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## **FLOTATION OF EGYPTIAN PETROLEUM COKE USING 4-PHENYL DODECYL BENZENE**

This paper aims to study monoisomeric 4-phenyl dodecyl benzene, a synthetic anionic surfactant as a collector in the flotation of an Egyptian petroleum coke to minimize its ash content as well as its heavy metals to be suitable for manufacture of electrodes. The structure of the surfactant was identified by IR, NMR and mass spectra. The surface properties and the thermodynamic characters of this surfactant were also studied. With raising the temperature from 25 to 55 °C the value of critical micelle concentration (cmc) increases slightly while the surface excess concentration at the surface saturation ( $\Gamma_{\max}$ ) shows a small reduction. This is accompanied by an increase in area occupied per molecule at the interface at surface saturation ( $A_{\max}$ ). The results reveal also that the values of the free energy change upon adsorption ( $\Delta G_{\text{ads}}$ ) are all negative at different temperatures. This indicates that the adsorption of this collector at the aqueous solution/air and the aqueous solution/solid interface is spontaneous. The flotation tests were carried out using a laboratory "Denver" flotation cell at 10 wt.% solids using sodium silicate as a depressant. Different parameters affecting flotation of coke such as the dosage of the surfactant, its mode of addition, dosage of depressant and pH, were studied. The results showed that at the optimum conditions for flotation a coke concentrate assaying only 0.19% ash in comparison with 1.38% in the feed samples is obtained. At the same time, the tail fractions had high ash contents (12.5%) and it was very contaminated with heavy metals. The flotation results are in good agreement with that of the surface properties and the thermodynamic characters of the surfactant.

### **INTRODUCTION**

Petroleum coke is obtained by thermal cracking of heavy petroleum oil. It contains about 85–92% of carbon, in addition to sulfur, hydrogen, nitrogen and some metals, depending on the quality of the feed stock used (Ahmed and Kamil 1982). Such coke is usually used as a fuel. It is also used in many industries. For example, it can be applied in blast furnaces, foundries, chemicals production and electrodes manufacture (The Standard Research Institute, 1971; Foster 1980). In the latter case the petroleum coke should be of high purity (Ahmed and Kamil 1982).

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Upgrading the petroleum coke by the physical methods of separation have been successively applied to remove its associated ash and pyrite (Pai and Nayak 1978; Viswanathan and Nayak 1980). Sredniawa (1991) reviewed different methods employed for this purpose including jigging, classification and flotation. These methods were found to be more economical than flue gas desulphurisation, sorbent injection in fluidized and combustion or chemical desulphurisation (Sredniawa, 1991).

Application of flotation process using a collector with right structure can greatly enhance selectivity. In fact, the performance of surfactants in most interfacial processes depends mainly on their concentration and orientation at the interface. The modification of the lyophobic and lyophilic groups in the structure of the surfactant may become necessary to maintain surface activity at a suitable level (Smith 1989). In this respect, the present paper aims to study the amenability of upgrading an Egyptian petroleum coke sample to be suitable for application in electrodes manufacture that are used in aluminum industry. The paper also aims to investigate the efficiency of 4-phenyl dodecyl benzene sulphonate as a collector for the petroleum coke sample. The main operating parameters affecting the flotation process were investigated. The surface and thermodynamic properties of the collector were also studied.

## EXPERIMENTAL

### Surfactant preparation

A sodium salt of 4-phenyl dodecyl benzene sulphonate was prepared in the laboratory according to the synthesis scheme and methods which had been reported by other authors (El-Mergawy 1988; Omar 1994). The chemical structure and the purity of the surfactant were verified by IR, NMR, and mass spectroscopy (Omar 1994).

### Flotation feed sample

A technological sample of the Belayim petroleum coke was kindly supplied by the Suez Refining Co. Yard sampling of the thoroughly mixed ore was carried out by coning and quartering methods. Ore pulverization was carried out using a “Wedag” jaw crusher in a closed circuit with a 25 mm screen. This was followed by secondary crushing in a “Denver” roll crusher to 100% below 0.5 mm. The product was then dry screened on 0.063 mm sieve the undersize of which was rejected. The  $-0.5 \pm 0.063$  mm fraction (representing about 97.5 wt% of the sample) was then subdivided, after thoroughly mixing, to 0.5 kg batches and stored in a closed container as a flotation feed.

