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> PHYSICAL CHEMISTRY OF SURFACE PHENOMENA

Effective Purification of Water from Iron Ions and Potentially Pathogenic Microorganisms Using a Montmorillonite Composite Sorbent

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Abstract—The use of the developed composite sorbent based on the plant wastes (sunflower seed husks) and montmorillonite clay was shown to be highly effective for water purification from iron ions and potentially pathogenic microorganisms. It was shown experimentally that the developed composite sorbent at concentrations of up to 50 mg/L is capable of purifying water from iron ions under static and dynamic conditions. When a suspension of *Escherichia coli* was filtered at a rate of 10 mL/min, 1 g of the composite sorbent adsorbed 556 million *E. coli* colony-forming units (CFUs), showing that the sorbent is 1.7 times more effective than the complex sorbent of the replaceable Barrier-4 filter cartridge.

Keywords: montmorillonite, plant waste, sunflower seed husks, composite sorbent, iron ions, potentially pathogenic microorganisms

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The quality of drinking water, preservation of water resources, and their protection from chemical and microbiological contamination are worldwide challenges.

The hazardous pollutants of the hydrosphere include heavy metals, among which iron is very common. In water bodies it can be of both natural and technogenic origin. In underground and deep waters, the iron concentration can be very high (up to a few dozens mg/L). Significant amounts of iron come from sewage waters of metallurgical, metalworking, textile, paint and varnish, and other industries.

According to the results of the analysis of the Federal Sanitary and Hygienic Monitoring Database of the Russian Federation, iron that enters the centralized water supply system (CWSS) from the water supply source during water transportation because of pipe corrosion is one of the main pollutants of drinking water [1]. In the Belgorod region, which is one of Russia's large iron ore regions, where iron ore is mined openly, there are mining and metallurgical industries; as a result, iron in CWSS exceeds the hygienic standard for total iron (0.3 mg/L) [2, 3]. The high iron content in water causes dry skin, impairs the taste of drinking water, and its accumulation in the human body leads to dysfunction of liver, pancreas, endocrine glands, and cardiovascular system [4]. In view of this adverse effect of large amounts of iron in water of the Belgorod region, it is very important to develop methods for water deironing, which is a special method for improving the quality of water.

One of the most common procedures for deironing water is as follows. The iron compounds are oxidized and converted, on a special catalytic material, into insoluble flakes (e.g., of well hydrolyzable iron(III) hydroxide or iron(III) salts) well removable by filtration [5].

The iron content in water can be reduced by adsorption using a special clay sorbent. Research has been pursued for several years at the Belgorod State University to study the sorption properties of mont-morillonite-containing clays from various deposits with respect to iron and other heavy metal ions [6-11].

In addition to pollution of water reservoirs with heavy metals, another important environmental problem is their contamination with pathogenic bacteria as a result of the entry of contaminated sewage (household and industrial wastes, wastes from livestock, etc.). The sources of microbiological contamination of surface and ground water can also be unauthorized landfills for solid household waste. At the same time, as is known, some bacterial infections such as typhoid fever, dysentery, cholera, and tularemia have water ways of transmission. For some viral infections, for example, poliomyelitis, the possibility of transmission through the aquatic environment was also proven [12, 13]. To free water from pathogenic bacteria, it is detoxicated or disinfected. For this purpose, various chemical reagents and a number of physical and physicochemical methods are used. They differ in popularity and scale of application. Some of them are used on an industrial scale, while others in individual treatment plants. Each method has both advantages and disad-

Chlorination is the most common technique of water disinfection. It uses liquid or gaseous chlorine, chlorine dioxide, sodium and calcium hypochlorites, lime chloride, and chloramines. From the hygienic viewpoint, ozonation is one of the best methods of disinfecting the drinking water. These oxidative methods (chlorination, ozonation, etc.) have a significant drawback: they do not extract contaminants from the aquatic environment, but only convert them into other compounds.

vantages.

The physical methods for drinking water disinfection include boiling, UV irradiation, electric pulse method, treatment with ultrasound and high-frequency currents, magnetic treatment, and gamma irradiation. They are mostly expensive and have not found wide use.

Sorption is also one of the physicochemical methods of disinfection. Unlike the oxidants, the sorbents do not modify the pollutants, but extract them from water. Therefore, their use is safer from the sanitary viewpoint. As sorbents, various antibacterial materials can be used.

