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ORIGINAL ARTICLE

Ferrocene-Based Cationic Surfactants: Surface and Antimicrobial Properties

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Abstract A novel series of ferrocenyl surfactants was synthesized by the reaction of ferrocene disulfonic acid with different primary and tertiary fatty amines to produce the corresponding ammonium salts $Fc[SO_3^-\ {}^+NH_3(CH_2)_n CH_3]_2$, where $n = 9$, 11, or 15 and $Fc[SO_3^-\ {}^+NH(CH_3)_2 (CH_2)_nCH_3]_2$, where $n = 7$ or 11, respectively, and where Fc = ferrocene. Chemical structures were confirmed by microelemental analysis, FTIR, and ^{1}H NMR spectroscopy. The critical micelle concentration of each prepared surfactant was determined using equilibrium surface tension. Furthermore, air/water interface parameters including effectiveness (π_{CMC}), efficiency (Pc_{20}), maximum surface excess (Γ_{max}), and minimum surface area (A_{min}) were determined at 30, 40, and 50 °C. Thermodynamic parameters ($\Delta G°$, $\Delta S°$, and $\Delta H°$) for both micellization and adsorption processes were recorded. The new synthesized surfactants were screened as antimicrobial agents against different bacterial and fungal organisms.

Keywords Ferrocenyl surfactants · Surface activity · Ferrocene amine salts · Antimicrobial activity

Introduction

In designing a metal-containing surfactant system, the metal may be directly bound to the head group [1–3] or to the counterion of the cationic surfactant [4–6].

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In addition, surfactants containing ferrocene (Fc) moiety have been of considerable interest for several years [7]. $Fc(CH_2)_{11}SO_3^-$ can be reversibly oxidized to the zwitterionic state $Fc^+(CH_2)_{11}SO_3^-$. The surface and bulk properties of $Fc(CH_2)_{11}SO_3^-$ and $Fc^+(CH_2)_{11}SO_3^-$ in aqueous solutions have been reported [8].

Recent studies have demonstrated that $Fc(CH_2)_{11} N^+(CH_3)_3Br^-$ can be studied with electrochemical methods to form the basis of an experimental system that permits spatial and temporal control over the surface tensions of aqueous solutions [9].

This paper reports the synthesis, surface, and antimicrobial properties of novel compounds of ferrocene-based cationic surfactants.

Experimental

Materials

1. Hexadecyl amine and dodecyl amine were obtained from Acros Organics and decyl amine from MP Biomedicals.
2. Ferrocene, *N*,*N*-dimethyloctyl amine, and *N*,*N*-dimethyldodecyl amine were purchased from Aldrich.
3. Chlorosulfonic acid was purchased from Fluka.
4. Chloroform, ethanol, and 1,4-dioxane were purchased from ADWIC.

Analysis

1. Elemental analyses were carried out at the Micro Analytical Center (Cairo University).
2. A FTIR spectrophotometer (ATI Mattson Genesis Series) was used with KBR pellets.

3. ^{1}H-NMR was carried out using a Varian Gemini 200 MHz spectrophotometer. The samples were dissolved in DMSO and TMS. An internal standard was used.
4. The melting points were determined using an electrothermal MEL-TEMP 3.0 apparatus.

Synthesis of Ferrocene Disulfonic Acid (I)

Ferrocene disulfonic acid was prepared through dropwise addition of chlorosulfonic acid (0.05 mol) to a rapidly stirred solution of ferrocene (0.025 mol) in acetic anhydride (75 ml) over 3 min. The temperature was raised from 25 to 40 °C. The mixture was stirred for 16 h and set aside for a further 6 h. The precipitated ferrocene disulfonic acid was filtered off and washed with acetic anhydride (15 ml) (yield % = 83) [10].

Synthesis of Fatty Ammonium Ferrocene Disulfonates ($I_{a, b, c, d, e}$)

The required fatty ammonium salts were obtained by dissolving 0.1 mol of each fatty amine in ethanol, then adding the solution to ferrocene disulfonic acid (0.05 mol) dissolved in ethanol and stirring for 1 h. The products were decyl, dodecyl, and hexadecyl primary ammonium salts of ferrocene disulfonic acid, as well as *N*,*N*-dimethyloctyl and *N*,*N*-dimethyl dodecyl tertiary ammonium salts of ferrocene disulfonic acid.

