

Selective Adsorption of Organic Compounds from Solutions on Hyper-Cross-Linked Polystyrenes with Ultimate Degrees of Cross Linking

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Abstract—The adsorption selectivities of a series of organic compounds from solutions on hyper-cross-linked polystyrene networks (HPNs) with ultimate degrees of cross linking were studied by high performance liquid chromatography (HPLC). The thermodynamic characteristics of adsorption (TCA) of substances under study from water–organic solutions on such HPNs in the Henry region were determined. The TCAs of organic compounds on the HPNs under study as a function of their molecular structures are discussed. It was established that the magnitude of the Gibbs energy change upon adsorption increases with increasing the number of π -electrons in the adsorbed molecules in a series of both aromatic hydrocarbons and their sulfur-containing heterocyclic analogs. As an example, a model mixture of organic compounds was separated on HPNs by HPLC. It was shown that middle and heavy oil fractions can be fractionated on HPNs into the groups of mono-, di-, and tricyclic aromatic compounds.

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INTRODUCTION

At the present time, high-performance liquid chromatography (HPLC) is wide-spread both in the practice of analytical chemistry and in physicochemical investigations aimed at the study of processes at the solution–solid interface [1]. Further progress in the analytical possibilities of HPLC is directly related to the design of new high-selectivity adsorbents allowing separation of mixtures of substances, which were difficult to separate previously. Among such promising materials that can be used as stationary phases in HPLC are hyper-cross-linked polystyrenes. A considerable number of papers [2–7] deal with the adsorption properties of hyper-cross-linked polystyrene networks (HPNs) in liquid-phase systems. Also, works where authors studied the adsorption properties of HPNs modified with ethylene diamine, aniline, and sulfo groups [8–11] appeared in recent years. The study of adsorption from a solution on HPNs allowed deep insight into physicochemical regularities forming the basis for adsorption from a liquid phase on these materials [12, 13]. Based on analysis of the compensation effect upon adsorption of heterocyclic compounds on hyper-cross-linked polystyrene, we have shown that the mechanism of retention on this adsorbent depends on the capability of adsorbed molecules

to enter into π - π - and π - d -interactions with its aromatic fragments [13].

One of the trends in subsequent development of the themes associated with adsorption application of HPNs is application of hyper-cross-linked polystyrene with extremely high degrees of cross linking in selective adsorption and HPLC [14, 15]. These adsorbents, in contrast to hyper-cross-linked polystyrenes with low and moderate degrees of cross linking, are characterized by a high rigidity of openwork three-dimensional network and specific donor-acceptor properties [5]. Such HPNs represent a class of polymeric adsorption materials that have not been studied earlier as stationary phases in liquid chromatography. For this reason, physicochemical regularities forming the basis for adsorption on these adsorbents are unknown and, as a consequence, no methods for controlling their adsorption selectivities are developed. Therefore, it is necessary to perform detailed thermodynamic studies of adsorption from solutions on HPNs with ultimate degrees of cross linking.

The aim of the present work is to study selective adsorption of a series of organic compounds from solutions on hyper-cross-linked polystyrenes with ultimate degrees of cross linking and separation of model and real mixtures of substances on these sorbents.

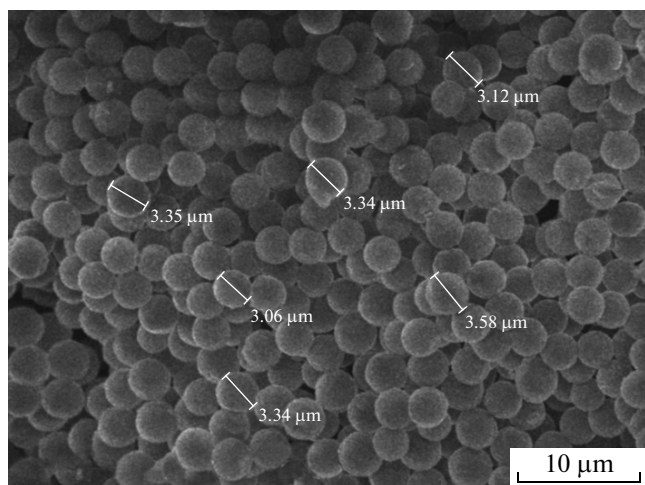


Fig. 1. Electron photomicrograph of the hyper-cross-linked polystyrene powder with $X = 500\%$.

