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**EXTRACTION OF PHENOL FROM AQUEOUS SOLUTIONS  
BY METHYL TERT-BUTYL ETHER**

**ЭКСТРАКЦИЯ ФЕНОЛА ИЗ ВОДНЫХ РАСТВОРОВ  
МЕТИЛ-ТРЕТ-БУТИЛОВЫМ ЭФИРОМ**

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**Abstract.** Extraction of phenol from aqueous media, including wastewater, is a very important problem nowadays, due to both high toxicity of phenols and absence of the extractants for their recovery satisfying the requirements for extraction capacity, accessibility, cost, fire and explosion hazards, and so on.

The article considers the application of different extractants for removal of phenol from aqueous solutions and waste water. The efficiency of the extractants is discussed in relationship with the processes of their regeneration, chemical stability and other technological parameters. It is shown that methyl-tert-butyl ether

(MTBE) can be an effective phenol extractant as an alternative to the solvents applied.

The authors identified a phenol distribution constant from aqueous solutions in MTBE, which proved to be significantly higher than that of aliphatic ethers and close to butyl acetate. The dependency of phenol extraction degree on the volume ratio between organic and aqueous phases as well as on the number of extraction stages was established.

A phenol salting-out constant was identified in the process of its extraction from aqueous solutions by MTBE. The effect of LiCl, NaCl, KCl, CaCl<sub>2</sub> on phenol distribution constants in MTBE was studied. It was established that calcium chloride has maximum salting-out effect with phenol distribution constants significantly increasing in MTBE.

The perspectiveness of methyl tert-butyl ether application for phenol extraction from aqueous solutions is shown.

**Аннотация.** Задачи выделения фенола из водных сред, включая сточные воды, являются весьма актуальными, что связано как с высокой токсичностью фенолов, так и отсутствием экстрагентов для их извлечения, которые бы в полной мере удовлетворяли предъявляемым к ним требованиям по экстракционной способности, доступности, стоимости, пожаро- и взрывоопасности и др. В статье рассмотрено применение различных экстрагентов для выделения фенола из водных растворов и сточных вод. Эффективность экстрагентов обсуждена во взаимосвязи с процессами их регенерации, химической устойчивостью и другими технологическими параметрами. Показано, что, альтернативой используемым растворителям – эффективным экстрагентом фенола может явиться метил-трет.бутиловый эфир (МТБЭ).

Авторами определена константа распределения фенола из водных растворов в МТБЭ, которая оказалась существенно выше, чем у простых алифатических эфиров и близка к бутилацетату. Установлена зависимость степени извлечения фенола от соотношения объемов органической и водной фаз и числа ступеней экстракции.

Определены константы высаливания фенола при его экстракции из водных растворов МТБЭ. Изучено влияние  $\text{LiCl}$ ,  $\text{NaCl}$ ,  $\text{KCl}$ ,  $\text{CaCl}_2$  на константы распределения фенола в МТБЭ. Установлено, что максимальный высаливающий эффект имеет хлорид кальция, константы распределения фенола из которого в МТБЭ заметно возрастают.

Показана перспективность применения метил-трет-бутилового эфира для экстракции фенола из водных растворов.

**Key words:** extraction, phenol, methyl tert-butyl ether, distribution constant, salting-out.

**Ключевые слова:** экстракция, фенол, метил-трет-бутиловый эфир, константа распределения, высаливание.

Extraction of phenol from aqueous media, including wastewater, is a very important problem nowadays, due to both high toxicity of phenols and absence of the extractants for their recovery satisfying the requirements for extraction capacity, accessibility, cost, fire and explosion hazards, and so on. The systematic study of the mechanism of phenol extraction and the pattern of formation of H-complexes with organic reagents [2] is a scientific basis for selection of the most effective phenol extractants.

Fundamental series of studies on phenol extraction equilibria and its derivatives were conducted by Russian researchers. Ya.I.Korenman and his colleagues studied

the main regularities of phenol extraction from aqueous solutions by hydrocarbon solvents, ethers and esters, alcohols and their compositions [3, 4]. Technological aspects of extracting phenolic compounds from waste water were discussed in the monograph by G.D. Kharlampovich and Yu.V Churkin (Volga-Vyatka publishing house) [5].

Earlier on, members of the Institute of Org. Chem. URC RAS showed a high extraction capacity relative to phenolic compounds of sulfur - and phosphorus containing sulfoxide- and tributyl phosphate-based organic reagents due to their high electron-donor properties and ability to form stable H-complexes [6, 7]. A high phenol extraction capacity was demonstrated by phosphine oxides and N-oxides [8, 9].

