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# Removal of Phenol from Wastewater by Different Separation Techniques

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## Removal of phenol from wastewater by different separation techniques

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### Abstract

Application of membrane techniques (pervaporation and membrane-based solvent extraction) and adsorption to the removal of phenol from solutions modelling wastewater from phenol production by cumene oxidation process was investigated. The transport and separation properties of composite membranes PEBA, PERVAP 1060 and PERVAP 1070 in pervaporation of water–phenol mixtures were determined. It was found that the best removal efficiency of phenol was obtained using the PEBA membrane. MTBE, cumene and the mixture of hydrocarbons were applied in the membrane-based phenol extraction. Extra-Flow contactor with Celgard X-30 polypropylene hollow-fiber porous membranes was used in the experiments. MTBE was found the most efficient extractant. Adsorption of phenol on the different Amberlite resins was also investigated. Among the Amberlite resins of various grades used, the Amberlite XAD-4 had the best properties in the phenol removal from the aqueous solutions. It was shown that regeneration of the adsorbent bed could be effectively performed with sodium hydroxide solution.

**Keywords:** Phenol; Wastewater treatment; Pervaporation; Adsorption; Hybrid processes

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### 1. Introduction

Nowadays phenol is an important raw material in petrochemical, pharmaceutical, plastic and pesticide chemical industry. The global production

capacity of phenol reached 7.8 million of tonnes in 2001 [1].

Since 1952 the cumene oxidation process, called also the Kellong, Brown & Root (KBR) phenol process, is a commonly used technology for the manufacture of phenol and acetone [2,3].

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This process consists in oxidation of isopropyl benzene (cumene) with air, followed by cleavage of the formed cumene hydroperoxide in the presence of an acid catalyst. However the cumene oxidation process is also a source of wastewater. Depending on the process conditions, up to 0.6 tonne of liquid wastes is generated per tonne of the phenol produced. The wastewater contains 2–3% phenol, 3–6% acetone, up to 0.1% aromatic hydrocarbons (mainly cumene and  $\alpha$ -methylstyrene) and 2–3% sodium salts (mainly formate and sulphate). Taking into account the high toxicity and hazardous character of phenol, the importance of decontamination of these effluents before their discharge into sewage system and the environment is obvious. Recently several papers dealing with the feasibility of different separation processes to recover phenol from wastewaters have been published [4–6].

The conventional treatment of the cumene oxidation process effluents is presented schematically in Fig. 1. Briefly, it consists of two operation steps: 1) distillation of acetone and hydrocarbons from raw wastes and 2) phenol adsorption on polymeric resins or phenol extraction with organic solvent. However, the presently used solution exhibits some essential disadvantages: 1) distillation is an energy consuming technique, 2) high phenol content in liquors directed to the adsorption step

involves either a frequent regeneration of the adsorbent bed or the demand of its high capacity, 3) the use of combustible acetone as regenerant of the adsorbent.

The present work was aimed at developing an alternative hybrid process which would involve both conventional separation methods (distillation, adsorption) and membrane separation techniques (pervaporation and/or membrane-based solvent extraction) for the treatment of effluents from the cumene oxidation process.

The membrane techniques should allow removal of a considerable part of the organic pollutants, whereas adsorption should lower the phenol concentration of the treated effluent to the level acceptable by wastewater treatment plants. Pervaporation is an energy saving membrane technique used to separate liquid mixtures [7]. Membrane-based solvent extraction (membrane contactor) can be used as an alternative technique to the classical solvent extraction [8,9].

In the present paper we describe the results of our studies on the removal of phenol using different methods:

- pervaporation with hydrophobic membranes,
- membrane-based solvent extraction with porous hydrophobic membranes,
- adsorption on polymeric resins.

