Relationship of Structure to Properties of Some Anionic Surfactants as Collectors in the Flotation Process. 2. Effect of Phenyl Group

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Surface and thermodynamic properties of four synthetic anionic surfactants are studied. The properties of these surfactants are correlated with their flotation performance to upgrade a petroleum coke sample to minimize its impurities. The surfactants are isomers for dodecylbenzenesulfonate of the same molecular weight, but the benzene ring is attached to different carbon atoms along the dodecyl chain. The results show that the position of benzene ring, attached to the carbon chain, affects their surface and thermodynamic properties. The values of critical micelle concentration (cmc), minimum area per molecule ($A_{\min}$), effectiveness (Π_{cmc}), and efficiency (pC_{20}), to reduce surface tension of water, are decreased with changing the position of benzene ring from carbon atom number 3 to 5. Increasing temperature is accompanied by an increase in each of cmc, minimum area per molecule ($A_{\min}$), Gibbs energy of micellization ($\Delta G^{\circ}_{\mathrm{mic}}$), and adsorption ($\Delta G^{\circ}_{\mathrm{ad}}$). Meanwhile, the values of surface tension (γ_{cmc}) at cmc, surface excess concentration ($\Gamma_{\max}$), and effectiveness (Π_{cmc}) to reduce surface tension of water are decreased. There is a good relationship between the structure of a surfactant and its efficiency as a collector. Changing the position of the phenyl group along the alkyl chain of a surfactant toward its central carbon atom (6ϕC12) improves its efficiency as a collector.

Introduction

Surfactants are widely used in processing of minerals, effluent, and other particulate systems. Considerable effort has been made in developing different types of surfactants and other specialty chemicals to treat ores containing many components, often finely distributed in ill-defined and variable matrices.

Most of the innovations in processing of low-grade complex ores—a current world phenomenon—are based on more effective and efficient use of chemical reagents for beneficiation and disposal of waste generated. For these reasons, the possibility of using new chelating, modifying agents and/or new surfactants with a strong affinity for minerals themselves has been investigated (Usoni et al., 1971; Nagaraj and Sumasundaran, 1981; Nagaraj, 1982; Marabini et al., 1988, 1989). Nevertheless, selection of a collector (surfactant) with the right structure can greatly enhance selective flotation in any number of practical flotation separations (Smith, 1987, 1989). For example, the structural variations of sulfonate surfactants in terms of chain length and branching and incorporation of phenyl, ethoxyl, and multiple functional groups are found to produce specific effects during their adsorption on alumina (Sumasundaran et al., 1984).

As part of a study of the relationship between the chemical structure of well-purified surfactants and their surface and thermodynamic properties, a number of cationic and anionic surfactants with different hydrocarbon chain are synthesized and their properties are investigated (Omar and Abdel-Khalek, 1998; Abdel-Khalek et al., 1999). The effect of the presence of different numbers of ethyl groups attached to the carbon chain on their surface properties, and in turn on their efficiency as collectors, has been studied (Omar and Abdel-Khalek, 1998). Also, the relationship between surface properties of some sulfonate surfactants of different carbon chain length and their performance as collectors in the flotation process has been investigated (Abdel-Khalek et al., 1999).

This paper aims to study the surface and thermodynamic properties of some other surfactants to find the relationship between their chemical structure and their performance as collectors in the flotation process. The surfactants are monoisomeric alkylbenzenesulfonates in which a benzene ring is attached to the carbon chain at different positions. Application of these surfactants in the flotation of a petroleum coke sample is tested.

Materials and Methods

Four anionic surfactants of sodium salt of monoisomeric alkylbenzenesulfonate are prepared according to recommended procedures (El-Mergawy, 1988; Omar, 1994). The prepared surfactants have the same molecular weight (i.e., the same alkyl structure of phenyldodecylsulfonate), but they contain a phenyl group attached at a different position along the carbon chain. They are 3-phenyldodecylsulfonate (3ϕC12), 4-phenyldodecylsulfonate (4ϕC12), 5-phenyldodecylsulfonate (5ϕC12), and 6-phenyldodecylsulfonate (6ϕC12). The chemical structure and purity of these prepared surfactants are verified by different techniques such as IR, NMR, and mass spectra (Omar, 1994).

