Fig.4 as surface tension isotherms. As is seen from the figure, surface tension decreases as the concentration of all four surfactants increase. After reaching a certain concentration, the surface tension values are stabilized. The concentration corresponding to the beginning of this stabilization is taken as the CMC value.

In table 1, the CMC values of the synthesized surfactants are given. These values for C_9HMPB, C_{10}HMPB, C_{12}HMPB and C_{14}HMPB ionic-liquid surfactants are respectively as following: 3.40, 2.73, 1.27 and 0.355 mM. As is seen from the table, with elongation of the alkyl chain from C_9 to C_{14}, the CMC values decrease. A similar regularity is observed in the case of the cationic surfactants with another type head-group [26,27]. With lengthening of hydrocarbon chain, hydrophobicity of the ionic-liquid surfactants rises. So, their CMC values are lowered. The nature of surfactant head-group is one of the main factors impacting stability of micelles. For a homological series of single alkyl chain-surfactants with such a chain of varying length, CMC obeys Stauff-Klevens rule [28] prognosticating a logarithmic dependence between log CMC and the number of carbon atoms in the alkyl group:

$$\log\mathrm{CMC} = A - Bn \quad (1)$$

where A and B are constants for a certain homological series of surfactants at a given temperature. Constant "A" varies depending on the nature and number of hydrophilic groups. "B" is the constant describing an impact of each methylene group, added to the alkyl chain, on the CMC value. The value of "B" is ~0.5 for nonionic surfactants, 0.28-0.30 for the surfactants containing paraffinic chain and ionic head-group. In Fig. 5, for the synthesized ionic-liquid surfactants, the dependence of CMC (in logarithm from $\mathrm{mol{\cdot}dm^{-3}}$ value) on the number of carbon atoms in the alkyl chain (at

25 °C) is illustrated. As is seen from the graph, the dependence is a straight line with a slope (B) 0.2153, intercept (A) 0.4242 and a correlation coefficient 0.9994. Similar results are observed for n-alkyl-3-methylpyridinium bromides [29] and n-alkyl-N-(2-hydroxypropyl)-N,N-(2-hydroxyethyl) ammonium bromides [30] type cationic surfactants.

In Table 2, surfactivity parameters for the surfactants having N-methylmorpholinium bromide [31] and N-(2-hydroxypropil)dimethylammonium bromide [32] head-groups and C_{12} or C_{14} alkyl chains. From comparison of the CMC values from Tables 1 and 2, the following consequency will be derived: $C_nMMB > C_niPrOH > C_nHMPB$. When methyl group in N-methylmorpholinium fragment is replaced by 2-hydroxypropyl group, CMC decreases, i.e., it is smaller for C_nHMPB-type surfactants.

For the synthesized ionic-liquid surfactants the value of maximum surface excess concentration (Γ_{max}) was determined using Gibbs adsorption isotherm equation (2):

$$\Gamma_{max} = -\frac{1}{nRT}\lim_{C\to C_{CMC}}\frac{d\gamma}{dlnC} \quad (2)$$

Area of minimal surface occupied by the polar group A_{min} was computed by equation (3) [33]:

$$A_{min} = 10^{16}/N_A\Gamma_{max} \quad (3)$$

where R is the universal gas constant (8.314 J/mol K), T is the absolute temperature, and C is the concentration of surfactant, N_A is the Avogadro number. The value of n is accepted as 2 as there is one counter-ion linked with each cationic head group.

The calculated values of Γ_{max} and A_{min} are given in Table 1. As is seen from this table, lengthening of the alkyl chain leads to a rise of Γ_{max} values whereas A_{min} diminishes. The same regularity is observed for cationic surfactants with other head-groups [26, 30]. From comparison of Γ_{max} and A_{min} values of the same alkyl chain-surfactants included into Tables 1 and 2, it is noticeable that Γ_{max} and A_{min} values for C_nHMPB series are between those of C_niPrOH and C_nMMB-type surfactants.

For characterization of surfactivity, important parameters are surface pressure (π_{CMC}) and adsorption efficiency (pC_{20}). These parameters are calculated using the following formulas:

$$\pi_{CMC} = \gamma_0 - \gamma_{CMC} \quad (4)$$

$$pC_{20} = -\log C_{20} \quad (5)$$

where γ_0 is the surface tension of solvent without a surfactant and γ_{CMC} is the surface tension in the presence of the surfactant at CMC [33].

C_{20} is the concentration of a surfactant consumed for lowering surface tension by 20 mN m^{-1}. In Table 1, the values of π_{CMC} and pC_{20} for the synthesized ionic-liquid surfactants are presented. The surface pressure values diminish in the following series: $C_{12}HMPB > C_{10}HMPB > C_9HMPB > C_{14}HMPB$. The values of pC_{20} rise with elongation of the alkyl chain. Similar correlation is noticed in the surfactants having trimethylammonium head-group [33, 34].

