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Nonlinear Fitting of Iron Sorption on Bentonite to Theoretical Isotherm Models

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A comparative analysis of adsorption models application for the description of experimental isotherms of iron adsorption on two samples: natural bentonite and bentonite irradiated with microwave irradiation. Sorption isotherms are described using the theories of Langmuir, Freundlich, Redlich-Peterson and Langmuir-Freundlich. The constants and parameters of these equations are determined. Nonlinear fitting of experimental data to the theoretical models of isotherms showed that adsorption by native bentonite corresponds to the three-parameter Redlich-Peterson model, and microwave irradiated - to the Freindlich isotherm. This modeling allows to predict the maximum adsorption capacity, which is 37.7 mg/g of natural bentonite and 64.4 mg/g of the modified sample.

Keywords: bentonite, microwave, adsorption, iron, non-linear fitting.

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Introduction

Iron is one of the most widespread natural elements. Its presence in natural water is a common problem, as it causes a lot of problems, both for industrial and residential users. Iron can get into sewage from pickling and galvanic units, operations of metal surfaces preparation, sections of fabrics dyeing and other productions. In the aqueous medium it is most often present in the form of iron oxide, bicarbonate, sulfide.

Currently wastewater treatment of various industries is the issue of the day due to the growing anthropogenic pressure on the environment. Heavy metal ions are one of the most common environmental pollutants, which lead to changes in the physicochemical properties of wastewater, disrupt the processes of biological self-purification of water bodies, affect the health of humans and living organisms [1, 2]. Known methods of wastewater treatment from heavy metal ions are based on colloidal chemical processes of flocculation, adsorption, precipitation, etc. However, most of them are expensive, difficult to use, need expensive equipment and scarce reagents. In this regard, inexpensive and efficient methods of wastewater treatment based on the use of industrial waste and local raw materials as a sorbent are of much-needed [3-6]. The efficiency of microwave treatment of bentonite for improving of its adsorption capacity against heavy metal ions Zn [7, 8], Pb [9], Cu [10] was described earlier. This paper presents the results of adsorption of Fe ions from aqueous solutions under the action of microwave irradiation.

Adsorption is a process that is widely used to remove contaminants from wastewater. In order to design the adsorption process and its further efficient operation, it is necessary to establish the parameters of the equilibrium state of the process for its further application in kinetic and mass transfer models. These models can be used to predict the efficiency of adsorption in different operating conditions.

The main purpose of this research is to establish the type of isotherms giving the best description of the sorption on an investigated material. Sets of isotherm parameters were determined using minimizing a number of error functions.

I. Materials and methods

1.1. Experimental research

According to X-ray phase analysis, the natural bentonite consists of four main minerals: hydromica (about 27 %), montmorillonite (about 25 %), quartz (about 22 %), chlorite (about 20 %) and some impurities (calcite, sylvinite and other minerals). pH of the aqueous extract of the clay sorbent is 8.92; bulk density – 1244 kg/m^3; swelling factor ~ 2.

Sorption properties of bentonite were investigated under static conditions. Adsorption was performed in two series: at room temperature and under the microwave irradiation.

The first series of experiments was obtained as follows. Ten 150 ml beakers were filled with 100 ml of working solution of appropriate concentration and 1.0 g of bentonite was added, stirred and left for 24 hours. The solutions were then filtered and analyzed for the content of iron (III) ions.

The second series of samples after mixing with the adsorbent was treated with microwave EMI of medium power in three stages of 120 sec each. After each step, the solution was stirred. After cooling, the solution was filtered and followed by determination of equilibrium concentrations of iron (III) ions in the solution.

The content of Fe^{3+} ions in the solutions was determined by the atomic absorption method. All experiments were repeated three times.

