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Micellar extraction of phenols from carbolic oil

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Abstract—A new method, called micellar extraction has been proposed for extractive recovery of phenols from carbolic oil using the solubilization phenomenon in aqueous solutions of anionic surfactants. The efficiency of a one-step extraction was determined depending on the kind of surfactants and their concentration. The curve of extraction equilibrium in the system: carbolic oil–phenols–0.25 M solution of sodium laurate was also defined. The results obtained were compared with those for standard extractive agents given in the literature.

INTRODUCTION

PHENOLS are mainly produced during dry distillation of coal. During redistillation the phenols pass to light (upper b.p. 200°C) and middle (b.p. between 200 and 300°C) oil fractions. Because of the high demand for phenols, extraction methods for recovery of these compounds from the oil fractions are widely developed. On an industrial scale, solutions of compounds reacting selectively with the phenols (i.e. sodium hydroxide [1, 2], ethanolamines [1, 3–5], ammonia [1] or solutions of compounds increasing phenols' solubility (i.e. 70–80 wt% methanol solution [1], glycols and glycerine [1, 3–5]) are mainly used as extraction eluents. Another known method is the extraction of phenols with pure water at elevated temperature (up to 160°C) and under pressure of up to 20 atm. [1].

The group of compounds increasing the solubility of phenols in water include surface active agents. Their solutions in the concentration exceeding critical micelle concentration (CMC) have the ability to dissolve substances that are usually nonsoluble or hardly soluble in pure solvent. This phenomenon, called solubilization [6–12], has found a number of practical applications, e.g. in the pharmaceutical industry for condensation of some drugs that hardly dissolve in pure water [7], in the cosmetic industry [7], and in production of herbicides, fungicides and insecticides required for agriculture [7]. Solubilization is also widely applied in catalytic reactions (so-called micellar catalysis) [12]. Presence of micelles in environment is a factor influencing the rate of various organic as well as inorganic reactions.

Solubilization of phenols

Solubilization of cresols for preparation of disinfectants, such as Kresolum saponatum (Ph. Nord), Saponated Solution of Cresol (U.S.P.) and Lysol (B.P.) [13], has been used since the last decade of the 19th century. This led many scientists to perform extensive studies on the solubilization of different antiseptic phenols and their properties in aqueous solutions.

Several authors [14, 15] have described homogeneous solutions of phenol in aqueous solutions of soaps and alkali metal alkyl sulfates. Other authors [15, 16] however, have reported that surfactants of Tweens and Myrjs types (trademarks of Atlas Chemical Industries Inc., Wilmington, Delaware for polyoxyethylene sorbitan fatty acid esters and polyoxyethylene stearates, respectively) in aqueous solutions interact with phenol to give two different phases.

The first investigations on solubilization of cresols were carried out by Bailey [17] in 1923. He reported a positive effect of sodium oleate on solubilities of separate cresols in water. Shortly later Jenčič and Udovč [18] published their data on the solubility of a mixture of cresol isomers in aqueous solutions of some twenty fatty acid soaps. Angelescu and Popescu [19] investigated experimentally solubilization of *o*-cresol in sodium stearate, palmitate and oleate solutions and found that the solubility increased several times with respect to that in pure water. The data on solubilization of cresols and xylenols in aqueous solutions of long-chain alkyl sulfates are also available [14, 19].

Taking into account the experimental data on solubilization of phenols and applications of solubilization in technology, it seems that aqueous solutions of anionic surfactants may be used for extraction of phenols from oil fractions.

The purpose of this paper is to select appropriate surfactants and establish their concentrations suitable for extraction of phenols from oils. For the compounds exhibiting the best extraction effects the equilibrium curves in the system: carbolic oil–phenols–aqueous solution of the individual surfactant, have been defined and compared with the corresponding ones given in the literature.

ATTEMPTS TO DEFINE THERMODYNAMICAL PRINCIPLES OF MICELLAR EXTRACTION

If the system considered is in equilibrium and the temperature T and pressure p are constant, it is described by the known equation:

$$\delta g = 0 \qquad (1)$$

NOMENCLATURE

a	activity [kMol kMol^{-1}]
c	concentration [kMol m^{-3}]
g	thermodynamical potential [J]
HLB	hydrophile–lipophile balance
k	equilibrium constant
m	mass [kg or kMol]
n	number of molecules
N	number of micelles
p	pressure [N m^{-2}]
R	gas constant [8314 J kMol^{-1} K^{-1}]
T	temperature [K]
v	molar volume of solvent [m^3 kMol^{-1}]
x	concentration [kMol kMol^{-1} or kg kg^{-1}].