EXPERIMENTAL

Hyper-cross-linked polystyrene adsorbents with nominal degrees of network cross linking of 300, 400, and 500% were prepared by additional cross linking of dichloroethane-swollen monodisperse microspherical beads of styrene–divinylbenzene copolymer by its reaction with 1.5, 2.0, and 2.5 mmol of chloromethyl methyl ether, respectively. In the presence of Friedel–Crafts catalyst, each molecule of a cross linking agent combines two spatially approximated phenyl rings of the polymer through the methylene group. Theoretically, at the degrees of cross linking of 300, 400, and 500%, all phenyl rings of the starting polystyrene chains must become tetra-, penta-, and six-substituted.

Hyper-cross-linked polystyrenes with degrees of cross linking $X = 300, 400,$ and 500% were packed by the suspension method into steel chromatographic columns with a diameter of 3 mm ($X = 300, 500\%$) and 4 mm ($X = 400\%$) and a length of 50 mm. The columns were packed using suspensions of polymers in *n*-heptane. The adsorbent weights in the column were equal to 0.0922 g ($X = 300\%$), 0.1639 g ($X = 400\%$), and 0.0922 g ($X = 500\%$). It follows from the photomicrographs of studied HPN powders obtained on a JSM-6390A scanning electron microscope (JEOL, United States) that the adsorbents under study have regular-shaped spherical particles with a size of $\sim 3.5 \mu\text{m}$ (Fig. 1). The apparent specific internal surface areas of HPNs under study determined according to the BET theory by the thermal desorption of argon in the helium flow were $880 \text{ m}^2/\text{g}$ ($X = 300\%$), $1100 \text{ m}^2/\text{g}$ ($X = 400\%$), and $684 \text{ m}^2/\text{g}$ ($X = 500\%$). The polymeric adsorbents under study were characterized by developed microporous structure [15].

Selective adsorption of a series of organic compounds (Table 1) from water–organic media on polymeric adsorbents was studied by HPLC in the temperature range of 313–333 K at 10 K intervals under

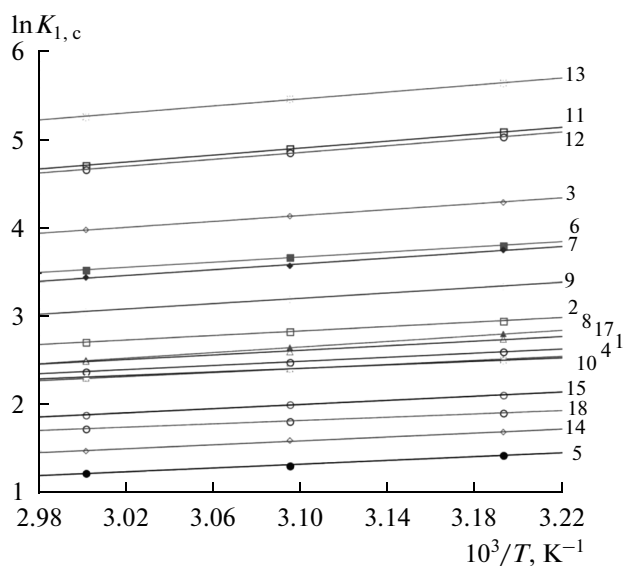

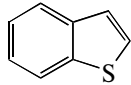
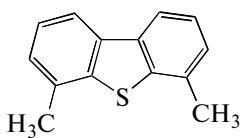
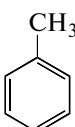
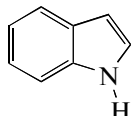
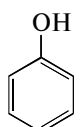
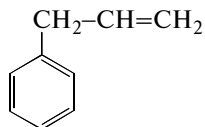
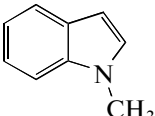
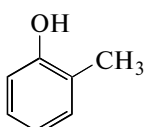