According to Table 2, π_{CMC} and pC_{20} values of the surfactants containing N-methylmorpholinium and N-(2-hydroxypropyl)dimethylammonium head-groups are smaller than those of the surfactant with N-(2-hydroxypropyl)morpholinium head-group.

Critical packing parameter-CPP values of these surfactants are based on morphology of the formed aggregates in water and calculated by the following formula:

$$CPP = \frac{V_H}{a_0 \times l_C} \quad (6)$$

where V_H is the effective volume of hydrophobic chain, a_0 is the area of the polar head-group surface, l_C is the length of the alkyl chain. At the maximum level of concentration, a_0 may be replaced by A_{min}. The value of V_H changes depending on the number of carbon atoms in the hydrophobic chain. It is calculated by the following formula:

$$V_H = 27.4 + 26.9 \times (n - 1)\ \text{Å}^3 \quad (7)$$

The value of l_C may be computed by the following formula depending on the alkyl chain length:

$$l_C \leq 1.5 + 1.265 \times (n - 1)\ \text{Å} \quad (8)$$

In Table 3, the values of CPP are shown. As is noticed from the table, the CPP values are in the interval 0 - 0.33. Therefore, in aqueous solution, the micelles of these ionic-liquid surfactants are in spheroidal form.

Size of the aggregates

The dimensions of the aggregates, which formed by the synthesized cationic surfactants in the aqueous solution were determined by the DLS method (Fig. 6). As can be seen from the images, a peak is observed in the C_9HMPB surfactant at both of CMC and in the concentration, which 3 times higher than CMC. The size of the aggregates of this surfactant at CMC is 260 nm and at the density with 3 times higher than CMC is 1000 nm. Two peaks were observed in surfactants with an alkyl chain length greater than C_{10}. The first peak in CMC is 75 nm, 150 nm and 225 nm, respectively for C_{10}HMPB, C_{12}HMPB and C_{14}HMPB surfactants. The second peak is 1150 nm for the C_{10}HMPB, 1500 nm for the C_{12}HMPB and 1730 nm for the C_{14}HMPB. The first peak is the presence of small vesicles, and the second peak is the presence of large vesicles or bilayer systems. Aggregates diameter of surfactants in aqueous solution increase about two times with increasing their alkyl length chains. This is because β increases with increasing alkyl chain length. As the β increases, the micelles neutralize with the opposite ions, in the meantime they aggregate and form large vesicles [35, 36]. In surfactants C_{10}HMPB, C_{12}HMPB and C_{14}HMPB is observed the 3 times higher concentration decreasing of the aggregates diameters than the CMC.

Specific electrical conductivity of the synthesized ionic-liquid surfactants

Specific electrical conductivity of the synthesized ionic-liquid surfactants was determined by conductometric method at 25 °C. Based on the obtained values κ vs concentration (C) plots were built (Figs. 7 and 8). As is seen from the figures, with an increase of concentration of the ionic-liquid surfactants in water, the value of κ rises. κ vs C plots are characterized by two different linear dependences at low and high concentrations. The point of intersection of these straight lines is CMC. The ration of the slope after CMC (S_1) and before CMC (S_2) is equal to a dissociation degree-α of the surfactant counter-ion:

$$\alpha = S_1/S_2 = 1 - \beta \quad (9)$$

where β is the binding degree of the counter-ion. In Table 1, the values of β of the synthesized ionic-liquid surfactants are given. As is clear from the Table 1, the value of β rises as the alkyl chain in the surfactant lengthens. Its reason is lowering of charge density on the surface of the micelles [37]. Longer alkyl chain-surfactants form larger aggregates. In very large structures, the ratio of surface and volume is small. Therefore, polar groups become packed more tightly and are surrounded by more counter-ions.

