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Adsorption of Metal Ions on Aluminosilicates

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Abstract—The ODM-2F sorbent based on diatomite with a porosity of ~75% is used as a filter material for effective wastewater treatment at a thermal power plant. At pH 6.0–8.0, when metals Ni, Zn, Cu are in the form of Me^{2+} and $MeON^+$ cations, the process of their separation per MM is described by two types of adsorption isotherms – Langmuir and S-shaped. For unmodified MM in the Ca-form, S-shaped isotherms are characteristic. For MM, partially and completely converted to the Na-form, metal adsorption isotherms acquire a Langmuir appearance. An increase in the salt background of the solution (100–800 mg/dm^3 NaCl) does not reduce the sorption capacity for copper (120–130 mg/g), unlike organic resins. The adsorption of nickel, zinc, and copper cations at a pH of 7–8 per MM in the Ca-form (11–46 mg/g) is lower than in the Na-form (17–75 mg/g), which is due to the high energy costs when replacing Ca^{2+} with metal counterions compared to Na^+.

Keywords: montmorillonite, intercalation, polynomial, electro kinetic potential, adsorption isotherm, Langmuir equation

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INTRODUCTION

Sorption technologies are widely used for purification and separation of components of complex solutions. At the same time, synthetic ion-exchange resins were initially used. To isolate Cd^{2+} ions in the solid-phase extraction system, the Amberchrom CG-300m polymer resin was used as a sorbent, which is a styrene-acrylic ester modified by impregnation with ditizon (3.5 mg/g) to increase its efficiency by ~47%. The capacity of the modified resin depends on pH, initial metal concentration, temperature and duration, not exceeding 0.55 mg Cd^{2+}/g or 0.055 wt %/g, which is low [1]. When sorption of Cd^{2+} and Pb^{2+} ions on chelated resin (P-DETATA) in the Na-form obtained by fixing polysiloxane material on the initial DETATA, equilibrium adsorption of Pb/Cd = 0.25/0.17 mg/g or 0.025/0.017 wt %/g is achieved over a long period of time (100 min) in a narrow pH range 6.1/6.5 for both studied ions [2]. To ensure selectivity and improve sorption characteristics of polycondensation thiourea-formaldehyde (TF) resin, preformed magnetite particles (MTF) were included in its composition, followed by ultrasonic treatment (UT) to extract anionic forms of dyes: yellow titanium (TY) and bengal rose (RB), which increases the cost of reagent production. UT shortens the duration of reaching equilibrium (240–30 min) and increases the sorption capacity (from 0.3–0.6 to 0.9–1 mmol of dye/g) at pH 3. However, the presence of a magnetic component does not improve the kinetics of the process, and the stability of sorption characteristics is confirmed only during five cycles [3]. Hafnium-amino trisomethylene phosphonate (Hf-ATMP), which is an organic-inorganic hybrid sorbent for actinides, was exposed to ^{60}Co gamma rays with a dose of 2.4 mGy to increase the sorption capacity by 58% according to Th(IV), due to the conversion of P–O–H groups to P–O–Hf. Hf-ATMP sorbent is characterized by a complex production technology and a limited scope of use [4].

For the sorption of two rare-earth elements La^{3+} (light) and Dy^{3+} (heavy), the amidoximation of algae/polyethylenimine beads is used. In binary solutions, Dy^{3+} exhibits a slightly higher affinity for the sorbent than La^{3+}, without significant differences in selectivity. With the help of the EDTA complexing agent, the selectivity coefficient La/Dy can be increased to 40–50 with a gradual decrease in the sorption capacity over 3 cycles, which reduces the practical significance of the reagent [5].

An alternative to organic ion-exchange resins are mineral sorbents, which may include inorganic compounds of various classes (oxides, hydroxides, sulfides, phosphates, aluminosilicates, heteropoly acids and their salts, ferrocyanides, etc.), providing the necessary selectivity. Heavy metals are extracted by activated carbons, zeolites, clay minerals, solid waste. The ODM-2F sorbent based on diatomite with a porosity of ~75% is used instead of activated carbon BAU as a filter material for effective wastewater treatment at a thermal power plant. The reagent has an increased duration of use due to its high mechanical and chemical resistance, has a high sorption capacity for iron, manganese, heavy metals [6]. Fine clay particles with a diameter of <1 μm (EDCM) effectively immobilized heavy metals. The sorption process was controlled by internal diffusion, dominated by chemisorption. The maximum sorption capacity of Pb^{2+} was 263–588 mg/g or 26–59 wt %/g, depending on the size of clay particles, surface area, organic-mineral composition, and active functional groups [7]. Radionuclide cations $^{134}Cs^+$ and $^{152}Eu^{3+}$ are sorbed on negatively charged clay minerals (illite).