A technological sample of Belayim petroleum coke, Egypt, is kindly supplied by the Suez Refining Co. Yard sampling of thoroughly mixed ore is carried out by coning and quartering methods. Ore pulverization is carried out

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Table 1. Effect of Position of the Phenyl Group on Surface Properties of the Prepared Surfactants at 25 °C

compound	cmc × 10^{-2}/(mol·L^{-1})	γ_{cmc}/(mN·m^{-1})	Γ_{max} × 10^6/(mol·m^{-2})	$A°_{min}$ × 10^2/(nm^2·mol^{-1})	Π_{cmc}/(mN.m^{-1})	pC_{20}
3ϕC12	136.5	27.58	3.338	49.733	44.42	5.146
4ϕC12	122.8	27.84	3.435	48.338	44.14	5.143
5ϕC12	114.6	28.30	3.528	47.055	43.70	5.117
6ϕC12	159.2	27.18	3.531	47.072	44.82	5.063
C12[a]	124		2.93	56.7	33.0	2.36

[a] Dahanayake et al. (1986).

using “Wedag” jaw crusher in closed circuit with a 25 mm screen. This is followed by secondary crushing in a “Denver” roll crusher to 100% below 0.5 mm. The product is then dry screened on 0.063 mm sieve, the undersize of which is rejected. The fraction $-0.5 + 0.063$ mm (representing about 97.5% of the sample) is then subdivided, after thoroughly mixing, to 0.5 kg batches and is stored in a closed container as a flotation feed. The chemical analysis of the flotation feed shows that it contains about 87.2% fixed carbon and 14.5% volatile matter. The ash content is 1.38% while the total sulfur is 4.2%. The Ni, V, Si, Ca, and Na are the major metallic inclusions of the coke, whereas Fe, Co, Mo, and Cu are present in minor concentrations (Abdel-Khalek et al., 1997).

The flotation tests are carried out using a laboratory “Denver D12” flotation cell, with a 5 L. container, at 10% solids and at room temperature (~25 °C). The sample is conditioned at 1500 rpm with sodium silicate, as an ash depressant, for 5 min. This is followed by another 5 min for conditioning with surfactant before aeration. The pH is maintained constant at pH 7 during flotation tests using H_2SO_4 or NaOH (BDH products). The flotation speed is kept constant at 1200 rpm. The froth (concentrate) and the tail fractions are collected, dried, weighed, and analyzed.

The surface tension measurements are carried out using a “Dagnon Abribot” tensiometer, and the points of intersection are determined using a linear regression analysis technique (Drapper and Smith, 1968). The uncertainties in measurements of surface tension, temperature, and concentration of surfactants are 0.10, 0.10, and 0.001% respectively. From surface tension−concentration isotherms, the other surface and thermodynamic properties are calculated (Rosen et al., 1982; Abdel-Khalek et al., 1997). Maximum surface excess concentration (Γ_{max}), in mol cm^{-2}, and area per molecule (A_{min}), in nm^2, at the aqueous−air interface are calculated from the relationships

$$\Gamma_{max} = 1/2.303RT\,(-\mathrm{d}\gamma/\mathrm{d}\log C)_T \tag{1}$$

$$A_{min} = 10^{14}/N\Gamma \tag{2}$$

where $(-\mathrm{d}\gamma/\mathrm{d}\log C)_T$ is the slope of $\gamma - \log C$ plotted at constant temperature, T, $R = 8.314$ J mol^{-1} K^{-1}, and N is Avogadro's number.

The surface pressure at cmc (Π_{cmc}) is calculated from the relationship

$$\Pi_{cmc} = \gamma_0 - \gamma_{cmc} \tag{3}$$

where γ_0 is the surface tension of water at zero surfactant concentration and γ_{cmc} the surface tension of solution at cmc.

Results and Discussion

Surface Properties of the Prepared Surfactants. The prepared surfactants have the same molecular weight (sodium dodecylbenzenesulfonate), but they differ in the position of phenyl group along the hydrocarbon chain, which is changed from carbon atom number three to number six. The completely soluble sodium dodecylbenzenesulfonate isomers have been employed because the solubility of these compounds is an essential property in the flotation processes.