However, it should be noted that sulfur- and phosphorus- nitrogen-containing reagents are high boiling organic compounds, regeneration of which by methods of distillation is either very difficult or associated with high energy costs. It also impedes the practical use of the compounds for phenol extraction. Apart from distillation, regeneration of phenol extracts can be carried out by re-extraction of phenols, for example, by alkalis with formation of corresponding phenolate concentrates. However, if the end product is phenol but not phenolate, a necessity to block carbonization of phenols arises. But it is not always rational, if, for example, productiveness of the unit for phenol utilization is poor.

High phenol extraction capacity was shown by methyl derivatives of six-membered cyclic esters, for example, 4-methyltetrahydropyran [10], where phenol distribution constant was 102. However, the solvent production has not been mastered so far.

This paper shows the possibility of phenol extraction from thermal cracking waste water by oil and its mixtures with gas condensates followed by compounding the extracts obtained with the products to be subsequently refined in accordance with the existing process flow schemes. Despite the relatively low extraction

capacity of oils ( $R_o = 2-10$ ) and the formation of sufficiently stable emulsions in the process of extraction, this approach can be considered appropriate in many cases, as it does not involve any subsequent technological stages connected with recovery of extractants and production of commercial phenol or phenolates.

At the same time the method of phenol and acetone production waste water treatment by cumene process considered here can be recognized as economically unjustified, because phenol concentration in local drains is relatively high reaching 5-10 g/l, and thus it is necessary not only to extract phenol, but dispose of it. For this purpose, extraction with benzene, butyl acetate (BA) and diisopropyl ether (DIPE) is commonly used. Among these solvents the most effective phenol extractants are BA and DIPE, distribution constants of which are respectively 44 and 23. However, BA is partially hydrolyzed to form highly corrosive acetic acid, and DIPE produces explosive peroxides [12].

As will be shown later, methyl tert-butyl ether (MTBE) may be an alternative to these solvents and an effective phenol extractant. MTBE is mainly applied as a high-octane additive to motor fuels [13].

A phenol distribution constant from aqueous solutions in MTBE has been identified and proved to be significantly higher than those of aliphatic ethers, close to BA and was  $P_o = 54$ .

According to GLC [14], the purity of the MTBE applied was 98.6%. Phenol was qualified as a "reagent grade".

The extraction was performed in thermostated bedded test tubes or separating funnels at  $t = 20^{\circ}\text{C}$  for 10 min. The preliminary experiments showed that the extraction equilibrium is achieved within 2-3 minutes of phase contact.

The phenol content in the initial solutions ( $C_o$ ) in equilibrium aqueous (X) and organic (Y) phases after extraction was determined spectrophotometrically using a "Specord UV VIS" spectrophotometer in quartz cells at a wavelength of 271 nm. The molar phenol absorption coefficient in dilute acidic aqueous solutions in that

case was 1500. The relative error in determining phenol concentration did not exceed 3-5%. The extracts had been diluted 50 times with a 0.01N HCl solution prior to photometering, 2% aqueous solution of MTBE in 0.01N HCl was placed in a reference cell.

The distribution coefficient was calculated using  $D_i = Y/X$  formula.

Table 1 shows degrees of phenol extraction by MTBE from aqueous solutions with a phenol concentration of 5 g/l and a different volume to volume ratio of organic and aqueous phases ( $V_{org}/V_{water}$ ) in conditions of one-stage (N=1) and two-stage (N=2) extractions.

Table 1. Phenol extraction degree ( $E_{ex}$ , %) dependence on the ratio of organic and aqueous phase volumes ( $V_{org}/V_{water}$ ) and the number of extraction stages (N)

| Rate of extraction, ( $E_{ex}$ , %) |       |       | Rate of extraction, ( $E_{ex}$ , %) |       |       |
|-------------------------------------|-------|-------|-------------------------------------|-------|-------|
| $V_{org}/V_{water}$                 | N=1   | N=2   | $V_{org}/V_{water}$                 | N=1   | N=2   |
| 1/1                                 | 97.90 | 99.96 | 1/3                                 | 93.56 | 99.67 |
| 1/2                                 | 95.85 | 99.85 |                                     |       |       |
| 1/2.5                               | 95.12 | 99.78 | 1/5                                 | 88.52 | 99.0  |

Further increase of the phenol distribution constant is possible if salting-out agents are injected into the aqueous phase – as a rule, inorganic salts, strongly hydrated in water, facilitating dehydration of the distributed compounds [15]. There is no information about salting-out effects in inter-phase phenol distribution between the aqueous phase and MTBE in the literature available. Therefore, we studied the effect of LiCl, NaCl, KCl,  $CaCl_2$  on phenol distribution constants in MTBE.