Our previous studies revealed that modified montmorillonite-containing clays are as effective in killing pathogenic microorganisms as modern antibiotics [14–16]. Unlike the antibacterial agents, however, clay itself remains chemically inert and hence harmless to the human organism. A promising approach to the preparation of antibacterial drugs is the introduction of a coal component in the clay matrix. As is known, activated carbons effectively inhibit the development of pathogenic microflora [17]. Nanomaterials are also widely used in catalytic purification [18–28], and it would be interesting to study their efficiency in cleaning aqueous solutions.

The purpose of this study was to determine the efficiency of water purification from potentially pathogenic microorganisms and heavy metal ions (iron) using the composite sorbent developed by the authors on the basis of plant waste from the agroindustrial complex (sunflower seed husks) and montmorillonite clay. The method for the preparation of this composite sorbent was described in [29–31].

EXPERIMENTAL

To study the efficiency of water purification from iron ions and potentially pathogenic microorganisms, we took the samples of native montmorillonite-containing clay (NC) from the Nelidovka deposit, Korocha district, Belgorod region and the composite sorbent (CS) obtained from plant waste from the agroindustrial complex (sunflower seed husks) and montmorillonite clay.

The sorption characteristics with respect to Fe^{3+} ions were studied spectrophotometrically on the experimental samples under static and dynamic conditions. In the course of the experiment, it was checked that the solution had no acid anions that give more stable complexes with Fe^{3+} than rhodanides: phosphates, acetates, arsenates, fluorides, borates, and significant amounts of chlorides and sulfates [32]. In this method, ammonium iron alum was used. Distilled water was used as a solvent.

To determine the sorption of Fe³⁺ ions under static conditions, a series of model solutions with concentrations of 0.5–5 mg/L were prepared. The light wavelength in the region of maximum absorption of the solution containing Fe³⁺ ions was 480 nm. The concentration of solutions after sorption was calculated using the regression equation of the calibration curve constructed from the obtained data: y = 0.1192x + 0.0156.

The sorption ability of the experimental samples with respect to Fe^{3+} ions under static conditions was studied in the following way. A solution (100 mL) containing Fe^{3+} ions at a concentration of 5 mg/L was poured on a clay sample (1 g) and held for 1, 5, 15, 30, and 60 min. The iron(III) content in solution after sorption was determined spectrophotometrically on a Specord 210 Plus spectrophotometer; then the sorption rate was calculated by the equation

$$v = \frac{C_0 - C_1}{\Delta t},\tag{1}$$

where C_0 and C_1 is the concentration of Fe³⁺ ions (mg/L) at the start and end of a certain interval of time during sorption Δt .

The adsorption of Fe^{3+} ions under dynamic conditions was studied on a granular composite sorbent. The solution containing the adsorbate was passed at a fixed rate through a column containing the adsorbent. The initial Fe^{3+} concentration was 50 mg/L. The height of the sorbent layer was 16 cm, the sorbent sample 20 g, the height of the liquid layer above the sorbent 2 cm, and the column diameter 1 cm. The solution filtration rate was kept constant at 10 mL/min. The experiment was performed with adsorbent of different particle sizes. The sorbent was ground and divided into fractions with particle sizes: $d > 200 \,\mu\text{m}$, $67 \,\mu\text{m} < d < 200 \,\mu\text{m}$, and $d < 67 \,\mu\text{m}$. The experiment was performed at a temperature of $20 \pm 2^{\circ}\text{C}$.

The filtrate was sampled in 25 and 100 mL portions until the equilibrium set in. The filtrate solution was analyzed.

The concentration of Fe^{3+} ions in each filtrate sample was determined by the spectrophotometry.

In this experiment, the total dynamic exchange capacity (TDEC (D_t) , mg/g) was calculated by the equation

$$D_{\rm t} = C_0 V/m, \tag{2}$$

where C_0 is the initial concentration of Fe³⁺ ions, mg/L; *V* is the total volume of the filtrate, L; and *m* is the sorbent sample, g.

Studies on purification of model water solutions from the CWSS from the potentially pathogenic microorganisms were performed under laboratory conditions with water contaminated with *E. coli* (*E. coli* ATCC 25922 test culture). As a filtering element, a composite sorbent was used.

As control, we used the sorbing components of a Barrier-4 replaceable filter cartridge of a household filter for water purification. The cartridge included ion-exchange resins, coconut activated carbon, and coconut activated carbon treated with silver.