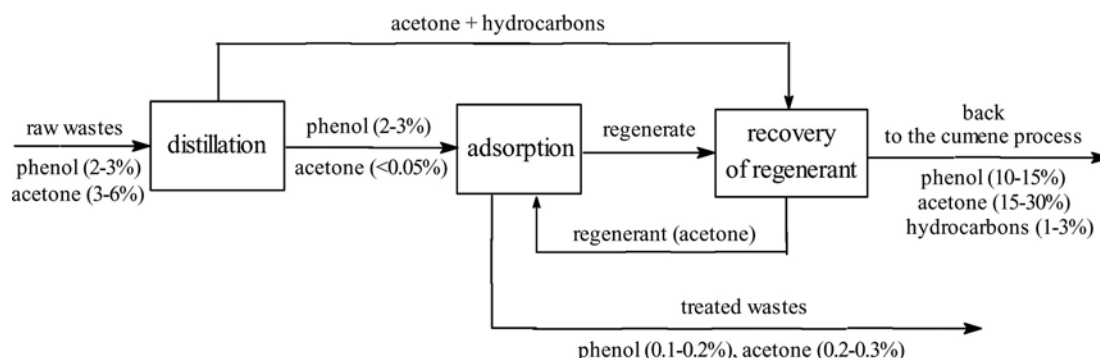


Fig. 1. Scheme of the cumene process wastewater treatment.

## 2. Experimental

### 2.1. Pervaporation

The experimental set-up used in pervaporation was described in the details elsewhere [10]. The membrane area was equal to 170 cm<sup>2</sup>. The experiments were carried out using composite membranes PERVAP–1060, PERVAP–1070 (Sulzer Chemtech Membrane Systems A.G.) and PEBA (GKSS-Forschungszentrum Geesthacht GmbH). Characteristics of the investigated membranes are presented in Table 1.

### 2.2. Membrane-based solvent extraction

The membrane-based solvent extraction experiments were carried out using a membrane cross-flow type contactor Extra-Flow 2.5"×8" supplied by the Hoechst Celanese. The contactor was equipped with hollow-fiber hydrophobic membranes Celgard X-30. The characteristics of the membrane and the contactor were presented in Tables 2 and 3. Aqueous phenol solution (0.17–2 wt.%) passed through the tube side hollow-fiber membranes with the volumetric flow within the

Table 2

Characteristics of the hollow-fiber membrane Celgard X-30

Membrane material	Polypropylene
Inner diameter, μm	240
Thickness, μm	30
Pore diameter, μm	0.03
Porosity, %	40

Table 3

Characteristics of the membrane contactor Extra-Flow 2.5"×8"

Inner diameter, mm	63
Number of hollow-fibers	12,000
Area/volume ratio, cm <sup>2</sup> ·cm <sup>-3</sup>	29.3

range 1.6–7.6 cm<sup>3</sup>·s<sup>-1</sup>. An organic extraction solvent was circulated in countercurrent flow on the shell side of the hollow-fiber membranes. Methyl-*ter*-butyl ether (MTBE), cumene and a mixture of hydrocarbons (cumene 45 wt.%, α-methylstyrene 55 wt.%) were used as extraction

Table 1

Characteristics of the investigated membranes according to the manufacturers' data

Membrane	Thickness of the selective layer, μm	Composition of the selective layer
PERVAP-1060	8	PDMS* $\left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{---Si---O---} \\   \\ \text{CH}_3 \end{array} \right]_n$
PERVAP-1070	10	Zeolite** filled PDMS $\left[ \begin{array}{c} \text{CH}_3 \\   \\ \text{---Si---O---} \\   \\ \text{CH}_3 \end{array} \right]_n$
PEBAX-4033	80	PEBA*** $\text{HO} \left[ \begin{array}{c} \text{C} \text{---} \text{PA} \text{---} \text{C} \text{---} \text{O} \text{---} \text{PE} \text{---} \text{O} \\    \quad \quad    \\ \text{O} \quad \quad \text{O} \end{array} \right]_n \text{---H}$

\* PDMS – poly(dimethylsiloxane)

\*\* Zeolite ZSM-5 Na<sub>n</sub>[Al<sub>n</sub>Si<sub>(96-n)</sub>O<sub>192</sub>] ~ 16 H<sub>2</sub>O, n < 27

\*\*\* Block copolymer polyether-polyamide (PE-PA)

solvents. The volumetric flow of the extraction solvent was maintained constant at  $0.33 \text{ cm}^3 \cdot \text{s}^{-1}$ . The pressure on the tube side was 31.0–37.9 kPa higher than that on the shell side but did not exceed the breakthrough pressure. Phenol content in the investigated mixtures was determined using the spectrophotometer UV-VIS Varian Carry 3E [11].