Antimicrobial properties of the synthesized ionic-liquid surfactants

The antimicrobial ability of the synthesized cationic surfactants was studied on Gram-positive (*Staphylococcus aureus, Bacillus anthracoides*) bacteria, Gram-negative (*Pseudomonas* *aeuroginosa, Escherichia coli, Klebsiella pneumoniae)* bacteria and fungi (*Candida albicans*). The obtained results are given in Table 4. As can be seen from the table, the antimicrobial capacity of the synthesized cationic surfactants is high and varies depending on the length of the alkyl chain. As can be seen from the table, the synthesized cationic surfactants have a more effective action against gram-positive bacteria. C_{14}HMPB has a higher effect than other surfactants. Therefore, cationic surfactants with a higher alkyl chain length have a higher impact. The antimicrobial effect depends on the following surface parameters of surfactants: CMC, ΔG_{ad}, accumulation of surfactants on the cell wall and diffusion size of surface active molecule. As can be seen from Table 1, with the increasing length of the alkyl chain, the CMC and ΔG_{ad} decrease. Therefore, there will be higher antibacterial capacity in the C_nHMPB class surfactants with the lower CMC and ΔG_{ad} values. The antibacterial ability of cationic surfactants is approximately close against the Gram-negative bacteria. The surfactant C_{14}HMPB showed the highest antibacterial properties against the *Escherichia coli* bacteria. The surfactant C_9HMPB, which has a small alkyl chain length, is more effective against the development of *Candida albicans*. As the length of the chain increases, this effect decreases slightly.

Thermodynamic parameters of the synthesized ionic-liquid surfactants

Using CMC and β values for the synthesized ionic-liquid surfactants, the standard Gibbs free energies for micellization process were calculated by the following formula:

$$\Delta G^{\circ}_{mic} = (1+\beta)RT\ln X_{CMC} \qquad (10)$$

where X_{CMC} is the CMC in molar fraction, X_{CMC} = CMC/55.4, where CMC is in mol/L, 55.4 originates from 1 L of water corresponding to 55.4 mol of water at 25 °C, β- is the binding degree of counter-ion, R is universal gas constant, and T is standard absolute temperature (298 K). The values of standard Gibbs free energy for adsorption process at water-air border were computed according to the following formula [33]:

$$\Delta G^{\circ}_{ad} = (1+\beta)RT\ln X_{CMC} - 0.6023\pi_{CMC}A_{CMC} \qquad (11)$$

where A_{CMC} has the unit Å^2 per molecule, and π_{CMC} denotes the surface pressure (in mN/m) at CMC at the border of surfactant in aqueous solution with air.

In Table 1, Gibbs free energy values for micellization process of the synthesized ionic-liquid surfactants in aqueous solution and adsorption process at water-air border are presented. It is seen that, for both micelle formation and adsorption processes, the values of standard Gibbs free energies are negative. It means that, with these surfactants, micellization and adsorption processes occur spontaneously. Mean while, more negative values of ΔG_{ad} show that the adsorption process precedes the micelle formation. In Fig. 9, graphs of dependence of ΔG°_{ad} and ΔG°_{ad} on the length of the alkyl chain of the ionic-liquid surfactants are illustrated. As is evident from the figure ΔG°_{mic} and ΔG°_{ad} values decrease with elongation of the alkyl chain. Lengthening of this chain enhance hydrophobic interaction between the alkyl chains and favour micelle formation.

Gibbs free energy of aggregation is contributed by transfer of the ionic liquid fragments from solution to the aggregates, and therefore, displays the relative input of ionic-liquid hydrophilic and hydrophobic parts to aggregation process. As was mentioned before [38], the value of ΔG°_{mic}consists of contributions from the surfactant head-group (ΔG°_{mic} head-group), methylene group of the hydrophobic chain ($\Delta G^{\circ}_{mic,\ CH_2}$) and CH_3- group from the alkyl chain top ($\Delta G^{\circ}_{mic,\ CH_3}$):

$$\Delta G^{\circ}_{\mathrm{mic}} = \Delta G^{\circ}_{\mathrm{mic,\ head\ group}} + \Delta G^{\circ}_{\mathrm{mic,\ CH_3}} + n_{CH_2}\Delta G^{\circ}_{\mathrm{mic,\ CH_2}} \quad (12)$$

Then, the following may be written for ΔG°_{ad}:

$$\Delta G^{\circ}_{\mathrm{ad}} = \Delta G^{\circ}_{\mathrm{ad,\ head\ group}} + \Delta G^{\circ}_{\mathrm{ad,\ CH_3}} + n_{CH_2}\Delta G^{\circ}_{\mathrm{ad,\ CH_2}} \quad (13)$$

where n_{CH_2} is the number of CH_2 groups in the hydrophobic chain of C_nHMPB.