Greek symbols

α	selectivity coefficient
δ	solubility parameter [cal$^{0.5}$ m$^{-1.5}$]
δ	differential (in eqs. (1) and (2))
ε_m	subdivision potential [J kMol^{-1}]
μ	chemical potential [J kMol^{-1}]
ϕ	volume ratio of solvent [m^3 m^{-3}].

Sub- and superscripts

E	extract
F	phenols
i	component
i	inert (in Fig. 6)
j	phase
m	micelle
o	oil
R	raffinate
s	surfactant
S	feed
w	water
α	number of components
β	number of phases
$^-$	standard condition.

which can be written in different form:

$$g = \sum_{j=1}^{\beta} \sum_{i=1}^{\alpha} \mu_i^j \cdot \delta m_i^j = 0. \tag{2}$$

Assuming that only one component is consecutively transferred from the first phase to the second one and further on, equation (2) may be rewritten as

$$\mu_2^1 = \mu_1^2 = \mu_1^3 = \cdots = \mu_1^\beta. \tag{3}$$

From the physicochemical point of view, the solubilization corresponds to the separation of substances into two nonmiscible aqueous and micellar phases. In addition, including the equilibrium at the water interface, the equation for one system (i.e. oil–phenol–aqueous solution of surfactant) will take the form

$$\mu_F^o = \mu_F^w = \mu_F^m. \tag{4}$$

Chemical potentials of phenol in individual phases are described by the following equations:

$$\mu_F^o = \bar{\mu}_F^o(p, T) + RT \ln a_F^o, \tag{5a}$$

$$\mu_F^w = \bar{\mu}_F^w(p, T) + RT \ln a_F^w, \tag{5b}$$

$$\mu_F^m = \bar{\mu}_F^m(p, T, \varepsilon_m) + RT \ln a_F^m. \tag{5c}$$

In contrast to the usual thermodynamics of large systems, where standard chemical potential depends on temperature and pressure exclusively, in thermodynamics of small systems, developed by Hill [20] and applied by Hall and Pethica [21] to micellar systems, the standard chemical potential is additionally a function of so-called 'subdivision potential' ε_m; defined as

$$\mu^m(p, T, \varepsilon_m) = \mu^m(p, T) + RT \ln x_m. \tag{6}$$

According to Hildebrand's theory [22, 23] of the 'regular solutions' the activity of dissolved substance is given by

$$\ln a_F = \ln x_F + \frac{v_F \phi^2 (\delta - \delta_F)^2}{RT}. \tag{7}$$

From equations (5)–(7) the relations (8a, b, c) take the form

$$\mu_F^o = \bar{\mu}_F^o(p, T) + RT \ln x_F^o + v_F \phi_o^2 (\delta_o - \delta_F)^2, \tag{8a}$$

$$\mu_F^w = \bar{\mu}_F^w(p, T) + RT \ln x_F^w + v_F \phi_w^2 (\delta_w - \delta_F)^2, \tag{8b}$$

$$\mu_F^m = \bar{\mu}_F^m(p, T) + RT \ln x_m + RT \ln x_F^m + v_F \phi_m^2 (\delta_s - \delta_F)^2. \tag{8c}$$

Substitution of equations (8a, b, c) into equation (4), describing the equilibrium condition yields

$$k_F^{o/w} = \frac{x_F^w}{x_F^o} = \exp\{[\bar{\mu}_F^o - \bar{\mu}_F^w + v_F \phi_w^2 (\delta_w - \delta_F)^2 - v_F \phi_o^2 (\delta_o - \delta_F)^2]/RT\}, \tag{9}$$

$$k_F^{o/m} = \frac{x_F^m}{x_F^o} = \exp\{[\bar{\mu}_F^o - \bar{\mu}_F^m - RT \ln x_m + v_F \phi_m^2 (\delta_s - \delta_F)^2 - v_F \phi_o^2 (\delta_o - \delta_F)^2]/RT\}. \tag{10}$$

