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ORIGINAL ARTICLE

Synthesis and Study of the Surface Properties of Alkylnaphthalene and Alkylphenanthrene Sulfonates

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Abstract Some alkylnaphthalene and alkylphenanthrene sulfonates were synthesized by means of a Wurtz–Fittig reaction. The HLB values for the prepared compounds were calculated, and the basic properties were studied in water at different temperatures, namely, 25, 35 and 45 °C. Through surface tension measurements, the following values were determined: the critical micelle concentration (CMC) and the surface tension at the CMC (γ_{CMC}). The following values were calculated: area per molecule at the CMC (A_{CMC}), standard free energy change of micellization (ΔG_{mic}), standard free energy of adsorption (ΔG_{ad}), and the efficiency of a surfactant in reducing surface tension (pC20). Furthermore, the partition coefficients of the synthesized compounds were also measured. The results show that *n*-alkylnaphthalene and *n*-alkylphenanthrene surfactants studied exhibit desirable properties that may be of value in some fields such as detergency. To confirm the detergency power of the prepared surfactants, some foam studies were performed.

Keywords CMC · Micelle aggregation number · Sodium α-(*n*-C6–C10) naphthalene sulfonate · Sodium (*n*-C6–C10) phenanthrene sulfonate

Introduction

Surfactants are widely used and find a very large number of applications because of their remarkable ability to influence the properties of surfaces and interfaces. They find many applications in the petroleum industry. They can be applied at all stages in petroleum recovery and processing, from oil well drilling up to transportation of petroleum emulsions [1–3]. Short-chain alkylnaphthalene sulfonates have been extensively applied in the printing, dyeing, and oil extraction industries [4, 5].

There are some reported researches on the surface and interfacial properties of long and short-chain alkylnaphthalene sulfonates [6–8].

The hydrotropic properties of a novel alkylnaphthalene sulfonate were investigated for a novel sodium diisopropylnaphthalene sulfonate (SDIPNS) which is both a hydrotrope and a surfactant [9].

Until now, some papers concerning alkylnaphthalene surfactants have been published. However, the reports about alkylphenanthrene surfactants are very few. Therefore, it is meaningful for this paper to describe the synthesis of sulfonate type anionic alkylnaphthalene and alkylphenanthrene surfactants.

Experimental Procedures

Reagents

Reagents were as following: twice distilled water, α-bromo naphthalene, *n*-hexyl bromide, *n*-octyl bromide, *n*-decylbromide, *n*-hexyl phenanthrene bromide, *n*-octyl phenanthrene bromide, *n*-decyl phenanthrene bromide, which were all of analytical reagent grade and supplied from Sigma–Aldrich for chemicals. The sodium metal was highly pure (≥99.5%) and also supplied by Sigma–Aldrich for chemicals. The solvents used throughout this work such as chloroform, ethanol, methanol, petroleum ether were purchased from the Acros Organics Company as ultra pure

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solvents and used as received. Anhydrous $MgSO_4$ (AR), sodium hydroxide NaOH (AR) were also from Acros.

Synthesis Procedures

Synthesis of Sodium α-(n-alkyl)Naphthalene Sulfonates and α-(n-alkyl)Phenanthrene Sulfonates

A dry, 250-mL, three-necked, round-bottomed flask was fitted with a reflux condenser, a drying tube, and a thermometer, which dipped into the reaction mixture. The flask was set up so that it could be cooled in an external cooling bath. In the flask was placed 4 g of sodium pellets dispersed in 50 mL ether. A mixture of 21.0 mL of α-bromonaphthalene and 25 mL of *n*-alkyl bromide was added to the flask. The flask was then placed in an ice bath to keep the reaction temperature as near to 40 °C as possible. The reaction started within 5 min. The mass acquired a yellow color. After 0.5 h, the mixture was boiled gently and was kept boiling gently for 2.5 h and then was held for a day at room temperature with continuous shaking. The liquid was decanted and washed several times with ether. The ether extract was dried over 4 g of anhydrous $MgSO_4$. The ether was then removed by rotary evaporation, The final product was a yellow liquid [about 6 g of *n*-alkyl $C_{10}H_7$].