The specifications and the elemental analyses of the synthesized ammonium salts ($I_{a, b, c, d, e}$) are shown in Table 1.

The purity of the investigated compounds was confirmed by the absence of minima near the critical micelle concentration (CMC) in the surface tension plots [7, 11, 12]. The chemical structures of the novel surfactants were confirmed using elemental analyses and FTIR spectroscopy (Table 2; Figs. 1, 2). In addition the chemical structures of I_b and I_e as examples for both primary and tertiary ammonium salts respectively were further confirmed using ^{1}H-NMR spectra (Table 3; Figs. 3, 4).

Surface Tension Measurements

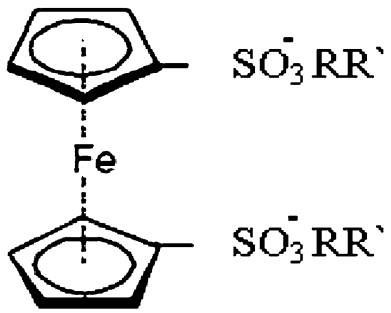
The determination of the surface tension was carried out using a Krüss-type K6 tensiometer equipped with a platinum–iridium Du Nouy ring. The solutions were transferred slowly into the double-walled vessel around which a thermostated liquid was circulated to maintain a constant temperature (30, 40, and 50 °C). Distilled water with a surface tension of 69 dyne/cm at 30 °C was used to prepare all solutions in the concentration range of 5×10^{-3} to 1×10^{-6} mol/L.

Antimicrobial Activities

The antibacterial activity of the synthesized surfactants was evaluated at the Micro Analytical Center (Cairo University) according to the diffusion disc method [13–16] against *Pseudomonas aeruginosa* and *Staphylococcus aureus*, while the fungicidal activity was evaluated against *Aspergillus flavus* and *Candida albicans*.

Results and Discussion

Primary and tertiary alkyl ammonium salts of ferrocene disulfonic acid may be denoted by



where R = NH_3 and R′ = $(CH_2)_nCH_3$, with n = 9, 11, 15 (I_a, I_b, I_c); or R = $NH(CH_3)_2$ and R′ = $(CH_2)_nCH_3$ with n = 7, 11 (I_d, I_e) and

Table 1 Specifications and elemental analyses of the synthesized ammonium salts ($I_{a, b, c, d, e}$)

Ammonium salts	Molecular formula	Yield %	Solvent of crystallization	Melting point (°C)	Elemental analysis (calculated %)			
					C	H	N	S
I_a	$C_{30}H_{56}FeN_2O_6S_2.2H_2O$	45	Chloroform	188	51.88 (51.72)	8.24 (8.62)	4.00 (4.02)	10.08 (9.19)
I_b	$C_{34}H_{64}FeN_2O_6S_2.2H_2O$	38	Ethanol	195	54.10 (54.25)	9.17 (9.04)	3.84 (3.72)	8.86 (8.51)
I_c	$C_{42}H_{80}FeN_2O_6S_2.H_2O$	33	Alcoholic H_2O	190	60.47 (59.57)	9.96 (9.69)	3.64 (3.30)	7.29 (7.56)
I_d	$C_{30}H_{56}FeN_2O_6S_2.2H_2O$	30	Purified in 1,4-dioxane	Viscous oil	51.04 (51.72)	9.02 (8.62)	4.00 (4.02)	8.70 (9.19)
I_e	$C_{38}H_{72}FeN_2\ O_6S_2.2H_2O$	32	Purified in 1,4-dioxane	Viscous oil	55.53 (56.43)	9.90 (9.40)	3.98 (3.46)	7.63 (7.92)

Table 2 FTIR spectroscopic analysis of the synthesized surfactants ($I_{a, b, c, d, e}$)

Functional group	IR bands (cm^{-1})				
	I_a	I_b	I_c	I_d	I_e
C-$H_{st.}$	2,920 and 2,851	2,918 and 2,850	2,920 and 2,851	2,926 and 2,856	2,918 and 2,849
C-$H_{def.}$	1,467	1,470	1,466	1,469	1,465
ν_s (S=O)	1,062	1,062	1,062	1,042	1,039
ν_{as} (S=O)	1,212	1,213	1,214	1,215	1,214
Asym. bending N^+H_3	1,594	1,594	1,593	–	–
Symm. bending N^+H_3	1,529	1,529	1,529		
N^+H	–	–	–	2,682	2,653