In equation (10) the 'solubility parameter' δ depends on the kind of surfactant, whereas x_m and ϕ_m are concentration dependent. The solubility parameter, according to Little [24], can be simply correlated with the HLB value of surfactants

$$\delta = \frac{118.8}{54 - HLB} + 6.0. \tag{11}$$

Equations (10) and (11) show that the higher the value of

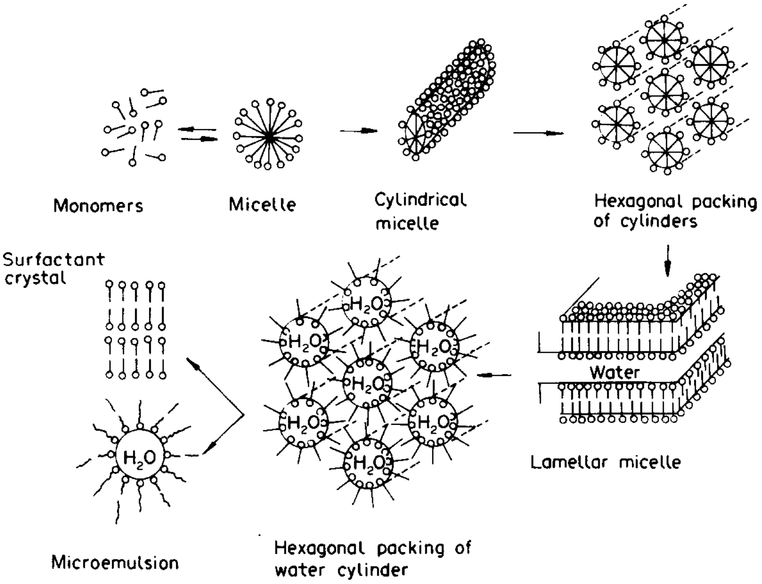

FIG. 1. Structure formation in surfactant solution.

HLB, i.e. the higher the surfactant's hydrophobicity, the higher is the concentration of phenols in the micelles. This fact means that the equilibrium is shifted towards water phase.

In the case of solubilization, i.e. micellar extraction the fraction of the solvent volume ϕ_m depends on the kind of micelles being formed. The kinds of micelles formed depending on the concentration of surfactant are shown in Fig. 1.

The molar ratio of micelles equals

$$x_m = \frac{N}{n_F^w + n_s^w + N}. \tag{12}$$

Its dependence on the total surfactant concentration is given by [21]

$$\left(\frac{\partial x_m}{\partial x_s}\right)_{p,T} = \frac{N \cdot x_m}{x_s^w + N^2 \cdot x_m}. \tag{13}$$

The influence of surfactant concentration on the equilibrium constant $k_F^{o/m}$ will be estimated by differentiating equation (10), and taking into consideration equation (13), to finally obtain

$$\begin{aligned}\left(\frac{\partial k_F^{o/m}}{\partial x_s}\right)_{p,T} &= \left[\exp\frac{\bar{\mu}_F^o - \bar{\mu}_F^m - v_F\phi_o^2(\delta_o - \delta_F)^2}{RT}\right] \\ &\times \left[-\frac{1}{x_m}\frac{N}{x_s^w + N^2 x_m}\exp\frac{v_F\phi_m^2(\delta_s - \delta_F)^2}{RT}\right. \\ &+ \frac{2v_F\phi_m(\delta_s - \delta_F)^2}{RT} \\ &\left.\times \exp\frac{v_F\phi_m^2(\delta_s - \delta_F)^2}{RT}\frac{\partial \phi_m}{\partial x_s}\right]. \end{aligned} \tag{14}$$

From this equation it follows additionally that equilibrium coefficient depends on the number of micelles and on concentration of surfactant monomers in water.

EXPERIMENTAL

The carbolic oil used was produced by the Hajduki Chemical Plant in Chorzów (Poland). According to the manufacturer's data [25] the average composition is:

aromatic hydrocarbons	60.6 wt%
phenol	18.8
o-cresol	4.5
m- and *p*-cresols	11.0
xylenols	1.5
pyridine base	3.6.

The efficiency and selectivity of the extraction process were investigated using the following surfactants: sodium laurate (NaL), sodium oleate (NaO), potassium palmitate (KP) all from P. O. Ch. Gliwice, Poland, sodium dodecylsulfate (Roth, West Germany), sodium dodecylbenzenesulfonate (NaDBS) and 'Kosulfonat'. The latter is a mixture of sodium dodecylbenzenesulfonate and sodium dodecylsulfate (NaDS) in a 3:1 ratio by wt (Z. Ch. G. Pollena, Wrocław, Poland). Concentrations of these surfactants varied from 1×10^{-3} to 5×10^{-1} kMol m^{-3}. All the experiments were made in a one-step mixer–settler extractor with phase ratio 1:1 by vol. Both phases, after being poured into the extractor, were first stirred (400 rpm) with a spade stirrer for 15 min, then transferred to the separator funnel and left for 24 h. All the experiments were carried out at room temperature, under atmospheric pressure. After the separation of phases their volumes and densities were measured. The latter measurement was performed using a DMA-40 densitometer (Anton Paar K. G., Austria). Viscosity of the extract phase was measured at 20°C using a Hoeppler viscosimeter. Phenols present in the extract were re-extracted with *n*-butyl acetate (P. O. Ch. Gliwice) using a two-step procedure and a phase ratio of 1:5 (water:acetate). Contents of phenols in re-