The extraction was carried out with the volume ratio of aqueous and organic phases  $O/A = 1/1$ . The solutions of salts were prepared gravimetrically with their concentration being subsequently checked by density.

The concentration of salts in the aqueous phase varied from 0.5 to 3.5-4 mol/l. The concentration of phenol varied in a wide range of  $5 \cdot 10^{-2}$  mol/l to  $7 \cdot 10^{-1}$  mol/l. It was found that the extraction isotherm of phenol is almost rectilinear, and distribution coefficients ( $D_i$ ) correspond to distribution constants ( $P_{oi}$ ). This allowed using a narrower variation interval of phenol concentration equal to  $5 \cdot 10^{-2}$ – $1 \cdot 10^{-1}$  mol/l in further experiments.

It is known that extraction does not have to depend on the phase of inter-phase distribution implementation [16]. Taking this into account, and in order to facilitate the study of salting-out effects it was considered appropriate to study the inter-phase transfer of phenol from MTBE into the aqueous phase, and not vice versa. For this purpose, a stock solution of phenol in MTBE was initially obtained by its extraction from dilute acidic solutions, which in further experiments was subjected to contact with acidified ( $\text{pH} < 3$ ) aqueous solutions of varying salt concentration. Slight acidification of the aqueous phase to  $\text{pH} = 2-3$  does not substantially change the ionic force of the solution of salting-out agents, and ensures complete inhibition of phenol dissociation ( $\text{pK}_a = 10$ ) and its transfer to the most extractable molecular shape, making it possible to obtain reliable figures of distribution constants, eliminating the errors resulting from production of ionic phenol forms.

Table 2 shows dependence of phenol distribution constants ( $P_{oi}$ ) between MTBE and water-salt solutions of LiCl, NaCl, KCl,  $\text{CaCl}_2$  on the molar concentration of salting-out agents.

As can be seen, calcium chloride has maximum salting out effect with phenol distribution constants in MTBE markedly increasing, reaching 360 with 3.5 mol/l  $\text{CaCl}_2$  solution being used. This agrees well with the maximum charge density of  $\text{Ca}^{+2}$  cation, ensuring its strong hydration.

These dependences are well described by the Sechenov equation (1), which was used for calculation of phenol salting constants ( $C_{sal}$ ):

$$\lg P_0/P_{oi} = C_{salting-out} C_{sal} \quad (1)$$

where  $P_0$ ,  $P_{oi}$  are phenol distribution constants from dilute acidic solutions without salting-out agents and with salting-out agents;  $C_{sal}$  is the concentration of salting-out agents, mol/l.

Table 2. Salting-out of phenol during its extraction from aqueous solutions by MTBE ( $P_0 = 54$ ,  $T=20^\circ\text{C}$ ,  $V_{org}/V_{water} = 1/1$ )

| № | $C_{sal}$ ,<br>mole/l | Salts             |              |                  |            |          |              |                  |            |          |              |                  |            |          |              |                  |            |
|---|-----------------------|-------------------|--------------|------------------|------------|----------|--------------|------------------|------------|----------|--------------|------------------|------------|----------|--------------|------------------|------------|
|   |                       | CaCl <sub>2</sub> |              |                  |            | NaCl     |              |                  |            | LiCl     |              |                  |            | KCl      |              |                  |            |
|   |                       | $P_{oi}$          | $P_{oi}/P_0$ | $\lg P_{oi}/P_0$ | $k_{high}$ | $P_{oi}$ | $P_{oi}/P_0$ | $\lg P_{oi}/P_0$ | $k_{high}$ | $P_{oi}$ | $P_{oi}/P_0$ | $\lg P_{oi}/P_0$ | $k_{high}$ | $P_{oi}$ | $P_{oi}/P_0$ | $\lg P_{oi}/P_0$ | $k_{high}$ |
| 1 | 0.5                   | 73                | 1.35         | 0.130            | 0.26       | 66       | 1.22         | 0.087            | 0.174      | 61       | 1.130        | 0.05             | 0.100      | 60       | 1.111        | 0.0453           | 0.091      |
| 2 | 1.0                   | 103               | 1.89         | 0.270            | 0.27       | 80       | 1.48         | 0.170            | 0.170      | 68       | 1.260        | 0.100            | 0.100      | 67       | 1.241        | 0.094            | 0.094      |
| 3 | 1.5                   | 128               | 2.37         | 0.345            | 0.23       | 100      | 1.85         | 0.267            | 1.178      | 78       | 1.441        | 0.160            | 0.110      | 75       | 1.390        | 0.143            | 0.0095     |
| 4 | 2.0                   | 178               | 3.30         | 0.519            | 0.26       | 125      | 2.32         | 0.364            | 0.182      | 92       | 1.703        | 0.231            | 0.116      | 86       | 1.604        | 0.260            | 0.100      |
| 5 | 2.5                   | 230               | 4.26         | 0.694            | 0.28       | 156      | 2.78         | 0.444            | 0.178      | 110      | 2.037        | 0.309            | 0.124      | 100      | 1.851        | 0.267            | 0.107      |
| 6 | 3.0                   | 280               | 5.18         | 0.747            | 0.25       | 180      | 3.33         | 0.523            | 0.174      | 125      | 2.315        | 0.364            | 0.121      | 115      | 2.130        | 0.328            | 0.110      |
| 7 | 3.5                   | 360               | 6.67         | 0.824            | 0.24       | 225      | 4.17         | 0.620            | 0.172      | 150      | 2.778        | 0.445            | 0.124      | 134      | 2.481        | 0.395            | 0.113      |