### Methods

The surface tension measurements were carried out using a “Dagnon Abribat” tensiometer and the points of intersection were determined using the linear regression

analysis technique (Drapper and Smith 1968). From the surface tension–concentration isotherm, other surface properties and thermodynamic parameters of the surfactant were calculated. The amount of material adsorbed per unit area at the interface ( $\Gamma N$ ) is calculated indirectly from the surface tension measurements as a function of equilibrium concentration of surfactant in the liquid phase by applying the Gibbs absorption equation (Leja 1982; Jaycock and Parfitt 1981; Gendy et al. 1994):

$$d\gamma = -RT\Gamma d \ln C \quad \text{or} \quad \Gamma = -\frac{C}{RT} \frac{d\gamma}{dc} \quad (1)$$

where  $d\gamma$  is the change in surface tension of the liquid,  $\Gamma$  is the surface excess concentration, which can be considered to be equal to the actual surface concentration without significant error.  $C$  is the molar concentration of the surfactant.  $R$  and  $T$  are the gas constant and absolute temperature, respectively. The values of  $d\gamma/dC$  were obtained by fitting the  $\gamma$  versus  $C$  data to the polynomial:

$$\gamma = a_3 C^3 + a_2 C^2 + a_1 C + a_0$$

using the least-squares method, where  $a_3$ ,  $a_2$ ,  $a_1$  and  $a_0$  are constants, and the differentiating the resulting polynomial (Gendy et al. 1994).

From the surface excess concentration ( $\Gamma$ ) the area per molecule ( $A$ ) is calculated from the relationship :

$$A = \frac{10^{18}}{\Gamma N} \quad (2)$$

where  $N$  is Avogadro's number and  $\Gamma$  is in mole/m<sup>2</sup> (Gendy et al. 1994).

The surface pressure ( $\Pi$ ) is calculated from the relationship:

$$\Pi = \gamma_0 - \gamma \quad (3)$$

where  $\gamma_0$  is the surface tension of water for zero surfactant concentration at a specified condition, and  $\gamma$  is the surface tension of aqueous surfactant solution (Gendy et al. 1994). The surface molecular area ( $A$ ) at the interface provides information on the degree of packing and the orientation of the absorbed surfactant molecule, while surface pressure is a measure of the surface tension reduction attained. The effectiveness ( $\Pi_{cmc}$ ) of the collector is the amount of surface reduction at cmc. The efficiency ( $pC_{20}$ ) of the collector, is a useful measure of a surfactant's efficiency number, is  $pC_{20} = \log 1/C_{\Pi=20}$  where  $C_{\Pi=20}$  is the bulk molar concentration required to reduce the surface tension by 20 mN/m (Rosen 1989; El-Kholy 1993).

Using the cmc values, the standard free energy change upon micelle formation,  $\Delta G_{mic}$ , can be calculated from the equation (Leja 1982; Jaycock and Parfitt 1981):

$$\Delta G_{mic} = RT \ln cmc \quad (4)$$

The standard free energy change upon adsorption,  $\Delta G_{ad}$ , is calculated from  $\Delta G_{mic}$  and  $I_{cmc}$  values using the equation (Leja 1982; Jaycock and Parfitt 1981):

$$\Delta G_{ad} = \Delta G_{mic} - I_{cmc} \quad (5)$$

The flotation tests were carried out using a laboratory “Denver D12” flotation cell with a 5 liter container, at 10 wt.% solids and at room temperature (~25 °C). The sample was conditioned at 1500 rpm with sodium silicate as an ash depressant for 5 minutes followed by another 5 minutes for conditioning with the surfactant (4-phenyl dodecyl benzene) as a collector before aeration. The pH was maintained constant during the test (unless otherwise mentioned) at pH = 7 using H<sub>2</sub>SO<sub>4</sub> or NaOH. The flotation speed was kept constant at 1200 rpm. The froth (concentrate) and the tail fractions were collected, dried, weighed, and analyzed.

Granulometric analysis of the secondary crushed sample was carried out by dry screening using a “Ro-tap” shaker and a set of British Standard sieves.