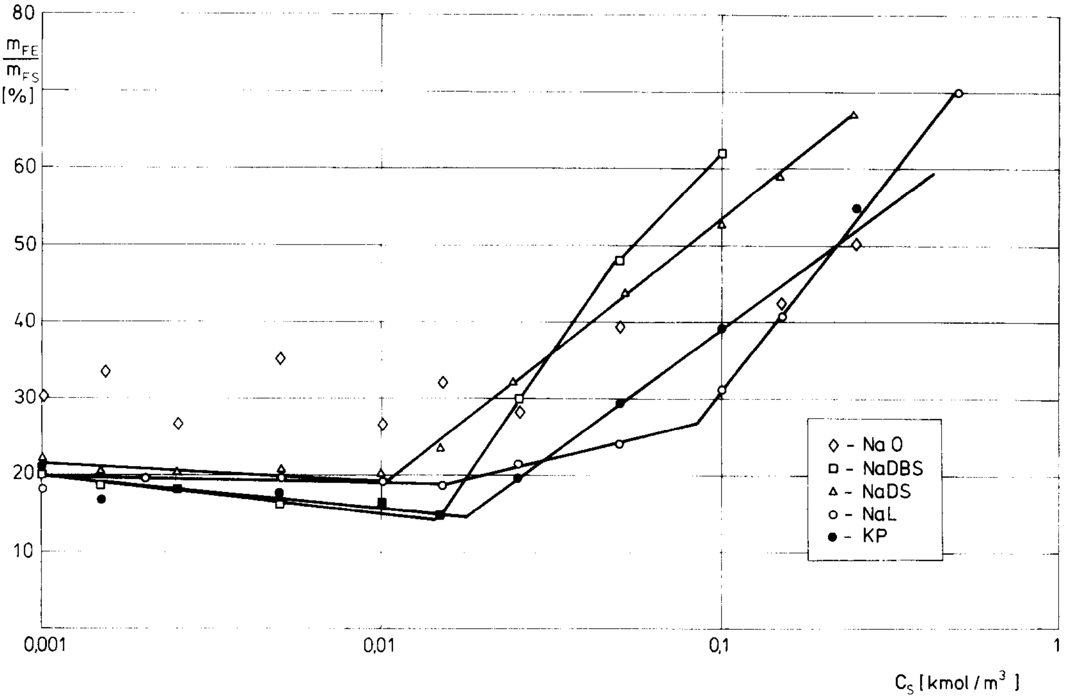

FIG. 2. The dependence of amount of extracted phenols on surfactant concentration.

extract and raffinate were determined by means of GLC, using a Chromatron GCHF 18.3 with an FID head. The analysis was performed in a metal column 1 m long of diameter 3×10^{-3} m, filled with 10 wt% DEGS (diethylene glycol succinate) and 3 wt% by weight orthophosphoric acid (80%) on to Chromosorb WAW 60–80 mesh. The temperature programmed ranged from 30 to 160°C; nitrogen was used as eluent. The concentrations of individual phenols in the sample were determined from the calibration curves.

In order to prove that it is advisable to apply the solutions of surfactants to extractions of phenols from the carbolic oil, the extraction of phenols by means of diethanolamine and ethylene glycol (both P. O. Ch. Gliwice, Poland) aqueous solutions was also studied. For the NaL solution (0.25 kMol m^{-3}) the extraction equilibrium curve was determined. The experiments were carried out for the following phase ratios o:w = 1:10, 1:8, 1:6, 1:4, 1:2, 1:1, 2:1, 4:1, 6:1, 8:1, 10:1. Analyses of the phenol content in both phases: extracts and raffinate were performed in the same way.

RESULTS AND DISCUSSION

The presence of surfactants in extracting medium influences both the quantity of the substance extracted and the selectivity of extraction. The relative quantity of extracted phenols vs concentration of surfactants is presented in Fig. 2. The quantities of the extracted phenols increase considerably with the increasing concentrations of all the surfactants examined, in some cases exhibiting the minima. The concentrations at which the dependence m_{FE}/m_{FS} on c_s changes its character are given in Table 1.

