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Original Theoretical Research

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Natural Sorbent Based on Zeolite-Containing Rocks of Tatarsko-Shatrashan Deposit for Treatment of Surface and Waste Water from Organic Pollutants

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Abstract
Introduction. The modern industrial and environmental challenges require the identification of optimal sorbents for water purification from organic contaminants. Sorbents like activated carbon and silicon dioxide have been widely used, but the problem of selecting the optimal sorbent that can adequately purify water remains relevant. There is information in the scientific literature about potential use of sorbents from natural zeolite-containing minerals for this purpose. However, this approach is not well developed; the materials are poorly studied and, as a result, are rarely used to solve environmental problems. This work aims to fill this gap by studying the sorption properties of a natural sorbent based on zeolite-containing rocks of the Tatarsko-Shatrashan deposit.
Materials and Methods. The method of ascending liquid column chromatography became the basis for this study. The sorption material was loaded into a chromatographic column with a length of 120 mm and an inner diameter of 3 mm. The model organic substances in the vial moved along the length of the sorption layer. Trichloroethane, ethyl acetate, methyl ethyl ketone, dichloroethane, and trichloroethylene were used as model compounds. We summarized the significant data in tables and visually represented it in graphs.
Results. The technological characteristics of natural sorbents obtained on the basis of zeolite-containing rocks of the Tatarsko-Shatrashan deposit have been experimentally investigated. The absolute retention time of the studied sorbates, as well as their sorption capacity in relation to zeolite-containing rocks of the Tatarsko-Shatrashansky deposit, were determined. The dependence of the retention time of model organic substances on the length of the sorption layer, which was determined by the physical-chemical nature of the sorbate under study, has been established. From the same point of view (as components of the dependence), the boiling points of model organic substances, dipole moments, refractive indices, and densities were considered. The experimental data were statistically processed, and the absolute and relative errors of a single measurement were determined. All sorbates considered in the framework of this scientific work showed significant or high sorption capacity. The recorded minimum was 34% (methyl ethyl ketone); the maximum was 72% (ethyl acetate). At the same time, ethyl acetate had an extremely short retention time in a 10-centimeter sorption layer (26 min). The longest retention time was for methyl ethyl ketone (314 min). Its sorption capacity was minimal (34%).
Discussion and Conclusion. The prospects of the studied material for the purification of surface and waste water from major pollutants in the natural environment have been experimentally proven. It has been determined that zeolite-containing rocks from the Tatarsko-Shatrashan deposit can adsorb 34–72% of organic compounds that pollute water. They can be used in technological processes for the purification of natural and wastewater from major environmental pollutants.

Keywords: zeolite-containing rocks, model organic substances, sorption capacity, liquid chromatography, water purification from organic pollutants

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Оригинальное теоретическое исследование

Природный сорбент на основе цеолитсодержащих пород Татарско-Шатрашанского месторождения для очистки поверхностных и сточных вод от органических загрязнителей

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Аннотация

Введение. Современная производственная и экологическая повестка ставит задачу выявления оптимальных сорбентов для очистки воды от органических загрязняющих веществ. Широкое практическое применение получили такие сорбенты, как активированный уголь, диоксид кремния и др. Однако сохраняет актуальность проблема поиска оптимального по селективности сорбента. Он должен приемлемо или хорошо очищать воду от главных органических загрязнителей. В научной литературе есть сведения о перспективах использования в рассматриваемых целях сорбентов из природных цеолитсодержащих минералов. Однако такой подход недостаточно проработан, материалы малоизучены и, как следствие, редко используются для решения экологических проблем. Представленная научная работа призвана восполнить этот пробел. Ее цель — исследование сорбционных характеристик природного сорбента на основе цеолитсодержащих пород Татарско-Шатрашанского месторождения.