To describe the equilibrium distribution of cations between the solution and clay the model of non-electrostatic surface complexation and cation exchange with two protolysis sites (2SPNE-SC/CE) [8] is used. The combination of clay minerals (goethite, kaolinite, and montmorillonite) and heavy metals (Cd^{2+}) contributed to the development of the Serratia marcescens S14 biofilm, which increases Cd(II) adsorption. Functional groups –OH, –NH, $-CH_2$, –SH, –COO involved in Cd(II) immobilization were identified in the biofilm [9]. The sorption of thallium(I) on Mn(IV)-birnesites at pH 4–8, consisting of the predominant mechanism of irreversible oxidation with retention of Tl(III), formed mainly on free areas of the layer, and the subsequent formation of mineral avicennite (Tl_2O_3) at high pH values was investigated [10]. Sorption of perfluoroalkyl substances (PFASs) was analyzed: carboxylates (PFCA) and sulfonates (PFSA) on mineral reagents with organic carbon (OC) content in the range from 1.6 to 41%. It was shown that the sorption mechanisms in each phase depend mainly on the length of the PFAS chain containing from 3 to 11 fluorinated carbons. The degree of PFASs extraction depends on the OC content, the silt/clay ratio and the length of the PFAS chain [11]. To extract phosphorus (P), composite biochars—biochar impregnated with $FeCl_3$ or $MgCl_2$—were used, since mineral profiles play an important role in the interaction with metal chlorides and determine sorption and precipitation of P. Intercalation of $FeCl_3/MgCl_2$ provided a P extraction rate of approximately 60–100/50–100%, respectively, due to electrostatic attraction or exchange of PO_4^{3-} with –OH/–COOH ligands, which was explained by the enhancement of positive charges and –OH/–COOH on materials by these metal chlorides. The inherent minerals inhibited the conversion of $FeCl_3$ to Fe_3O_4 during pyrolysis and promoted the formation of $Fe_4(PO_4)_3(OH)_3$ during P sorption [12].

The joint sorption of several radionuclides (RN) from the uranium decay chain: ^{238}U, ^{230}Th, ^{226}Ra, ^{210}Pb, and ^{210}Po on minerals (chalcopyrite, bornite, pyrite, and barite) was investigated. Identified selective sorption of RN by various minerals: ^{226}Ra and ^{210}Po are quantitatively absorbed by all substances; pyrite and barite showed increased affinity for ^{226}Ra. The behavior of ^{210}Po was found to be abnormal: in both sulfate and nitrate solutions, ^{210}Po had little affinity for barite compared to sulfides. The series of selectivity of RN to minerals from various media has been revealed [13]. Smectite treated with Fe(III) significantly increased the sorption of atrazine (AT) and promethrin (PY) at pH 4.0 and 6.0, but decreased at pH 8.0. Intercalation of Cu(II) promoted sorption of AT and PY under alkaline conditions, but was suppressed at pH 4.0. For illite and kaolinite, Fe(III) and Cu(II), as a rule, enhance the sorption of AT and PY [14]. Sorption of herbicides (atrazine, metribuzine, amethrin, promethrin) on natural zeolites (clinoptilite, and Fe-mordenite) and clays (kaolin, attapulgite) with microporosity revealed selectivity ranges: for zeolites/clays – corresponding to the order of increase in hydrophobicity/basicity of the sorbate due to the significant contribution of hydrophobic effect/cation exchange and electrostatic interactions [15].

Thus, inorganic sorbents modified to ensure selectivity make it possible to selectively extract transition and non-ferrous metal ions from complex industrial solutions and wastewater.

The aim of this work was to study the effect of the ionic form and nature of precursors on the adsorption activity of the initial and modified montmorillonites, as well as the pH values on the efficiency of purification of solutions from nickel, zinc, copper. The objectives of the research are to evaluate the mechanism of interaction of sorbate ions with each other and with functional groups of mineral sorbents.

METHODOLOGY

The studied model and real mono- and polycomponent solutions had the following chemical composition, when the elements and their concentration varied for different objects of research, mg/dm^3: 9.5–11.5 Si; 1.0–250.0 Mn; 0.2–250.0 Ni; 90–500 Ca; 55–150 Na; 30–150 Mg; 0.1–1.5 Cu; 0.01–100.0 Zn; 0.01–0.2 Fe; pH 4.5–12.5.

Inorganic sorbents were obtained based on layered aluminosilicates with an expanding structural cell formed by three-layer packages in which one grid of Al–Fe–Mg-octahedra is articulated with two grids of silicon-oxygen Si–O-tetrahedra. Montmorillonite of the composition $(Na,Ca)_{0.3}(Al,Mg)_2Si_4O_{10}(OH)_2 \cdot nH_2O$, which is a dioctahedral formation with a three-layer package thickness of 0.94 nm, has the optimal characteristics for the synthesis of sorbents (Fig. 1).

When montmorillonite is modified, its transition into the gel form occurs with a weakening of the interparticle interactions of the individual structural elements due to the acquisition of an additional negative

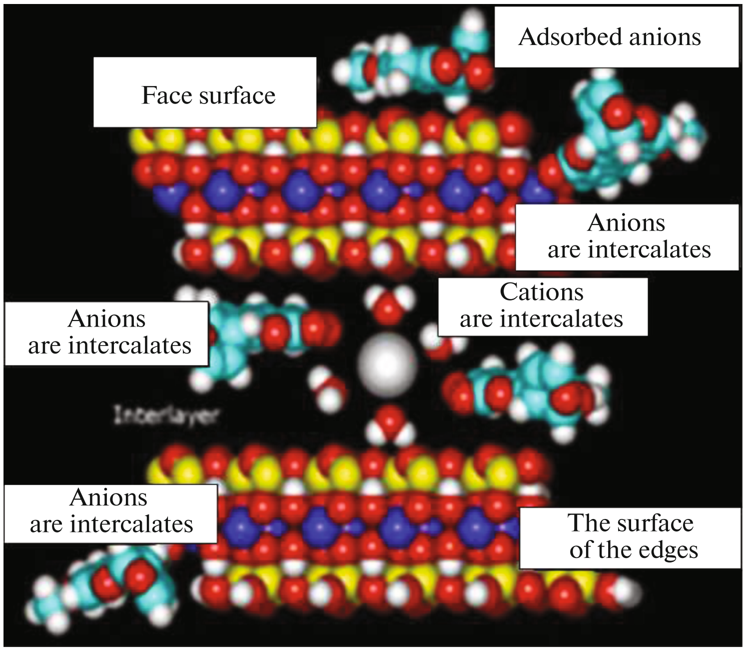

Fig. 1. Schematic representation of the intercalation modification of montmorillonite.