1.2. Isotherm fitting

The dynamic process of adsorption from a solution by a solid adsorbent depends on the characteristics of the equilibrium distribution of the substance between the two phases. The adsorption equilibrium is reached when the amount of adsorbed substance is equal to the amount of desorbed. At this point, the equilibrium concentration of the solution remains constant. The graphical dependence of the equilibrium concentration of a substance in the solid phase on its equilibrium concentration in solution presents an equilibrium adsorption isotherm. There are many theories of adsorption equilibrium.

The Langmuir isotherm model

Langmuir's isotherm theory assumes the formation of an adsorbate monolayer on the homogeneous surface of the adsorbent [11]. When the equilibrium state is reached, no further adsorption takes place. In this model, it is assumed that sorption occurs in certain homogeneous areas on the surface of the adsorbent. As soon as a certain molecule occupies one of the sites, further adsorption at this site does not occur. In equation (1), K_L is the Langmuir isotherm constant, which characterizes the affinity of the adsorbent and adsorbate, dm^3/mg; q_m is the maximum sorption capacity, mg/g of sorbent; C_e and q_e are the equilibrium concentrations of the component in the liquid and solid phases, respectively.

$$q_e = \frac{q_m K_L C_e}{1+K_L C_e} \tag{1}$$

The Langmuir equation is used to describe homogeneous absorption when the sorption of each molecule has the same activation energy.

The Freundlich isotherm model

The Freundlich model (2) is an exponential equation, and therefore it can be assumed that with increasing solution concentration, the concentration of adsorbate on the surface of the adsorbent increases. Theoretically, this equation allows an infinite adsorption process [12].

$$q_e = K_F C_e^{1/n_f} \tag{2}$$

In this equation, K_F is the Freundlich isotherm constant, which characterizes the adsorption capacity, mg/g of sorbent. When the value of K_F increases, the adsorption capacity grows. Since this equation is characterized by n_f heterogeneity coefficient, therefore the Freundlich isotherm model can be used for heterogeneous systems [13, 14]. The Freundlich's equation corresponds with Langmuir's theory in the range of low concentrations, but, unlike the Langmuir's model, it is not reduced to a linear isotherm (Henry's law) at low surface saturation. Both of these theories have one drawback: the values of equilibrium concentrations in a wide range of concentrations cannot be described by the same set of constants (McKay, 1980).

The Redlich-Peterson isotherm model

Redlich and Peterson [15] proposed a three-parameter empirical equation that can be used to describe adsorption equilibrium over a wide range of concentrations [16]. This is a hybrid isotherm model that includes Freundlich and Langmuir isotherms. Since this model is a combination of two models, the adsorption mechanism does not correspond to the ideal monolayer adsorption. This is a universal isotherm model that can be used for homogeneous and heterogeneous systems.

$$q_e = \frac{K_{RP} C_e}{1+a_{RP} C_e^{\beta}} \tag{3}$$

This equation is reduced to a linear isotherm at low concentrations of adsorbate on the surface, to a Freundlich isotherm at high concentrations of adsorbate, and to a Langmuir isotherm at $\beta = 1$.

The Langmuir-Freundlich isotherm model

At low concentrations of the adsorbate, the Langmuir-Freundlich isotherm model is reduced to the Freundlich isotherm, while at high concentrations it predicts the adsorption capacity of the monolayer, which is inherent to the Langmuir isotherm.

$$q_e = \frac{q_m (K_{LF} C_e)^{nLF}}{1+(K_{LF} C_e)^{nLF}}, \tag{4}$$

where q_m and K_{LF} are the adsorption capacity and the affinity constant, respectively, n_{LF} is the coefficient of heterogeneity or a measure of the adsorption intensity. If $n_{LF} = 1$, then equation (5) reduces to the Langmuir isotherm model.

Since the conversion of an isotherm into linearized forms leads to a change in the error structure of experimental data, nonlinear analysis has become indispensable because it provides an accurate method for determining adsorption parameters without changing the original form of isothermal equations. In addition, linear modeling is unsuitable for three-parameter isotherms. In the context of this work, five different error functions were investigated, and in each case the isotherm parameters were determined by minimizing the corresponding error function in the range of liquid phase concentrations using the "Solver" add-in in the MS Excel spreadsheet.