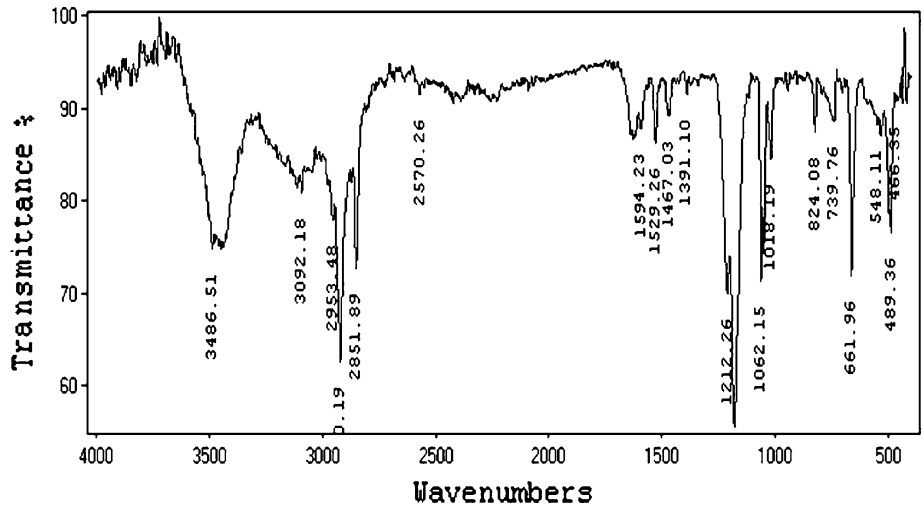


Fig. 1 FTIR spectrum of 1,1′-di-decyl ammonium ferrocene disulfonate (I_a)

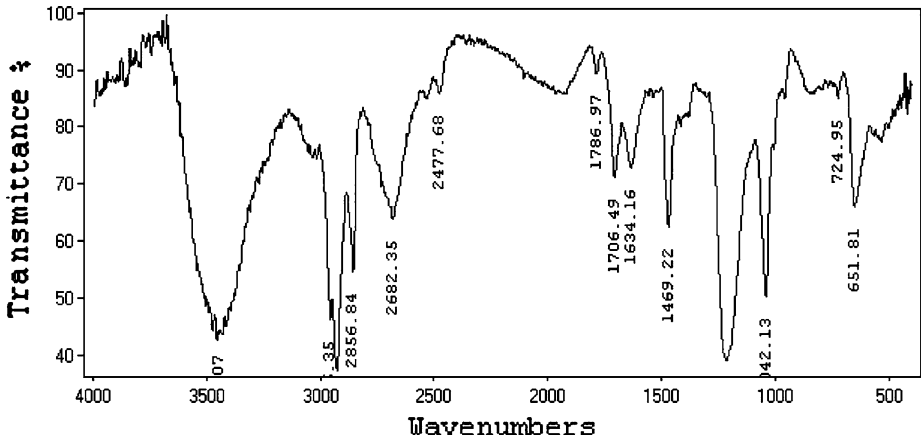


Fig. 2 FTIR spectrum of 1,1′-di-*N*,*N*-dimethyloctyl ammonium ferrocene disulfonate (I_d)

Table 3 ^{1}H-NMR spectral data for compounds I_b and I_e

Ammonium salt	^{1}H-NMR spectra data (δ ppm)
I_b	4.14, $4H_{ferrocene}$; 4.34, $4H_{ferrocene}$; 0.87, CH_3; 1.26, $2(CH_2)_8$; 1.53, $(CH_2)CH_3$; 2.78, $(CH_2)N^+$; 2.52, $(CH_2)CH_2N^+$
I_e	4.17, $2H_{ferrocene}$; 4.39, $2H_{ferrocene}$; 4.93, $4H_{ferrocene}$; 0.87, CH_3; 1.27, $2(CH_2)_8$; 1.56, $(CH_2)CH_3$; 2.84, CH_2N^+; 2.63, $(CH_2)CH_2N^+$; 3.42, $(CH_3)_2N^+$

