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Phenol Adsorption from Aqueous Solutions by Chemically Activated Carbon Sorbents

N. N. IVANOV, I. YU. ZYKOV, V. E. TSVETKOV, YU. N. DUDNIKOVA

*Federal Research Center for Coal and Coal Chemistry, Siberian Branch, Russian Academy of Sciences, Kemerovo, Russia**E-mail: zyak.kot@mail.ru*

Abstract

Phenol adsorption from aqueous solutions on carbon sorbents was studied. The carbon sorbents were prepared from Kuzbass coals by means of chemical activation of raw coals using potassium hydroxide. Coal and potassium hydroxide were mixed by means of impregnation (with a mass ratio of coal/alkali 1 : 1), followed by mixture carbonization at 800 °C with isothermal exposure for 1 h. Phenol adsorption on thus prepared sorbents was shown to be described by Langmuir equation ($R^2 \approx 0.999$). Sorption parameters point to the high efficiency of phenol adsorption on carbon sorbents, especially on the sorbent obtained from D coal. It was established that phenol adsorption from aqueous solutions by the sorbents is described by the pseudo-second-order adsorption kinetics model, and the limiting stage is the diffusion of phenol molecules in the microporous space of the sorbent.

Keywords: phenol adsorption, carbon sorbents, chemical activation, adsorption isotherm

INTRODUCTION

Substantial pollution of open water bodies is an urgent ecological problem for the Kemerovo Region. The excess over the maximum permissible concentrations (MPC) of petroleum products and organic substances, in particular phenol (up to 6 MPC), is observed for the Tom river, as well as for the Inya river near the cities of Belovo and Leninsk-Kuznetskiy. The high concentrations of phenol (up to 2.4 MPC) and manganese (up to 11.7 MPC) were detected in the waste waters from the Yagunovskaya mine under liquidation in Kemerovo [1]. The pollution of natural water reservoirs restricts their use as the sources for household and drinking water supply.

Phenol and its derivatives are among the most dangerous pollutants of water. These compounds are highly toxic, and even a slight increase of phenol concentration in water causes substantial worsening of its organoleptic parameters [2]. At the territory of the Russian Federation, the MPC of phenol in water is $0.001 \mu\text{g}/\text{cm}^3$ [3].

The major source of the pollution of water reservoirs with phenol is the waste waters from industrial enterprises and sewage. A substantial part of waste waters enters water bodies in non-purified or partially purified forms, which is due to insufficient efficiency of purifying facilities or their absence, and the use of inefficient purification technologies [4].

The most widespread method of water purification at the territory of the Kemerovo Region is chlorination [1]. However, in the case of phenol, this method has a substantial disadvantage: if the time of the process is short and the consumption of chlorinating reagent is standard, phenol and its derivatives are transformed into chlorinated compounds with several times higher toxicity [5]. An increase in the consumption of the chlorinating reagent allows a substantial decrease in the concentrations of phenol and its derivatives in water, but additional purification of water from excess chlorine is necessary. A more efficient method of oxidative purification is ozonation, which involves the oxidation of phenol derivatives into more safe

compounds. High energy consumption limits the use of this method, so it is usually used for additional purification of water [5].

To enhance the quality of water purification, it is reasonable to use adsorption-based methods that provide the high degree of purification of industrial and household waste waters, with the absence of secondary pollutants. Adsorption-based purification methods may be used either for preliminary reduction of the concentration of pollutants before chlorination/ozonation or for final additional purification of water from oxidation products (especially after chlorination). Among a wide range of sorption materials, the most frequently used ones are carbon sorbents possessing excellent adsorption characteristics [6, 7]. The preparation of carbon sorbents by means of carbonization of a mixture of coal and KOH at a temperature of 700–900 °C was described in [8–13]. The synthesized sorbents are distinguished by developed porous structure, which enhances the sorption activity of these materials with respect to organic substances.

Under equilibrium conditions, carbon sorbents demonstrate high adsorption activity in the recovery of phenol from aqueous solutions [8, 9, 12]. To develop technologies for adsorption-based purification of waste waters, calculate the parameters of industrial adsorbents and choose adsorbents, the data on the kinetics of the adsorption process and on the capacity of adsorbent monolayer are necessary [5–7, 14].

The goal of the work was to investigate the processes of phenol adsorption from aqueous solutions on carbon sorbents prepared from different marks of coal from the Kuznetsk basin by means of chemical activation with potassium hydroxide.