1. The sum of square errors (SSE). This widely used error function has one major drawback. The function provides better compliance with the calculated parameters in the upper range of liquid phase concentrations. This is due to the fact that the magnitude of the errors and, consequently, the square of the errors will increase with increasing concentration.

$$\sum_{i=1}^{n}\left(q_{e\_exp}-q_{e\_calc}\right)_i^2 \qquad (5)$$

where q_{e_exp} and q_{e_calc} are the content of adsorbate in the sorbent determined experimentally and calculated, respectively, mg/g of sorbent;

2. Hybrid fractional error function (HYBRID). This error function was developed [17] in order to improve the compliance of the SSE function in the range of low concentrations of the liquid phase. In this method, each SSE value was divided by the experimental value of the solid phase concentration. In addition, the divisor includes the degree of freedom of the system – the number of experimental points minus the number of parameters in the equation of the isotherm.

$$\frac{100}{n-p}\sum_{i=1}^{n}\left(\frac{(q_{e\_exp}-q_{e\_calc})^2}{q_{e\_exp}}\right)_i \qquad (6)$$

p is the number of model parameters to be determined.

3. Marquard's percent standard deviation (MPSD). This error function [18] is similar to a modified standard deviation to take into account the number of degrees of freedom of the system.

$$100\sqrt{\frac{1}{n-p}\sum_{i=1}^{n}\left(\frac{q_{e\_exp}-q_{e\_calc}}{q_{e\_exp}}\right)_i^2} \qquad (7)$$

4. Average relative error (ARE) [19]. This error function minimizes the distribution of fractional particles over the entire concentration range.

$$\frac{100}{n}\sum_{i=1}^{n}\left|\frac{q_{e\_exp}-q_{e\_calc}}{q_{e\_exp}}\right|_i \qquad (8)$$

5. The sum of absolute errors (SAE). In this error function, the approach is similar to the SSE method. The isotherm parameters determined by this method will be better matched as the magnitude of the errors increases, i.e. shifting towards the data from the high concentrations range.

$$\sum_{i=1}^{n}\left|q_{e\_exp}-q_{e\_calc}\right|_i \qquad (9)$$

For more accurate models evaluation, two experimental measurements were performed for experimental values that were not used to model the isotherms. For each model, the isotherm parameters were calculated by minimizing errors and calculating other error functions and the sum of normalized errors (SNE). The most suitable isotherms were selected based on the error values of the experimental data and were used to establish the adsorption mechanism and determine the maximum adsorption capacity of the adsorbent.

II. Results and their discussion

As a result of experimental research and calculation of the maximum sorption capacity by nonlinear modeling in the Langmuir model, it was found that bentonite treatment with microwave irradiation increases the sorption capacity of the material by 1.7 times. Accordingly, the saturation of natural bentonite from 37.7 increases to 64.4 mg/g of sorbent.

Isotherms parameters determined by minimizing error functions are given in Tables 1-4. It is obvious that the lowest value of the sum of normalized errors, in case of the sorption by natural bentonite, is achieved mainly by minimizing the sum of absolute errors and/or average relative error, which is inherent to the high concentration range. Instead, in the case of an irradiated sample, the

Table 1

The maximum sorption capacity q_m and the Langmuir isotherm constant K_L determined by the results of error analysis.