Table 1 shows the surface properties of these surfactants at 25 °C. It is obvious from these results that changing the position of phenyl group along the linear alkyl chain from carbon number 3 to carbon number 6 has little effect on the values of surface excess concentration (Γ_{max}), effectiveness (Π_{cmc}), and efficiency (pC_{20}), to reduce surface tension of water. For example, 3ϕC12, 4ϕC12, 5ϕC12, and 6ϕC12 have Γ_{max} = (3.338, 3.435, 3.528, and 3.531) 10^6 mol·m^{-2}, respectively, whereas the values of Π_{cmc} (43.7−44.82 mN·m^{-1}) and pC_{20} (5.146−5.063) are nearly constant. The values of Γ_{max} are slightly higher than 2.93 mol·m^{-2} × 10^6 for ($C_{12}H_{25}SO_3Na$) sodiumdodecylsulfonate (Dahanayka et al., 1986). The area per molecule (A_{min}) of these surfactants is slightly decreased from (49.733 to 47.072) 10^2 nm^2·mol^{-1} with changing position of phenyl group from carbon number three to the central carbon (C6). These values are smaller than 56.6 nm^2·mol^{-1} for ($C_{12}H_{25}SO_3Na$) sodiumdodecylsulfonate (Dahanayka et al., 1986). Meanwhile, the values of cmc of these prepared surfactants are decreased from (136.5 to 114.6) mol·L $^{-1}$ with changing the position of phenyl group from carbon number 3 to carbon number 5 (C5). However, moving the phenyl group to a more central position along the carbon chain, at carbon atom number 6, causes an increase in the value of cmc. Similar results have been mentioned by other authors who attributed such behavior to steric factors. In this case the carbon atoms on the shorter section of the chain have about half their usual effect on the cmc (Rosen, 1989). It seems from these results that addition of a phenyl group along the carbon chain of sodiumdodecylsulfonate might improve its surface properties by increasing its surface excess concentration (Γ_{max}) and decreasing its area per molecule (A_{min}). This may be positively reflected on the efficiency of these surfactants, as collectors, in the flotation process.

Effect of Temperature on Surface Properties of the Prepared Surfactants. Previous studies on both cationic and anionic surfactants showed that cmc values of all known ionic surfactants increase with increasing temperature (Abdel-Khalek et al. 1997; Omar and Abdel-Khalek 1998). From the data in Table 2, the cmc values are significantly increased with increasing temperature from 25 to 55 °C for the surfactant (3ϕC12) containing a phenyl group at carbon atom number 3. This tendency of increasing cmc with increasing temperature is gradually decreased by moving the phenyl group toward the central carbon atom (6ϕC12). Consequently, no drastic changes are expected in the behavior of the latter surfactant (6ϕC12) as a collector, within this range of temperature, i.e., during the flotation process. This may indicate that the surfactant with a phenyl group at its central carbon atom (6ϕC12) is more stable to the change in temperature, and in turn it may give better results as a collector in the flotation process than the other surfactants.

Table 2. Effect of Temperature on Surface Properties of Prepared Surfactants

		sulfonates			
	t/°C	3 ϕ C12	4 ϕ C12	5 ϕ C12	6 ϕ C12
cmc × 10^{-2}/mol·L^{-1}	25	136.5	122.8	114.6	159.2
	35	140.8	124.3	117.2	160.3
	45	155.1	129.6	120.1	157.8
	55	162.6	132.2	121.4	158.7
γ_{cmc}/mN·m^{-1}	25	27.58	27.84	28.30	27.18
	35	27.01	27.31	27.23	27.04
	45	26.53	26.65	26.38	26.75
	55	25.82	25.80	26.07	25.73
Γ_{max} × 10^6/mol·m^{-2}	25	3.338	3.435	3.528	3.531
	35	2.953	2.863	2.601	3.002
	45	1.491	2.787	2.245	2.608
	55	1.769	2.610	2.004	2.727
A° × 10^2/nm^2·mol^{-1}	25	49 .733	48.338	47.055	47.072
	35	49.219	57.986	63.827	55.343
	45	92.695	63.614	73.949	63.662
	55	93.887	69.969	82.832	60.874
Π_{cmc}/mN·m^{-1}	25	44.42	44.14	43.70	44.82
	35	39.99	39.69	39.77	39.97
	45	38.47	38.36	38.62	38.26
	55	37.18	37.21	36.93	37.27
pC_{20}	25	5.146	5.143	5.117	5.063
	35	5.065	5.146	5.239	4.985
	45	5.502	5.188	5.281	4.986
	55	5.423	5.143	5.339	4.989

Rosen (1989) has mentioned that factors that cause an increase in solubility with temperature (reflected in most cases by an increase in cmc) also work to decrease the effectiveness of adsorption, Γ_{max}, of the ionic surfactants. For the employed sulfonates, Γ_{max} decreases with increasing temperature. For example, 3ϕC12 sulfonate has Γ_{max} values of (3.338, 2.953, 1.491, and 1.769) 10^6 mol·m^{-2} at (25, 35, 45, and 55) °C, respectively. For 6ϕC12 the values of Γ_{max} decreased from (3.531 to 2.727) 10^6 mol·m^{-2} with raising the temperature from (25 to 55) °C. This reduction in the values of effectiveness of adsorption, Γ_{max}, is less significant than that noticed before with increasing length of the linear carbon chain from C12 to C14 for alkylbenzenesulfonate surfactants (Abdel-Khalek et al., 1999).