Материалы и методы. Базовым для данного исследования стал метод восходящей жидкостной колоночной хроматографии. Сорбционный материал загружали в хроматографическую колонку длиной 120 мм и внутренним диаметром 3 мм. Модельные органические вещества в виале перемещались по длине сорбционного слоя. В качестве модельных соединений использовали трихлорэтан, этилацетат, метилэтилкетон, дихлорэтан и трихлорэтилен. Значимые данные обобщены в виде таблиц и графически визуализированы.

Результаты исследования. Экспериментально исследованы технологические характеристики природных сорбентов, полученных на основе цеолитсодержащих пород Татарско-Шатрашанского месторождения. Определены абсолютное время удерживания исследуемых сорбатов, а также их сорбционная емкость по отношению к цеолитсодержащим породам Татарско-Шатрашанского месторождения. Установлена зависимость времени удерживания модельных органических веществ от длины сорбционного слоя, которая определяется физико-химической природой исследуемого сорбата. С этой же точки зрения (как составляющие зависимости) рассмотрены температуры кипения модельных органических веществ, дипольные моменты, показатели преломления и плотности. Экспериментальные данные статистически обработали, определили абсолютную и относительную погрешность единичного измерения. Все сорбаты, рассмотренные в рамках данной научной работы, показали значимую или высокую сорбционную емкость. Зафиксированный минимум — 34 % (метилэтилкетон), максимум — 72 % (этилацетат). При этом у этилацетата экстремально малое значение времени удерживания в 10-сантиметровом сорбционном слое (26 мин). Самое продолжительное время удерживания — у метилэтилкетона (314 мин). Его сорбционная емкость — минимальная (34 %).

Обсуждение и заключение. Экспериментально доказана перспективность исследуемого материала для очистки поверхностных и сточных вод от главных загрязнителей природной среды. Установлено, что цеолитсодержащие породы Татарско-Шатрашанского месторождения могут адсорбировать 34–72 % загрязняющих воду органических соединений. Их можно использовать в технологических процессах очистки природных и сточных вод от основных загрязнителей окружающей природной среды.

Ключевые слова: цеолитсодержащие породы, модельные органические вещества, сорбционная емкость, жидкостная хроматография, очистка воды от органических загрязняющих веществ

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Introduction. One of the conditions for environmental safety is the protection of water bodies from anthropogenic and natural pollution. A special case of this problem is the negative impact of organic compounds such as petroleum products, phenols, aromatic hydrocarbons, and organochlorine [1]. These pollutants spread relatively quickly in water over considerable distances and harm the environment even in neighboring regions, which can lead to irreversible changes in the ecosystem [2]. Phenol and its derivatives are especially dangerous for the environment and humans, as many of them are mutagens and teratogens capable of disrupting the endocrine system [3]. In addition, under certain conditions, the phenol molecule is transformed into compounds with a higher hazard class [4]. Biodegradation of phenol can accelerate in summer, when aerobic microorganisms intensively oxidize organic compounds [5]. The limiting stage of this process is the mass transfer of oxygen molecules from the gas phase to the aqueous phase [6]. In addition to phenols, other classes of organic compounds can be found in surface waters: aromatics and alkylaromatics, alkanes, carboxylic acids, hexachlorane, hexachlorobenzene, benz(a)pyrene, acetatanaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benz(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, indeno(1,2, 3-cd)pyrene [7].

Source [8] provides information collected over 15 years on the contamination of organic compounds in the bottom sediments of the Belaya River. Analysis of the ecological status of surface waters in various regions of Russia [9] has revealed that in many cases, water resources do not meet the standards. As a rule, cities rely on surface water for their water supply. Water utilities treat and chlorinate this water [10], which can lead to the formation of organochloride compounds that pollute drinking water. These compounds can be removed through various methods, such as sorption, which uses activated carbon and synthetic zeolite materials to remove toxic impurities from water. Recent studies indicate that natural zeolite minerals have promising potential for this purpose due to their well-developed systems of macro- and micropores that effectively remove organic compounds from water [11]. A system of cavities and channels run through the three-dimensional alumino–silico–oxygen framework of natural zeolites. The huge inner surface and cavernous structure of zeolite-containing rocks ensures strength of adsorption processes, which is sufficient for efficient wastewater treatment by industrial enterprises from toxic impurities. However, natural zeolite rocks are not well studied, which hinders their widespread use in solving environmental problems [12].