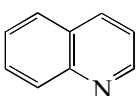
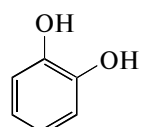

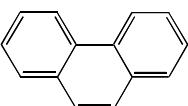
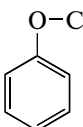
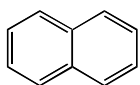
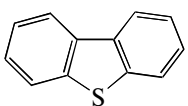
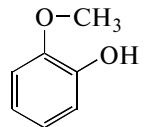


Fig. 2. Plots of the logarithmic Henry constant for adsorption of compounds under study from a MeCN–H₂O solution (60 : 40) on the hyper-cross-linked polystyrene with $X = 300\%$ as a function of reciprocal temperature.

dynamic conditions. The chromatographic equipment is described in details in [16]. All aromatic compounds under study and organic solvents for the preparation of mobile phases (MeCN, *i*-PrOH, MeOH) were commercially available reagents. Water for the preparation of mobile phase was obtained using a DME-1/B deionization device (BMT, Russia). In the study of adsorption thermodynamics, the mobile phases were MeCN–H₂O with a MeCN concentration Φ_{MeCN} from 0.40 to 0.70 parts by volume, as well as *i*-PrOH–H₂O and MeOH–H₂O with an organic component concentration $\Phi_{i\text{-PrOH}} = \Phi_{\text{MeOH}} = 0.60$ parts by volume. The flow rate of mobile phase was 500 $\mu\text{L}/\text{min}$. The void volume of the column was equal to 277.5 μL ($X = 300\%$), 375 μL ($X = 400\%$), and 277 μL ($X = 500\%$). Extremely diluted solutions of adsorbates in the corresponding mobile phase were used as the samples.

Based on the data from HPLC, the Henry adsorption constant $K_{1,c}$ was calculated by the known formula [17]. The thermodynamic characteristics of adsorption (TCA) of compounds under study from water–organic solutions on the studied HPN samples with ultimate degrees of cross linking were calculated from the plot of logarithmic Henry adsorption constant $\ln K_{1,c}$ against reciprocal temperature $1/T$. Coefficients of determination r^2 for the corresponding dependences $\ln K_{1,c} - 1/T$ were at least 0.99 (Fig. 2). The calculated TCAs from a MeCN–H₂O solution with $\Phi_{\text{MeCN}} = 0.60$ on the HPNs with $X = 300, 400,$ and 500% at the average experimental temperature $T_{\text{av}} = 323 \text{ K}$ are given in Table 2.

Table 1. Structural formulas of compounds under study

No.	Structural formula	No.	Structural formula	No.	Structural formula
1	 Benzene	7	 Benzothiophene	13	 4,6-Dimethyldibenzothiophene
2	 Toluene	8	 Indole	14	 Phenol
3	 Allylbenzene	9	 1-Methylindole	15	 <i>o</i> -Cresol
4	 Thiophene	10	 Quinoline	16	 Pyrocatechol
5	 Pyridine	11	 Phenanthrene	17	 Anisole
6	 Naphthalene	12	 Dibenzothiophene	18	 Guaiacol

RESULTS AND DISCUSSION

It follows from the negative values of the Gibbs energy change upon adsorption of compounds under study from a MeCN–H₂O solution with $\Phi_{\text{MeCN}} = 0.60$ on HPNs with ultimate degrees of cross linking (Table 2) that the liquid-phase adsorption equilibrium on the polymer adsorbents under study is shifted in favor of the formation of adsorption complexes in the case of all adsorbed substances and even in the case of highly polar phenols (adsorbates **14–18**), the molecules of which are the best soluble in water–organic solvents. The adsorption of organic molecules on such HPNs is exothermic in its nature (Table 2). Due to the fact that the changes in the entropy upon adsorption on the HPNs under study are negative virtually for all sub-

stances, the driving force for adsorption is obviously a more significant negative change in the enthalpy.