From Fig. 9, it is noticed that ($\Delta G^{\circ}_{\mathrm{mic,\ head\ group}} + \Delta G^{\circ}_{\mathrm{mic,\ CH_3}}$) equals −4.17 kJ/mol and, for each methylene group, $\Delta G^{\circ}_{\mathrm{mic,\ CH_2}}$ is equal to −3.09 kJ/mol. For N-alkyl-N-methylpyrrolidinium bromide [26] and N-alkyl-N-methylimidazolium bromide [38] class ionic liquid surfactants, $\Delta G^{\circ}_{\mathrm{mic,\ CH_2}}$ is −3.01 kJ/mol. Therefore, thermodynamics of methylene groups transfer from aqueous solution to micelle does not depend on cationic head-group. In the case of N-alkyl-N-methylmorpholinium bromide, N-alkyl-N-methylpyrrolidinium bromide and N-alkyl-N-methylimidazolium bromide class ionic-liquid surfactants, ($\Delta G^{\circ}_{\mathrm{mic,\ head\ group}} + \Delta G^{\circ}_{\mathrm{mic,\ CH_3}}$) equals respectively −3.50, −3.61 and −3.7 kJ/mol. For the synthesized N-alkyl-N-(2-hydroxypropyl)morpholinium bromide class ionic-liquid surfactants, ($\Delta G^{\circ}_{\mathrm{mic,\ head\ group}} + \Delta G^{\circ}_{\mathrm{mic,\ CH_3}}$) value is smaller (-4.17 kJ/mol). This fact shows that, with this head-group ionic-liquid surfactants, micellization occurs more readily. Therefore, in N-alkyl-N-methylmorpholinium bromide class surfactants, replacement of methyl group connected to nitrogen atom by 2-hydroxypropyl group enhances micellization capacity of the surfactant head-group. ($\Delta G^{\circ}_{\mathrm{ad,\ head\ group}} + \Delta G^{\circ}_{\mathrm{ad,\ CH_3}}$) equals −8.91 kJ/mol, for each methylene group $\Delta G^{\circ}_{\mathrm{ad,\ CH_2}}$ is −2.95 kJ/mol. The value of $\Delta G^{\circ}_{\mathrm{ad,\ CH_2}}$ is close to those of other classes of such surfactants [33].

Conclusions

On the basis of 1-(4-morpholinyl)-2-propanol and alkyl bromides (nonyl-, decyl-, dodecyl- and tetradecyl bromde), new ionic-liquid cationic surfactants were synthesized and characterized by spectroscopic (^{1}H, ^{13}C NMR and IR) and thermogravimetric analysis methods. Micellization and adsorption properties of these surfactants were studied by measurements of surface tension and specific electrical conductivity. It was established that, with an elongation of the alkyl chain of the ionic – liquid surfactants, the values of CMC, $A_{\min}$, ΔG°_{mic} and ΔG°_{ad} decrease whereas the values of β, $\Gamma_{\max}$ and pC_{20} rise. It was revealed that the CMC values are much less compared to surfactants with the N-methylmorpholinium and N-(2-hydroxypropyl)-N,N-dimethyl ammonium similar head-groups ionic-liquid surfactants. Antimicrobial properties of the obtained novel ionic-liquid surfactants were studied. It was determined that the surfactant with C_{14}-alkyl chain manifests the strongest antimicrobial properties.

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Compliance with ethical standards

Conflict of interest. The authors declare that they have no conflict of interest.

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Table 1. Surfactivity parameters of 1-(4-morpholinyl)-2-propanol-based ionic-liquid surfactants (25 °C)

Surfactants	CMC×10^3, mol·dm^{-3}	Γ_{max}×10^{10}, mol·cm^{-2}	A_{min}×10^2, nm^2	β	γ_{CMC}, mN·m^{-1}	pC_{20}	π_{CMC}, mN·m^{-1}	ΔG_{mic}, kJ·mol^{-1}	ΔG_{ad}, kJ·mol^{-1}
C_9HMPB	3.40	1.40	118.5	0.335	26.6	4.03	45.4	-32.08	-35.33
C_{10}HMPB	2.73	1.45	114.3	0.43	24.5	4.25	47.5	-35.14	-38.41
C_{12}HMPB	1.27	1.52	109.1	0.56	24.0	4.47	48.0	-41.30	-44.45
C_{14}HMPB	0.355	1.55	107.0	0.60	31.0	4.60	41.0	-47.41	-50.05

Table 2. Surfactivity parameters of some cationic surfactants with C_{12} and C_{14} alkyl chain (25°C)

n	CMC×10^3, mol·dm^{-3}		Γ_{max}×10^{10}, mol·cm^{-2}		A_{min}×10^2, nm^2		π_{CMC}, mN·m^{-1}		pC_{20}	
	C_niPrOH	C_nMMB	C_niPrOH	C_nMMB	C_niPrOH	C_nMMB	C_niPrOH	C_nMMB	C_niPrOH	C_nMMB
12	11.70	16.11	0.987	2.51	168.19	66.20	37.8	29.02	3.5	2.11
14	1.47	3.96	0.883	2.78	188.05	59.73	37.9	29.14	2.7	2.69