The same procedures were applied for the preparation of *n*-alkylphenathrene.

The equations of the reactions are shown in Fig. 1a, b.

Sulfonation Reaction

A mixture of 6 g of *n*-alkyl naphthalene and 12 mL of chloroform was added to a 50-mL three-necked, round-bottomed flask and cooled to between −10 and −5 °C in an ice bath. Chlorosulfonic acid (2 mL) was added slowly from a dropping funnel over a period of about 30 min. The temperature of the reaction increased slowly to +5 °C owing to heat of the reaction and was held as near 5 °C as possible. The reaction was continued for 2 h and then neutralized by adding 30% NaOH (AR). The crude product was recrystallized twice in an ethanol/water mixture (50:50, vol/vol) and then dried for 36 h under vacuum at 80 °C.

The equations of the sulfonation reaction are given in Fig. 2a, b. The designations of the prepared compounds together with some of their characteristics are listed in Table 1.

Characterization of the Prepared Compounds

Gel Permeation Chromatography (GPC)

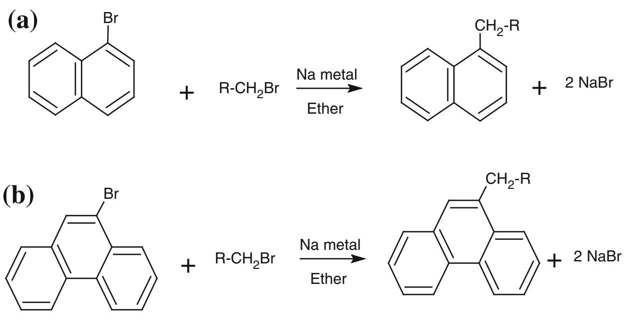
The molecular weights of the prepared surfactants were measured by GPC Water model 600 E. The mobile phase was toluene HPLC grade. The measured and calculated molecular weights are provided in Table 1.

Spectral Data of Alkylnaphthalene and Alkylphenanthrene Sulfonates

The chemical structure of the investigated sulfonates was confirmed via both NMR and IR spectral techniques. The NMR spectral results were obtained on a Shimadzu-400 NMR spectrograph (D_2O as solvent and TMS as the internal standard), the assignments of the protons of *n*-alkyl naphthalene and *n*-alkyl phenanthrene are given in Fig. 3a, b. For brevity, NMR spectra of SN6 and SP6 are supplied. Infrared (IR) spectra were obtained using a Perkin-Elmer FT-IR spectrometer. The data are shown in Tables 2 and 3.

Determination of the Partition Coefficient (PC)

The partition coefficient is the ratio of the solubility of a surfactant in the aqueous phase to its solubility in the oil



Where R=C_6H_{13}, C_8H_{17} or $C_{10}H_{21}$

Fig. 1 **a**, **b** The alkylation reactions of bromonaphthalene and bromophenanthrene