charge up to –(70–100) mV. When diluted, the gel spontaneously disperses into elementary packets ~10 nm thick. The process of "opening" the surface of the mineral increases the proportion of its outer region, which participates in the interaction with adsorbing ions from the solution. The introduction of certain types of modifiers leads to the appearance of active adsorption centers in the structure and on the surface of elementary packages of montmorillonite, creating a high sorption capacity. A number of modified samples of colloidal montmorillonite have been synthesized.

During the synthesis, sorption, electrosurface, rheological, and hydrophilic-oleophilic properties of natural aluminosilicates were directionally regulated. Intercallates (modifiers) determining the sorption properties of the reagent were Na_2CO_3 and organic substances taken in certain ratios with ether and carboxyl active groups in their composition.

Colloidal modified montmorillonite, dosed into the treated water, is a fairly thick gel-like dispersion that exhibits thixotropic properties. After daily swelling, the viscosity of 5% colloidal dispersion was 2.1–2.3 P. The formation of a thixotropic gel-like structure in montmorillonite dispersions can be considered as a connection of the edges of some layers with the flat surfaces of other layers to form a three-dimensional lattice system. These structures are destroyed to varying degrees when the system is stirred, but are formed again almost instantly when the stirring stops. The latter property of colloidal dispersions of montmorillonite turns out to be especially valuable in coagulation water purification due to the possibility of enlarging flakes of water contamination and coagulants by including them in the three-dimensional structures of the reagent.

A mixture of highly dispersed insoluble aluminosilicate with organic modifiers is an insoluble colloidal structure (sol) containing carboxyl groups and having a developed surface due to the small radius of the reagent particles, providing a high total surface area of the particles.

The chemical composition of the analyzed solutions was determined by inductively coupled plasma atomic emission spectrometry (Optima 4300 DV) and X-ray fluorescence spectrometry (S4 Explorer).

RESULTS

Experimental samples of sorbents from various deposits were studied: CB, Cherkasskoye (Ca-form); CBS, Cherkasskoe (Na-form); KB, Zyryanovskoe (Ca-form); KBS, Zyryanovskoe deposit (Na-form) obtained by modification of natural montmorillonites of Cherkasskoe/Zyryanovskoe deposits of composition, %: 51.9/54.9 SiO_2; 17.1/16.1 Al_2O_3; 7.9/6.3 Fe_2O_3; 1.2/1.6 MgO; 1.5/2.2 CaO; 0.2/0.4 Na_2O; 0.3/0.7 K_2O; 8.8/8.5 H_2O, involving several stages:

– treatment with Na_2CO_3 solutions to replace Ca^{2+} with Na^+ with the final content, %: 1.6–2.3 Na_2O; 0.25–0.45 CaO (Fig. 2a);

– intercalation of montmorillonites in the Na-form (IKBS) using anionic and amphoteric precursors of organic and inorganic nature, in particular sodium aluminate (Fig. 2b), followed by a test of the strength

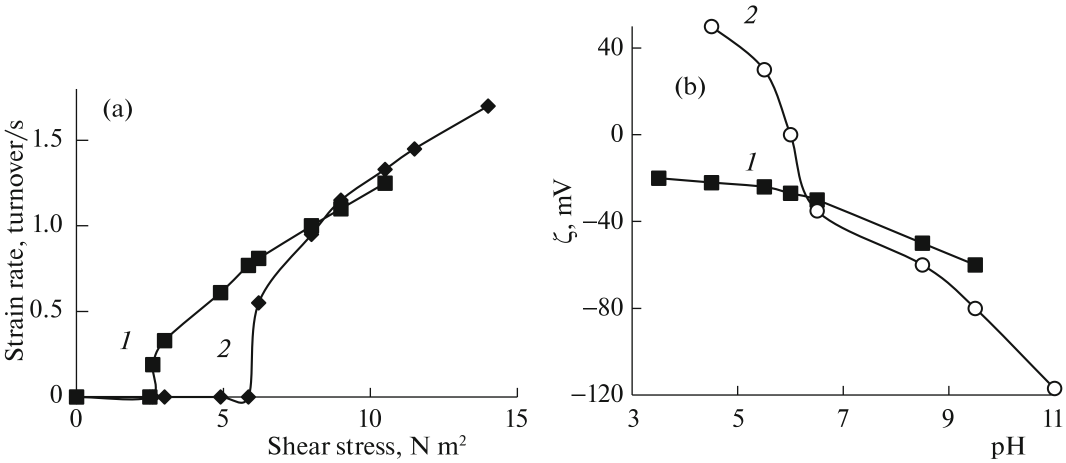

Fig. 2. Dependences of the strain rate on the shear stress (a) and the zeta-potential on the pH (b) for natural (*1*) and modified (*2*) montmorillonite.