Note.C_nMMB–N-alkyl-N-methylmorpholinium bromides [31]
C_niPrOH–N-alkyl-N-(2-hydroxypropyl)-N,N-dimethyl ammonium bromides [32]

Table 3. CPP of 1-(4-morpholinyl)-2-propanol-based ionic-liquid surfactants

Surfactants	CPP
C_9HMPB	0.18
C_{10}HMPB	0.18
C_{12}HMPB	0.19
C_{14}HMPB	0.20

Table 4. Antimicrobial properties of the synthesized surfactants by disk-diffusion method

Bacteria and fungi	C_9HMPB	C_{10}HMPB	C_{12}HMPB	C_{14}HMPB
	Diameter of inhibition zone (mm)			
Escherichia coli	12	13	11	14
Pseudomonas aeruginosa	-	13	12	10
Klebsiella pneumoniae	7	14	15	9
Staphylococcus aureus	17	19	16	23
Bacillus anthracoides	11	18	16	22
Candida albicans	20	17	17	18

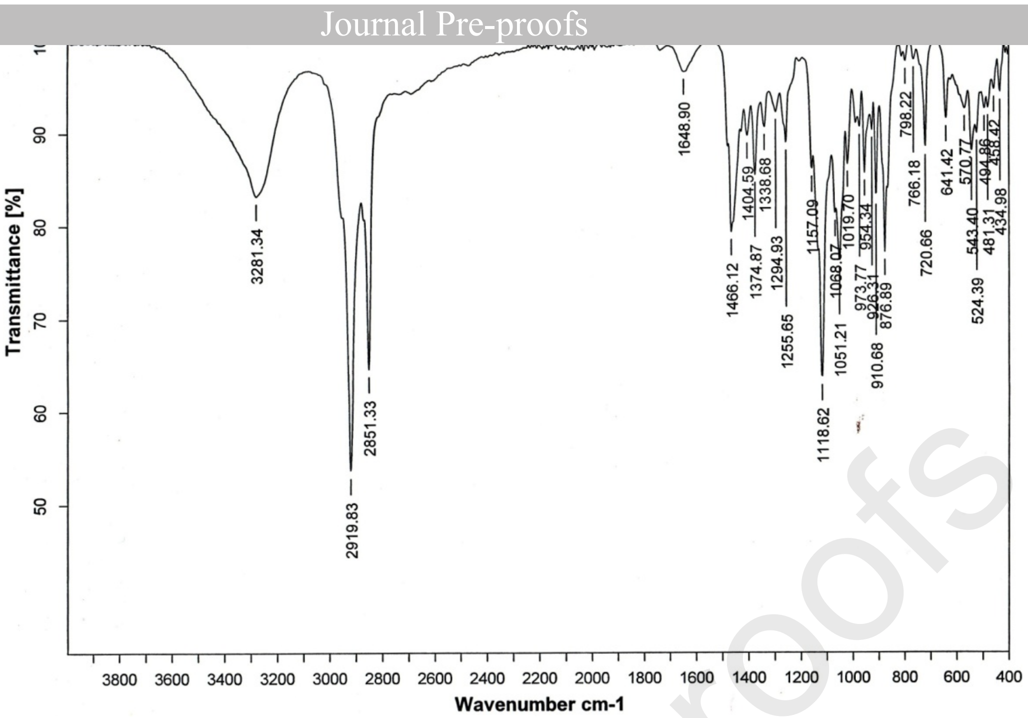

Fig. 1. IR spectrum of C_{12}HMPB

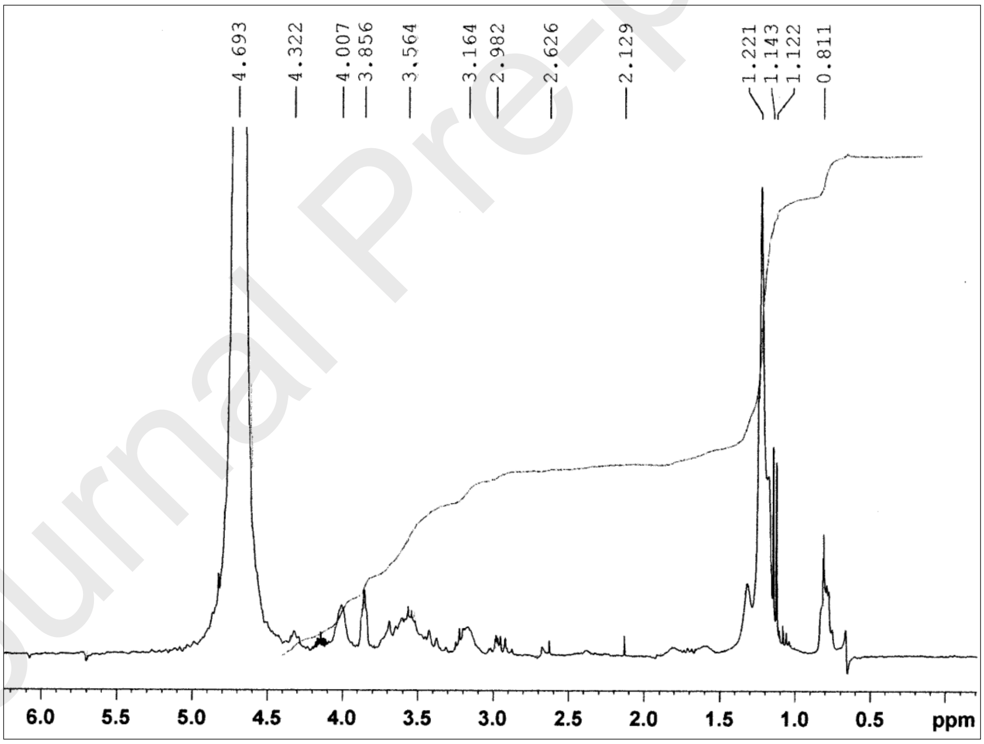

Fig. 2. ^{1}H NMR spectrum of C_{12}HMPB

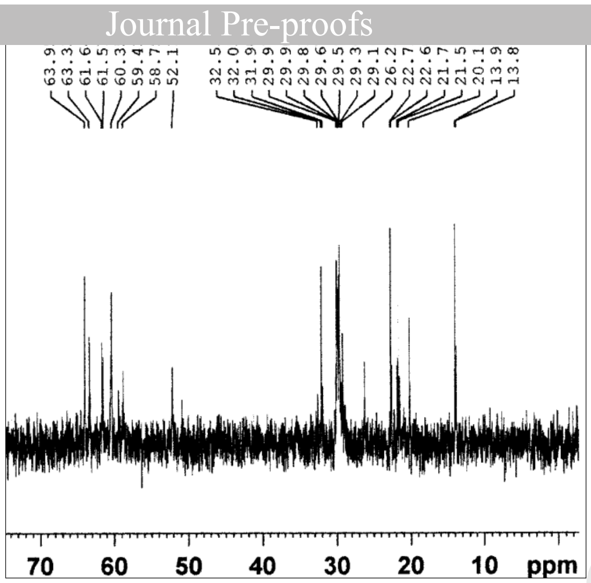

Fig. 3. ^{13}C NMR spectrum of C_{12}HMPB

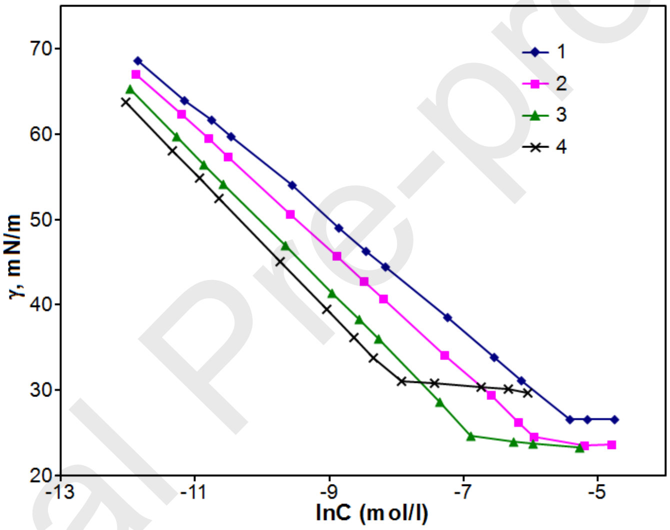

Fig. 4. Surface tension plots of C_9HMPB (1), C_{10}HMPB (2), C_{12}HMPB(3) and C_{14}HMPB(4) in aqueous solution at 25°C versus natural logarithmic concentration of surfactants