### 2.3. Adsorption

The Amberlite resins XAD-4, XAD-7, XAD-16 manufactured by Rohm and Hass Co. were applied in adsorption experiments. The properties of the adsorbents were presented in Table 4. Prior to use, all the adsorbent samples were standardized using the following procedure: the resin sample ( $100 \text{ cm}^3$ ) was placed in a column and the adsorbent bed was rinsed using distilled water ( $500 \text{ cm}^3$ ), acetone ( $200 \text{ cm}^3$ ) and distilled water ( $1000 \text{ cm}^3$ ) with the volumetric flow rate  $200 \text{ cm}^3 \cdot \text{h}^{-1}$ .

Adsorption experiments under static conditions using three Amberlite resins were carried out by the batch method [12]. The samples of an adsorbent (0.6–25 g) were shaken with  $100 \text{ cm}^3$  of aqueous phenol solution ( $10 \text{ g} \cdot \text{dm}^{-3}$ ). When adsorption reached equilibrium, phenol concentration in the solution was determined. The amount of phenol in the adsorbent was calculated from concentrations of solution before and after adsorption.

The column method [12] was applied to determine adsorption properties under dynamic conditions using the Amberlite XAD-4 resin. The model feed solutions containing different amounts of phenol (i.e. 3 and  $5 \text{ g} \cdot \text{dm}^{-3}$ ) and sodium sulphate ( $30 \text{ g} \cdot \text{dm}^{-3}$ ) passed through the resin bed with

volumetric feed rate of  $2 \text{ BV} \cdot \text{h}^{-1}$ . After a breakthrough of the column the model solution was removed and resin was regenerated (proportioning rate of a regenerant was  $1 \text{ BV} \cdot \text{h}^{-1}$ ). The following liquid mixtures were used as regenerants: water, sodium sulphate solution ( $30 \text{ g} \cdot \text{dm}^{-3}$ ), treated model solution (phenol concentration  $0.08 \text{ g} \cdot \text{dm}^{-3}$ ) and sodium hydroxide solution ( $200 \text{ g} \cdot \text{dm}^{-3}$ ).

## 3. Results and discussion

### 3.1. Pervaporation

All investigated hydrophobic membranes were selective toward phenol, however the selectivity was dependent on the kind of the membrane used for the separation (Figs. 2,3).

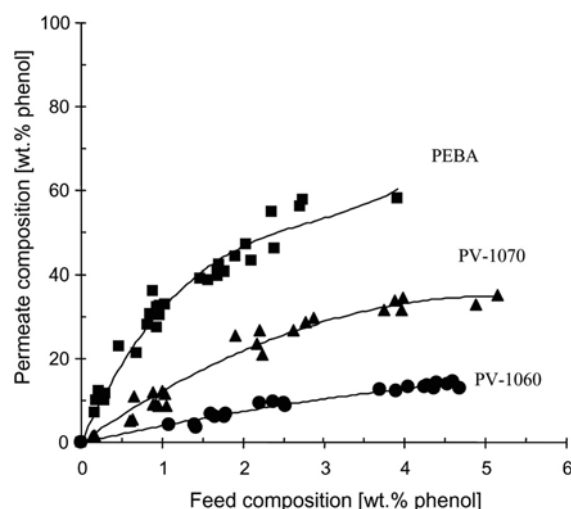


Fig. 2. Separation diagram of hydrophobic membranes investigated in contact with binary water–phenol mixtures.

Table 4  
Characteristics of the Amberlite resins

Resin	XAD-4	XAD-7	XAD-16
Copolymerisate	Styrene-divinylbenzene	Acrylate-divinylbenzene	Styrene-divinylbenzene
Specific surface, $\text{m}^2 \cdot \text{g}^{-1}$	750	450	750
Porosity, $\text{cm}^3 \cdot \text{cm}^{-3}$	0.65–0.70	0.55	0.58–0.63
Bulk density, $\text{g} \cdot \text{cm}^{-3}$	0.62–0.63	0.62	0.61
Particle size, mm	0.3–1.2	0.3–1.2	0.3–1.2