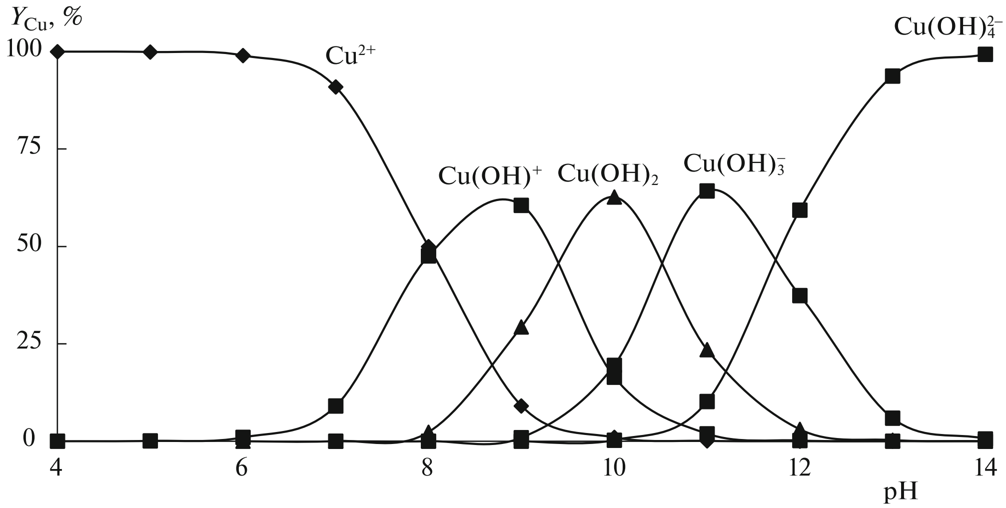

Fig. 3. Dependences of the content of copper hydroxocomplexes (Y_{Cu}) on the pH of the solution.

of the intercalate fixation in the fine-porous structure of montmorillonite.

In accordance with the values of the stability constants of hydroxocomplexes, the content of ionic forms of copper (Fig. 3) and previously nickel, zinc was calculated depending on the pH of the solution.

Figure 4 additionally illustrates the dependences of the metal hydroxide content (Y_{Me}, mg/dm^3), depending on the pH value of the solution (X_1) and the initial concentration of the corresponding metal (X_2, mg/dm^3) which are satisfactorily described by second-degree polynomials:

$$Y_{Ni} = -7.629 + 1.371X_1 + 0.056X_1X_2 - 0.060X_1^2 + 0.215X_2^2,$$

$$Y_{Zn} = -6.786 + 1.345X_1 + 0.058X_1X_2 - 0.066X_1^2 + 0.267X_2^2,$$

$$Y_{Cu} = -4.188 + 0.838X_1 + 0.011X_1X_2 - 0.041X_1^2 + 0.459X_2^2.$$

According to the distribution of hydroxocomplexes diagrams, the pH regions of the existence of cationic forms of Me^{2+} and $MeOH^+$ were revealed: 6.0–8.0 Zn, Cu; 7.0–8.5 Ni. Sorption of cationic forms of metals on modified montmorillonites corresponds to different types of isotherms, namely, S-shaped and Langmuir (Fig. 5). The value of the maximum adsorption of metal ions on montmorillonite depends on its location form (Table 1).