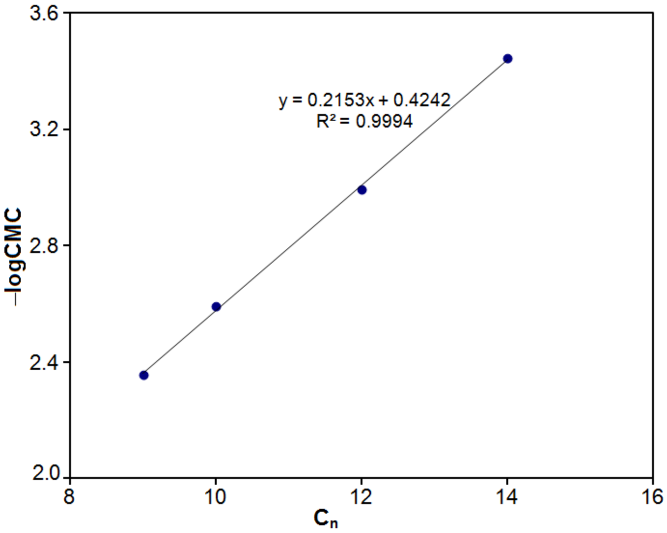

Fig. 5. Dependence of -log(CMC) for C_nHMPB on the number of carbon atoms in the alkyl chain

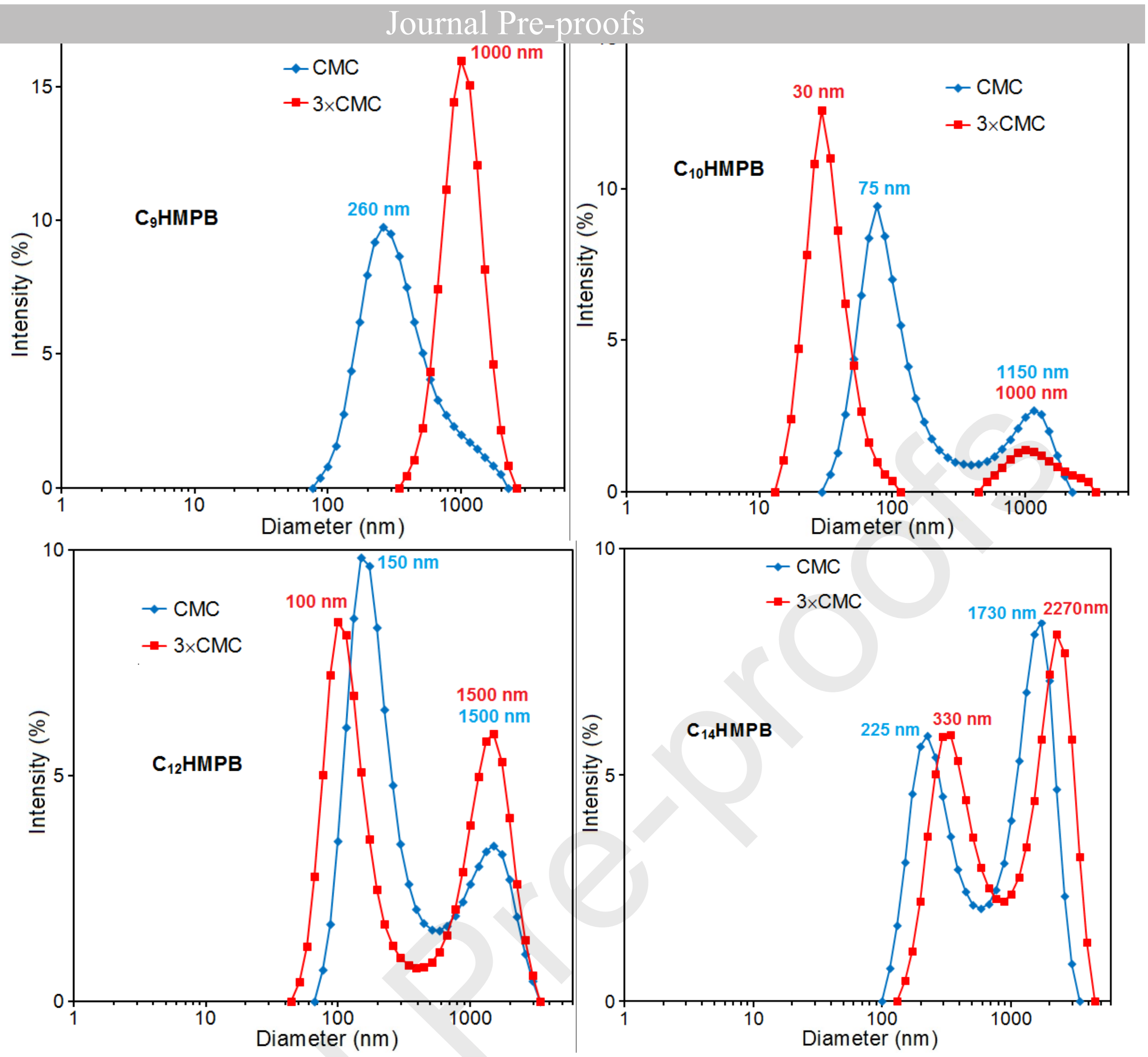

Fig. 6. The results of DLS measurements (25 ºC) for synthesized ionic liquid surfactants at various concentrations