## RESULTS AND DISCUSSION

### Characterization of petroleum coke sample

Table 1 shows the chemical analysis of the Belayim petroleum coke sample. It is clear that the sample contains about 87.2% of fixed carbon and 14.5% volatile matter. The ash content is 1.38% while the total sulphur is 4.2%. 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. Table 2 depicts the size distribution of the coke sample. It is clear that the sample has more than 52% by weight of grain sizes above 0.297 mm while the fraction below 0.063 mm represents only 2.50% by weight. The ash content progressively increases with decreasing size. The lowest fraction (-0.063 mm) contains the highest ash content (2.8%) and consequently it is rejected. The size fraction -0.5+0.063 mm is taken as a flotation feed.

Table 1. Chemical analysis of the petroleum coke sample

Constituents	%	Metal content	ppm
Ash	1.38	Mo	2
Volatile matter	14.50	Cu	11
Nitrogen	2.00	Ti	14
Fixed Carbon	87.20	Ca	110
Sulphur	4.20	Cr	9
		Na	130
Metal Content	ppm	Si	410
Ni	380	Co	1
V	500	Fe	83

Table 2. Size analysis of Balayim petroleum coke sample

Size, mm	wt, %	Cum. wt.%, passed	Ash, %	Distribution Ash, %
-0.5 + 0.297	52.04	100	1.15	44.87
-0.297 + 0.21	13.48	47.96	1.20	12.13
-0.21 + 0.105	18.81	34.48	1.60	22.56
-0.105 + 0.063	13.17	15.69	1.70	16.79
-0.063	2.50	2.50	2.80	3.65

### Surface and thermodynamic properties of the prepared surfactant

Table 3 shows the surface properties of the prepared surfactant (4-phenyl dodecyl benzene sulphonate). It is clear that the value of critical micelle concentration (cmc) increases slightly with raising the temperature from 25 to 55 °C. Consequently, no drastic changes are expected in the behaviour of this surfactant within this range of temperature, i.e. during the flotation process. Also, the surface excess concentration at the surface saturation ( $\Gamma_{\max}$ ) shows a slight reduction (from 3.435 to 2.610 mole/m<sup>2</sup>·10<sup>6</sup>) with raising the temperature from 25 to 55 °C. The surface excess concentration is a useful measure of effectiveness of adsorption of collector at the liquid/air, liquid/liquid and liquid/solid interface since it is the maximum value which system can attain (Rosen 1989; Omar 1994).

For a wide variety of surfactants, the area occupied per molecule at the interface at surface saturation ( $A_{\max}$ ) is inversely proportional to the effectiveness of adsorption ( $\Gamma_{\max}$ ) (Rosen 1989). This is confirmed in Table 3 where the increase in  $A_{\max}$  is accompanied by a decrease in  $\Gamma_{\max}$  and vice versa. It is obvious that an increase in temperature results in an increase in  $A_{\max}$  presumably due to the increased thermal motion which consequently causes a decrease in  $\Gamma_{\max}$  values (Rosen 1989; Omar 1994; El-Mergawy 1988).

Meanwhile, the calculated efficiency (pC<sub>20</sub>) and effectiveness ( $II_{\text{cmc}}$ ) values (Table 3) indicated that no significant changes have been observed by changing temperature from 25 to 55 °C since temperature has a rather minor effect on these quantities (Rosen 1989; Omar 1994). Many investigators have reached a general conclusion that increasing effectiveness ( $II_{\text{cmc}}$ ) tends to decrease efficiency and vice versa (Rosen 1989; Omar 1994). The effectiveness of surface tension reduction ( $II_{\text{cmc}}$ ) is usually considered to depend on the cohesiveness of the collector molecule (low cohesiveness means high effectiveness) and the magnitude of cmc (high cmc promotes effectiveness), since a surface tension reduction requires the presence of dispersed collector molecules at the surface. A comparison of the effectiveness values with those reported in literature shows that the prepared surfactant has a fairly good effectiveness compared with most surfactants of similar efficiency (Omar 1994)