The results in Table 2 also illustrate that the values of A_{min} increase with increasing temperature especially for the surfactants 3ϕC12 and 4ϕC12, presumably owing to the increased thermal motion that consequently cause a decrease in Γ_{max} values. For example A_{min} is significantly increased by about 44.2 × 10^2 nm·mol^{-1}, while Γ_{max} is decreased from 3.338 to 1.769 mol·m^2, with increasing temperature from (25 to 55) °C.

The change in efficiency (Π_{cmc}) and effectiveness (pC_{20}) of surfactants, to reduce surface tension of water, with changing temperature from 25 to 55 °C is also presented in Table 2. These results reveal that temperature has a minor effect on effectiveness of surfactants. For instance, the 6ϕC12 sulfonate surfactant has effectiveness values of 44.82, 39.97, 38.26, and 37.27 at (25, 35, 45, and 55) °C, respectively. This surfactant has efficiency values between 4.989 and 5.063 at the same temperatures. Many investigators (Rosen, 1989; El-Mergawy, 1988) have reached a general conclusion that increasing effectiveness tends to decrease efficiency and vice versa.

Some Thermodynamic Parameters of the Prepared Surfactants. The standard Gibbs energy change upon micellization is calculated, using the cmc values, from the following equation:

$$\Delta G°_{mic} = RT \ln \mathrm{cmc} \qquad (4)$$

Table 3. Some Thermodynamic Parameters of Prepared Surfactants

		sulfonates			
ΔG/(kJ·mol^{-1})	t/°C	3ϕC12	4ϕC12	5ϕC12	6ϕC12
ΔG_{mic}	25	−32.02	−32.28	−32.05	−32.63
	35	−33.40	−33.77	−33.59	−32.84
	45	−33.83	−35.17	−34.50	−33.79
	55	−35.32	−37.09	−36.06	−35.20
ΔG_{ad}	25	−45.32	−45.13	−44.13	−44.33
	35	−46.96	−48.83	−48.83	−46.17
	45	−55.30	−51.70	−51.70	−48.46
	55	−56.33	−50.44	−54.48	−48.86

The Gibbs energy change upon adsorption, ΔG_{ad}, which depends on the mole fraction of Gibbs energy change upon adsorption, is calculated from ΔG_{mic}, Π_{cmc}, and A_{max} values using the following equation (Rosen et al., 1982; Abdel-Khalek et al., 1997):

$$\Delta G°_{ad} = \Delta G°_{mic} - \Pi_{cmc} A_{cmc} \qquad (5)$$

Table 3 depicts the results obtained for ΔG_{mic} and ΔG_{ad} values. It is clear that ΔG_{mic} appears to become more negative with increasing temperature from (25 to 55) °C regardless the position of the phenyl group along the carbon chain of surfactants. This may be attributed to the fact that the amount of water structured by the hydrophobic chain and the amount of water bound by the sulfonate headgroup in the nonmicellar species decrease with increasing temperature (Rosen, 1989). Meanwhile, the variation in ΔG_{mic} values with changing position of the phenyl group along the carbon chain of surfactants is not significant, indicating that the amount of water structured by the tail group, and in turn the surfactants hydrophobicity, is not changed.

The data in Table 3 reveal also that ΔG_{ad} values are negative. These negative ΔG_{ad} values indicate that adsorption of these surfactants at the aqueous solution−air and the aqueous solution−solid interfaces is spontaneous (Rosen, 1989). In the flotation process, properties that deal with the adsorption of collector molecules at the aqueous solution−solid interface are highly considered (Abdel-Khalek et al., 1999).

Flotation Efficiency of the Prepared Surfactants. Application of the prepared surfactants as collectors in the flotation process has been investigated on a petroleum coke sample to minimize its impurities (1.38% ash content). The flotation tests are performed at the predetermined optimum conditions (Abdel-Khalek et al., 1997). The tests are performed at two different levels of 20 and 100 g·ton^{-1} of each surfactant. The results are shown in Figure 1.