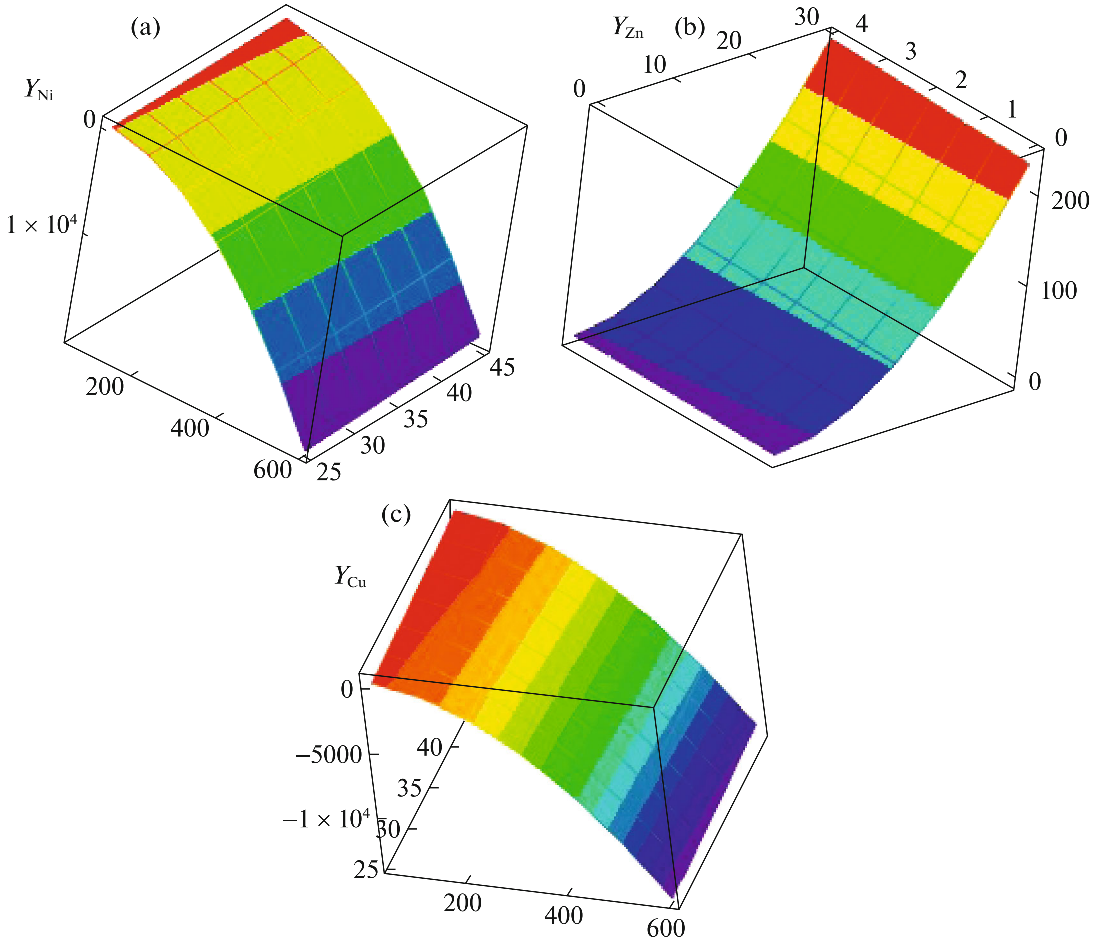

Fig. 4. Dependences of the content of metal hydroxides (Y_{Me}, mg/dm^3): nickel (a), zinc (b), copper (c), on the pH value of solutions (X_1) and the initial concentration of metals (X_2, mg/dm^3).

Further studies were limited to intercalation of the Na-form of montmorillonite by precursors, in particular, modification by a mixture of anionic surfactants (Figs. 5i–5k) illustrates the results of the conducted studies.

A fork of isotherms for the sodium and intercalated forms of montmorillonite was identified at pH 6.0 (Fig. 6).

With an increase in pH, the Langmuir type of isotherms turns into an S-shaped one (Fig. 7).

DISCUSSION

The Na-form of montmorillonite differs from the Ca-form by a large shear stress (Fig. 2a), an increase in the values of the specific surface area and zeta potential of the particles and, as a consequence, better adsorption characteristics.

The nature of the adsorption isotherms is determined by the physico-chemical properties of the surface of the adsorbents and the forms of the metals in solution. Natural unmodified montmorillonites (Figs. 5a, 5b, 5e, 5f) are characterized by S-shaped isotherms. For montmorillonites partially (Figs. 5c, 5g) and completely (Figs. 5d, 5h) converted into the Na-form, metal adsorption isotherms acquire a Langmuir appearance. The values of the limiting adsorption and the adsorption equilibrium constants were graphically determined from the Langmuir equation in linear form in the coordinates $1/a$–$1/C$.

In the case of S-shaped isotherms, due to the complexity of the mathematical determination of the maximum adsorption, the latter were determined approximately, as the maximum adsorption obtained experimentally (Figs. 5a, 5e).

By the example of copper, the effect of the salt background of the solution on the extraction of metal ions was studied (Fig. 5k), the increase of which ($C \leq 500$–800 mg/dm^3 NaCl) does not significantly affect the sorption capacity in comparison with conventional ion exchange resins. For comparison, with an increase in the ionic strength of the solution, the