When sodium laurate was used two inflexion points were observed. The first one appeared at the surfactant concentration of 0.015 kMol m^{-3}. This value is comparable with the CMC amounting to 0.024 kMol m^{-3} [12]. A decrease of this value may be due to weak electrolytes, i.e. phenols and pyridine base present in the medium. It is well known that the presence of electrolytes [7, 8, 12] decreases the CMC value. In all other compounds the value of CMC is one order of magnitude lower than that of sodium laurate, and this could be the reason why only one inflexion point of the $m_{FE}/m_{FS} = f(c_s)$ curve was observed. The second inflexion point occurring in all surfactants is probably related to critical macromicellar concentration, at which solubilization strongly increases. This concentration corresponds probably to the change in the micellar structures, i.e. to their transition from the spherical shape to the lamellar one. This fact can be confirmed by the strongly increasing viscosity of the extract phase, that in concentrations above inflexion

Table 1. Concentrations at which the dependence m_{FE}/m_{FS} on c_s changes its character

Surfactant	Concentration (kMol m^{-3})	
Sodium laurate	0.015	0.085
Potassium palmitate		0.018
Sodium dodecylbenzenesulfonate		0.014
Sodium dodecylsulfate		0.012

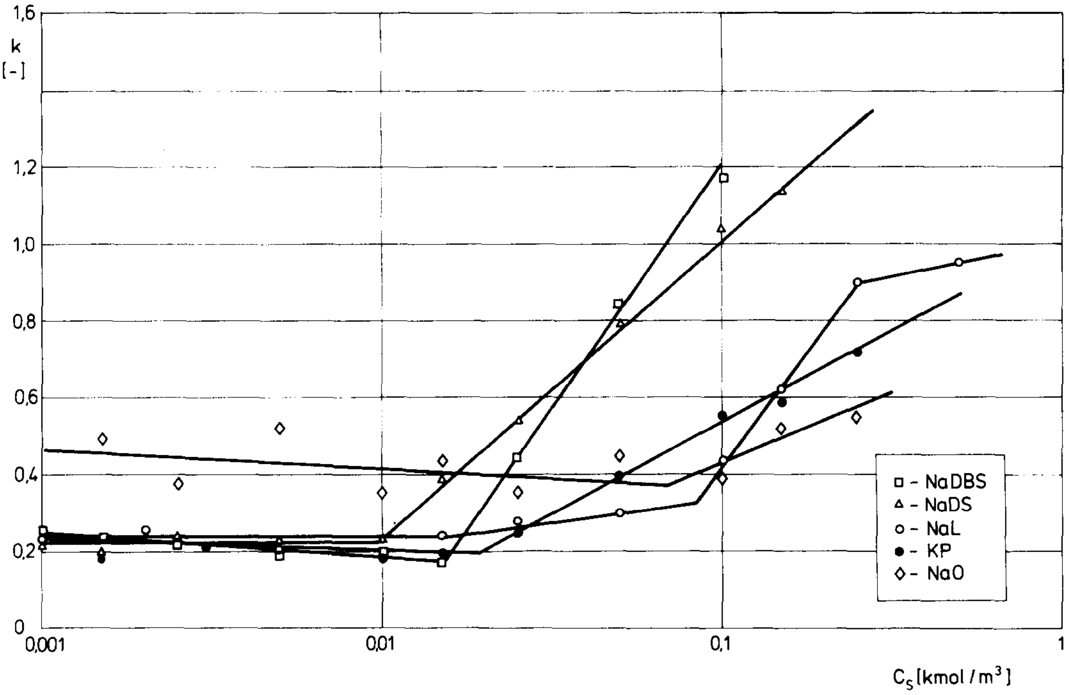

FIG. 3. The dependence of equilibrium constant of phenols on surfactant concentration.

point reaches 24.8×10^{-3} Ns m^{-2} for 0.5 kMol m^{-3} sodium laurate solution. The increase of viscosity in micellar systems caused by shape of micelles, was the highest for the lamellar ones [11].