EXPERIMENTAL

Carbon sorbents were obtained through chemical activation of coal samples of marks B (brown

coal), D (long-flame coal), SS (low-caking coal) and T (lean coal), mined at the territory of eth Kuzbass. The characteristics of coal samples involved in the investigation are listed in Table 1 in the order of increasing metamorphism stage.

To carry out chemical activation, a portion of coal dried to a constant mass (50 g) composed of the particles 0.2–0.5 mm in size was placed in the evaporation cup, mixed with KOH (50 g) and distilled water (50 cm³). The resulting mixture was kept at room temperature for 24 h, then dried and carbonized in a muffle furnace. Carbonization mode: heating rate 10 °C/min, carbonization temperature 800±5 °C, isothermal exposure for 1 h. The resulting carbonizate of the coal-alkali mixture was washed with distilled water and 1 % HCl solution to achieve pH 7.

Thus obtained samples of carbon sorbents were marked in the following manner: SBK-1, SDK-1, SSK-1, STK-1 – carbonizates of coal – alkali mixture with coal B/KOH, coal D/KOH, coal SS/KOH, coal T/KOH, respectively. Specific surface (S_{BET} , m²/g), total pore volume (V_{Σ} , cm³/g), the volume of mesopores (V_{meso} , cm³/g) and micropores (V_{micro} , cm³/g), and average pore diameter (D , nm) were calculated from the isotherms of nitrogen adsorption-desorption recorded at –195.75 °C and a pressure <0.133 Pa, with the help of ASAP 2020 analyzer (Micromeritics Instrument Co., USA). The conditions of the preparation of materials under investigation were indicated in [13].

The experimental data on phenol adsorption were obtained at a temperature of 25±1 °C according to the procedure described in [10]. A portion of sorbent dried to the constant mass (0.5 g) was put in a flask 100 cm³ in volume, then 50 cm³ of freshly prepared phenol solution with the given initial concentration ($C_0 = 0.1–4.0$ mg/cm³) was added, and mixing was carried out with a LOIP LS110 shaker (JSC LOIP, Russia) for 5–120 min. Phenol concentration was determined

TABLE 1
Characteristics of initial coal samples

Coal mark	Technical analysis, %			Elemental analysis, % (per daf)			Atomic ratio	
	W ^a	A ^d	V ^{daf}	C	H	(O + N + S)	H/C	O/C
B	11.5	10.4	46.7	70.4	4.4	25.2	0.75	0.27
D	4.5	3.4	43.3	80.2	5.6	14.2	0.84	0.13
SS	1.9	6.0	19.5	87.5	4.5	8.0	0.62	0.07
T	0.5	8.4	14.1	90.3	4.1	5.6	0.54	0.05

Note. Here and in Table 2: W^a is analytical humidity, A^d is ash content, V^{daf} is the yield of volatile substances, daf is dry ash-free state of the sample.

by means of photometry using a PE-5400UF spectrophotometer (LC EKROSKHIM, Russia) at the wavelength of $\lambda = 540$ nm. The determination procedure is based on the interaction of phenol with 4-aminoantipyrine during oxidation with potassium hexacyanoferrate (III) in the ammonia buffer solution (pH 10.0 \pm 0.3) [15]. The calibration dependences of optical density on the initial concentration of phenol in solution (C_0) were obtained within the concentration range 0–5 mg/cm³. The square mean deviation was 5.1 %, the maximal error was 10.4 %. The adsorption activity of sorbents with respect to phenol (A_{ph}) was calculated using equation

$$A_{ph} = (C_0 - C_p)V/m$$

where C_0 is the initial concentration of phenol in solution, mg/cm³; C_p is phenol concentration in solution after sorption, mg/cm³; V is solution volume, cm³; m is the mass of sorbent portion, g.

RESULTS AND DISCUSSION

Results of the technical and chemical analyses of the obtained carbon sorbents are presented in Table 2. The yield of volatile substances (V^{daf}) from sorbents is substantially lower than for initial coal samples (see Table 1) and is within the range of 5.4–8.7 %. The change of ash content (A^d) as a result of alkaline activation is less substantial; there are trends both to a decrease in high ash content (coal marks B, SS, T) and to an increase in low ash content (coal mark D). The elemental composition of sorbents is practically

the same, while substantial changes in the composition in comparison with initial coal samples are observed. Carbon content in the sorbents is ~93.5 %, while heteroatoms account for ~6.0 % independently of the origin of initial coal (see Table 2).