	Minimized error				
	SAE	ARE	SSE	MPSD	HYBRID
Natural bentonite					
q_m	37.700917	37.700932	37.424889	36.581272	36.856051
K_L	0.108400236	0.108397008	0.10145356	2.303186403	0.127669975
SAE	**19.437**	19.562	19.437	29.066	21.900
ARE	14.083	**12.996**	13.387	16.503	14.084
SSE	142.397	132.457	**131.247**	155.527	134.553
MPSD	36.8278	35.531	35.657	**34.611**	35.233
HYBRID	163.116	150.868	151.164	153.230	**149.642**

Table 1(continuation)

SNE	4.328452	4.325778	**4.322607**	4.3623269	4.3378071
Microwave-treated bentonite					
q_m	64.636792	64.641621	64.297085	63.867789	64.439166
K_L	0.042587553	0.042580848	0.046920508	0.046580696	0.04552693
SAE	19.942	19.942	22.193	21.615	21.483
ARE	13.082	13.082	14.018	13.781	13.729
SSE	156.639	156.639	**150.405**	151.647	150.946
MPSD	35.6880	35.6880	35.623	**35.610**	35.616
HYBRID	157.709	157.709	156.565	156.542	**156.321**
SNE	4.354765	4.354765	4.354089	4.3545008	**4.3538925**

Table 2

The heterogeneity coefficient n_f and Freundlich isotherm constant of K_F determined by the results of error analysis.

	Minimized error				
	SAE	ARE	SSE	MPSD	HYBRID
Natural bentonite					
n_f	6.8480727	6.85	7.5109224	6.8002076	7.0680319
K_F	15.14552989	15.15	16.39741252	15.17407098	15.6358158
SAE	**12.206**	12.206	12.507	12.388	12.335
ARE	4.173	**4.173**	5.231	4.223	4.577
SSE	25.071	25.071	**21.101**	25.151	22.594
MPSD	5.608	5.608	7.244	**5.551**	5.827
HYBRID	10.448	10.448	11.093	10.352	**9.868**
SNE	**3.448**	**3.448**	3.498	3.480	3.585
Microwave-treated bentonite.					
n_f	4.3010416	4.26	4.7303056	4.1520125	4.303366
K_F	15.53661922	15.32	17.49209459	14.70767554	15.53722861
SAE	**32.942**	33.343	34.060	35.522	32.961
ARE	9.235	**9.219**	11.573	10.001	9.241
SSE	206.431	210.282	**190.320**	224.939	206.432
MPSD	15.300	15.152	18.238	**14.949**	15.296
HYBRID	76.796	76.913	86.341	78.943	**76.794**
SNE	3.597	3.582	3.778	**3.544**	3.597

Table 3

Redlich-Peterson isotherm coefficients determined by the results of error analysis.

	Minimized error				
	SAE	ARE	SSE	MPSD	HYBRID
Natural bentonite					
β	0.8834793	**0.8834793**	0.8821875	0.8705815	0.8760855
a_{RP}	13.77	**13.77**	14.00	22.88	17.84
K_{RP}	251.29139	**251.29139**	251.27828	383.58885	308.8414
SAE	**9.321788809**	9.321788809	10.0388077	10.59500348	10.33177327
ARE	3.377237862	**3.377237862**	3.611364056	3.598404691	3.591417813
SSE	16.39990674	16.39990674	**15.60777264**	17.16392933	16.04958861
MPSD	4.758315152	4.758315152	4.497875417	**4.193481495**	4.260172455
HYBRID	9.322208184	9.322208184	8.611214248	8.538878227	**8.325524995**
SNE	3.59	**3.59**	3.67	3.67	3.70
Microwave-treated bentonite.					
β	0.779656	0.7798172	0.7886096	**0.759176**	0.7722366
a_{RP}	36.21	36.21	9099.98	**10814.25**	36.83
K_{RP}	586.3722	586.25741	159187.91	**159071.37**	586.92939
SAE	**32.34694435**	32.35095474	34.05624852	35.51954385	32.83360906
ARE	9.130463759	**9.127700765**	11.57085179	10.00173148	9.417313945
SSE	210.8315159	211.9887439	**190.3015599**	224.9036963	200.3522502
MPSD	13.78904694	13.79265236	16.08224763	**13.18476915**	13.66332899
HYBRID	92.82348394	93.11731244	100.7171522	92.09544296	**89.23610611**
SNE	3.79	3.78	3.95	**3.71**	3.79

Table 4

The adsorption capacity q_m, the affinity constant K_{LF} and the coefficient of heterogeneity n_{LF} of Langmuir-Freundlich isotherm model determined by the results of error analysis.