The concentrations at which the extraction equilibria change have been also determined from the plots of equilibrium constants vs surfactant concentration, Fig. 3. In all the cases these concentrations were identical with those determined from the curves $m_{FE}/m_{FS} = f(c_s)$. When sodium oleate solution was used as the extracting medium it yielded a considerable dispersion of the experimental points (Figs. 2 and 3) which seems to be due to the structure of micelles. Sodium oleate has a double bond in the hydrophobic group and because of a higher polarity it will tend to be placed at the micelle surface, thus affecting the mechanism of phenol solubilization. Phenol, as a polar substance, is usually built up into the palisade layer of the hydrophobic group of the micellizing surfactant. Its hydroxyl moiety is directed outside the micelle. Since the double bond of the hydrophobic group of the oleate occupies some areas on the micelle surface and creates a steric hindrance, phenol will be probably absorbed deeper in the micelle core, so that its solubility will decrease. The application of 'Kosulfonat' as the extracting medium also gave results that differed considerably from those obtained for other surfactants. In practice the quantity of the extracted phenols was constant regardless of the concentration of the 'Kosulfonat'. 'Kosulfonat', being a mixture of sodium dodecylbenzenesulfonate and sodium dodecylsulfate, forms (in aqueous solutions) mixed micelles the properties of which differ significantly from those of micelles composed of a single chemical compound [9, 10].

The dependence of selectivity coefficient defined as

$$\alpha = \frac{x_{FE}}{x_{oE}} \frac{x_{oR}}{x_{FR}} \tag{15}$$

on surfactants' concentration is presented in Fig. 4. Selectivity of the extraction process increases with the concentration of surfactant to reach a steady state (sodium laurate, dodecylsulfate and dodecylbenzenesulfonate) or (potassium palmitate, sodium oleate) to slightly decrease at high concentrations. The concentration at which the value of the selectivity coefficient is established corresponds to the concentration yielding the minimum on the curve illustrating the quantity of extracted phenols vs concentration of surfactant (Fig. 2).

The highest 'yield' of the extraction process was observed for aqueous solutions of sodium dodecylbenzenesulfonate and dodecylsulfate used as extractants (Figs. 2, 3).

This fact is in accordance with the proposed model of equilibrium. Both the compounds are characterized by relatively high solubility parameters. These parameters for the surfactants used, and based on the literature [22–24] data are given in Table 2.

To prove the applicability of surfactant solutions to extraction of phenols from carbolic oil, extractions of these compounds by the methods described in the literature [1, 3–5] were also carried out. The results of experiments for mono- or diethanolamine or ethylene glycol solutions used as extracting media, were

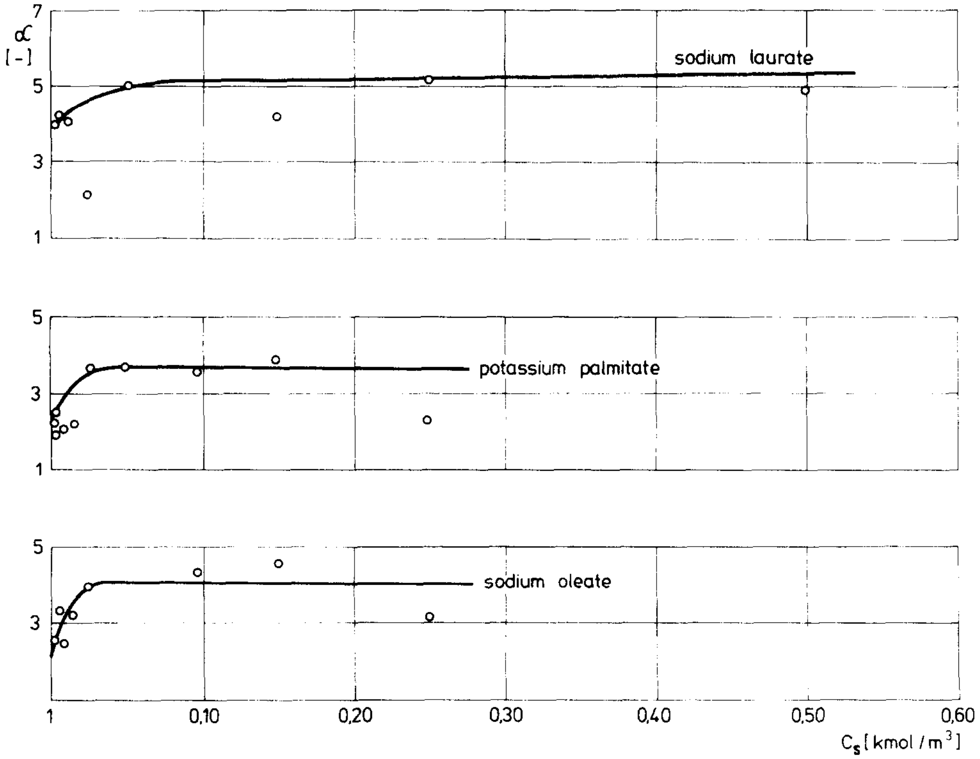

FIG. 4. The influence of the surfactant concentration on selectivity coefficient.

compared with those obtained for sodium dodecylbenzenesulfonate and laurate, and the results are shown in Fig. 5. In all the cases the extraction efficiency of standard procedures was lower than that obtained for surfactant solutions. Slightly higher values of selectivity coefficient were obtained only for monoethanolamine solutions.