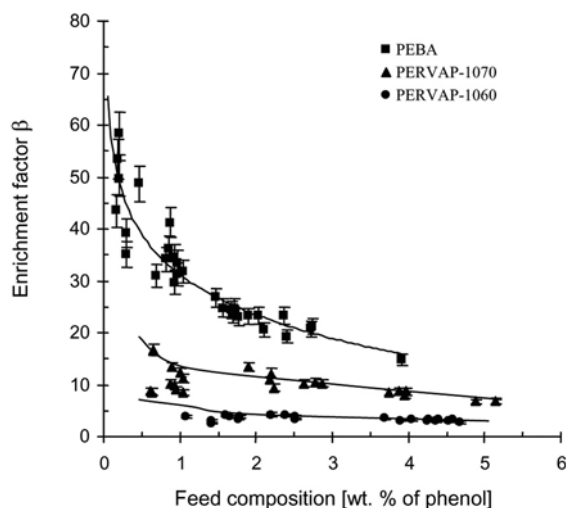


Fig. 3. Enrichment  $\beta$  factor of hydrophobic membranes investigated in contact with binary water–phenol mixtures.

In contact with aqueous phenol solutions, the PEBA membrane, made of poly(ether block amide) polymer, showed the highest selectivity. Both PDMS membranes were less selective, however the zeolite filling of PERVAP–1070 membrane improved the selectivity of this membrane (Figs. 2,3). The high selectivity of PEBA membranes in contact with water–phenol mixtures have been already reported by Kondo et al. [13] and by Boeddeker et al. [14]. The enrichment factor  $\beta$  (i.e. a ratio of mass fractions of the component preferentially transported in permeate and in feed, respectively) [7] found for the PEBA membrane (Fig. 4) was in the same range as found by Boeddeker et al. [14].

The transport properties of the investigated membranes in contact with water–phenol mixtures are presented in Figs. 4,5. The permeate flux of the organic component through the membranes was linearly dependent on the feed composition (Fig. 4). The highest flux of phenol was found for the PEBA membrane, e.g. for this membrane in contact with 2 wt.% phenol feed solution the phenol permeate flux was  $0.150 \text{ kg m}^{-2} \text{ h}^{-1}$  (Fig. 4). The permeate flux of water was also substantial

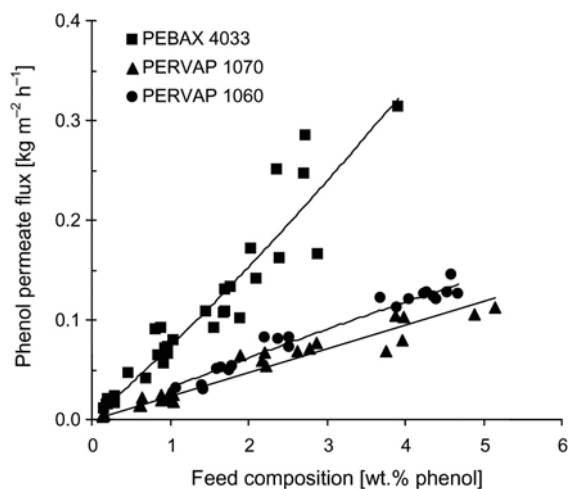


Fig. 4. Permeate phenol flux through the investigated membranes investigated in contact with binary water–phenol mixtures.

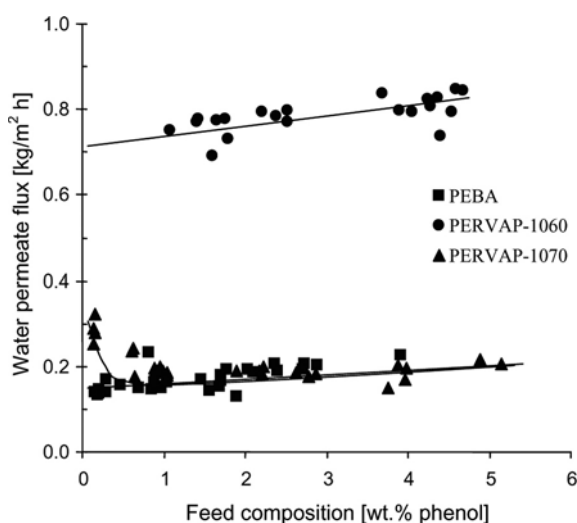


Fig. 5. Permeate water flux through the investigated membranes in contact with binary water–phenol mixtures.

(around  $0.2 \text{ kg m}^{-2} \text{ h}^{-1}$  for PEBA and PERVAP–1070 membranes and  $0.6\text{--}0.8 \text{ kg m}^{-2} \text{ h}^{-1}$  for the PERVAP–1060 membrane) but practically independent on the feed composition (Fig. 5).