The aim of this study is to experimentally investigate the technological properties of natural sorbents based on zeolite-containing rocks of the Tatarsko-Shatrashansky deposit.

Materials and Methods. A natural sorbent based on zeolite-containing rocks from the Tatarsko-Shatrashansky deposit has been studied (Table 1).

Table 1

Chemical composition of the natural sorbent [12]

Elements	% wt
Silicon dioxide SiO_2	66.00
Titanium dioxide TiO_2	0.35
Aluminum oxide Al_2O_3	6.19
Iron oxide Fe_2O	2.65
Manganese oxide MnO	0.01
Calcium oxide CaO	17.00
Magnesium oxide MgO	1.45
Sodium oxide Na_2O	0.16
Potassium oxide K_2O	1.43
Phosphorus oxide P_2O_5	0.13

The natural elements under consideration are thermally stable and acid resistant. The total cation exchange capacity — 130.0 mg-eq / 100 g. Calcium plays a major role in the metabolic process. Table 2 provides the main characteristics of the material under study.

Table 2

Characteristics of the natural sorbent under study

Indicators	Physical and mechanical properties
Appearance	Granules of light gray or white color
Porosity	37.25–55.72%
Density	2.03–2.37 g/cm^3
Mechanical crushing strength	At 200 °C — 46 kg/cm^2, at 2500 °C — 59 kg/cm^2
Vibration wear	0.96%
Bulk weight	0.4–1.2 g/cm^3
Volume weight	1.10 g/cm^3
Effective pore diameter	0.4 Nm (4A°)
Thermal resistance	Higher than 450°C
Solubility in water	Insoluble

To prepare the sorbent, rocks containing zeolite from the Tatarsko-Shatrashansky deposit were mechanically activated. They were crushed in a ball mill; the fractions were separated, and then treated with a 1:1 solution of hydrochloric acid. Next, the resulting material was washed with water until a neutral reaction was achieved and subjected to heat treatment at 450–500°C for 5 hours. The sorbent obtained was placed in 150 mm long glass chromatographic columns with an inner diameter of 3 mm. Vials containing the model liquids under study, organic substances with various physical and chemical properties, were attached to the bottom of the columns. As the model organic substances passed through the sorption layer of the zeolite-containing rock, their movement up through the channels and pores was recorded. The time of passage was measured every 10 millimeters of the sorbent and kinetic curves were plotted for the dependence of the retention of model organic substances on the height of the sorption layer.

Sorption properties were determined by the formula:

$$A = \frac{m_2 - m_1}{m_1} \cdot 100\%, \quad (1)$$

where A — sorption capacity of the material under study, %; m_1 — mass of the initial adsorbent; m_2 — mass of the adsorbent saturated with an organic solvent.

For statistical processing of experimental data, the absolute and relative errors of a single measurement were determined with a confidence probability of 0.95. Table 3 provides the results for hexane sorption.

Table 3

Absolute and relative errors in the measurement of hexane on the tested sorbents

Measurement criteria	Error	
	absolute, Δ	relative, δ %
5	7.16	13.64
10	6.40	12.12
20	5.29	11.96
30	5.15	10.78

Results. Table 4 provides information on physical-chemical properties of model organic substances (sorbates).