	Minimized error				
	SAE	ARE	SSE	MPSD	HYBRID
Natural bentonite					
q_m	60.51720934	72.50	57.59	72.64	60.5171511
K_{LF}	0.014440097	0.002845539	0.020050544	0.002601976	0.01315058
n_{LF}	0.2464876	0.2220414	0.2648458	0.2217918	0.2506052
SAE	**7.603379498**	7.879	8.019	8.616	8.009653603
ARE	3.558172654	**3.531**	4.228	3.898	3.960733997
SSE	10.21882702	11.015	**9.313**	10.799	9.474606668
MPSD	5.869405142	5.378	6.082	**5.195**	5.551314518
HYBRID	9.085876707	8.667	8.600	8.316	**8.119882882**
SNE	**3.871132**	3.901336	4.0419979	4.0500803	4.0833367
Microwave-treated bentonite.					
q_m	64.474981	232.37	232.37079	82.818686	141.7157
K_{LF}	0.042793202	4.77568E-05	6.43714E-05	0.02187358	0.001135538
n_{LF}	1.0146113	0.2642641	0.2810225	0.4905086	0.3250694
SAE	**19.81523714**	30.316	27.632	31.795	29.75217404
ARE	16.7817071	**11.526**	15.955	12.483	13.1539591
SSE	156.6931986	185.247	**134.353**	189.683	154.6600378
MPSD	47.38382313	20.817	29.267	**20.093**	21.48811219
HYBRID	277.4234244	131.125	151.461	127.166	**121.2695626**
SNE	**4.02837**	4.230994	4.3494794	4.1869799	4.2911517

Table 5

The results of the errors analysis based on the calculation of the deviation of the experimental results from those calculated from different isotherm models.

q_m obtained by theoretical isotherm models		Minimized error					SNE
		SAE	ARE	SSE	MPSD	HYBRID	
Natural bentonite – coordinates of additional point [324.4;37.12]							
Langmuir	36.31	0.798714	2.15171	0.637944	2.151708495	1.718600115	5.270306
Freundlich	35.24	1.885445	5.07932	3.554904	5.079324932	9.576789904	6.652946
Redlich-Peterson	35.78	1.343486	3.61931	1.804956	3.619305986	4.862488304	**0.351033**
Langmuir-Freundlich	35.95	1.171207	3.15519	1.371725	3.155190114	3.695379392	1.612857
MW-treated bentonite – coordinates of additional point [146.3;54.9].							
Langmuir	56.02737293	-1.127373	-2.0535	1.27097	2.053502606	2.315063251	**4.323193**
Freundlich	48.86942764	6.030572	10.9846	36.3678	10.98464911	66.24372131	4.329199
Redlich-Peterson	48.86948399	6.030516	10.9845	36.36712	10.98454647	66.24248342	4.329085
Langmuir-Freundlich	55.79815121	-0.898151	-1.636	0.806676	1.635976696	1.469354443	4.335091

best approximations can be achieved by minimizing the Marquard's percent standard deviation and/or the hybrid fractional error function. A significant advantage of these functions is the consideration of degrees of freedom.

As a result of comparing the obtained SNEs in modeling within 4 theoretical models, it was concluded that the process of iron absorption in natural and modified samples is best described using the Freundlich model (Table 1-4).