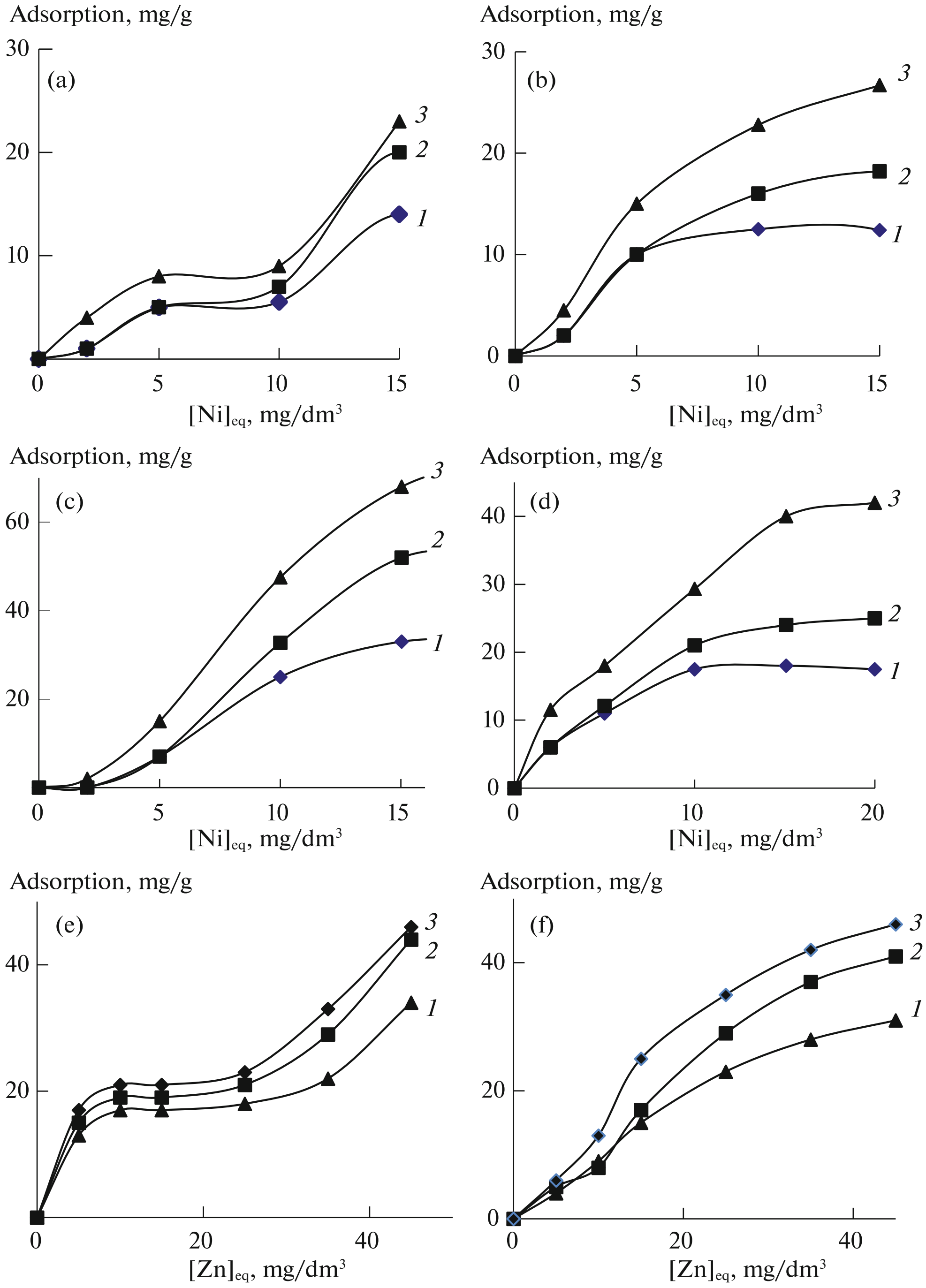

Fig. 5. Adsorption isotherms of Ni (a–d), Zn (e–h), Cu (i–k) samples: KB (a, e), CB (b, f), KBS (c, g, i), CBS (d, h) after intercalation of IKBS (i–k) at pH: 6.0 (*1*); 6.5 (*4*–*7*); 7.0 (*2*); 8.0 (*3*); 9.0 (*4*), and concentration of NaCl, mg/dm^3: 100 (*5*); 300 (*6*); 500 (*7*); 800 (*8*).

absorption of copper by the selective sorbent ST-1 decreases from 30 to 10–16 mg/g. The value of the limiting adsorption of metal ions on montmorillonite depends on its location form: with the Na-form of montmorillonite, the absorption of elements is significantly greater than for the Ca-form (Table 1). This is evident, since the substitution of calcium counterions for nickel, zinc, and copper ions in the structure of montmorillonite proceeds with high-energy costs, compared to the substitution of sodium counterions for metal ions.

For mineral montmorillonites in different forms (Ca, Na), the different type of isotherms is explained by the energy disparity of the surface of thin pores,