Table 4

Characteristics of model sorbates*

Model sorbates	Formula	$T_{кип}$	*d*	n_D^{20}	t_{10}	*A*	μ
Trichlorethane	$CHCl_3$	74	1.453	1.4463	152	45	1.15
Ethyl acetate	$C_4H_8O_2$	77.1	0.900	1.3720	26	72	2.48
Methyl ethyl ketone	C_4H_8O	79.6	0.805	1.3800	314	34	2.84
Dichloroethane	$C_2H_4Cl_2$	83.5	1.253	1.4400	61	62	1.80
Trichloroethylene	C_2HCl_3	87.2	1.464	1.4800	88	56	0.85
Hexane	C_6H_{14}	68.0	0.660	1.416	59	46	0.05

where **d* — density, g/cm^3; Ткип — boiling point, °C; n_D^{20} — refractive indices at 20°C; t_{10} — retention time of model organic substances in a 10-centimeter sorption layer, min; *A* — sorption capacity, %; μ — dipole moment, *D*

As a result of the experiments (Table 4) the retention time of ethyl acetate was extremely low (26 min). Trichloroethane, which had a lower boiling point (74°C), was washed out of the column later than ethyl acetate with a boiling point of 77.1°C. Obviously, this could be explained by the higher molecular weight of trichloroethane (M = 119.4 g/mol) compared to ethyl acetate (M = 88.11 g/mol). In this case, the order in which the components exit was not determined by their boiling points. This dependence was more complicated. It took into account the chemical nature of the model organic substances used, as well as the possibility of their adsorption and desorption by the pores of the material under study. At the same time, despite the low retention time of ethyl acetate, its sorption capacity was relatively high (72%) compared to other organic substances studied. The sorption capacity of all the studied organic substances was quite high (34–72%), which made it possible to use these materials to purify water from organic compounds.

To evaluate the kinetic characteristics of the sorption process, dependencies were constructed linking the retention time of model sorbates and the height of the sorption layer in the ranges from 0 to 5 cm (Fig. 1) and from 5 to 10 cm (Fig. 2).

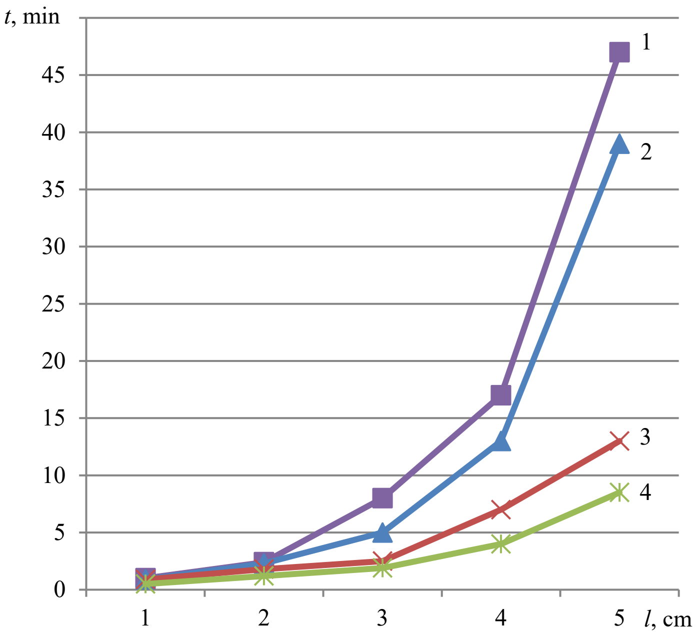

Fig. 1. The effect of retention of model sorbates on the nature of sorption processes (sorbent layer from 0 to 5 cm): 1 — methyl ethyl ketone; 2 — trichloroethane; 3 — trichloroethylene; 4 — dichloroethane

As it can be seen from Figure 1, the dependence had a parabolic character. Obviously, this was due to the fact that at the initial moment of time (up to 5 mm of the sorption layer) there was no equilibrium between the organic liquid and the solid. It was established after sorption, and an almost linear pattern was observed at a distance of more than 5 cm. The exception was methyl ethyl ketone, which had a sufficiently high value of the dipole moment (μ = 2.84D)