The experimental and control samples of sorptionfiltering substances in the same amount (3 g each) were weighed on OHAUS PA-114C analytical balance (OHAUS) and placed in funnels with Schott glas frit filters (VF-3 filter funnel, State Standard (GOST) 25366-82), through which the model suspensions of E. coli were filtered. The pore size in the Schott filter was 16-40 µm (GOST 25336-82). The concentration of E. coli in the contaminated model suspensions exceeded the SanPiN standard 2.1.4.10749-01 "Drinking water. Hygienic requirements to the quality of water." The coli-titer was 100 thousand, 1 million, 10 million, 100 million, and 1 billion times higher than the norm (300 mL of drinking water contained 100 thousand, 1 million, 10 million, 100 million, and 1 billion colony-forming units (CFUs) of *E. coli*).

At the start of the main experiment, we checked the ability of the Schott filter to let a suspension of *E. coli* in the drinking water pass through the pores with a diameter of $16-40 \,\mu\text{m}$. Before the start of our study on the ability of the experimental and control samples of sorption-filtering substances to purify water from potentially pathogenic microflora, the samples were placed as a uniform layer on the surface of the Schott filter, and 100 mL of sterile isotonic sodium chloride solution was passed through them. This led to a swell-

ing of the crystal lattice of dioctahedral smectite and the removal of coal dust microparticles from the sorbents under study capable of passing through the filter pores.

To prepare the model bacterial suspensions, *E. coli* were grown on meat-and-peptone agar (MPA) "For growing bacteria" (Medgamal, Moscow), and cultivation was carried out at 37°C for 18 h in a TS-1/80 SPU dry air thermostat (Smolensk SKTB SPU, Russia). After cultivation, the test culture was washed off the MPA surface with a sterile isotonic sodium chloride solution.

The concentration of microorganisms was determined using a Densi-La-Meter device for determining the turbidity of bacterial suspension (Erba Lachema, Czech Republic), whose operation is based on the optical absorption of a suspension (the result of measurement is given in McFarland units).

The contaminated drinking water was supplied to the filter funnels containing the sorption-filtering substances under study with the help of dropping tubes. The filtration rate was 10 mL/min. The amount of drinking water contaminated once with a known amount of *E. coli* and filtered through the test and control sorbent samples during the experiment was 900 mL. Each 100-mL portion of filtered model suspensions of *E. coli* was centrifuged for 3 min at 3000 rpm. An ELMI CM-6MT (ELMI, Latvia) desktop centrifuge was used in the experiment.

The resulting centrifugate was suspended in 1 mL of drinking water. Then smears were taken from the resulting suspension. They were fixed over the flame of an alcohol lamp and stained with carbol fuchsin. Before the use, fuchsin was diluted with distilled water in a ratio of 1:10. Then the smears were microscoped in a Lomo Micromed-5 microscope at large magnification (×18 eyepiece) (LOMO, St. Petersburg, Russia). The microbial cells were counted in five microscope fields, and the average was calculated. To obtain reliable results, the experiment was repeated three times.

RESULTS AND DISCUSSION

At the preliminary stage, the sorption capacity of the composite sorbent with respect to Fe^{3+} ions under static conditions was studied.

At the initial stage of sorption (15 min), all the samples actively absorbed Fe³⁺ ions. Then the process slowed down and tended to equilibrium (Fig. 1). The maximum amount of Fe³⁺ ions was adsorbed within 15 min. On the composite sorbents (fractions with $d > 200 \ \mu m$ and 67 $\mu m < d < 200 \ \mu m$), the maximum



Fig. 1. Variation of the concentration of Fe^{3+} ions with time when in contact with adsorbent samples: (1) NC, (2) CS ($d > 200 \,\mu\text{m}$), and (3) CS ($67 \,\mu\text{m} < d < 200 \,\mu\text{m}$).



Fig. 2. Dependence of the sorption rate of Fe^{3+} ions on the duration of sorption on the adsorbent samples. For notation, see Fig. 1.



Fig. 3. Sorption curves of Fe³⁺ ions under dynamic conditions by composite sorbents with fractions of (1) $d > 200 \,\mu\text{m}$ and (2) 67 $\mu\text{m} < d < 200 \,\mu\text{m}$.

amount of Fe^{3+} ions was adsorbed within the first 5 min, which indicates that CSs are more effective and characterized by shorter contact time for complete recovery of heavy metals from the aqueous solution.

According to the data in Fig. 2, the maximum adsorption rate is reached within the first 5 min for all samples.

The dependence of the efficiency of sorption of Fe^{3+} ions on the volume of transmitted eluent was studied under dynamic conditions on composite sorbents with fractions with 67 µm < *d* < 200 µm and *d* > 200 µm (Fig. 3).