The texture characteristics of carbon sorbents are listed in Table 3. The S_{BET} for the sorbents is 780–1270 m²/g, V_{Σ} varies within the range 0.33–0.63 cm³/g. The materials under study are mainly microporous ($V_{micro} = 0.27$ – 0.48 cm³/g), which is characteristic of carbon sorbents obtained from black coal [11]. An exception is the SBK-1 sample (from brown coal) for which the fraction of mesopores is 0.42. For sorbents from black coal, specific surface and total pore volume are observed to increase with a decrease in the metamorphism stage of initial coal, that is, with a decrease in carbon content and an increase in the yield of volatile products (see Tables 1, 3). The sorbent from brown coal (SBK-1) possesses smaller specific surface area and pore volume than the sorbents from D coal (SDK-1) and SS coal (SSK-1), which may be due to the high ash content of initial brown coal (see Table 1). It is known that the adsorption of organic substances proceeds mainly in micropores [7]. Because of this, it may be assumed that these sorbents would exhibit high adsorption activity in the extraction of phenol from aqueous solutions

Experimental isotherms of phenol adsorption on carbon sorbents are presented in Table 1. Their shapes allow attributing them to type I,

TABLE 2

Characterization of the obtained sorbents

Sample	Technical analysis, %			Elemental analysis, % (per daf)			Atomic ratio	
	W ^a	A ^d	V ^{daf}	C	H	(O + N + S)	H/C	O/C
SBK-1	2.5	8.4	8.7	92.9	0.54	6.1	0.08	0.05
SDK-1	1.7	5.8	5.4	93.8	0.62	5.9	0.06	0.05
SSK-1	1.6	4.7	6.1	93.5	0.50	5.5	0.10	0.04
STK-1	2.3	7.4	6.8	93.3	0.40	6.1	0.05	0.05

Note. For designations, see Table 1.

TABLE 3

Texture characteristics of the obtained carbon sorbents

Sample	S_{BET} , m ² /g	V_{Σ} , cm ³ /g	V_{micro} , cm ³ /g	V_{meso} , cm ³ /g	D , nm
SBK-1	910	0.48	0.28	0.20	1.7
SDK-1	1270	0.63	0.49	0.10	2.0
SSK-1	1010	0.43	0.35	0.03	1.7
STK-1	780	0.33	0.27	0.03	1.5

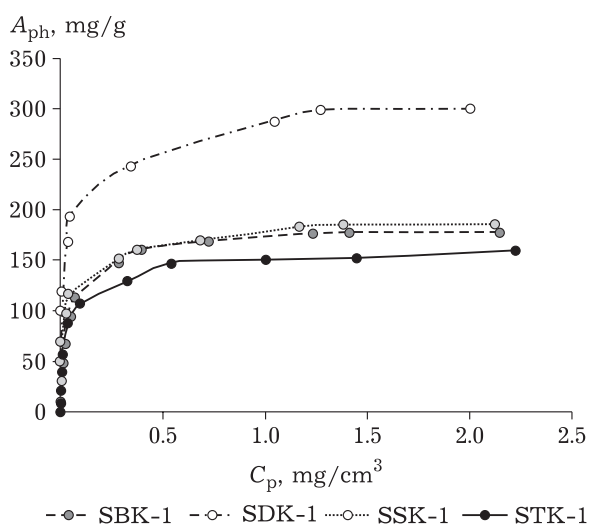


Fig. 1. Isotherms of phenol adsorption from aqueous solutions on the samples of coal sorbents.

which described monomolecular adsorption in microporous systems. The plots exhibit a smooth increase in adsorption activity (A_{ph}) within the range of equilibrium phenol concentrations $C_p = 0.1$ – 1.0 mg/cm^3 . For $C_p \geq 1.5$ mg/cm^3 , A_{ph} values approach the limit. The adsorption activity of sorbents increases in the sequence $\text{STK-1} < \text{SBK-1} < \text{SSK-1} < \text{SDK-1}$. The highest limiting A_{ph} value is observed for SDK-1 sorbent (289.1–300.1 mg/g), the lowest one for STK-1 (152.4–160.1 mg/g), the limiting values for sorbents SSK-1 (183.8–186.1 mg/g) and SBK-1 (177.1–178.1 mg/g) differ from each other insignificantly. The limiting A_{ph} values are achieved for initial phenol concentration $C_0 = 3.0$ mg/cm^3 .