Of note is the specificity of the effects of molecular structures of adsorbates on the TCA from a MeCN–H₂O solution with $\Phi_{\text{MeCN}} = 0.60$ on the HPNs with $X = 300, 400,$ and 500% (Table 2). For example, the increase in the number of aromatic rings in the adsorbed molecules results in an increase in Henry adsorption constant $K_{1,c}$ and, consequently, in the magnitude of Gibbs energy change upon adsorption $|\Delta_a G^*|$. Such an increase in the above-mentioned TCA is likely caused by the effect of enthalpy factor, since it is the $|\Delta_a H^*|$ value, which changes generally in a sym-bate mode with increasing the number of aromatic rings, while the $|\Delta_a S^*|$ value is less susceptible to the

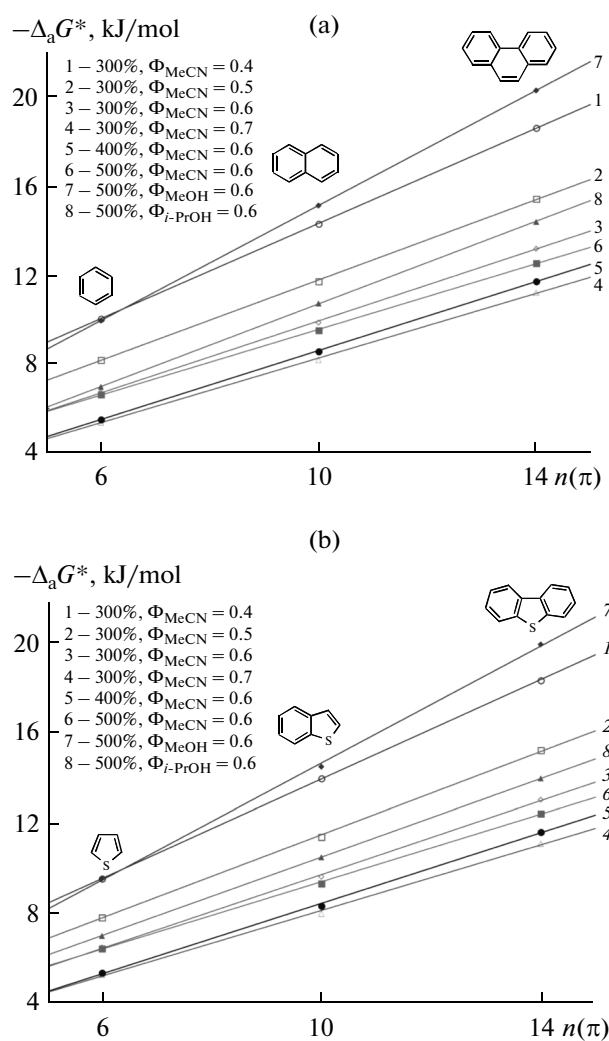


Fig. 3. Plots of the Gibbs energy change upon adsorption of (a) aromatic hydrocarbons and (b) their sulfur-containing heterocyclic analogs from water–organic solutions of different compositions on hyper-cross-linked polystyrenes with different X at $T_{av} = 323$ K vs. the number of π -electrons in the molecule.

change in the molecular structures of adsorbates (Table 2).