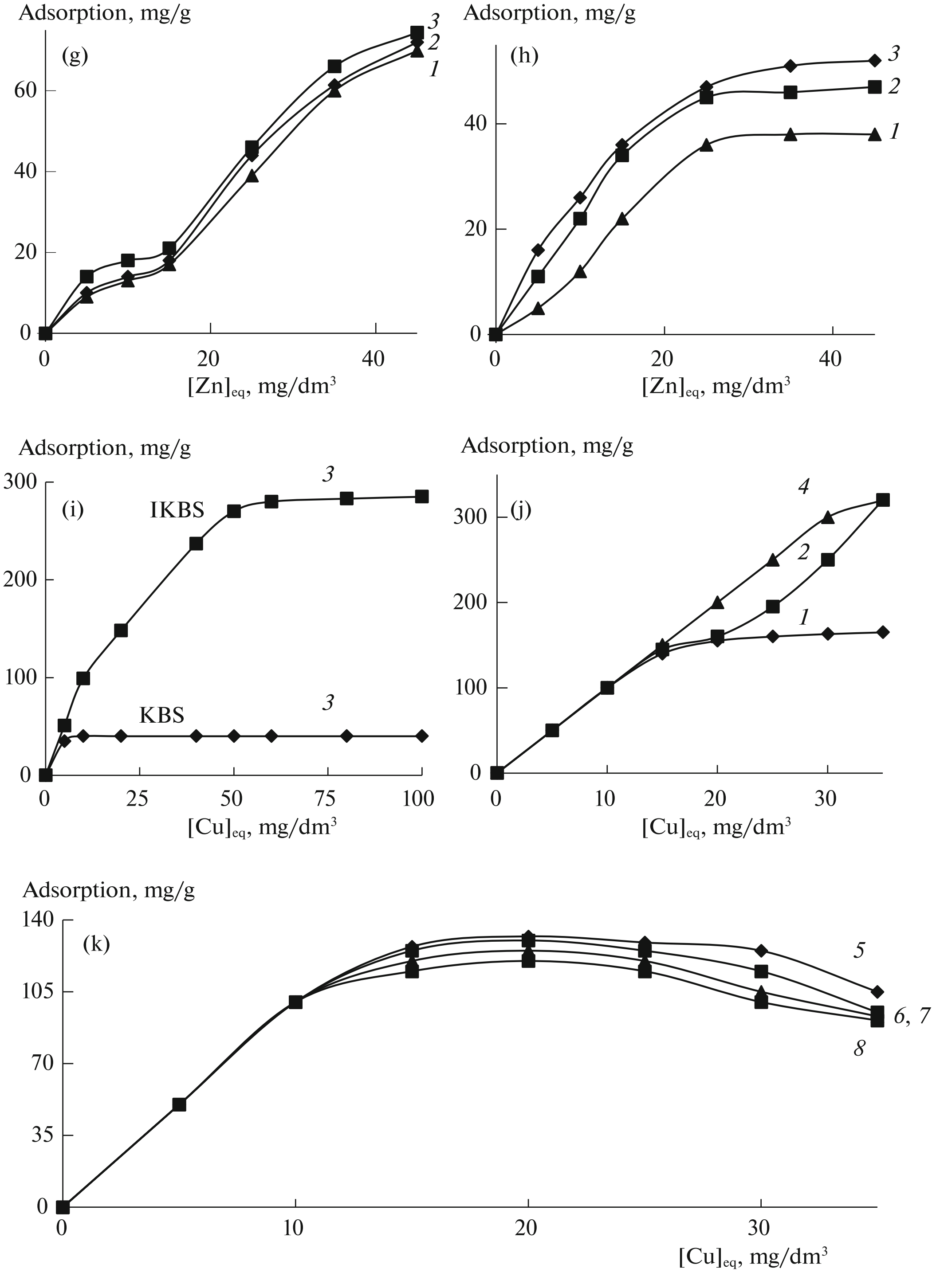

Fig. 5. (Contd.)

with different adsorption sites by nature. As a result of the modification of the surface, which consists in the complete replacement of calcium ions with sodium, it becomes energetically equivalent. As a result, monomolecular surface layers are formed during the adsorption of metal cations, which is typical for Langmuir-type isotherms.

When transferring bentonite from calcium to sodium form, not only the type of adsorption isotherms changes but also the maximum adsorption A_∞ increases with an increase in pH to a value of 8.0.

Further studies were limited to intercalation of the Na-form of montmorillonite with precursors, in par-

Table 1. The values of the maximum adsorption (A_∞) and the type of nickel isotherm

Sorbent	A_∞, mg/g			Type of isotherm
	Ni	Zn	Cu	
KB	14.0 (pH 7.0) 22.0 (pH 7.5) 22.0 (pH 8.0)	34.0 (pH 7.0) 44.0 (pH 7.5) 44.2 (pH 8.0)	11.2 (pH 7.0) 17.0 (pH 7.5) 19.3 (pH 8.0)	S-Shaped
CB	12.5 (pH 7.0) 18.0 (pH 7.5) 27.0 (pH 8.0)	31.0 (pH 7.0) 41.0 (pH 7.5) 46.0 (pH 8.0)	10.4 (pH 7.0) 16.2 (pH 7.5) 23.7 (pH 8.0)	S-Shaped
KBS	33.0 (pH 7.0) 54.0 (pH 7.5) 71.0 (pH 8.0) 64.0 (pH 8.5) 57.0 (pH 9.0)	69.9 (pH 7.0) 74.4 (pH 7.5) 75.0 (pH 8.0) 67.0 (pH 8.5) 60.0 (pH 9.0)	27.5 (pH 7.0) 34.4 (pH 7.5) 40.2 (pH 8.0) 35.3 (pH 8.5) 31.1 (pH 9.0)	S-Shaped and Langmuir
CBS	18.0 (pH 7.0) 25.0 (pH 7.5) 42.0 (pH 8.0)	38.0 (pH 7.0) 47.0 (pH 7.5) 52.0 (pH 8.0)	16.7 (pH 7.0) 22.3 (pH 7.5) 37.8 (pH 8.0)	Langmuir

ticular, modification with a mixture of anionic surfactants—alkylcarboxysiloxanes $(-R_2Si_3O_5)_{70-80}$. Figures 5k and 6 illustrate the results of the conducted studies.

It is shown that as a result of modification of the montmorillonite surface, the sorption capacity increases significantly compared to the Na-form of montmorillonite: the maximum adsorption of metals on sorbents subjected to intercalation reaches 175 mg/g (5.96 mg-eq/g Ni), 152 mg/g (4.65 mg-eq/g Zn), 285 mg/g (8.9 mg-eq/g Cu), while the maximum adsorption on sorbents in the Na-form is 71 mg/g (2.41 mg-eq/g Ni), 75 mg/g (2.30 mg-eq/g Zn), 40 mg/g (1.2 mg-eq/g Cu). In comparison with organic resins, synthesized mineral nanodisperse sorbents have a commensurate or greater sorption capacity. For example, the maximum static exchange capacity (SEC) of microporous anionite AN-401 is 4.5–5.6 mg-eq/g.

It is established that the Langmuir type of isotherms is observed on both the sodium and intercalated form of montmorillonite at pH 6.0 (Fig. 6). With an increase in pH, the Langmuir type of isotherms turns into an S-shaped one (Fig. 7), which may be due to the transition of metal cations Me^{2+} into hydroxoforms $MeOH^+$, as well as with the intensification of polycondensation processes of various forms of metal ions in the thin pores of the adsorbent and with the appearance of a multilayer adsorption.