The experimental isotherms of phenol adsorption were analyzed in the coordinates of the Langmuir (1) and Freundlich (2) equations of monomolecular adsorption [7, 16–18]:

$$A_{ph} = \frac{KA_{max}C_p}{1 + KC_p} \quad (1)$$

TABLE 4

Parameters of Langmuir and Freundlich equations for phenol adsorption from aqueous solutions on carbon sorbents

Sample	Langmuir equation				Freundlich equation		
	A_{max} , mg/g	K , cm^3/mg	R_L	R^2	K_f , $(\text{mg}/\text{g}) \cdot (\text{cm}^3/\text{mg})^{1/n}$	$1/n$	R^2
SBK-1	181.3	27.08	0.27	0.9997	194.9	0.34	0.8916
SDK-1	301.1	56.45	0.15	0.9998	343.3	0.29	0.8789
SSK-1	187.6	34.61	0.22	0.9989	202.5	0.30	0.8699
STK-1	160.1	24.70	0.29	0.9989	170.2	0.34	0.8327

Note. Here and in Table 5: R^2 – determination coefficient.

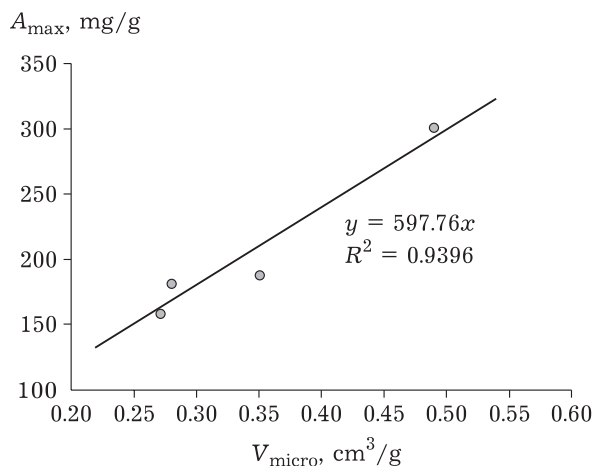


Fig. 2. Dependence of the capacity of carbon sorbent monolayer (A_{max}) on the volume of its micropores (V_{micro}) for phenol adsorption from aqueous solutions.

$$A_{ph} = K_f C_p^{1/n} \quad (2)$$

Here A_{ph} is adsorption value (adsorption activity), mg/g ; C_p is the equilibrium concentration of phenol in solution, mg/cm^3 ; A_{max} is the capacity of the adsorbent monolayer, mg/g ; K is the constant of adsorption equilibrium, cm^3/mg ; K_f and $1/n$ are the constants of the Freundlich equation.

The calculated parameters of the Langmuir and Freundlich equations are presented in Table 4. It was established that the experimental adsorption isotherms for equilibrium phenol concentration 0.001–2.5 mg/cm^3 are described most precisely with the Langmuir equation (determination coefficient $R^2 = 0.9989$ – 0.9998). The values of adsorbent monolayer capacity (A_{max}) calculated from Langmuir equation correlate with the experimental values of limiting adsorption activity. The calculated values of monolayer capacity increase in the sequence of sorbents: $\text{STK-1} < \text{SBK-1} < \text{SSK-1} < \text{SDK-1}$.

The dependence of the capacity of adsorption monolayer on micropore volume is presented in Fig. 2. The dependence is satisfactorily described with the linear function, which is the evidence of data correlation. The capacity of the monolayer increases proportionally to an increase in the volume of micropores in the samples.

The efficiency of phenol adsorption was evaluated relying on the degree of extraction X and the value of separation coefficient R_L [16–18]:

$$X = \frac{C_0 - C_p}{C_0} \cdot 100 \% \quad (3)$$

$$R_L = 1/(1 + KC_0) \quad (4)$$

where C_0 is the initial concentration of phenol in solution, mg/cm³; C_p is the equilibrium concentration of phenol in solution; K is the constant of adsorption equilibrium from Langmuir equation, cm³/mg.

Separation coefficient R_L allows one to estimate the nature of the sorption process. For instance, for $R_L = 0$ irreversible adsorption is observed, in the case of $0 < R_L < 1$ adsorption is reversible, with high extraction degree; for $R_L = 1$ adsorption of the substance under consideration is absent [16–18]. The calculated R_L values are within the range 0.15–0.27 (see Table 4), therefore, phenol adsorption from aqueous solutions on carbon sorbents under consideration proceeds rather efficiently. The value of X for the obtained sorbents (for initial concentration of phenol in solution $C_0 = 1.0$ mg/cm³) was 88–99 %, which is also the evidence of the high efficiency of adsorption.