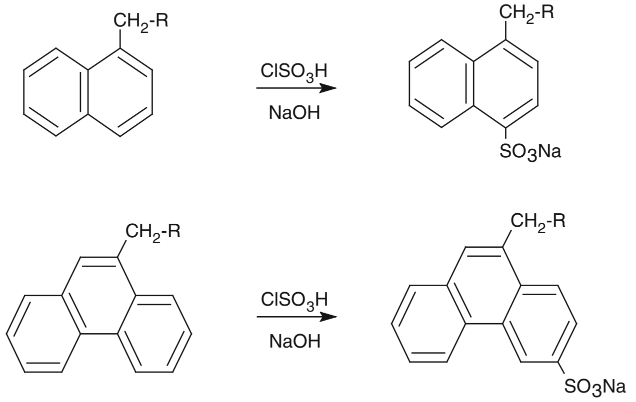
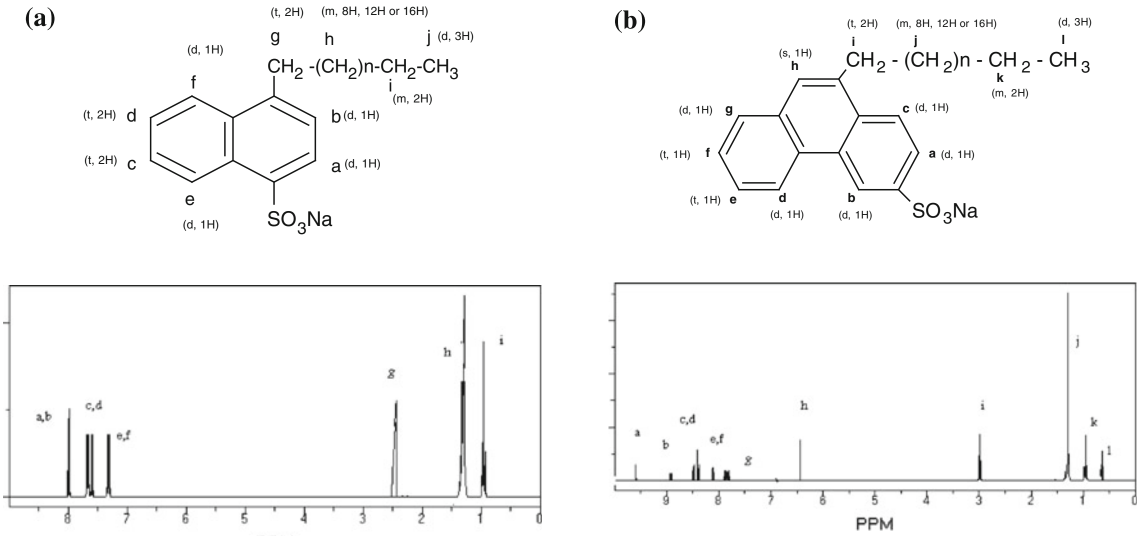


Fig. 2 **a**, **b** The sulfonation reactions of alkylnaphthalene and alkylphenanthrene

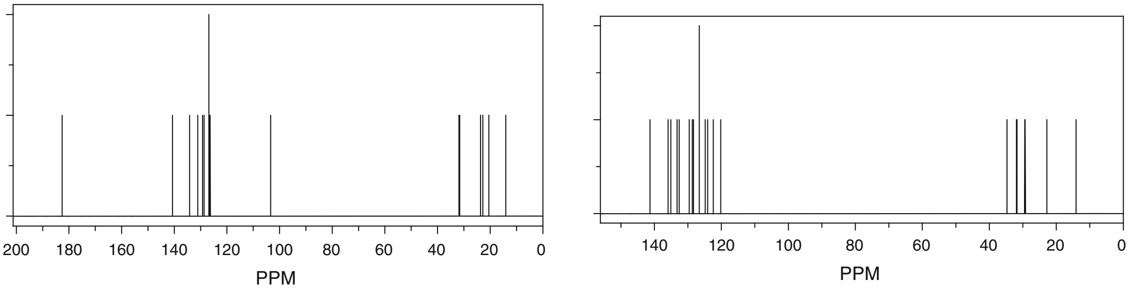
Table 1 Designations, M.Wt. and HLB values for the prepared compounds

The compound	Designation	Yield (%)	M.Wt. ($gmol^{-1}$)		HLB	PC
			Cal.	Det.		
Sodium *n*-hexyl naphthalene sulfonate	SN6	65	328	330	23.19	5.02
Sodium *n*-octyl naphthalene sulfonate	SN8	67	360	363	22.24	4.22
Sodium *n*-decyl naphthalene sulfonate	SN10	60	392	393	21.29	3.92
Sodium *n*-hexyl phenanthrene sulfonate	SP6	68	350	351	21.53	3.87
Sodium *n*-octyl phenanthrene sulfonate	SP8	75	382	384	20.58	3.21
Sodium *n*-decyl phenanthrene sulfonate	SP10	71	414	417	19.63	2.11