The detailed comparison of fluxes of water and phenol molecules through both PERVAP–1060

(i.e. PDMS membrane) and PERVAP–1070 (i.e. zeolite-filled PDMS membrane) allowed explanation of the role of zeolite filling in the separation of water–phenol mixtures (Figs. 2–5). In general, a decrease of water flux would be expected due to the increase in the diffusion pathway through the polymer matrix, in the presence of the hydrophobic zeolite particles. On the other hand, the flux of organic component can increase or decrease, depending on the balance between the loss in flux due to the increase of a tortuosity pathway and augmentation of organic compound sorption in the zeolite particles [15]. In the case of water–phenol mixture, zeolite fillings caused the substantial decrease of the water flux only (Fig. 5), whereas the flux of phenol remained practically unaltered (Fig. 4). As a consequence, the selectivity of the PERVAP–1070 membrane exceeded that of the PERVAP–1060 one.

### 3.2. Membrane-based solvent extraction

The phenol concentration in an organic phase after single-stage extraction as a function of the phenol concentration in aqueous phase is presented in Fig. 6. The distribution coefficients calculated from slope of these straight lines are collected in Table 5. MTBE was found the best extraction

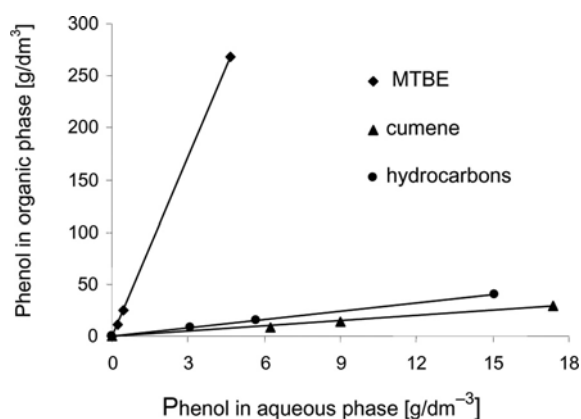


Fig. 6. Extraction equilibria in systems with various extraction solvents: MTBE, cumene and the mixture of hydrocarbons.

Table 5

Distribution coefficient of phenol between water and the extraction solvents

Extraction solvent	Distribution coefficient
MTBE	57.4
The mixture of hydrocarbons	2.7
Cumene	1.6

solvent. The distribution coefficients for cumene and the mixture of hydrocarbons were comparable.

In Fig. 7 the dephenolization degree DG [%] was plotted against volumetric flow of aqueous feed solution ( $Q_w$ ). The dephenolization degree was calculated according to the following equation:

$$DG = \frac{c_w^{\text{in}} - c_w^{\text{out}}}{c_w^{\text{in}}} 100\% \quad (1)$$

where  $c_w^{\text{in}}$ ,  $c_w^{\text{out}}$  are phenol concentrations in the aqueous phase at the beginning and the end of the contactor, respectively, g·dm<sup>-3</sup>. As expected from the comparison of the distribution coefficients (Table 5) MTBE was the most efficient extractant of phenol. Over 98% of phenol was removed in a single-stage extraction carried out at  $Q_w \approx 1.5 \text{ cm}^3 \cdot \text{s}^{-1}$ . The dephenolization degree decreased

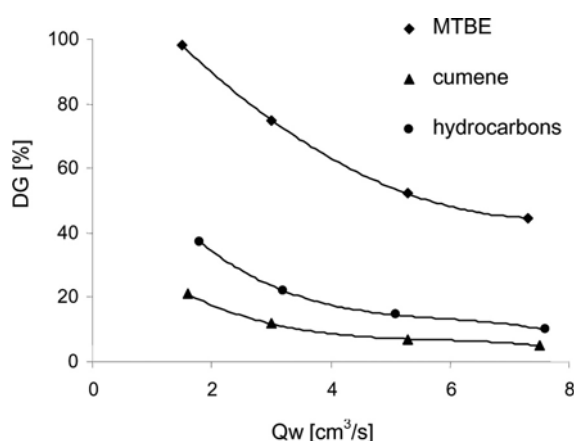


Fig. 7. Dephenolization degree (DG) vs. volumetric aqueous solution flow ( $Q_w$ ). The volumetric flow of the extraction solvent was equal  $0.33 \text{ cm}^3 \cdot \text{s}^{-1}$ .



to 45% when  $Q_w$  increased to  $\approx 7.5 \text{ cm}^3 \cdot \text{s}^{-1}$ . For the mixture of hydrocarbons 200 and cumene as extractants, the DG varied within 180 the range 37–10% and 21–5%, respectively.