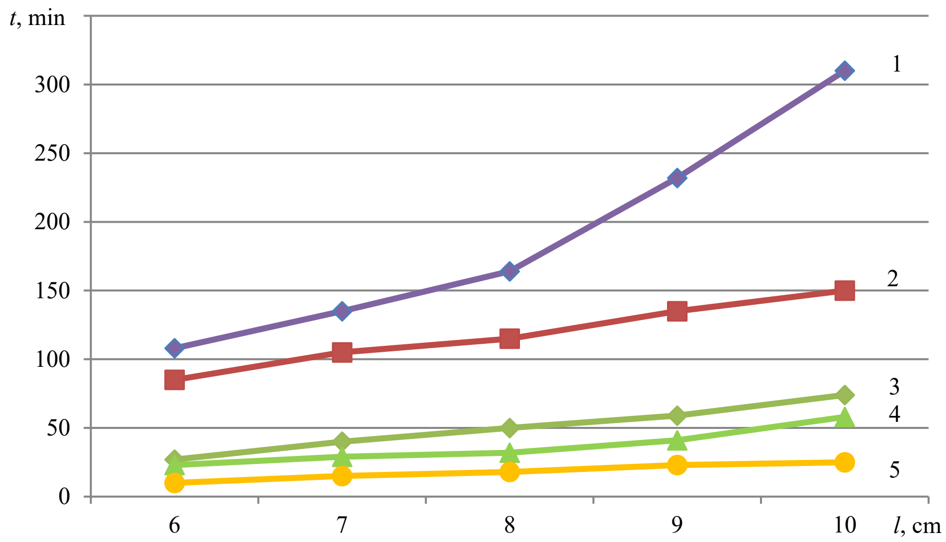

Fig. 2. Dependence of the retention time of model organic substances on the height of the sorption layer of zeolite-containing rock (from 5 to 10 cm): 1 — methyl ethyl ketone; 2 — trichloroethane; 3 — trichloroethylene; 4 — dichloroethane; 5 — ethyl acetate

It should be noted that for all the organochlorine compounds studied (trichloroethane, dichloroethane, trichloroethylene), the angles of inclination of straight lines were almost identical (Fig. 2). This indicated the additivity of the sorption process for organochlorine compounds.

An important parameter of an organic molecule is the dipole moment. It characterizes the asymmetry of charge distribution in an electrically neutral molecule, which makes it possible to form electric dipoles with the same charge values $+g$ and $-g$. The dipole moment of a molecule is determined as a result of vector addition of the dipole moments of individual bonds. The dipole moments of organic compounds characterize the polar properties of a molecule, as well as determine the direction and strength of intermolecular electrostatic interactions and the sorption properties of porous materials.

Figure 3 shows the dependence of the retention time of model sorbates ($t_{уд}$) on their boiling points ($T_{кип}$, °C) and dipole moments (μ, D).

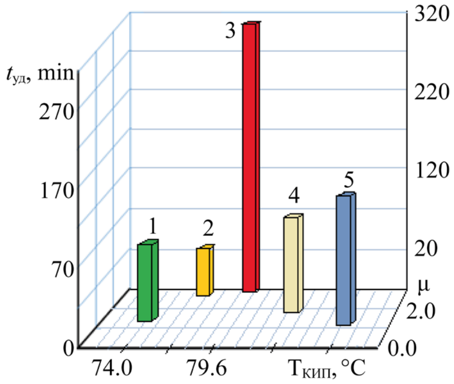

Fig. 3. Dependence of the retention time of model organic substances on their boiling points and dipole moments: 1 — trichloroethane; 2 — ethyl acetate; 3 — methyl ethyl ketone; 4 — dichloroethane; 5 — trichloroethylene

As it can be seen from Figure 3, the release time of ethyl acetate (2) and methyl ethyl ketone (3) differed significantly, although they had similar values of dipole moments and boiling points. Methyl ethyl ketone retained significantly longer ($t_{уд} = 314$ min) than ethyl acetate ($t_{уд} = 26$ min), dichloroethane ($t_{уд} = 61$ min), trichloroethylene ($t_{уд} = 88$ min), and trichloroethane ($t_{уд} = 152$ min). Let us note that according to the Rorschneider classification in the sorbate—sorbent system, methyl ethyl ketone determines the dispersion interaction. Obviously, the Van der Waals forces play a leading role in this process, and the sorption time is more significant. This is also due to the fact that methyl ethyl ketone is characterized by a higher polarity (dipole moment — μ = 2.84D), as well as a higher boiling point ($T_{кип} = 79.6$°C).