The comparison of SNE obtained by calculating the error functions in the case of comparing the experimental data, which was not used in isotherm fitting, with that calculated for each of the selected models (Table 5), indicates that the iron absorption on natural bentonite fits the three-parameter Redlich-Peterson model (Fig. 1), and on the microwave irradiated - the Freundlich isotherm the best (Fig. 2). The Redlich-Peterson model combines center models with the same sorption potential [15]. The

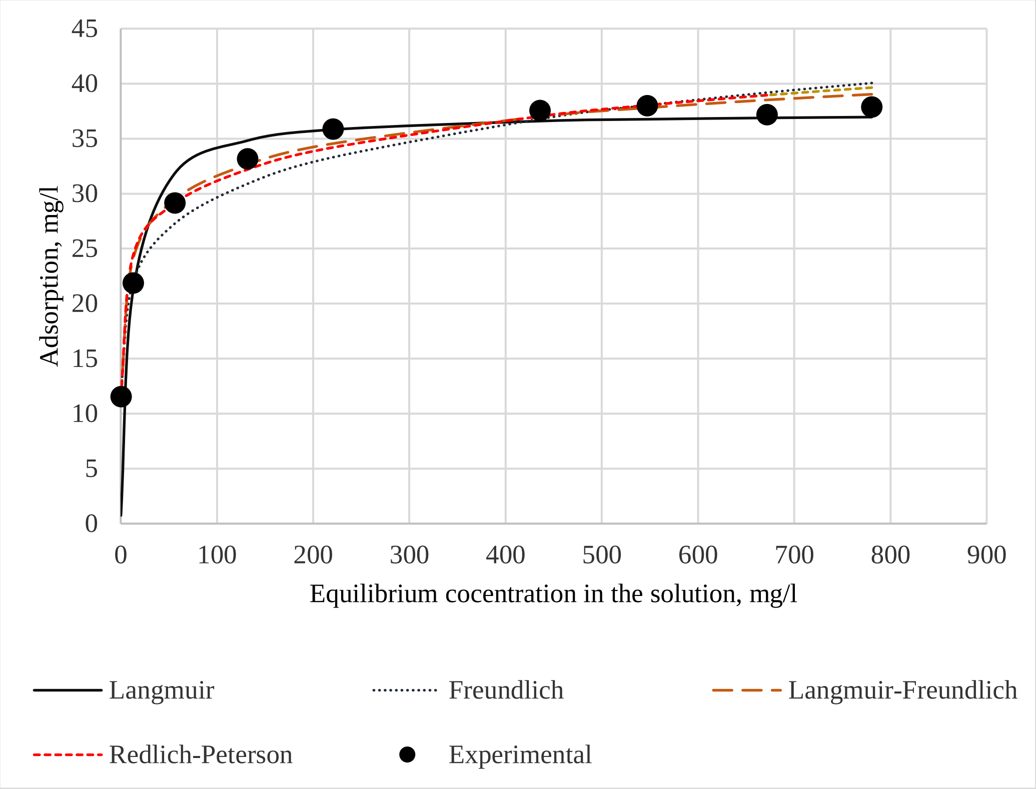

Fig. 1. Isotherms of iron adsorption on the natural bentonite

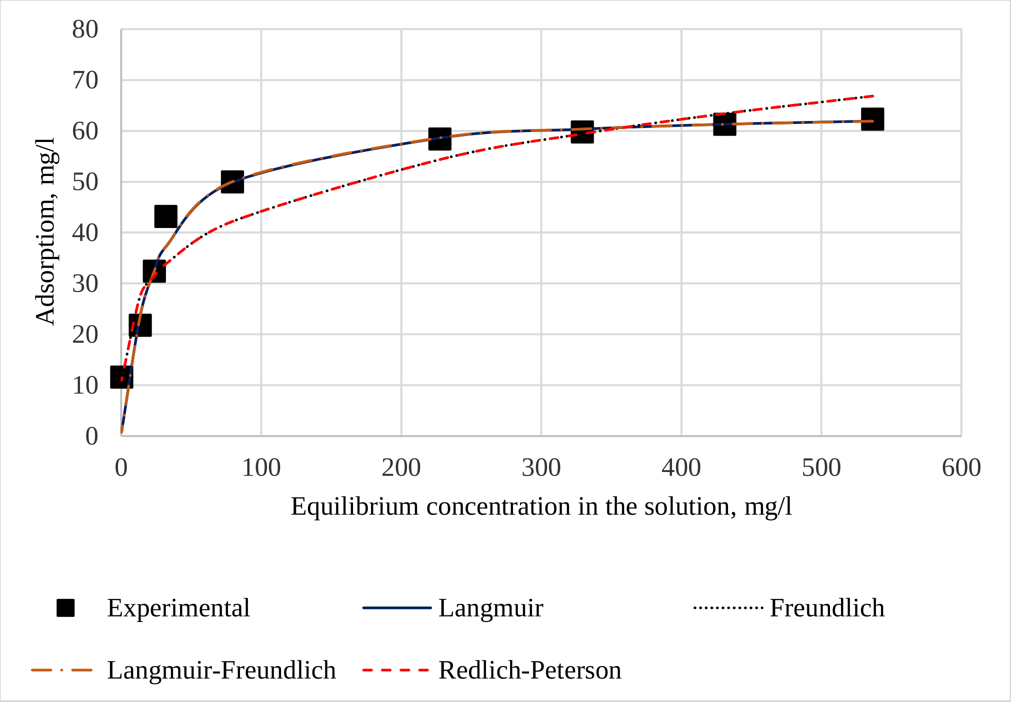

Fig. 2. Isotherms of iron adsorption on the MW-treated bentonite.

Freundlich isotherm model is used to describe sorption on a heterogeneous surface. Since the sorption centers according to this model are of different energy, first of all the active sorption positions are filled with the maximum energy [12]. Therefore, we can assume that due to the microwave irradiation of the bentonite new adsorption centers are formed on its surface.

Conclusion

1. The adsorption of iron on natural bentonite irradiated with microwave irradiation in stationary conditions has been studied. Adsorption isotherms were plotted to determine the maximum sorption capacity of the samples, which are 37.7 and 64.4 mg/g of sorbent, respectively.