According to the experimental data, the Fe³⁺ ions appear in the eluate at a transmitted volume of 1000 mL for the sorbent with particle sizes 67 μ m < d < 200 μ m and 25 mL for the sorbent with d > 200 μ m. After some time during dynamic sorption, the sorbent becomes saturated. Then the concentration of Fe³⁺ ions in the eluate increases. The sorption was stopped when the concentration of Fe³⁺ ions in the eluate became equal to that in the eluent.

Our study of sorption of Fe³⁺ ions on a composite sorbent under dynamic conditions with different particle sizes showed that the sorbent with $d < 67 \,\mu\text{m}$ is too finely dispersed. It passes into the filtrate and is thus difficult to handle. The sorbent with $d > 200 \,\mu\text{m}$ does not pass into the filtrate, but has the worst adsorption properties. The sorbent with 67 $\mu\text{m} < d <$ 200 μm has optimum texture and adsorption characteristics. The dynamic exchange capacities presented in Table 1 show that D_n of the sorbent with medium particle sizes ($67 \,\mu\text{m} < d < 200 \,\mu\text{m}$) is eight times larger than that of the sorbent with large particles (d >200 μm).

The prevailing mechanism of sorption kinetics can be roughly assessed from the shape of the curve. On the initial section of the curve of the composite sorbent with 67 μ m < d < 200 μ m, C/C_0 gradually slightly increases; at a volume above 1000 mL, it increases significantly. For the composite sorbent with large particle sizes (d < 200 μ m), the curve is characterized by a sharp increase in C/C_0 as the volume of the solution passed through the sorbent layer increases on the initial section (Fig. 3). The observed difference in the shape of the curve can be explained by the fact that for the same sorbent mass, the sample with smaller particles has a larger total surface area than the sample with larger particles (Fig. 3).

It was found that the composite sorbent with particle sizes of 67 μ m < d < 200 μ m is optimum for water purification from Fe³⁺ ions.

In the preliminary experiment, free passage of *E. coli* cells through the Schott filter was recorded. The detection of *E. coli* in smears from the centrifugate obtained after the 100 mL of *E. coli* suspension at a concentration of 100 thousand CFUs in 300 mL of

Table 1. Dependence of the total dynamic exchange capacity (D_n) on the sorbent particle size

V _{filt} , mL	$R_{\rm dil}$	A	$C_{\mathrm{Fe}^{3+}}, \mathrm{mg/L}$	C/C_0					
$d > 200 \mu\text{m}, D_n = 0.6 \text{mg/g}$									
0	0	0.00	0.0000	0.00					
25	0	0.66	5.4060	0.11					
50	4	1.41	11.6980	0.23					
75	4	2.03	16.8993	0.34					
100	10	2.87	23.9463	0.48					
125	10	3.56	29.7349	0.59					
150	10	4.21	35.1879	0.70					
175	10	4.88	40.8087	0.82					
200	10	5.43	45.4228	0.91					
225	10	5.95	49.7852	1.00					
250	10	1.00							
$67 \mu\text{m} < d < 200 \mu\text{m}, D_n = 4.8 \text{mg/g}$									
0	0	0.00	0.0000	0.00					
100	0	0.02	0.0000	0.00					
200	0	0.02	0.0000	0.00					
300	0	0.02	0.0000	0.00					
400	0	0.02	0.0000	0.00					
500	0	0.02	0.0000	0.00					
600	0	0.02	0.0006	0.00					
700	0	0.02	0.0008	0.00					
800	0	0.02	0.0017	0.00					
900	0	0.04	0.2047	0.00					
1000	0	0.09	0.6242	0.01					
1100	0	0.14	1.0436	0.02					
1200	0	0.23	1.7987	0.04					
1300	0	0.37	2.9732	0.06					
1400	0	0.45	3.6443	0.07					
1500	2	0.74	6.0772	0.12					
1600	4	1.32	10.9430	0.22					
1700	4	2.56	21.3456	0.43					
1800	10	5.71	47.7718	0.96					
1900	10	5.98	49.9501	1.00					
2000	10	6.03	49.9561	1.00					

 C_0 is the initial concentration of Fe³⁺ ions in the model solution, C is the concentration of Fe³⁺ ions in the filtrate, V_{filt} is the volume of filtrate, R_{dil} is the degree of dilution, A is the optical density, and D_n is the dynamic exchange capacity.

drinking water passed through the Schott filter was explained by the large pore size, which exceeded the bacteria size 40- to 50-fold and by the mobility of *E. coli* cells.