Table 3. Surface and thermodynamic properties of the surfactant

Temperature °C	CMC μmole/l	$\gamma_{cmc}$ mN/m	$\Gamma_{max} \cdot 10^6$ mole/m <sup>2</sup>	$A_{max} \cdot 10^2$ nm <sup>2</sup> /mole	$\Pi_{cmc}$ mN/m	pC <sub>20</sub>	$\Delta G_{mic}$ KJ/mole	$\Delta G_{ads}$ KJ/mole
25	122.8	27.84	3.435	48.338	44.14	5.143	-32.28	-45.13
35	124.3	27.31	2.863	57.986	39.69	5.146	-33.77	-48.83
45	129.6	26.65	2.787	63.614	38.36	5.188	-35.17	-51.70
55	132.2	25.80	2.610	69.969	37.21	5.143	-37.09	-50.44

The data in Table 3 show the values of the standard free energy change upon micelle formation ( $\Delta G_{mic}$ ) at different temperatures. It is clear that  $\Delta G_{mic}$  appears to become more negative with increasing temperature from 25 to 55 °C. 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 sulphonate head group in the non-micellar species decreases with increasing temperature (Rosen 1989). On the other hand, the results in Table 3 reveal also that the values of the free energy change upon adsorption ( $\Delta G_{ads}$ ) are all negative at different temperatures. This indicates that the adsorption of this collector at the aqueous solution/air and the aqueous solution/solid interface is spontaneous (Rosen 1989). In flotation process, properties that depend on the adsorption of collector molecules at aqueous solution/solid interface are very important.

### Optimization of some parameters affecting coke flotation

**Effect of dosage of the collector.** Figure 1 shows the effect of changing the dosage of the collector on the yield and ash contents of the coke concentrate. The tests were performed at constant concentration of sodium silicate (0.30 kg/ton). The results indicate that the addition of a small quantity of the surfactant (~ 20 g/t) was enough to reduce the ash content from 1.38% in the feed to 0.35% only in the concentrate, with a yield of about 90 wt%. Further addition of the surfactant increases the yield of concentrate to about 95 wt% at the expense of its quality. The ash content increases gradually from 0.35% to 1.25% with increasing the dosage up to 0.20 kg/t. Both the ash content and the yield are not changed at the higher dosage (0.30 kg/ton). It is clear that 20 g/t is the optimum dosage required to remove about 77.2% of the total ash.

It is well known that coal and petroleum coke has a native hydrophobicity. Flotation of the petroleum coke sample without addition of the surfactant gives a product of 1.30% ash with a yield of 78 wt%. However, a concentrate of better grade was obtained in presence of 20 g/t of such surfactant and 0.30 kg/t of sodium silicate. This confirms the good surface properties of the 4-phenyl dodecyl benzene sulphonate shown before in Table 3.

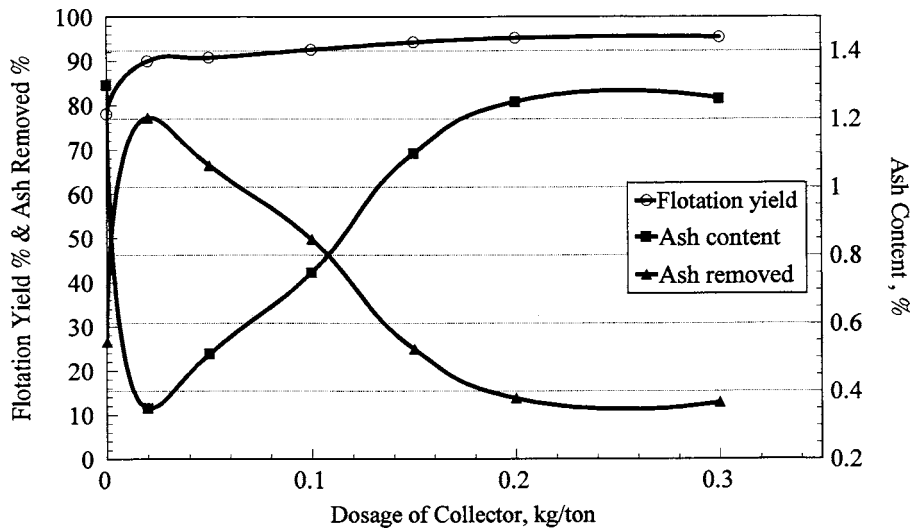


Fig. 1. Effect of dosage of collector on the flotation of petroleum coke

**Effect of dosage of sodium silicate.** The effect of using different amounts of sodium silicate as a depressant for the ash containing minerals was studied at pH 7 using 20 g/t of the collector. Figure 2 shows the results of these tests. The results show that the ash content gradually decreases, at the expense of the yield, with increasing the dosage of sodium silicate. At a dosage of 1 kg/t of sodium silicate a concentrate with the minimum ash content (0.19%) and yield of about 80 wt% is obtained. This dosage is considered as the optimum where about 89% of the total ash is removed.