Figure 3 shows the plots of the Gibbs energy change upon adsorption of aromatic hydrocarbons and their sulfur-containing heterocyclic analogs from water–organic solutions of various composition on the HPNs with $X = 300, 400,$ and 500% against the number of π -electrons in the molecules of adsorbates. As follows from these plots, the enhancement in the adsorption with increasing the number of aromatic rings in the molecules is caused by the increase in the number of π -electrons in the molecules of compounds under study, which directly points out the defining role of adsorbate–adsorbent π - π interactions in the energetics of adsorption on the HPNs under study. Probably, the increase in the energy of π - π interactions of adsorbate with the aromatic fragments of polymer net-

work due to electron density redistribution in the molecule also causes multiple increase in the $K_{1,c}$ value for the HPN with $X = 300\%$ from 12.2 to $63.6 \mu\text{L}/\text{m}^2$ on going from benzene (adsorbate **1**) to allylbenzene (adsorbate **3**).

The fact that electron density redistribution in the aromatic fragment of molecules plays a significant role in their adsorptions on the HPNs also follows from the following examples. Upon passing from benzene (adsorbate **1**) to toluene (adsorbate **2**), the $K_{1,c}$ value for the HPN with $X = 300\%$ changes from 12.2 to $17.2 \mu\text{L}/\text{m}^2$. The effect of methyl radical on the adsorption on the HPN is also exemplified by a strong change in the $K_{1,c}$ value from 129.4 to $237.2 \mu\text{L}/\text{m}^2$ on passing from dibenzothiophene (adsorbate **12**) to 4,6-dimethyldibenzothiophene (adsorbate **13**). Such a noticeable change in the adsorption interaction force due to introduction of methyl into the adsorbate molecule can be understood taking into account the electron-donating properties of methyl radical, which increases the electron density in the aromatic fragment of the molecule and, consequently, increases the adsorbate–adsorbent π - π interactions.

High adsorption selectivities of the HPNs with ultimate degrees of cross linking with regard to compounds differing in the number of π -electrons in their molecules are demonstrated by the chromatograms shown in Fig. 4. For example, we succeeded in separating a model mixture consisting of acetone, benzene, naphthalene, and anthracene within 10–15 min on the columns with the HPNs under study. Moreover, Fig. 4 shows that the chromatographic systems under study have a certain reserve selectivity allowing also separation of more complex mixtures including, for example, alkylated analogs of the compounds under consideration.

The HPNs under study were also found to possess certain adsorption selectivity with regard to aromatic hydrocarbons and their sulfur-containing analogs. This is apparent from the comparison of the plots of Gibbs energy change upon adsorption versus the number of π -electrons in the molecules of adsorbates (Fig. 5). It follows from Fig. 5 that the above-mentioned selectivity remains for both monocyclic and bi- and tricyclic aromatic compounds. While in the case of quasi-normal-phase HPLC on the HPNs the sulfur-containing analogs were typically retained more strongly than the corresponding aromatic hydrocarbons, which we associated to the adsorbate–adsorbent π - d interactions [4], conversely, in the reversed-phase HPLC, the sulfur-containing analogs were retained more weakly than the corresponding aromatic hydrocarbons (Fig. 5, Table 2). This fact is likely caused by the presence of dipole moment in the sulfur-containing heterocyclic compounds (adsorbates **4, 7, 12, 13**) in contrast to the corresponding aromatic hydrocarbons (adsorbates **1, 6, 11**) and, as a consequence, by the orientation interactions of these heterocycles with components of water–organic solvents.