^{1}HNMR of SN6

^{1}HNMR of SP6



^{13}CNMR of SN6

^{13}CNMR of SP6

Fig. 3 **a** and **b** ^{1}H NMR and ^{13}C NMR for SN6 and SP6

phase [10]. The procedure was carried out by dissolving 0.25 g of the surfactant sample in 25 ml benzene which represents the oil phase; 25 ml of distilled water was added, and the mixture was shaken by an automatic flask shaker for 0.5 h. The flask was tightly stoppered and left in a water bath at 25 °C to separate into two layers. The upper layer (benzene) was taken off, and its UV absorbance was determined. The concentration of the dissolved surfactant in the benzene was then estimated from a calibration curve representing the intensity of the absorbance as a function of concentration. By knowing the concentration of the surfactant in the benzene phase, the concentration of the surfactant in the aqueous phase was computed from the initial concentration.

Table 2 NMR and IR data for the SN series

Compound	NMR, δppm	IR cm^{-1}
SN6	*^{1}H NMR* The aromatic ring protons: a 8.01–7.9(d,1H), b 7.84–7.72(d,1H), c 7.51–7.49 (t, 1H) and d 7.47–7.44(t, 1H), e&f 7.19–7.12(d, 2H) The methylenes protons: g 2.47–2.45(t, 2H), h 1.70–1.40(m, 8H), I 1.2–0.9(m,2H) The methyl protons: J 0.70–0.60(t,3H) *^{13}C NMR* at 105–140 aromatic rings carbons, at 182 C–SO$_3$Na, at 31 CH$_3$, at 20–25 CH$_2$ carbons and at 15 CH$_2$–C=C	IR (KBr) [3030(C–H ar)] [2928, 2852(C–H aliph.)], 1465(CH$_2$)], [1450 and 1375(CH$_3$)1512(C = C), [1201, 1163, 1071(S=O)], [758(oop C–H ar.)]
SN8	The aromatic protons: a 8.58–8.42(d,1H), b 7.84–7.81(d,1H), c 7.48–7.44,(t,1H), d 7.20–7.17(t,1H), e&f 6.78–6.74(d, 2H) The methylenes protons: g 2.49–2.46(t, 2H), h 1.80–1.70(m, 12H), I 1.40–1.10(m, 2H), The methyl protons: J 0.90–0.80(t, 3H)	IR (KBr) [3070(C–H ar)] [2924, 2852 (C–H aliph.)], 1470(CH$_2$)], [1445 and 1375(CH$_3$)1512(C=C), [1218, 1198, 1071 (S=O)], [757(oop C–H ar.)]
SN10	The aromatic protons: a 8.80–8.70(d, 1H), b 8.12–8.11(d,1H), 8.07–8.04(d,1H), 7.58–7.54(m, 2H), 7.34–7.31(d,1H) The methylenes protons: 3.11–3.07(t,2H), 1.80–1.70(m,2H), 1.4–1.1(m,16H) The methyl protons: 0.88–0.80(t, 3H)	IR (KBr) [3090(C–H ar)] [2921, 2851 (C–H aliph.)], 1475(CH$_2$)], [1460 and 1370(CH$_3$)1511(C=C), [, 1201, 1164, 1072 (S=O)], [761(oop C–H ar.)]