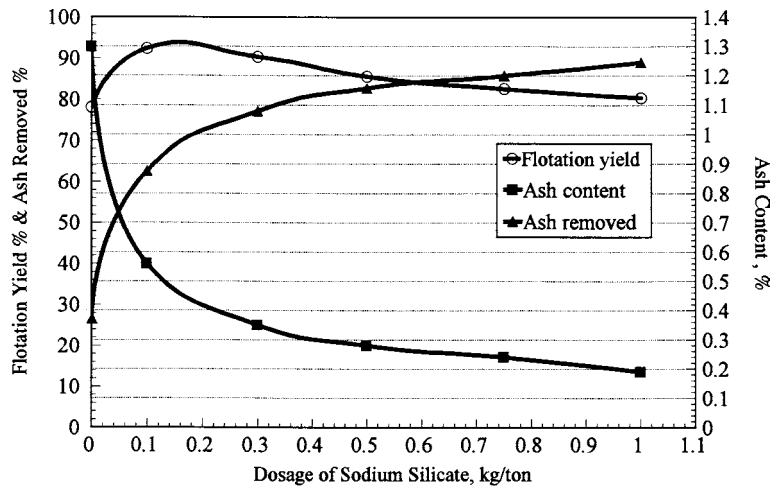


Fig. 2. Effect of dosage of sodium silicate on the flotation of petroleum coke

The structure of sodium silicate with  $\text{SiO}_2/\text{Na}_2\text{O}$  ratios ranging from 1.61:1 to 7.74:1 was studied by Lendz (1964). Sodium silicate was classified by the contents of  $\text{SiO}_4$ ,  $\text{Si}_2\text{O}_7$ ,  $\text{Si}_3\text{O}_{10}(\text{SiO}_3)_4$  and polysilicate structures. Sodium silicate solutions contain monomeric silicate ions, polysilicate ions and polymeric silicate micelles (Falcone 1981). The main dispersive action of sodium silicate is produced by electrostatic repulsion. Dho and Iwasaki (1990) had attributed the beneficial role of sodium silicate to: 1 – the removal of impurity minerals from the surface of the floating mineral by dispersing them, 2 – dry and more persistent froth, 3 – higher specific flotation rates and increased selectivity of separation. Thus, with increasing the dosage of sodium silicate the particles of the ash forming minerals, and also the locked particles, will become more hydrophilic due to the increase of the negative charges of their surfaces leading to their depression in the pulp. This gives the concentrate with lower ash content and lower yield, Fig.2.

**Effect of changing pH.** It is known that flotation of coal can be difficult in certain ranges of pH. Both recovery and quality of the produced coals can be affected by the pH. The optimum pH for coal flotation is usually near  $\text{pH} = 7$  and recovery frequently drops off at lower or higher values. The recovery is highest when the pH is between 6