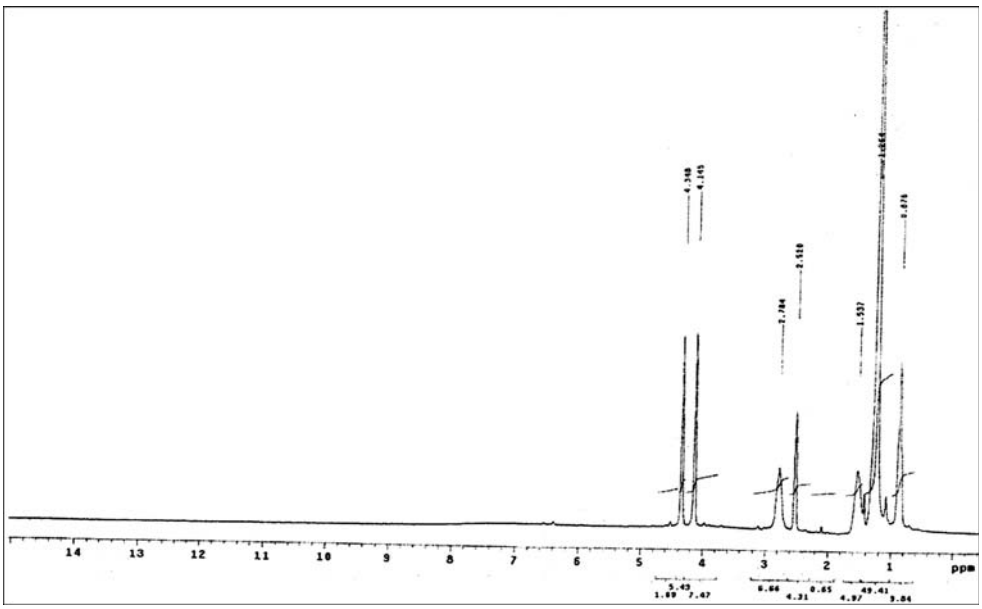


Fig. 3 ^{1}H-NMR spectrum of 1,1′-di-dodecyl ammonium ferrocene disulfonate (I_b)

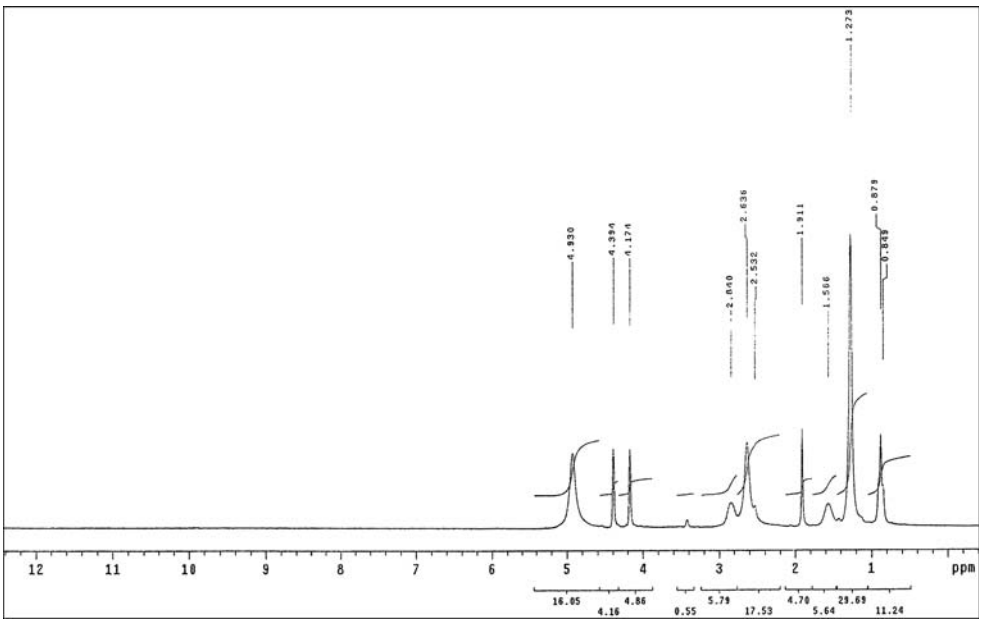


Fig. 4 ^{1}H-NMR spectrum of 1,1′-di-*N*,*N*-dimethyl dodecyl ammonium ferrocene disulfonate (I_e)