The length of transfer unit (LTU) as a function of volumetric aqueous solution flow ( $Q_w$ ) is presented in Fig. 8. The term length of transfer unit corresponds to the height of transfer unit used in classical solvent extraction with packed towers. It can be regarded as a measure of the dimensions of the contactor. The lower LTU is the smaller contactor is needed. Generally, the lower distribution coefficient of water–extraction solvent system the higher the length of the transfer unit. For MTBE LTU ranged 7–22 cm. For other extraction solvents used it was twice (the mixture of hydrocarbons) or even about 9 times (cumene) higher.

The results suggested that membrane-based solvent extraction using MTBE would be the best solution. However, it should be taken into account that after the extraction step it would be necessary either to return the mixture phenol–extraction solvent to the cumene oxidation process or to separate it in order to recycle the extraction solvent. One should also remember that MTBE dissolves relatively well in water (up to 5%) and therefore should be regarded as a potential pollutant [16,17].

### 3.3. Adsorption

#### 3.3.1. Equilibrium adsorption

Sorption isotherms of phenol on the Amberlite resins (XAD series) are plotted in Fig. 9 enabling the comparison of properties of various adsorbents and the choice of the most efficient one. The Amberlite XAD-4 and XAD-16 resins made of styrene-divinylbenzene copolymer showed higher affinity to phenol than the XAD-7 one with methyl acrylate-divinylbenzene copolymer matrix. The poly(styrene-divinylbenzene) resins were known as efficient phenol adsorbent [18]. Amberlite XAD-4 was successively used for adsorption of various organic compounds [19, 20], among them aromatic ones [21,22]. Our results confirmed also

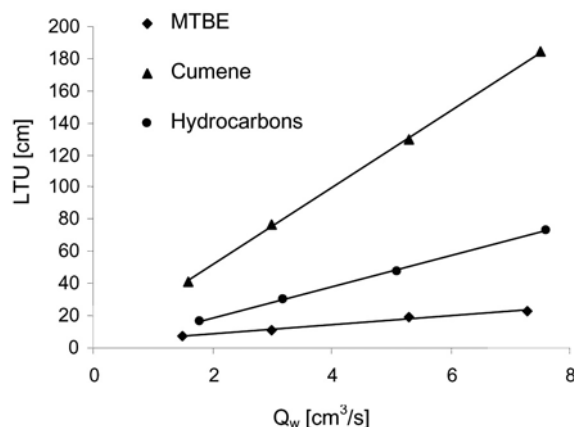


Fig. 8. Length of transfer unit (LTU) vs. volumetric aqueous solution flow ( $Q_w$ ). The volumetric flow of the extraction solvent was equal  $0.33 \text{ cm}^3 \cdot \text{s}^{-1}$ .

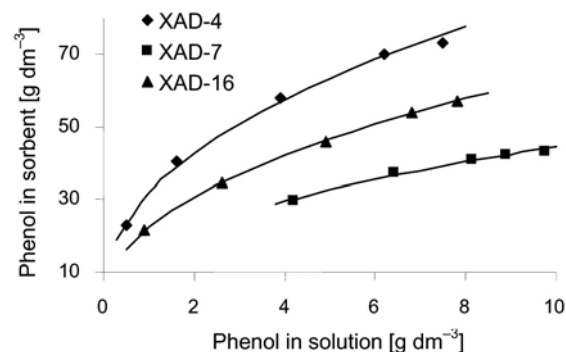


Fig. 9. Adsorption isotherms of phenol on the Amberlite resins of various grades.

the earlier results obtained by Li et al. for sorption of phenol traces by Amberlite XAD-4 (phenol concentration below  $1 \text{ g} \cdot \text{dm}^{-3}$ ) [23, 24]. Additional experiments, which were carried out for ternary solutions containing various amounts of sodium sulphate (up to  $50 \text{ g} \cdot \text{dm}^{-3}$ ), indicated that phenol sorption on the Amberlite XAD-4 increased with the increase of  $\text{Na}_2\text{SO}_4$  content in solution.