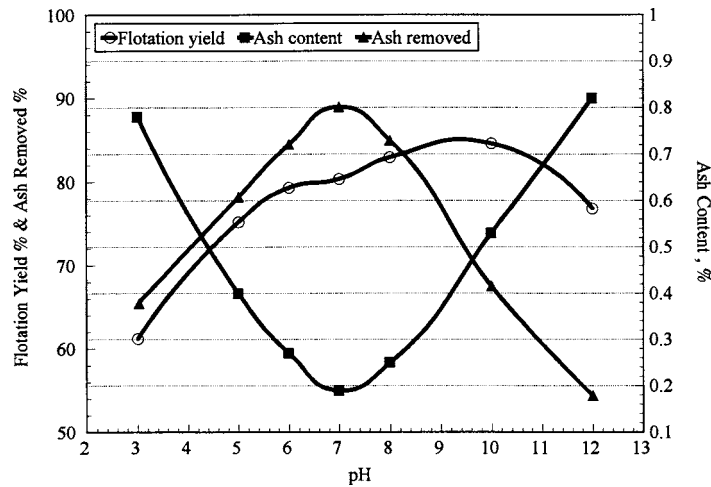


Fig. 3. Effect of changing pH

and 8. High pH inhibits the flotation of pyrite. The ash content in the concentrates, however, increases as the pH value is raised (Aplan 1976). Such a flotation behaviour has also been confirmed in the flotation of petroleum coke as shown in Figure 3. The tests are carried out using 20 g/t of surfactant and 1 kg/t of sodium silicate. It is clear that the pH of the medium plays a major role in determining the selectivity of the flotation of petroleum coke. Both the strongly acidic and alkaline pH's deteriorate the



selectivity of the flotation process where the highest ash content is obtained. This could be related to the presence of the petroleum coke particles at such conditions in a highly charged form thereby making their surfaces more hydrophilic and in turn deteriorates their floatability. The selectivity of the flotation process is best when is carried out in a neutral medium (pH = 7) probably due to the absence of potential determining ions ( $H^+$  and  $OH^-$ ) which make the surface of coke hydrophilic. At such conditions a concentrate with a minimum ash content (0.19%), and a yield of about 80 wt% is obtained. It is clear that at pH 7 the majority of the associated ash (~89%) is removed.

**Effect of multi-stage addition of the surfactant.** The mode of addition of the collector sometimes plays either a positive or negative role on the results. Table 4 shows the effect of addition of the optimum dosage (20 g/t) of the collector stepwisely in equal portions. The addition of the dosage in two steps rather than in one results in an increase of the yield of the concentrate from 80% to 89.7% without affecting its grade (ash content 0.18%).

Table 4. Effect of multi-stage addition of the surfactant

No. of additions	Concentrate wt %	Tail wt %	Concentrate ash, %	Tail ash, %
1	80.0	20.0	0.19	6.14
2	89.7	10.3	0.18	10.20
4	93.1	6.9	0.19	12.40

Increasing the number of stepwise addition of the surfactant to four, improves further the efficiency of the flotation process where a final concentrate assaying 0.19% ash with a yield of about 93.1% by weight is obtained (Table 4). Similar results have been obtained in the case of flotation of the Polish coals (Abdel-Khalek 1989). Such improvement in the efficiency of coke flotation while adding the surfactant stepwisely could be related to the decreasing of flotation of the ash forming minerals by the entrainment mechanism with the hydrophobic floated coke particles. This is indicated by the successive reduction in the weight percent of tailing (Table 4).

#### Characterization of the final concentrate and its tailing

Table 5 shows a complete chemical analysis of the final concentrate and its tailing obtained at the above mentioned optimum conditions. It is clear that the metallic impurities in the concentrate are significantly reduced in comparison with the flotation feed. A concentrate with only 0.19% ash content with removal of about 89% of the mass of ash is obtained.

The carbon content is significantly improved to 96.1% in comparison with 87.2% in the feed. The tailing fraction is heavily contaminated with Ni, V, Fe and Cu metals. The percentage of SiO<sub>2</sub> is about 17.5 in the tailing. This clearly confirms that a good selectivity is obtained in the flotation process when 4-phenyl dodecyl benzene sulphonate surfactant is used as a collector. This should encourage its application as a collector in the flotation of other minerals.

Table 5. Chemical analysis of the final concentrate and its tailing

Constituents	Concentrate %	Tail %	Constituents	Concentrate %	Tail %
Ash	0.19	12.4	Fe	60	2000
Nitrogen	0.50	2.1	Co	trace	trace
Fixed Carbon	96.10	71.7	Mo	2	trace
Sulphur	4.10	4.1	Cu	11	1060
SiO <sub>2</sub>	0.02	17.5	Ti	10	83
Metal Content	ppm	ppm	Ca	100	trace
Ni	250	1690	Cr	8	trace
V	260	1000	Na	100	trace