Table 3 NMR and IR data for the SP series

Compound	NMR, δppm	IR cm^{-1}
SP6	*^{1}H NMR* The aromatic ring protons: a 9.51–9.5(d,1H), b 8.88–8.84(d,1H), c 8.52–8.50(d,1H), d 8.42–8.4 (d,1H), e&f 8.2–8.1(t, 1H), g 7.90–7.88(d,1H), h 6.5(s,1H) The methylenes protons: I 3.01–2.91(t, 2H), j 1.40–1.20(m, 8H), k 1.1–0.9(m,2H) The methyl protons: 0.70–0.60(t,3H) *^{13}C NMR* at 120–140 aromatic rings carbons, at 142 C–SO$_3$Na, at 35 CH$_3$, at 20–33CH$_2$ carbons and at 17 CH$_2$–C=C	IR (KBr) [3100(C–H ar)] [2923 2850 (C–H aliph.)], 1475(CH$_2$)], [1455 and 1365(CH$_3$)1516(C=C), [1209, 1168,1077 (S=O)], [758,661,640 (oop C–H ar.)]
SP8	The aromatic ring protons: a 9.2–8.91(d,1H), b 8.88–8.78(d,1H), c 8.64–8.41 (d,1H), d 7.94–7.88(d,1H), e & f 7.66–7.57(t, 1H) and 7.41–7.35 (t, 1H), g 7.22-7.19(d,1H), h 6.83–6.75 (s,1H) The methylenes protons: I 2.54–2.50(t, 2H), j 1.70–1.40(m, 12H), k 1.1–0.9(m,2H) The methyl protons: 0.81–0.75(t,3H)	IR (KBr) [3050(C–H ar)] [2929 2859 (C–H aliph.)], 1520(CH$_2$)], [1450 and 1375(CH$_3$)1516(C=C), [1212, 1170,1080, (S=O)], [760,665,644 (oop C–H ar.)]
SP10	The aromatic ring protons: a 9.3–8.1(d,1H), b 8.98–8.84(d,1H), c 8.74–8.63 (d,1H), d 7.55–7.50(d,1H), e&f 7.47–7.43(t, 1H) and 7.23–7.20(t, 1H), g 7.17–7.13(d,1H), h 6.83–6.75 (s,1H) The methylenes protons: I 2.33–2.21(t, 2H), j 1.50–1.30(m, 16H), k 0.9–0.7(m,2H) The methyl protons: 0.60–0.40 (t,3H)	IR (KBr) [3070(C–H ar)] 2930 2855, (C–H aliph.)], 1518(CH$_2$)], [1460 and 1375(CH$_3$)1516(C=C), [1214, 1171,1082, (S=O)], [761,664,645 (oop C–H ar.)]

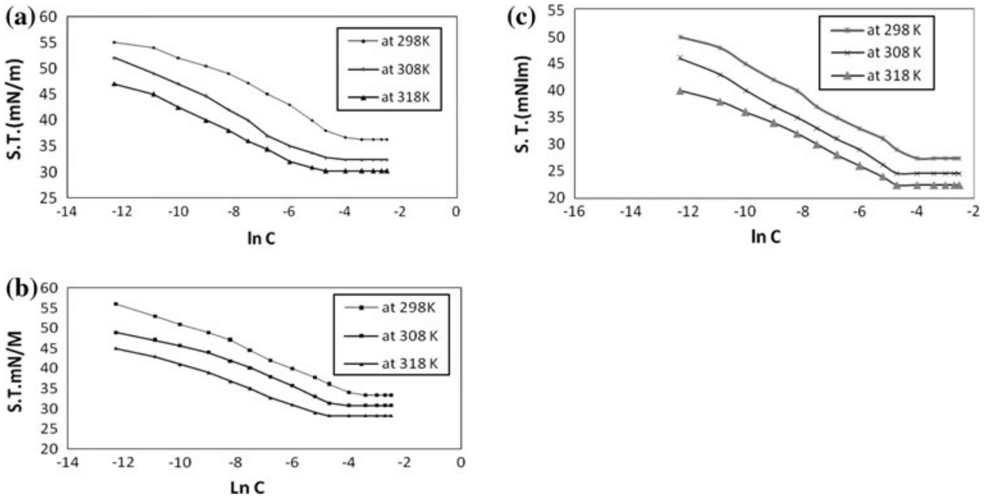


Fig. 4 **a** S.T. conc. isotherm of SN6 at different temperatures, **b** S.T. conc. isotherm of SN8 at different temperatures, **c** S.T. conc. isotherm of SN10 at different temperatures