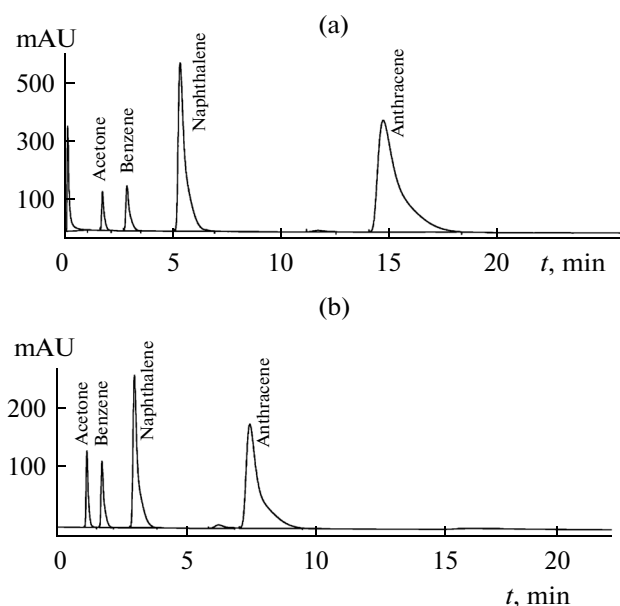


Fig. 4. Chromatograms for a model mixture of compounds on the hyper-cross-linked polystyrenes with $X =$ (a) 400 and (b) 500%. Separation conditions: the mobile phase was MeCN–H₂O (90 : 10), the flow rate was 0.3 mL/min, the UV detection wavelength was 254 nm, and the column temperature was 20°C.

Specific intermolecular interactions of adsorbates with mobile phase components are likely responsible for weaker retentions of pyridine (adsorbate **5**) compared to benzene (adsorbate **1**) and quinoline (adsorbate **10**) compared to naphthalene (adsorbate **6**), as well as highly polar phenols (adsorbates **14–16**, and **18**) compared to benzene (adsorbate **1**). Substances with molecules that contain the pyridine nitrogen atom (adsorbates **5** and **10**) and phenols with molecules that include a labile proton (adsorbates **14–16**, and **18**) are probably involved in hydrogen bonding with the molecules of both water and mobile phase organic components.

The hydrogen bonding between the molecules of adsorbate and components of water–organic solvent also results in a smaller adsorption of indole (adsorbate **8**), for example, compared to naphthalene (adsorbate **6**) and benzothiophene (adsorbate **7**). The matter is that the indole molecule contains a labile pyrrole hydrogen atom that possesses acidic properties and, therefore, can form hydrogen bonds with the components of used water–organic solvents to increase thereby the affinity of indole for the mobile phase compared to naphthalene and benzothiophene (Table 2). Blocking of the acidic properties of the indole molecule by substitution of methyl for the hydrogen atom results in a noticeable increase in the $K_{1,c}$ value on passing from indole (adsorbate **8**) to 1-methylindole (adsorbate **9**) from 14.3 to 24.7 $\mu\text{L}/\text{m}^2$ (Table 2). The same increase in the adsorption properties due to substitution of methyl for the labile proton

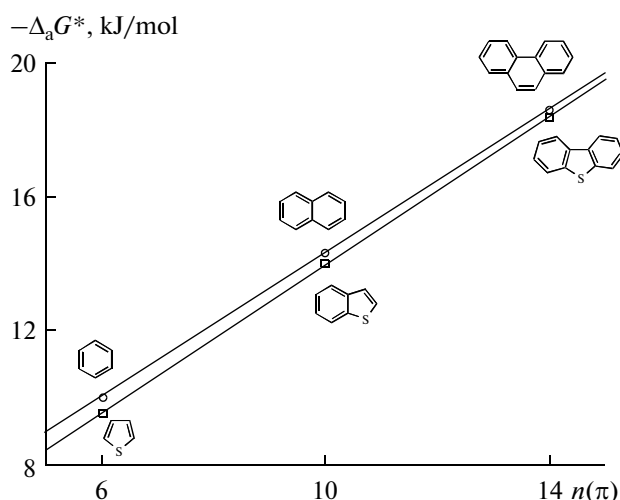


Fig. 5. Adsorption selectivity upon separation of aromatic hydrocarbons and their sulfur-containing heterocyclic analogs on the hyper-cross-linked polystyrene with $X = 300\%$ upon elution with the MeCN–H₂O (40 : 60) mobile phase at $T_{av} = 323$ K.

in the adsorbate molecule is demonstrated by the increase in the $K_{1,c}$ value on passing from phenol (adsorbate **14**) to anisole (adsorbate **17**) from 5.0 to 13.7 $\mu\text{L}/\text{m}^2$ (Table 2).