I_a = 1,1′-Di-decyl ammonium ferrocene disulfonate
I_b = 1,1′-Di-dodecyl ammonium ferrocene disulfonate
I_c = 1,1′-Di-hexadecyl ammonium ferrocene disulfonate
I_d = 1,1′-Di-*N*,*N*-dimethyloctyl ammonium ferrocene disulfonate
I_e = 1,1′-Di-*N*,*N*-dimethyldodecyl ammonium ferrocene disulfonate

^{1}H-NMR spectra of the compound I_b showed that in the $N^{+}H_3$ group, the proton exchange is suppressed and the signal disappeared, due to coupling with ^{14}N with $J_{NH} = 50$ Hz [17].

Surface Parameters

Surface Tension (γ) and Critical Micelle Concentration (CMC)

Figures 5 and 6 represent the relationship between the surface-tension values and concentration of 1,1′-didecyl ammonium ferrocene disulfonates (I_a) and 1,1′-di-*N*,*N*-dimethyloctyl ammonium ferrocene disulfonates (I_d) at 30, 40, and 50 °C. The surface tension values of the surfactant 1,1′-dihexadecyl ammonium ferrocene disulfonate (I_c) could not be determined due to its low solubility in an aqueous medium.

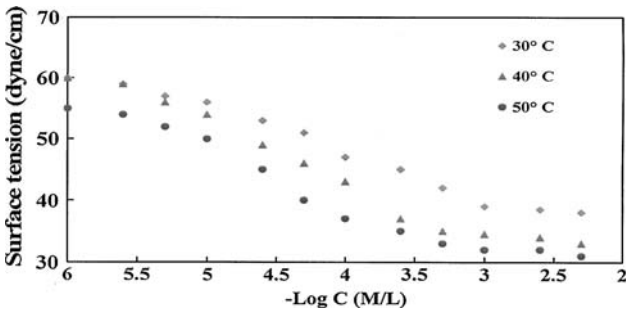


Fig. 5 Surface-tension profile of 1,1′-di-decyl ammonium ferrocene disulfonates (I_a) at different temperatures

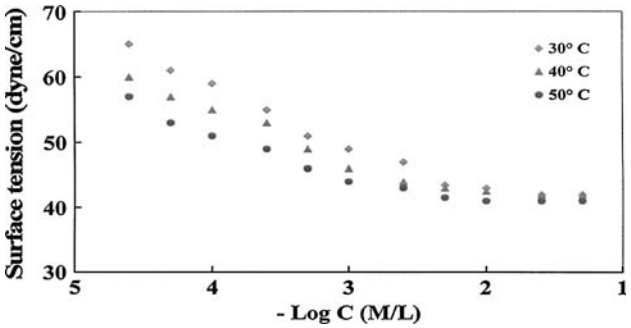


Fig. 6 Surface-tension profile of 1,1′-di-*N*,*N*-dimethyloctyl ammonium ferrocene disulfonates (I_d) at different temperatures

The CMC values of both primary and tertiary disubstituted series decreased with increasing alkyl chain length as shown in Table 4, which increases the hydrophobicity of the molecules [18], leading to increased repulsion between the polar medium (H_2O) and the nonpolar chains so that the molecules tend to aggregate at lower concentrations [19].

In general, increasing the temperature has a lowering effect on the CMC values of the investigated surfactants because increasing the temperature decreased the hydration of the hydrophilic group, favoring micellization [20].

Effectiveness (π_{CMC})

The effectiveness determines the surface activity of the surfactant molecules at their CMC. The effectiveness values are listed in Table 4 and may be attributed to the difference in aggregation type of each of the prepared surfactants as well as their hydrophobicity. Increasing the hydrophobic chain length along the prepared amine disulfonic acid salts increases their effectiveness.

The most effective surfactant is the one producing the lowest surface tension at CMC. According to the results of π_{CMC}, I_b and I_e are found to be more effective as shown in Table 4.

Efficiency Pc_{20}

From Table 4, it is clear that the efficiency of adsorption (Pc_{20}) increases increasing numbers of the carbon atoms of the hydrophobic group.