The experimental dependence of adsorption activity A_{ph} on the time (t , min) of adsorbent/adsorbate interaction for $C_0 = 1.0$ mg/cm³ is presented in Fig. 3. The obtained experimental curves were analyzed using two kinetic models [2, 7–9, 14, 16–19]: pseudo-first order (Lagergren model) (5) and pseudo-second order (6)

$$\ln(A_{eq}^{exp} - A_{ph}) = \ln(A_{eq}) - k_1 t \quad (5)$$

$$\frac{t}{A_{ph}} = \frac{1}{k_2 A_{eq}^2} + \frac{t}{A_{eq}} \quad (6)$$

where A_{ph} is adsorption value (adsorption activity) at a time moment t (min), mg/g; A_{eq}^{exp} is the equilibrium adsorption activity achieved in the experiment, mg/g; A_{eq} is the calculated equilibrium adsorption of phenol, mg/g; k_1 , k_2 are the rate constants of adsorption for the models of pseudo-first and pseudo-second order, respectively.

Results of calculations according to the indicated kinetic models are presented in Table 5. The experimental data obtained in the present work are described best of all by the second-order kinetic model ($R^2 = 0.9991$ – 0.9995), the calcu-

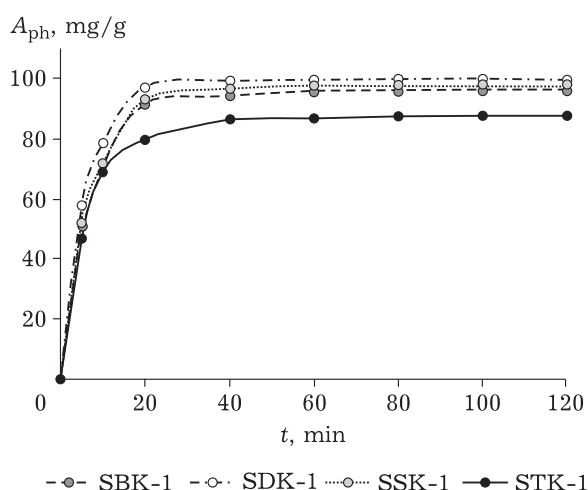


Fig. 3. Kinetic dependences of phenol sorption from aqueous solutions on the samples of carbon sorbents.

lated values of equilibrium adsorption A_{eq} rather well coincide the experimental values of A_{eq}^{exp} (see Table 5). The use of the pseudo-first order kinetic model for the description of experimental data is limited to the initial regions of curves (5–40 min). However, for the SSK-1 sample, the values of calculated equilibrium adsorption within the frameworks of two kinetic models are close to the experimentally determined values.

The adsorption of organic substances on carbon sorbents includes several stages, for example, mass transfer of the adsorbate from solution to the surface of the sorbent; diffusion of adsorbed molecules from the surface into the pore space. To evaluate the mechanism of the adsorption process and determine the limiting stage of phenol adsorption from aqueous solutions, the experimental kinetic curves were analyzed within the coordinates of the equation of the Weber and Morris diffusion model (7) [19]:

$$A_{ph} = k_{id} t^{0.5} + c \quad (7)$$

where A_{ph} is the value of phenol adsorption at the moment of time t (min), mg/g; k_{id} is the rate constant of adsorption inside the particle; c is a constant connected with the thickness of diffusion boundary layer.

The kinetic dependences of A_{ph} on are presented in Fig. 4. Two straight-line regions are distinguished in the obtained plots. The slope of the first region is substantially stronger than that of the second one, which is connected with the high rate of adsorption process at the initial stage. It may be assumed that the first region of the kinetic curve describes the diffusion of phenol to the surface of carbon sorbent.

TABLE 5

Kinetic constants determined according to the models of pseudo-first and pseudo-second order, for phenol adsorption from aqueous solutions on carbon sorbents

Sample	Kinetic model						
	A_{eq}^{exp} , mg/g	pseudo-first order (Lagergren model)			pseudo-second order		
		A_{eq} , mg/g	k_1 , min ⁻¹	R^2	$k_2 \cdot 10^3$, g/(mg · min)	A_{eq} , mg/g	R^2
SBK-1	97	45	0.0769	0.9436	3.24	99	0.9994
SDK-1	100	36	0.0888	0.8751	4.53	102	0.9994
SSK-1	98	110	0.1457	0.9681	3.53	101	0.9991
STK-1	88	41	0.0738	0.9588	3.55	90	0.9995

Note. For designations, see Table 4.