The equilibrium curve was determined for the chosen surfactant, e.g. Fig. 6, for sodium laurate. The concentrations of phenol in surfactant solutions were about three times higher than those for 15 wt% monoethanolamine solution, while the concentration of the surfactant was three times lower than that of monoethanolamine.

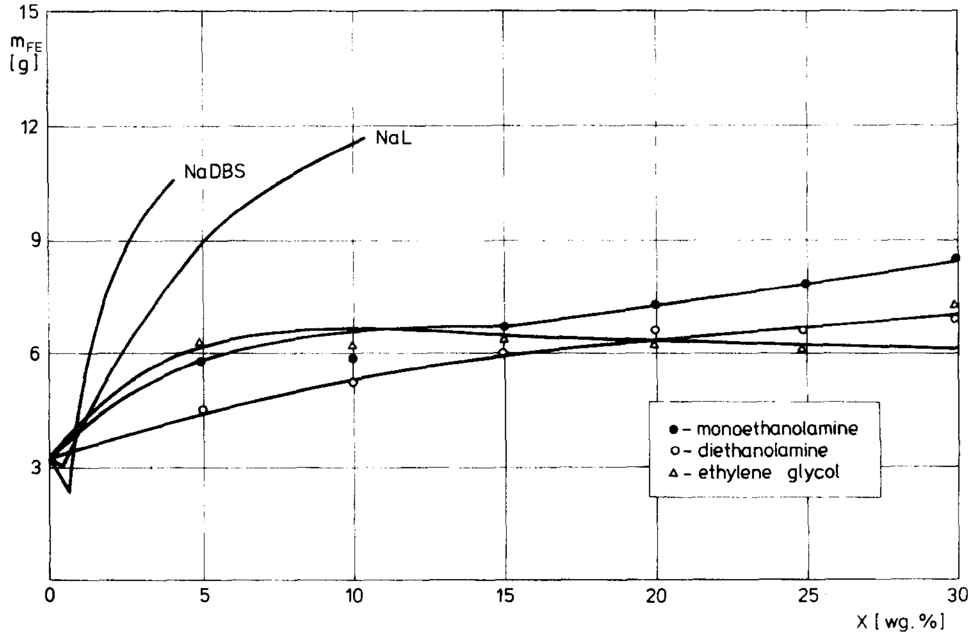

FIG. 5. The comparison of extraction effect of standard solvents with those for soap solutions.

Table 2. Surfactant parameters

Surfactant	δ $\mathrm{cal}^{0.5}\,\mathrm{m}^{-1.5}$
Sodium dodecylsulfate	14.1
Sodium dodecylbenzenesulfonate	13.0
Sodium laurate	9.6
Potassium palmitate	9.5
Sodium oleate	9.4

CONCLUSIONS

Studies on the micellar extraction of phenols from carbolic oil presented in this paper have led to the following conclusions.

(1) The application of solutions of surfactants (alkali metal salts of fatty acids, alkylsulfates and alkylbenzenesulfonates) to the extraction of phenol from the carbolic oil proved to be advisable.

(2) The quantity of the extracted phenols significantly increases with the surfactant concentration, exhibiting in some cases more or less significant minimum.

(3) The selectivity of the extraction process increases with the concentration of surface active compound, then it reaches a steady state or—in some cases—slightly decreases.

(4) The validity of the proposed model of extraction equilibrium, which predicts the inflexion point on the $m_{FE}/m_{FS} = f(c_s)$ curve, has been confirmed. The highest extraction efficiency was found for the dodecylsulfate and dodecylbenzenesulfonate, i.e. for compounds characterized by the highest value of solubility parameter.