For all the prepared surfactants, the efficiency also increased with increasing temperature. This increase was due to the decrease in the surface tension values by heating, leading to a decrease in the concentration, resulting in a decrease in the surface tension of the solvent decreased by 20 mN m^{-1} cm^{-1}.

Maximum Surface Excess (Γ_{max})

Increasing the temperature increases the interaction between the polar solvent and the surfactant molecules, which directs them towards the bulk of the solution leading to a decrease in the surface excess. Also increasing the hydrophobic chain length leads to the complete coverage of

Table 4 Surface parameters of the prepared surfactants ($I_{a,\ b,\ d,\ e}$) at different temperatures

Surfactant	Temperature (°C)	CMC (M/L)	π_{CMC} (mN/m)	Pc_{20}	$\Gamma_{max} \times 10^{-10}$ (M/cm^2)	A_{min} (nm^2)
I_a	30	1×10^{-3}	30	4.20	1.55	1.07
	40	0.56×10^{-3}	32	4.39	1.66	0.99
	50	0.4×10^{-3}	32	4.60	1.61	1.03
I_b	30	0.39×10^{-3}	33	5.00	1.35	1.23
	40	0.28×10^{-3}	34	5.30	1.27	1.30
	50	0.25×10^{-3}	33	5.30	1.22	1.36
I_d	30	5.01×10^{-3}	25.5	3.00	1.47	1.12
	40	3.09×10^{-3}	24	3.00	1.31	1.26
	50	2.18×10^{-3}	23.5	3.00	1.29	1.28
I_e	30	1×10^{-3}	32	4.60	1.35	1.22
	40	0.83×10^{-3}	31	4.60	1.16	1.42
	50	0.53×10^{-3}	30	5.00	1.13	1.46

the surfactant solution by adsorbed molecules at lower concentrations (shifts Γ_{max} to lower concentrations).

Minimum Surface Area (A_{min})

The minimum area occupied by classical surfactants [$P(CH_2)_nCH_3$, where P is a polar head group, e.g., $N^+(CH_3)_3$ or SO_4^{-2}] at the surface of water is determined, in part, by a competition between Van der Waals forces among aliphatic chains and repulsive interactions (e.g., electrostatic or hydration) between polar head groups. Therefore, an increase in aliphatic chain length results in a decrease in the minimum area per molecule at the surface of an aqueous solution [21].

In our case, A_{min} increased with the length of the aliphatic chain of the surfactant. The balance of forces leading to the organization of the ferrocenyl surfactants on the surfaces of aqueous solutions is, therefore, different from the balance of forces governing the assembly of classical surfactants on the surfaces of aqueous solutions. This result agrees with a previously reported work in ferrocene surfactants [22].

Increasing the temperature increased the surface coverage area of the molecules, which can be explained by the increase in the number of adsorbed molecules at the interface; hence the molecules occupied a larger area (Table 4).

By comparing the A_{min} values of I_b and I_e, we observed that A_{min} of I_e is slightly greater than that of I_b. This could be attributed to the fact that branching has a small effect and a small increase in the area per molecule at the interface [20].

Thermodynamic Parameters of Micellization and Adsorption of the Prepared Surfactants

According to Gibbs' adsorption equations (Eqs. 1–6) and following the methodology of [21], the thermodynamic parameters of micellization and adsorption of the synthesized surfactants were calculated at 30, 40, and 50 °C. The results are listed in Table 5. For micellization:

$$\Delta G^{\circ}_{(mic)} = RT\ln(CMC) \quad (1)$$

$$-\Delta S^{\circ}_{(mic)} = d(\Delta G^{\circ}_{(mic)})\Big/\Delta T \quad (2)$$

$$\Delta H^{\circ}_{(mic)} = \Delta G^{\circ}_{(mic)} + T\Delta S^{\circ}_{(mic)} \quad (3)$$

For adsorption:

$$\Delta G^{\circ}_{(ads)} = \Delta G^{\circ}_{(mic)} - 6.023 \times 10^{-1} \times \pi_{CMC} \times A_{min} \quad (4)$$

$$-\Delta S^{\circ}_{(ads)} = d(\Delta G^{\circ}_{(mic)})\Big/\Delta T \quad (5)$$

$$\Delta H^{\circ}_{(ads)} = \Delta G^{\circ}_{(ads)} + T\Delta S^{\circ}_{(ads)} \quad (6)$$