#### 3.3.2. Adsorption under dynamic conditions

Adsorption under dynamic conditions was performed in order to investigate properties of the chosen adsorbent Amberlite XAD-4 during



adsorption and regeneration steps. The solutions modelling partially dephenolized wastes contained phenol ( $3\text{--}5\text{ g}\cdot\text{dm}^{-3}$ ) and sodium sulphate ( $30\text{ g}\cdot\text{dm}^{-3}$ ). Four kinds of incombustible liquors were used to regenerate the resin bed.

The breakthrough curve for the adsorption step enabled to estimate the volume of the eluate in the breakthrough point ( $V_s$ ), which corresponded to the maximum volume of decontaminated eluate. On the other hand, the regeneration (elution) curve indicated the volume of a regenerant ( $V_R$ ) at which pollutant concentration decreased to a desired low value. This volume of regenerant was necessary to elute phenol out from the bed resin. The difference  $\Delta V = V_s - V_R$  was regarded as a measure of the efficiency of the regenerant used. The higher  $\Delta V$  value was, the more efficient the regenerant was.

The breakthrough and regeneration curves obtained at 368 K using water, sodium sulphate solution ( $30\text{ g}\cdot\text{dm}^{-3}$ ) and treated model solution (with phenol concentration of  $0.08\text{ g}\cdot\text{dm}^{-3}$ ) are presented in Fig. 10. Phenol concentration in the feed was equal  $5\text{ g}\cdot\text{dm}^{-3}$ . It was found that the highest volume of the treated waste ( $\Delta V \approx 6\text{ BV}$ )

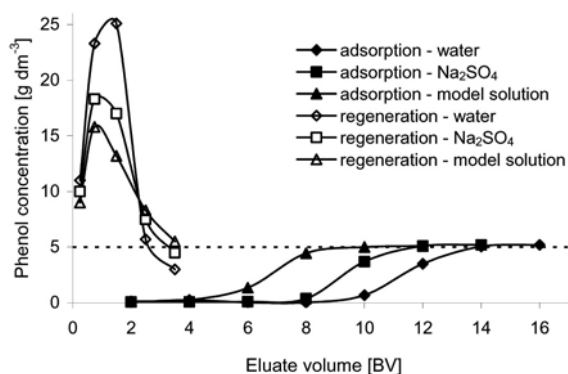


Fig. 10. The breakthrough curve for adsorption and the regeneration (elution) curve. The Amberlite XAD-4 bed regenerated at 368 K using water, sodium sulphate solution and treated model solution. The dashed line indicates phenol concentration in the feed ( $5\text{ g}\cdot\text{dm}^{-3}$ ). The eluate volume on the abscissa axis is expressed in the volume of resin bed (BV).

was obtained when the adsorbent bed was regenerated with hot water. On the other hand sodium sulphate solution and treated model solution were the less efficient regenerants ( $\Delta V$  approximately equal to 4 BV and 1 BV, respectively).

The regeneration step can be carried out efficiently with hydroxide solution due to the chemical reaction between phenol and hydroxide. The phenolate formed was not adsorbed on the resin and as the consequence the low concentration of phenol in solution changed the sorption–desorption equilibrium enhancing desorption of phenol from the bed. It is seen from Fig. 11 that about 8 BV of the model waste solution (i.e. solution containing phenol at the concentration  $3\text{ g}\cdot\text{dm}^{-3}$ ) could be purified in one adsorption–regeneration cycle. Moreover it would be possible to use sodium hydroxide solution ( $200\text{ g}\cdot\text{dm}^{-3}$ ) several times as an efficient regenerant, which could additionally decrease the total volume of the concentrate. The breakthrough curves proved also that the repeatedly usage of sodium hydroxide solution did not influence the efficiency of the adsorption of phenol during the next adsorption steps (Fig. 11).

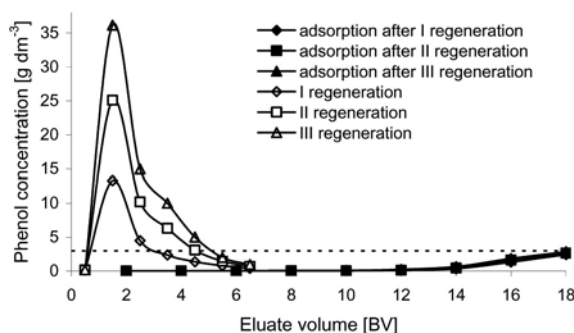


Fig. 11. The breakthrough curve for adsorption and the regeneration (elution) curve. The Amberlite XAD-4 bed regenerated at 333 K using sodium hydroxide solution ( $200\text{ g}\cdot\text{dm}^{-3}$ ). The dashed line indicates phenol concentration in the feed ( $3\text{ g}\cdot\text{dm}^{-3}$ ). The eluate volume on the abscissa axis is expressed in the volume of resin bed (BV).