The fact that the selectivity of an HPN-based HPLC system can be changed additionally by varying the composition and nature of a mobile phase allows performing such separation that cannot be performed in principle upon adsorption from the gas phase. For example, Fig. 6 shows the example of adsorption separation of middle and heavy oil fractions, i.e., straight-run diesel oil cut and light catalytic gas oil, into the groups of mono-, bi-, and tricyclic aromatic compounds. The patterns of elution curves given demonstrate the absence of selectivity of chromatographic systems used towards homologs of aromatic compounds, which is suppressed considerably due to the fact that the increase in the energy of dispersion interaction between adsorbate and HPN with elongating the hydrocarbon chain is compensated by the increase in the energy of dispersion interaction between the adsorbate and nonpolar fragments of components of the *i*-PrOH–CH₂Cl₂ (80 : 20) mobile phase. As a result, the HPLC system loses selectivity with regard to homologs, since the tendencies mentioned above act in opposite directions and the defining role upon separation is played by the adsorbate–adsorbent π - π interactions. Thus, Fig. 6 shows that it is possible in principle to fractionate completely the oil fractions into the groups of mono-, bi-, and tricyclic aromatic compounds on the HPN columns in 30 min.

CONCLUSIONS

We studied the adsorption selectivities of a series of organic compounds from solutions on hyper-cross-

Table 2. Thermodynamic characteristics of adsorption of compounds under study from a MeCN–H₂O solution (60 : 40) on hyper-cross-linked polystyrenes with ultimate degrees of cross linking at $T_{av} = 323$ K

No.	X = 300%				X = 400%				X = 500%			
	$-\Delta_a H^*$, kJ/mol	$-\Delta_a S^*$, J/(mol K)	$-\Delta_a G^*$, kJ/mol	$K_{1,c}$, $\mu\text{L}/\text{m}^2$	$-\Delta_a H^*$, kJ/mol	$-\Delta_a S^*$, J/(mol K)	$-\Delta_a G^*$, kJ/mol	$K_{1,c}$, $\mu\text{L}/\text{m}^2$	$-\Delta_a H^*$, kJ/mol	$-\Delta_a S^*$, J/(mol K)	$-\Delta_a G^*$, kJ/mol	$K_{1,c}$, $\mu\text{L}/\text{m}^2$
1	9.7	9.2	6.7	12.2	8.6	9.4	5.5	7.8	9.1	7.7	6.6	11.7
2	10.6	9.2	7.6	17.2	9.3	9.0	6.4	10.8	9.8	7.3	7.4	15.8
3	13.9	8.6	11.1	63.6	13.3	11.0	9.8	38.1	13.4	8.9	10.6	51.4
4	9.5	9.4	6.5	11.3	8.5	9.8	5.4	7.4	8.7	7.0	6.5	11.1
5	8.9	16.5	3.6	3.8	—	—	1.8	2.0	—	—	3.1	3.2
6	12.1	6.8	9.9	39.6	11.4	8.7	8.6	24.3	11.9	7.5	9.5	34.5
7	13.7	12.4	9.6	36.2	11.2	8.8	8.3	22.4	11.5	6.7	9.3	32.2
8	13.1	18.4	7.1	14.3	10.7	14.9	5.9	8.8	10.7	11.5	7.0	13.6
9	12.5	12.1	8.6	24.7	10.4	9.8	7.3	15.1	12.2	12.1	8.3	22.2
10	8.2	5.4	6.5	11.3	4.5	−1.2	4.9	6.1	5.5	−1.2	5.9	9.1
11	16.4	9.9	13.2	135.7	14.5	8.6	11.7	78.6	15.4	8.8	12.5	106.0
12	16.1	9.5	13.1	129.4	14.9	10.0	11.6	76.4	15.0	7.9	12.5	103.4
13	16.5	5.5	14.7	237.2	15.7	8.2	13.1	131.8	14.3	1.7	13.8	167.7
14	9.2	15.3	4.3	5.0	8.0	15.0	3.2	3.3	6.4	6.3	4.4	5.1
15	9.8	13.6	5.4	7.5	8.2	12.3	4.2	4.8	7.0	5.2	5.3	7.1
16	—	—	1.3	1.6	—	—	0.9	1.4	—	—	1.5	1.8
17	10.8	11.7	7.0	13.7	9.5	11.6	5.7	8.5	8.4	5.0	6.8	12.6
18	7.8	8.9	4.9	6.2	8.7	14.5	4.0	4.4	6.0	3.4	4.9	6.2