2. Experimental data were processed by the method of nonlinear modeling using Langmuir, Freundlich, Redlich-Peterson and Langmuir-Freundlich isotherm models. The constants of these equations are determined. It is shown that the three-parameter Redlich-Peterson model fits the best the iron absorption process on natural bentonite, and the Freundlich isotherm – the process on the microwave irradiated sorbent.

3. The microwave irradiated sample shows a high adsorption capacity for iron and, thus, is a promising material for wastewater treatment. Microwave stimulation is advantageous compared to chemical modification because it does not require additional reagents and an additional processing stages.

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Нелінійне моделювання сорбції іонів Феруму бентонітом в рамках теоретичних моделей

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Проведено порівняльне дослідження придатності адсорбційних моделей для опису експериментальних ізотерм адсорбції іонів Феруму на двох зразках: природного бентоніту та бентоніту опроміненого НВЧ-опроміненням. Ізотерми сорбції описані за допомогою теорій Ленгмюра, Фрейндліха, Редліха-Петерсона та Ленгмюра-Фрейндліха. Визначені константи та параметри зазначених рівнянь. Нелінійне моделювання експериментальних даних в рамках теоретичних моделей ізотерм продемонструвало, що адсорбція нативним бентонітом відповідає трипараметричній моделі Редліха-Петерсона, а НВЧ-опроміненим – ізотермі Фрейндліха. Таке моделювання дає змогу передбачити максимальну адсорбційну здатність, що складає 37,7 мг/г природного бентоніта та 64,4 мг/г модифікованого зразка.

Ключові слова: бентоніт; надвисокочастотне випромінювання; адсорбція; мідь; нелінійне моделювання.