As a result of filtration of the model suspensions of *E. coli* through the developed composite sorbent and the set of sorbing components in the Barrier-4 replaceable filter cartridge for the household water purification filter, the sorption of *E. coli* was found to directly depend on the number of its CFUs in the model suspensions (Table 2). This regularity illustrated in Table 2 was revealed in a series of experiments where the concentration of *E. coli* in model suspensions ranged from 100000 to 100 million CFUs in 300 mL of drinking water.

It is noteworthy that purification of drinking water from *E. coli* by the test and control samples of sorption-filtering substances obeys the law of equilibrium concentration. The detection of *E. coli* in the smears from the filtrates of its aqueous suspensions (1 billion in 300 mL) in the sixth test and fourth control samples suggests that the available resources of the test and control samples of sorption substances were completely exhausted. According to Table 2, 3 g of the test sorbent and 3 g of the control sorbent are capable of adsorbing 1 billion 667 million and 998 million CFUs of *E. coli*, respectively.

A comparison of the obtained data shows that the efficiency of adsorption of *E. coli* of the composite sorbent exceeds that of the Barrier-4 sorbent 1.7-fold. The increased efficiency of adsorption of the composite sorbent obtained by pyrolysis of agricultural waste (sunflower seeds husks) with montmorillonite-containing clays during water purification from potentially pathogenic microflora can be explained by the high specific surface area (up to 200.0 m²/g), small particle size (100 μ m-3 mm), and the presence of micro- and macropores (total porosity 0.351 cm³/g).

Thus, the developed composite sorbent obtained by energy-efficient pyrolysis from agricultural waste (sunflower seed husks) showed high antibacterial activity. When a suspension of *E. coli* was filtered at a rate of 10 mL/min, 1 g of the composite sorbent adsorbed 556 million *E. coli* CFUs. It was proven that the efficiency of the composite sorbent in adsorbing *E. coli* during purification of infected water was 1.7 times higher than that of the complex sorbent of the replaceable Barrier-4 filter cartridge. Due to its low cost (75 rubles/kg), the developed composite sorbent can find wide application in national economy for purification of sewage and surface waters contaminated with potentially pathogenic microflora. The developed composite sorbent obtained by pyrolysis of

		Experiment 1			Experiment 2				Experiment 3				
No.	Number of CFUs of <i>E. coli</i> in 300 mL	sam- ple no.	filtered volume of <i>E. coli</i> suspension,	numl <i>E. ce</i> smear centri	ber of o <i>li</i> in s from fugate	sam- ple no.	filtered volume of <i>E. coli</i> suspension,	num E. co smear centri	ber of b <i>li</i> in s from fugate	sam- ple no.	filtered volume of <i>E. coli</i> suspension,	numl <i>E. ce</i> smear centri	per of p <i>li</i> in s from fugate
			mL	test	control		mL	test	control		mL	test	control
1	100 thou-	1	100	0	0	4	100	0	0	7	100	0	0
	sand	2	100	0	0	5	100	0	0	8	100	0	0
		3	100	0	0	6	100	0	0	9	100	0	0
2	1 million	1	100	0	0	4	100	0	0	7	100	0	0
		2	100	0	0	5	100	0	0	8	100	0	0
		3	100	0	0	6	100	0	0	9	100	0	0
3	10 million	1	100	0	0	4	100	0	0	7	100	0	0
		2	100	0	0	5	100	0	0	8	100	0	0
		3	100	0	0	6	100	0	0	9	100	0	0
4	100 million	1	100	0	0	4	100	0	0	7	100	0	0
		2	100	0	0	5	100	0	0	8	100	0	0
		3	100	0	0	6	100	0	0	9	100	0	0
5	1 billion	1	100	0	0	4	100	0	1.6	7	100	3.4	8.0
									± 0.86			± 0.86	± 1.50
		2	100	0	0	5	100	0	3.4	8	100	—	
			100	_			100		± 1.07		100		
		3	100	0	0	6	100	$\begin{vmatrix} 2.6 \\ \pm 1.07 \end{vmatrix}$		9	100		

Table 2. Results of adsorption of *E. coli* from model suspensions by the composite sorbent

0-no Esherichia in smears from the centrifugate; "-" no study has been performed.

agricultural waste (sunflower seed husks) with montmorillonite-containing clays can be recommended for complex water purification from heavy metal ions (Fe^{3+}) and potentially pathogenic microorganisms.

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