It is evident that changing the position of phenyl group toward the central carbon atom of the linear hydrocarbon chain substantially increases the flotation yield of the coke concentrate while its ash content decreases. This means the both the selectivity and efficiency of the flotation process are improved by changing the position of the phenyl group toward the central carbon atom along the linear hydrocarbon chain. Addition of 20 g·ton^{-1} of 6ϕC12 as a collector in flotation of a petroleum coke sample gives a concentrate of a minimum ash content (∼0.1%) with a percent removal of 89% of its ash. Increasing the dosage of 6ϕC12 surfactant from 20 to 100 g·ton^{-1} increases the flotation yield from ∼ 85 to 95% without affecting greatly the ash content, Figure 1.

In the meantime, the marginal reduction in ΔG_{ad}, the standard Gibbs energy of adsorption, by moving the phenyl group position to carbon number 6 showing relatively better surface activity, should be taken into consideration in

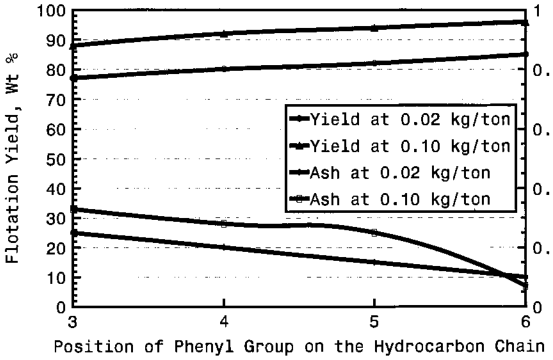

Figure 1. Effect of changing the position of the phenyl group along the alkyl chain length: *X*-axis, position of phenyl group on the carbon chain; *Y*1-axis, flotation yield %; *Y*2-axis, ash content %, O, yield % using 20 g·ton^{-1} ▲, yield % using 100 g·ton^{-1}; ◆, ash % using 20 g·ton^{-1}; □, using 20 g·ton^{-1}.

comparing the efficiency of these collectors. The quality of concentrates, in fact, will depend on the chemical constitution of the alkylbenzenesulfonate, which has a direct impact on its physical and surface properties.

It seems that the good selectivity obtained in the flotation process while using such prepared surfactants, especially 6ϕC12, may encourage their application as collectors in the flotation of other minerals.

Conclusions

For sodium dodecylbenzenesulfonate surfactants isomers, changing the position of their phenyl group along the linear alkyl chain from carbon number 3 to carbon number 6 has a little effect on the values of surface excess concentration (Γ_{max}), effectiveness (Π_{cmc}), and efficiency (pC_{20}), to reduce surface tension of water. The value of Γ_{max} is decreased with increasing temperature. The values of Γ_{max} are slightly higher than that of sodium dodecylsulfonate ($C_{12}H_{25}SO_3Na$). The area per molecule (A_{min}) of these surfactants is slightly decreased with changing the position of phenyl group from carbon number 3 to the central carbon (C6). These values are smaller than that of sodium dodecylsulfonate ($C_{12}H_{25}SO_3Na$). Meanwhile, the values of cmc are decreased with changing position of phenyl group from carbon number 3 to carbon number 5 (C5). Addition of a phenyl group to sodium dodecylsulfonate might improve its surface properties by increasing its surface excess concentration (Γ_{max}) and decreasing its area per molecule (A_{min}).

For sodium alkylbenzenesulfonate surfactants, ΔG_{mic} appears to become more negative with increasing temperature regardless position of phenyl group. Meanwhile, the negative ΔG_{ad} values of these surfactants indicate that the adsorption of such collectors, at the interfaces, is spontaneous.

The dosage of such surfactants required for flotation of a petroleum coke sample is small (~20 g·ton^{-1}). Using such dosage of the surfactant 6ϕC12 gives the best selectivity where a concentrate of about 0.10% ash content, with a yield of about 85%, is obtained from a feed containing 1.38% ash content. Increasing the dosage of 6ϕC12 from (20 to 100) g·ton^{-1} can increase the flotation yield to ~ 95% without affecting greatly the ash content.

The prepared surfactants of sodium alkylbenzenesulfonate showed a high selectivity in the flotation of petroleum coke. This higher selectivity in the flotation of petroleum coke while using such prepared surfactants, specially 6ϕC12, will encourage their application as collectors in flotation of other minerals.

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