#### 4. Conclusions

Poly(ether block amide) membrane (PEBA) had the best separation properties in the pervaporative removal of phenol from water. Partial permeate fluxes of phenol through both PDMS membranes were practically the same. It means that the presence of zeolite filling of PERVAP-1070 membrane did not influence the phenol transport. On the other hand flux of water was much smaller through the PDMS membrane with the zeolite filling (PERVAP-1070) compared to the pure PDMS membrane (PERVAP-1060).

The results obtained in the solvent-based membrane extraction showed that extraction of phenol using MTBE gave the best results for this technique. However, one should remember that after extraction step it would be necessary either to return the mixture phenol–extraction solvent to the cumene oxidation process or to separate it and recycle the extraction solvent. For that reason, in the suggested hybrid system only membrane pervaporation followed by adsorption step was taken into account (Fig. 12).

The best sorption properties were found in the case of the Amberlite XAD-4. Using this adsorbent, made of cross-linked styrene/divinylbenzene copolymer, the removal of phenol from model solution containing phenol (3–20 g/dm<sup>3</sup>) and sodium sulphate (30 g/dm<sup>3</sup>) was carried out

by the column method. Non-combustible liquors: distilled water, sodium sulphate solution (30 g/dm<sup>3</sup>), eluate and sodium hydroxide solution (200 g/dm<sup>3</sup>) were used to regenerate the sorbent bed. It was found that the regeneration of the sorbent bed using NaOH solution enabled the most effective removal of phenol from the low concentrated solutions.

Pervaporation and adsorption results obtained with water-phenol mixtures suggested that the hybrid system, schematically presented in Fig. 12, could be used to the efficient decontamination of the effluents from the cumene oxidation process.

#### 5. Symbols

$c$	— Concentration, g·dm <sup>-3</sup>
$J_k$	— Permeate flux of species $i$ , kg·m <sup>-2</sup> ·h <sup>-1</sup>
$k, n$	— Coefficients of the Freundlich equation
$Q_n$	— Volumetric flow of extraction solvent, cm <sup>3</sup> ·s <sup>-1</sup>
$Q_w$	— Volumetric flow of aqueous solution, cm <sup>3</sup> ·s <sup>-1</sup>
$q$	— Adsorption capacity, g·dm <sup>-3</sup>
BV	— Volume of resin bed
LTU	— Length of transfer unit, cm
$\alpha$	— Separation factor
$\beta$	— Enrichment factor

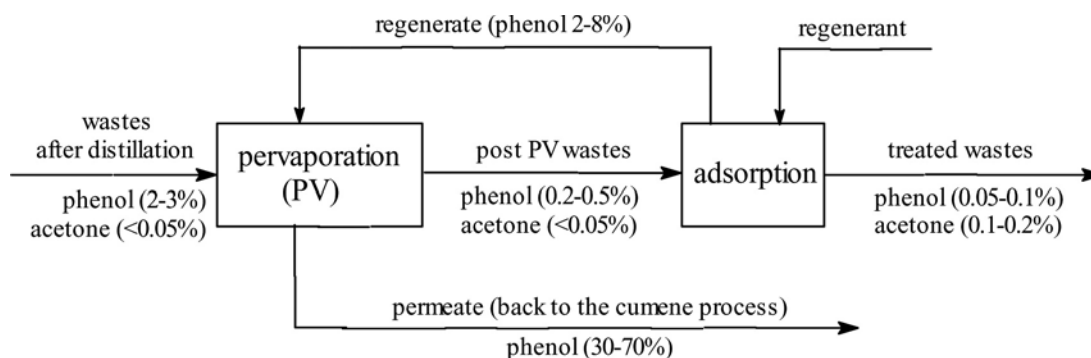


Fig. 12. The proposed hybrid pervaporation–adsorption process for treatment of wastewaters containing phenol.

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