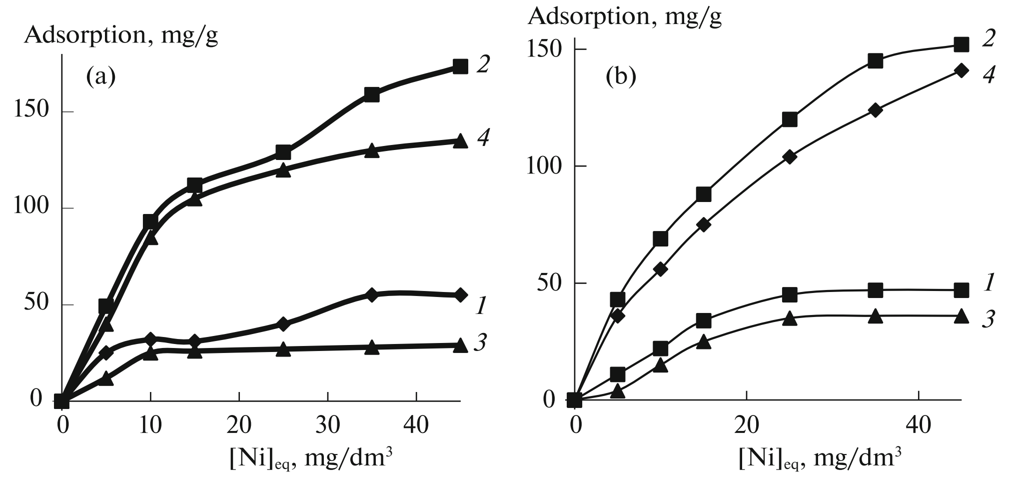

Fig. 6. Isotherms of adsorption of metals: Ni (a); Zn (b) from solutions on montmorillonite: Na-form (*1*, *3*); modified (*2*, *4*) at pH values: 6.0 (*3*, *4*) and 7.5 (*1*, *2*).

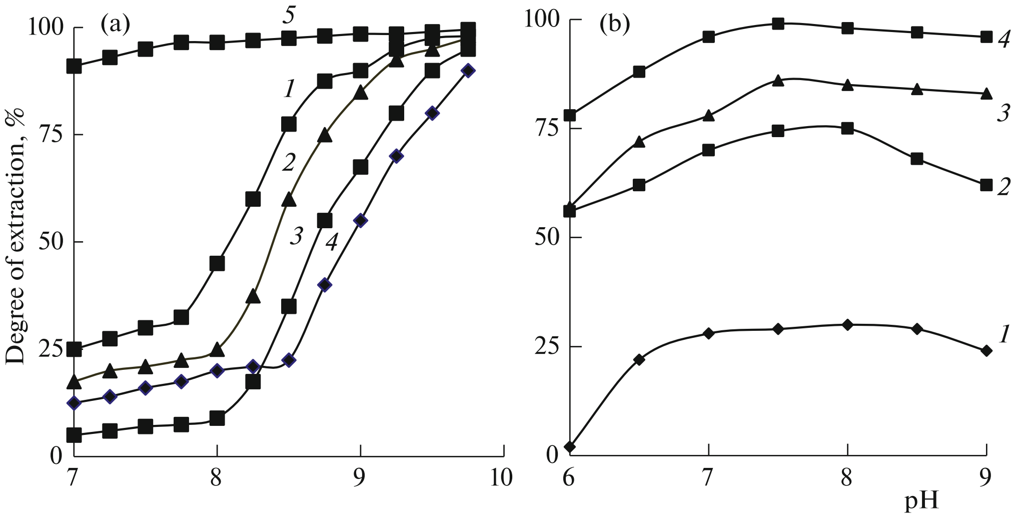

Fig. 7. Dependences of the degree of extraction of nickel (a), zinc (b) from the solution on the pH for KBS (*1*); CBS (*2*); KB (*3*); CB (*4*); and after intercalation of IKBS (*5*).

CONCLUSIONS

(1) At pH values (7.0–8.5 Ni; 6.0–8.0 Zn, Cu), in which metals are in the form of Me^{2+} and $MeOH^+$ cations, the process of their separation from solutions on highly dispersed montmorillonites is described by two different types of adsorption isotherms—Langmuir and S-shaped. The nature of the adsorption isotherms is also determined by the physicochemical properties of the adsorbent surface: natural unmodified montmorillonites are characterized by S-shaped isotherms. For montmorillonites partially and completely converted into the Na-form, metal adsorption isotherms acquire a Langmuir appearance.

(2) An increase in the salt background of the solution (100–800 mg/dm^3 NaCl) does not significantly affect the sorption capacity when extracting copper ions (120–130 mg/g) in comparison with conventional ion exchange resins.

(3) The adsorption of nickel, zinc, and copper cations at pH 7–8 on montmorillonite in the Ca-form (11–46 mg/g) is lower than in the Na-form (17–75 mg/g), which is due to the high-energy costs when replacing Ca^{2+} with metal counterions, compared with Na^+.

(4) With subsequent intercalation of the Na-form of montmorillonite by precursors, in particular, modification with a mixture of anionic surfactants—alkylcarboxysiloxanes $(-R_2Si_3O_5)_{70-80}$, the capacity of the mineral sorbent increases from 40–71 to 152–285 mg/g, as well as the range of pH values corresponding to a high degree of metal extraction expands, which is due to the intensification of adsorption and polycondensation processes in the thin pores of the sorbent due to the increase of the energy affinity between the active surface areas and adsorbent ions.

(5) For the slightly alkaline region (pH >8.0) revealed:

– linear isotherm of copper adsorption due to the formation of $Cu(OH)_2$ hydroxide and its subsequent mutual coagulation with adsorbent particles in heterocoagulation mode (Fig. 5j, the curve *1*);

– at low nickel concentrations (~5 mg/dm^3), a polycondensation process occurs, leading to the formation of $Ni(OH)_2$ hydroxide in the sorbent phase which intensifies with further increase in pH values (Fig. 7a);

– expansion of the pH region with high nickel extraction (Fig. 7a, the curve *5*), due to intercalation of montmorillonite by one of the amphoteric precursors;

– a slight decrease in the degree of zinc extraction (Fig. 7b) associated with the formation of zinc ions $Zn(OH)^{3-}$ in the surface layers of montmorillonite, the particles of which are treated with alkaline precursors, when zinc ions are desorbed from the solid phase into the liquid like amphoteric metals (copper, aluminum, etc.).

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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