Partition coefficient = Solubility in an aqueous phase/ solubility in an oil phase

A calibration curve was obtained for each surfactant by measuring the UV absorbance of a series of concentrations of the surfactant dissolved in benzene. The following concentrations were prepared: 1, 0.8, 0.6, 0.4, 0.2, and 0.1%. The absorbance was measured by a Shimadzu UV spectrophotometer, model 120.02, adjusted at 355 nm representing maximum absorbance. The values of the partition coefficient of the prepared compounds are given in Table 1.

Measurement of Surface Tension

Tensiometric measurements were carried out using ring method on Kruss K11 tensiometer. All surface-tension measurements were conducted at 25, 35and 45 °C. The results are shown in Fig. 4a–c for SN group and Fig. 5a–c for SP group, whereas the surface properties of the prepared compounds are supplied in Table 4.

Foaming Technique

The apparatus and procedure used were those of ASTM method Dl173-53 [11, 12], with the exception that the assembly used in the ASTM method at the top of the column to hold the pipet in place was replaced by ordinary flask clamps, and positioning of the pipet was accomplished with a spirit level. The foam was made for 0.1% aqueous solution of the prepared samples. The foam cylinder used had an internal diameter of 5 cm. All tests were

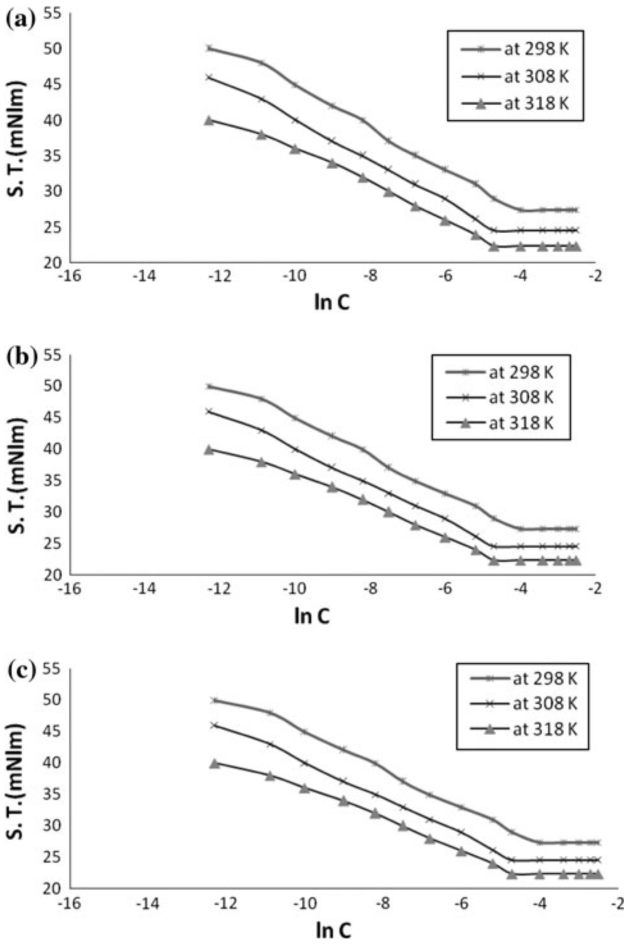


Fig. 5 **a** S.T. conc. isotherm of SP6 at different temperatures, **b** S.T. conc. isotherm of SP8 at different temperatures, **c** S.T. conc. isotherm of SP10 at different temperatures

Table 4 CMC and γCMC of sulfonated surfactants at 298, 308 and 318 K

The compound	CMC (mmol/L)			γCMC(mN/m)		
	298 K	308 K	318 K	298 K	308 K	318 K
SN6	27	13.6	6.7	36.3	32.4	30.2
SN8	20.7	10.2	4.1	33.4	30.8	28.3
SN10	15.4	9.3	1.4	30.5	28.9	22.4
SP6	25.6	11.4	5.5	34.7	31.4	28.7
SP8	18.3	9.1	3.5	30.2	27.1	25.4
SP10	12.2	5.3	0.54	27.4	24.6	22.4

conducted at 49 °C. Glassware was cleaned by immersion in a hot 1:9 HNO_3–H_2SO_4 mixture for 30 min., followed by rinsing with distilled water, redistilled acetone, and finally with quartz-distilled water. The data for the foaming test are given in Table 7.