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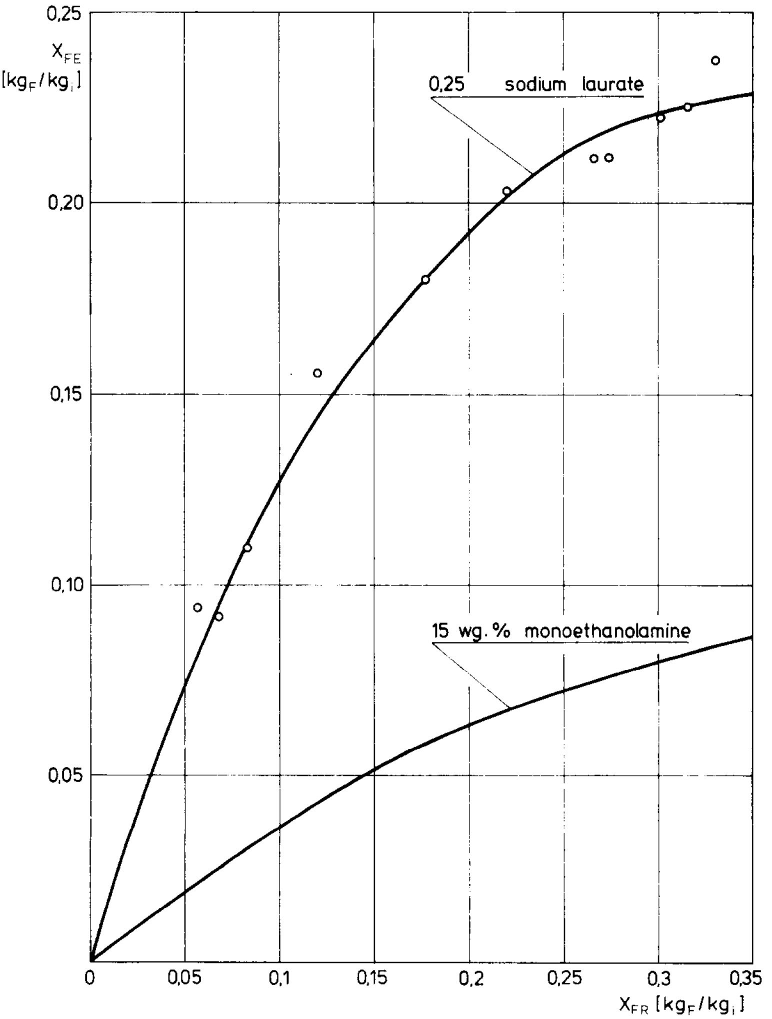

FIG. 6. The equilibrium curve of the system: carbolic oil–phenols–0.25 M solution of sodium laurate.

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EXTRACTION MISCELLAIRE DES PHENOLS A PARTIR D'HUILE CARBOLIQUE

Résumé—Une nouvelle méthode, appelée extraction miscellaire, est proposée pour la récupération extractive de phénols à partir d'huile carbolique en utilisant le phénomène de solubilisation dans des solutions aqueuses de surfactants anioniques. L'efficacité de l'extraction à un seul pas est déterminée selon les types de surfactants et leur concentration. On définit la courbe d'équilibre d'extraction dans le système: huile carbolique—phénols—0,25 M solution de laurate de sodium. Les effets obtenus sont comparés à ceux des agents extracteurs classiques donnés dans la bibliographie.

MIZELLENEXTRAKTION VON PHENOLEN AUS KARBOLÖL

Zusammenfassung—Ein neues Verfahren—die sogenannte Mizellenextraktion—wird für die Rückgewinnung von Phenolen aus Karbolöl durch Extraktion vorgeschlagen. Dabei wird die Löslichkeit von anionischen oberflächenaktiven Substanzen in wäßriger Form benutzt. Die Wirksamkeit der einstufigen Extraktion wurde in Abhängigkeit von der Art der Substanz und ihrer Konzentration bestimmt. Die Kurve des Extraktions-Gleichgewichts des Systems Karbolöl—Phenol–0,25 M. Lösung von Natrium-Laurat wird ebenfalls bestimmt. Die ermittelten Einflüsse werden mit denjenigen für Standard-Extraktionsstoffe aus der Literatur verglichen.

МИЦЕЛЛЯРНАЯ ЭКСТРАКЦИЯ ФЕНОЛОВ ИЗ ФЕНОЛЬНОГО МАСЛА

Аннотация—Предложен новый метод, называемый мицеллярной экстракцией, извлечения фенолов из фенольного масла на основе явления солюбилизации анионных поверхностно-активных веществ в водных растворах. Определена эффективность одноступенчатой экстракции в зависимости от вида поверхностно-активных веществ и их концентрации. Также получена кривая экстракционного равновесия в системе фенольное масло-фенолы-0,25 М раствора лаурата натрия. Проведено сравнение полученных результатов с опубликованными в литературе данными для стандартных экстрагирующих веществ.