## CONCLUSIONS

- The surface properties of the prepared surfactant (4-phenyl dodecyl benzene sulphonate) showed that the temperature change, within the range 25–55 °C, has little influence on its critical micelle concentration (cmc) and surface tension at cmc ( $\gamma_{cmc}$ ).
- The decrease in surface excess concentration at surface saturation ( $\Gamma_{max}$ ) with raising temperature is accompanied by an increase in the area occupied per molecule at the interface ( $A_{max}$ ) probably due to the increased thermal motion.
- The values of the standard free energy change upon micelle formation ( $\Delta G_{mic}$ ) become more negative with increasing temperature. This is attributed to the decrease of the amount of water bound by the sulphate head group and the water structured by the hydrophobic chain.
- The values of the free energy change upon adsorption ( $\Delta G_{ads}$ ) are all negative at different temperatures. This indicates that the adsorption of this collector at the aqueous solution/air and the aqueous solution/solid interfaces is spontaneous.
- The optimum dosage of 4-phenyl dodecyl benzene sulphonate as a collector for flotation of the petroleum coke sample is small (~20 g/t). Flotation of the coke sample using such dosage of surfactant and in presence of 1 kg/ton of sodium silicate and at pH 7 gave the best selectivity where a concentrate of only 0.19% ash content, with a yield of about 90 wt%, is obtained from a feed containing 1.38% ash content. Multi-

stage addition of the surfactant, in four steps rather than one, improved further the efficiency of the flotation process by increasing the yield of the final concentrate to about 93.1 wt% without affecting its grade.

- 4-phenyl dodecyl benzene sulphonate showed a high selectivity in the flotation of petroleum coke where the heavy metals are significantly reduced in the final concentrate while its tailing is heavily contaminated with heavy metals. A high selectivity of petroleum coke flotation while using the investigated surfactant will encourage its application as a flotation collector in flotation of other minerals.

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**Abdel-Khalek N.A., Omar A.M.A., Barakat Y.,** Flotacja egipskiego koksiku porafinacyjnego za pomocą 4-fenyldodecylobenzenu. *Fizykochemiczne Problemy Mineralurgii*, 31, 19–30 ( w jęz. angielskim)

Jako kolektora do badań flotacyjnych koksiku z rafinerii egipskich użyto syntetycznego surfaktantu 4-fenyldodecylobenzenu; celem flotacji było zminimalizowanie zawartości popiołu oraz ciężkich metali w koksiku, aby uczynić go zdatnym do produkcji elektrod. Strukturę surfaktantu określono za pomocą technik IR, NMR i spektroskopii masowej. Zbadano również właściwości powierzchniowe i termodynamiczne użytego kolektora. Stwierdzono, że przy podwyższaniu temperatury od 25 do 55 °C wartość krytycznego stężenia micelizacji (CMC) badanego surfaktantu wzrastało wolno, podczas gdy nadmiarowe stężenie powierzchniowe przy stężeniu nasycenia ( $\Gamma_{\max}$ ) nieznacznie malało. Wraz ze wzrostem temperatury wzrastał też obszar okupowany przez cząsteczkę kolektora na granicy fazowej przy adsorpcji nasycenia ( $A_{\max}$ ). Wyniki badań pokazały, że zmiana wartości swobodnej energii adsorpcji ( $\Delta G_{\text{ads}}$ ) jest ujemna w różnych temperaturach. Wskazuje to, że adsorpcja zastosowanego kolektora flotacyjnego na granicy faz roztwór wodny–powietrze i roztwór wodny–ciało stałe jest spontaniczna. Testy flotacyjne przeprowadzono w laboratoryjnej maszynie flotacyjnej typu Denver. Pulpa flotacyjna zawierała 10% wagowych koksiku, a jako depresantu użyto krzemian sodu. Badano różne parametry, które wpływają na flotację koksiku w tym stężenie surfaktantu, jego sposób dozowania, stężenie depresantu i pH. Wyniki badań wykazały, że optymalne warunki do flotacji koksiku prowadzą do otrzymania koncentratu zawierającego tylko 0,19% popiołu w stosunku do 1,38% popiołu w nadawie, a odpad zawierał 12,5% popiołu i był on bardzo zanieczyszczony metalami ciężkimi. Stwierdzono dużą zgodność wyników flotacyjnych z właściwościami powierzchniowymi i termodynamicznymi surfaktantu.