It was established that with the cation nature varied, salting-out constants change as follows:  $\text{CaCl}_2 > \text{NaCl} > \text{LiCl} > \text{KCl}$  (Table 2). The salting-out effect of NaCl in phenol extraction by MTBE was significantly higher than that of LiCl, despite the greater hydration of lithium cation [17], which is quite unusual, but, unfortunately, it is difficult to explain so far.

It should be noted that the use of salting-out agents, in particular  $\text{CaCl}_2$ , in the process of extraction of phenol trace amounts by MTBE, for example, with the purpose of its effective extraction concentrating and subsequent determination, proved to be convenient, as it allows phenol concentrating to be implemented in 5-25 times in one- two extraction stages.



Table 3 shows the degree of phenol extraction in the first stage ( $E_{ex1}$ ) and the second stage ( $E_{ex2}$ ) extraction modes, as well as in the two sequent distribution stages ( $E_{ex1-2}$ ), and the corresponding degrees of its concentration in these modes ( $N_1, N_{1-2}$ ), proceeding from the volume to volume ratio of aqueous and organic phases ( $V_{water}/V_{org}$ ).

Table 3. Phenol extraction degrees during its extraction from 3m/l  $CaCl_2$  solution by methyl tert-butyl ether

| № | $V_{org}/V_{water}$ | $E_{ex1}$ , % | $N_1$ | $E_{ex2}$ , % | $E_{ex1-2}$ , % | $N_{1-2}$ |
|---|---------------------|---------------|-------|---------------|-----------------|-----------|
| 1 | 5                   | 98.6          | 5.0   | 1.67          | 100             | 2.5       |
| 2 | 10                  | 97.3          | 9.7   | 2.62          | 99.92           | 5.0       |
| 3 | 20                  | 94.7          | 18.9  | 5.02          | 99.71           | 10.0      |
| 4 | 30                  | 92.3          | 27.6  | 7.10          | 99.40           | 14.9      |
| 5 | 40                  | 90.0          | 36.0  | 10.0          | 99.00           | 19.8      |
| 6 | 50                  | 87.8          | 43.9  | 12.2          | 98.50           | 24.6      |

To ensure a higher degree of phenol concentration it is necessary to increase the  $V_{org}/V_{water}$  ratio, i.e., to reduce the volume of the extractant used, which naturally leads to a decrease in the degree of extraction. As seen from Table 3, phenol concentration in almost twenty times and its almost complete (94.7%) extraction can be achieved in a single stage extraction, while concentration in 43.9 times during extraction with  $V_{org}/V_{water} = 50$  is characterized by a relatively low degree of phenol extraction ( $E_{ex1}=87.8\%$ ). Re-extraction of phenol from raffinate with the same volume ratio of the phases, followed by combination of the extracts, leads to virtually quantitative recovery of phenol, although the extent of its total concentration is significantly reduced (from 43.9 to 24.6). Thus, it is apparently impractical to carry out phenol extraction from 3.5 mol/L  $CaCl_2$  solutions with  $V_{org}/V_{water} > 20$ .

## Conclusions

Methyl tert-butyl ether with its availability, low cost, absence of explosive peroxides, simplicity of recovery and effectiveness of phenol extraction from aqueous solutions is the most technological solvent which can be recommended for local treatment of phenolic effluents.

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