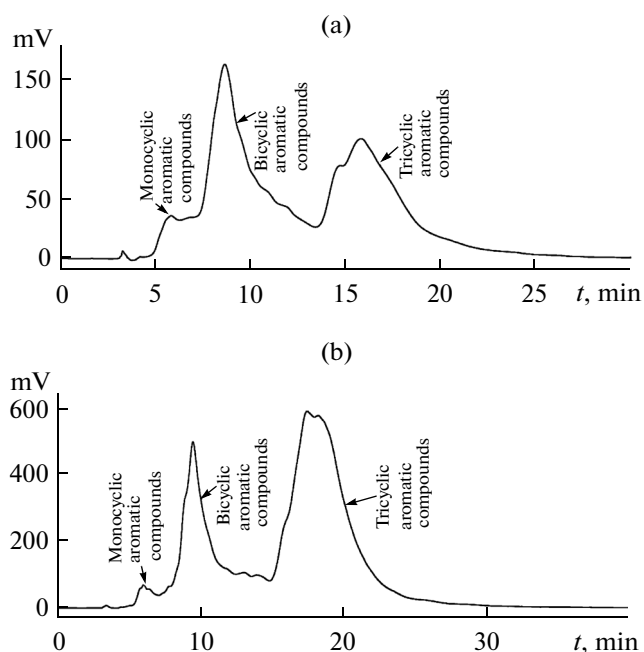


Fig. 6. Adsorption separation of the (a) straight-run diesel fraction and (b) light catalytic gas oil into the groups of mono-, bi-, and tricyclic aromatic compounds on the hyper-cross-linked polystyrene with $X = 150\%$. Separation conditions: the column dimensions were 4.6×250 mm, the mobile phase was i -PrOH– CH_2Cl_2 (80 : 20), the flow rate was 1 mL/min, the UV detection wavelength was 254 nm, the column temperature was 35°C , and the dilution ratio of samples under study in n -hexane was 1 : 100 by volume.

linked polystyrenes with ultimate degrees of cross linking by HPLC. The adsorption selectivities of polymeric materials under study was found to be determined mainly by π - π interactions between the adsorbate molecules and the aromatic fragments of openwork three-dimensional network. Of primary importance in separation is the nature and composition of the mobile phase. The unique adsorption selectivities of studied polymer networks open the way for separation of oil fractions thereon into the groups of mono-, di-, and tricyclic aromatic compounds, as well as the way for purification of oil fractions from hard-to-remove polycyclic aromatic components using the hyper-cross-linked polystyrene adsorbents.

The syntheses of hyper-cross-linked polystyrene samples, the preparation of chromatographic columns thereon, and the measurement of specific surface areas of polymer adsorbents by the low-temperature argon desorption were performed at the Nesmeyanov Institute of Organoelement Compounds of the Russian Academy of Sciences, and the scanning electron microscopy study of adsorbent powders, the thermodynamic study of liquid-phase adsorption on hyper-cross-linked polystyrenes by liquid chromatography, and group separation of oil fractions were performed at Samara State Technical University.

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