A small slope of the second region of the kinetic curve points to the low rate of phenol adsorption. According to [18, 19], the presence of the second region is connected with the diffusion of phenol molecules in the volume of micropores. Taking into account the low rate at the second stage of the kinetic curve, we may state that the limiting stage of phenol adsorption from aqueous solutions by the obtained sorbents is phenol diffusion in micropores.

CONCLUSION

Phenol adsorption by carbon sorbents is investigated. These sorbents were prepared by the chemical activation of coal samples of B, D, SS and T marks (800 °C, 1 h) with potassium hydroxide, which was introduced by means of impregnation (with the mass ratio of coal/alkali = 1 : 1). It was established that phenol adsorption on these sorbents is described by the Langmuir equation. The

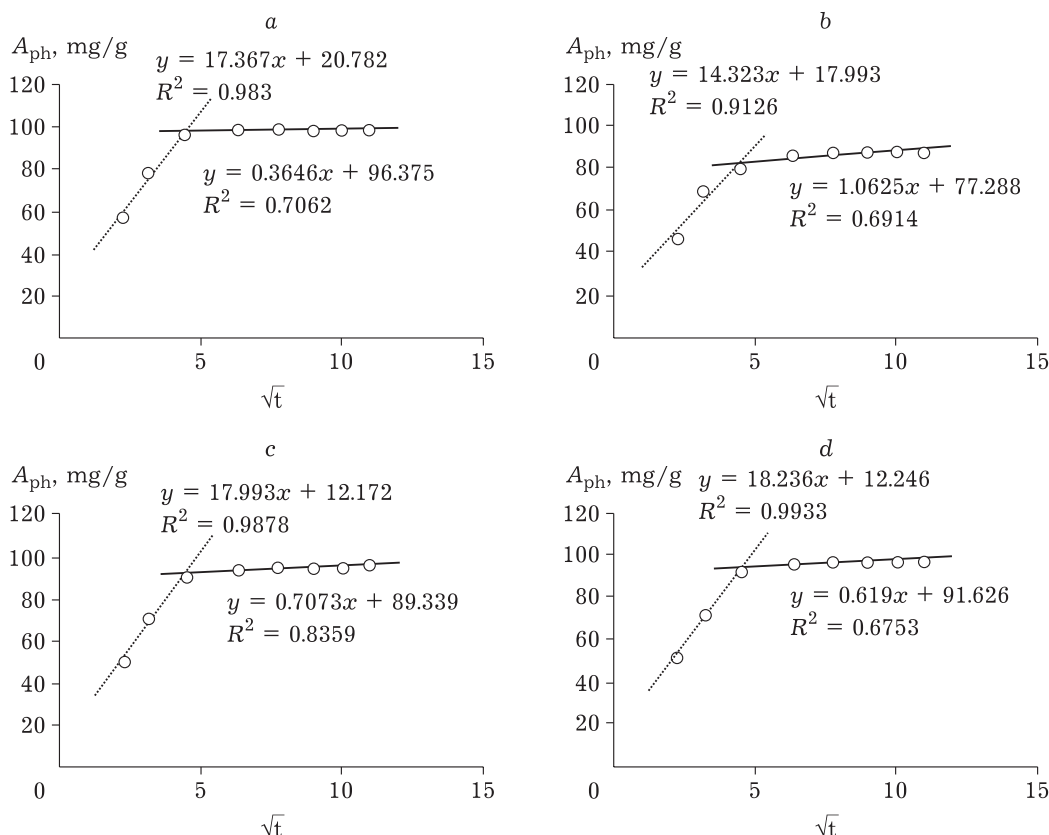


Fig. 4. Treatment of the kinetic dependences within the framework of diffusion model of adsorption: SDK-1 (a), STK-1 (b), SBK-1 (c), SSK-1 (d).

determined values of separation coefficient R_L allow us to state that the efficiency of adsorption is rather high. It was established that the adsorption process is described by the model of pseudo-second order adsorption kinetics, and the limiting stage is the diffusion of phenol molecules in the microporous space of the sorbents.

The obtained carbon sorbents may find application in the technological processes of the purification of water for household purposes from phenol.

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