The refractive index also determines the polarity of the model organic substances. It is related to molecular refraction, and is a measure of the electronic polarizability of the shell of a substance molecule.

Figure 4 shows the dependence of the retention time of the model sorbates on their boiling temperatures ($T_{кип}$, °C) and refractive indices (n_D^{20}).

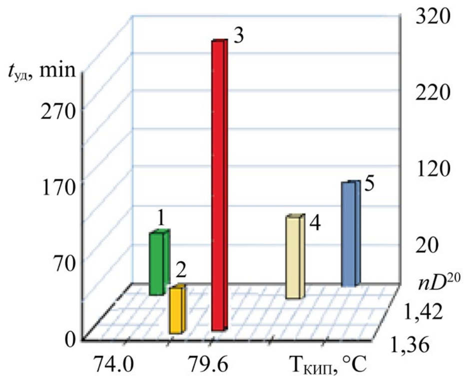

Fig. 4. Dependence of the retention time of model organic substances on their boiling points and refractive indices:
1 — trichloroethane; 2 — ethyl acetate; 3 — methyl ethyl ketone; 4 — dichloroethane; 5 — trichloroethylene

As it can be seen from Figure 4, ethyl acetate and methyl ethyl ketone were similar in two parameters:

– boiling points (77.1°C and 79.6°C respectively);

– refraction (1.37 and 1.38 respectively).

Methyl ethyl ketone is characterized by a higher retention time compared to other sorbates studied. Apparently, this is due to the higher energy of the orientational interaction of methyl ethyl ketone with the sorbent surface.

Figure 5 allows us to consider the density of sorbates in relation to retention time and boiling point.

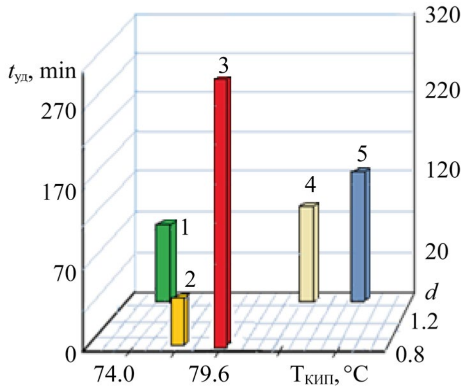

Fig. 5. Dependence of the retention time of model organic substances on their boiling points and density:
1 — trichloroethane; 2 — ethyl acetate; 3 — methyl ethyl ketone; 4 — dichloroethane; 5 — trichloroethylene

Thus, within the framework of this scientific work, it was found that the sorption capacity of the materials under consideration varied from 34% to 72%. Ethyl acetate ($C_4H_8O_2$) had a particularly high rate (72%). Its other characteristics were: dipole moment — 2,48; retention time of model organic substances in a 10-centimeter sorption layer — 26 min; refractive index at 20°C — 1.3720; boiling point — 77.1°C; density — 0.9 g/cm^3. Methyl ethyl ketone was similar to ethyl acetate in terms of boiling point, density, refraction, and dipole moment. However, among the sorbates considered, its sorption capacity was minimal (34%), and the retention time was the longest (314 min).

Discussion and Conclusion. The results of the study indicate that a high-quality sorbent can be produced using zeolite-containing rocks from the Tatarsko-Shatrashansky deposit. This material has the potential to purify water from major organic pollutants. It was found that, the best result in terms of sorption capacity was to remove ethyl acetate if necessary. The sorbent in question takes 72% of this substance from the water. The minimum is associated with methyl ethyl ketone. In this case, the lowest but acceptable sorption capacity (34%) is established.

Thus, the sorption properties of natural zeolites are considered to be good. The findings of this scientific study can be applied in technological processes for water purification from major contaminants.

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