The initial foam height was taken as the height of the foam 60 s after the last drop of solution had drained from the pipet. This interval was selected because rapid drainage of liquid from the foam had ceased by this time. Four runs were made with each solution; final foam heights are averages of values from these runs. The foam height tabulated is the difference between initial and final heights.

Results and Discussion

Synthesis and Characterization

Surfactants SN and SP were obtained in good yields as white crystalline solids that were highly water soluble as it can be seen from the values of the partition coefficient listed in Table 1. Other physical data are also summarized in Table 1.

The ^{1}H-NMR spectra are in accordance with the proposed structures and contain all expected chemical shifts for both the aromatic and alkyl moieties as it can be seen in Tables 2, 3 and Fig. 3a, b.

Hydrophilic–Lipophilic Balance (HLB) Values

Surfactants are often characterized by their hydrophilic/lipophilic balance or HLB. High HLB values indicate good water, or polar solvent solubility, of the surfactant while low HLB values are indicative of good solubility in non-polar systems, such as oil. It is a well-known fact that surfactant properties are greatly influenced by the nature of the hydrophilic and hydrophobic moieties. HLB values of individual surfactant molecules can be calculated applying the Davies formula [13]. These values serve as a guide to surfactant properties at room temperature, e.g., aqueous solubility, and they can be used as a first suggestion for potential applications [14]. The HLB values of the prepared sulfonates are given in Table 1.

The data reveal that HLB values are decreased by increasing the length of the alkyl chain and the HLB values for SP (based on phenanthrene) are less than those for SN (based on naphthalene).

Surface Properties of the Prepared Sulfonates

Some of the surface properties of the prepared surfactants at three temperatures are given in Tables 4 and 5. It was observed that both the CMC and γ_{CMC} values show a regular decrease with an increase in the number of methylene groups in the alkyl chain, as would be expected from the increase in hydrophobicity [15, 16]. This usually happens in a homologous series of cationic [16], anionic [17], and nonionic [18] surfactants. It is observed that the increase in temperature has a reducing effect on CMC and γ_{CMC} values.

Table 5 includes the values of pC20 which is a suitable measure of the efficiency of a surfactant in reducing surface tension and can be calculated from surface tension at the air–water solution versus log C. The larger the value of pC20, the more efficiently the surfactant is adsorbed at the interface and the more efficiently it reduces surface or interfacial tension. This means that a smaller bulk liquid phase concentration will be required either to attain

Table 5 Some surface properties of sulfated surfactants

The compound	pC20			$\Gamma_{max}10^{10}$ (mol cm^{-2})			A_{min} (nm^2/molecule)		
	298 K	308 K	318 K	298 K	308 K	318 K	298 K	308 K	318 K
SN6	5.4	6.2	7	0.78	0.76	0.75	0.213	0.215	0.218
SN8	7.08	7.73	9.3	0.82	0.67	0.75	0.2	0.24	0.22
SN10	8.2	8.4	9.7	0.9	0.99	1.1	0.18	0.16	0.15
SP6	6.30	6.73	7.00	1.3	1.2	0.74	0.12	0.13	0.21
SP8	7.08	7.73	9.33	0.77	0.80	0.99	0.21	0.20	0.24
SP10	9.7	8.25	9.77	0.57	0.39	0.76	0.29	0.42	0.218