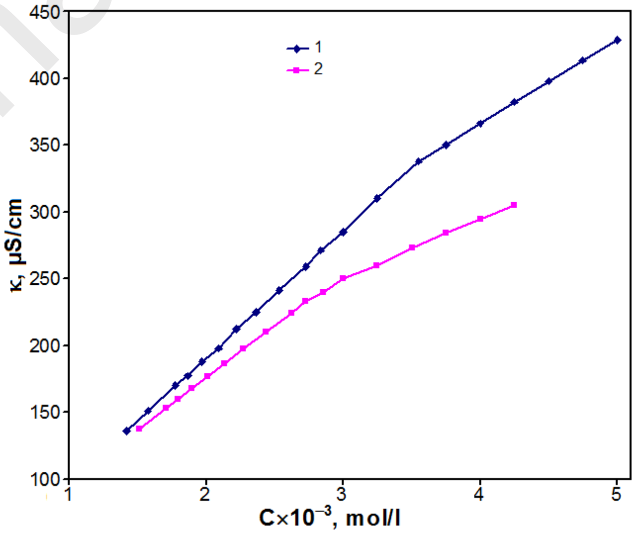

Fig. 7. Plots of specific electrical conductivity versus surfactant concentration for aqueous solutions of C$_9$HMPB (1) and C$_{10}$HMPB (2) at 25°C

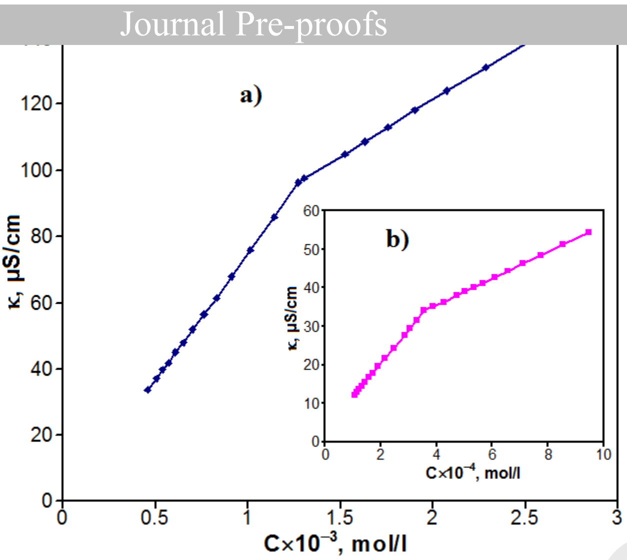

Fig. 8. Plots of specific electrical conductivity versus surfactant concentration for aqueous solutions of C_{12}HMPB(a) and C_{14}HMPB(b) at 25°C

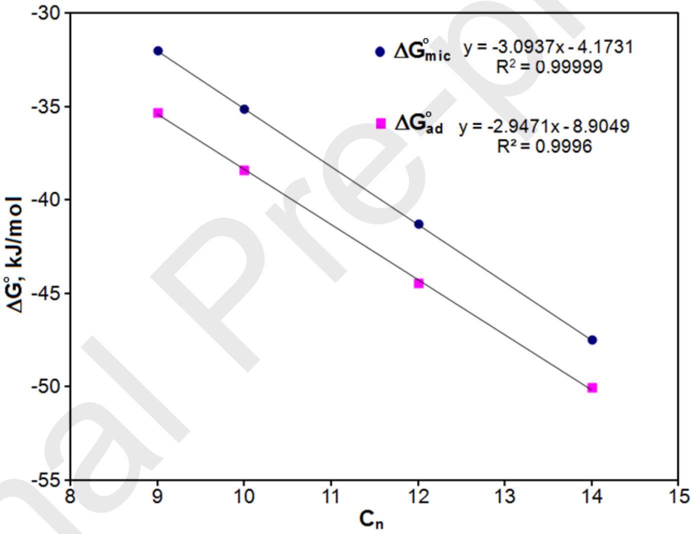

Fig. 6. Dependence of Gibbs free energy change for C_nHMPB on the number of carbon atoms in the alkyl chain

Credit Author Statement.

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Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

The authors declare with certain confidence that no financial interests, personal relationships or anything of similar nature that can have any kind of influence on the work presented in the current paper are known to them.

Highlights

- New cationic surfactants were obtained throughout quaternization of 1-(4-morpholinyl)-2-propanol.
- Physico-chemical parameters of the synthesized cationic surfactants have been determined.
- The synthesized cationic surfactants determined CMC, which decreased by increasing alkyl chain length.
- The prepared cationic surfactants have good antibiotic effect against microorganism.