Table 5 Thermodynamic parameters of micellization (mic) and adsorption (ads) of the prepared surfactants ($I_{a,\ b,\ d,\ e}$) at different temperatures

Cpd	T (°C)	$\Delta G°$ (KJ/mol)		$\Delta S°$ (KJ/mol·K)		$\Delta H°$ (KJ/mol)	
		Mic	Ads	Mic	Ads	Mic	Ads
I_a	30	−17.40	−36.73	–	–	–	–
	40	−19.48	−38.56	0.20	0.18	43.12	18.71
	50	−21.01	−40.86	–	–	–	–
I_b	30	−19.77	−44.21	–	–	–	–
	40	−21.28	−47.90	0.15	0.37	25.67	67.91
	50	−22.27	−49.30	–	–	–	–
I_d	30	−13.34	−30.54	–	–	–	–
	40	−15.04	−33.25	0.17	0.27	38.17	51.26
	50	−16.45	−34.56	–	–	–	–
I_e	30	−17.40	−40.91	–	–	–	–
	40	−18.46	−44.97	0.10	0.40	12.84	80.23
	50	−20.25	−51.02	–	–	–	–

The standard free energies of micellization and adsorption, $\Delta G°_{(mic)}$ and $\Delta G°_{(ads)}$, are always negative, indicating that these are spontaneous processes. There is more decrease in the negativity of $\Delta G°_{(ads)}$ than of micellization, showing the increasing tendency of surfactant molecules to be adsorbed at the interfaces. The preference for adsorption relates to the fact that the repulsion forces occurring between the hydrophobic molecules and the aqueous phase reach their minimum value when surfactant molecules are located at the interface (air/water).

The values of standard entropy changes of adsorption ($\Delta S°_{(ads)}$) show a greater increase than those of micellization [$\Delta S°_{(mic)}$] (Table 5), indicating greater randomness of the molecules in the adsorbed state than in the micellized one. This may be due to the compactness of the hydrophobes within the micelles, which offers a higher degree of constraint of molecules [23].

$\Delta H°_{(mic)}$ are all positive values indicating the endothermic nature of the micellization process with the surfactants under study. The values of the standard heat enthalpy of micellization, $\Delta H°_{(mic)}$, decreased with increasing hydrophobic chain length.

Antimicrobial Activity

The synthesized surfactants were evaluated as biocides for Gram-positive bacteria, Gram-negative bacteria, and fungi. The data are shown in Table 6.

In general, the inhibition-zone diameter values (mm) are classified as follows [24]: >15 mm = significant activity, 7–14 mm = moderate activity, and <7 mm = weak activity.

Table 6 Antibacterial and antifungal activities of the synthesized surfactants ($I_{a–e}$)

Sample	Inhibition zone diameter (mm)			
	Pseudomonae aeruginosa	*Staphylococcus aureus*	*Aspergillus flavus*	*Candida albicans*
I_a	20	20	13	15
I_b	16	16	13	15
I_c	11	11	10	10
I_d	12	12	12	11
I_e	12	11	10	11

According to Table 6, most of the compounds show moderate antibacterial and antifungal activities**,** while only I_a and I_b show significant antibacterial activity.

It is clear from the data that the antibacterial activities of the compounds decrease with increasing chain length in I_a, I_b, and I_c, which may be attributed to the greatly increased lipophilicity of the molecules resulting from both the chain and the counterion (ferrocene sulfonates), leading them to take more time to cross the cell membrane, so activity decreases [25]. The activities of I_d and I_e are not affected by the difference in the chain length.

The wall of Gram-positive bacterial cells is composed of a peptidoglycan chain of polysaccharide, teichonic acid, and phosphated sugar. Teichonic acids gave the Gram-positive bacterial cell wall a negative charge, which may be important in determining the types of substances attracted to the cell membrane [26].

Data in Table 6 show that these surfactants have approximately the same activity against *Pseudomonae aeruginosa* and *Staphylococcus aureus*. This means that their mechanism of action may depend on the counterion penetrating into the cytoplasm of the cell, where it inactivates essential metabolic proteins. The inactivation proceeded via oxidation of these proteins resulting in the bacterial cell death [27], while the cation portion of the molecule is attracted to the negatively charged cell membrane, resulting in neutralizing its charge and distorting its selective permeability as well [28].

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