Table 6 ΔG_{mic} and ΔG_{ads} of the sulfated alkyl naphthalene and phenanthrene at different temperatures

The compound	ΔG_{mic} (kJ/mol)			ΔG_{ads} (kJ/mol)		
	298 K	308 K	318 K	298 K	308 K	318 K
SN6	−23.145	−21.421	−20.779	−21.813	−19.957	−16.376
SN8	−25.010	−22.891	−20.460	−22.420	−20.124	−18.085
SN10	−26.339	−23.367	−20.057	−24.833	−21.397	−19.962
SP6	−22.225	−18.223	−15.258	−19.359	−17.070	−15.442
SP8	−24.102	−21.317	−19.154	−20.419	−19129	−17.107
SP10	−26.067	−22.449	−20.014	−25.979	−23.432	−21.136

saturation, adsorption, or to reduce the surface or interfacial tension by 20 dyn/cm. In Table 5, the order of pC20 values is decyl > octyl > hexyl. This agrees with the findings of Xiaoli et al. and it is also higher than that of sodium *n*-alkylbenzene sulfonate [15]. Thus, the most efficient surfactant structure for detergency must be the one with decyl group.

The surface excess concentration $\Gamma_{\max}$ is calculated by means of the Gibbs equation,

$$\Gamma_{\max} = \frac{1}{RT}\left(\frac{-\partial\gamma}{\partial \ln c}\right)_T \qquad (1)$$

where the derivative is the slope of the plot of γ versus ln c at a constant temperature (*T*), and *R* is the gas constant in $\mathrm{Jmol}^{-1}\ \mathrm{K}^{-1}$.

In Table 5, one can see that the increase in the alkyl chain length increases the area occupied by the molecule at the interface, $\mathrm{A}_{\mathrm{CMC}}$ which can be calculated from the equation,

$$\mathrm{A}_{\min} = \frac{10^{16}}{N\Gamma_{\max}} \qquad (2)$$

The standard free energy of micellization is calculated from the equation

$$\Delta G_{\mathrm{mic}} = 2RT\ \ln \mathrm{CMC} \qquad (3)$$

The standard free energy of adsorption (ΔG_{ad}) is calculated according to the following equation [12]:

$$\Delta G_{\mathrm{ad}} = -(2.303\,RT)\mathrm{pC20} - 6.023 \times 20\ \mathrm{A}_{\mathrm{CMC}} - 2.303\,RT\ \log\omega \qquad (4)$$

where ω is the number of moles of water per liter of water (=55.5)

From the data in Table 6, it can be seen that ΔG_{mic} become significantly more negative with increasing alkyl chain length. It is clear also that the values of ΔG_{mic} are all negative whereas those of ΔG_{ad} are all positives which leads to the speculation that the prepared surfactants prefer micellization than adsorption so that they are effective in detergent formulations [19–21]. It is also obvious that ΔG_{mic} for the sulfonated naphthalenes are somewhat greater than those for sulfated phenanthrenes which indicates that the naphthalene-based surfactants are better than the phenanthrene surfactants in detergency formulations.

Table 7 Some foam properties of the prepared surfactants at 49 °C

The compound	Foam height (cm)	Foam stability (min)
SN6	2.7	11
SN8	3.5	12.5
SN10	4.1	15.2
SP6	2.3	9
SP8	2.6	10.3
SP10	2.9	11.4

Foaming Studies of the Prepared Sulfonates

Foaming tests were carried out as described in the experimental section to investigate the detergency power of the prepared sulfonates. Two parameters were considered, namely, foam height and foam stability. The data are summarized in Table 7. It can be seen that the foams produced from sulfonated naphthalenes (with higher HLB values) are somewhat higher and more stable than the foams produced from sulfonated phenathrenes. The data obtained confirm the higher detergency power of sulfonated naphthalenes over sulfonated phenanthrenes.

Our findings run in harmony with those of Rosen et al. [22] who concluded that, the higher the HLB values of linear decyldiphenylether sulfonates, the more stable the foam.

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