



ОРИГИНАЛЬНЫЕ СТАТЬИ

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Adsorption study of Fe (III) on modified adsorbent: adsorption isotherms and kinetics

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Abstract. This work is dedicated to the adsorption of ferric (III) ions by maleic anhydride styrene copolymer (MASC) modified with 2-amino-4,6-disulphoacid. The influence of pH, time required for reaching of maximum adsorption capacity, ionic strength, and primary concentration of ferric (III) ions were studied during investigations. Results have shown that the maximum adsorption capacity of the sorbent with respect to ferric ions is 373.31 mg/g at pH=5. Equilibrium concentrations of ferric (III) ions in solution was set by using 2,2'-(1,1'-biphenyl)-4,4'-diilbis(diazen-2,1-diyl)bis(benzene-1,3,5-triol) C₁₈H₁₈O₆N₄ (R) as reagent. Absorbances of solutions were measured on KFK-2 with l=1 cm at λ_{max}=490 nm. Beer's law was obeyed in the range 0.056-0.12 mcg/g. Various adsorption isotherm and kinetic models have been studied. It was found that Langmuir model best explains the current adsorption process and pseudo-second order kinetic model best fits with results. The structure of adsorbent was studied by IR spectroscopy on Varian 3600 Fourier spectrometer in the region from 400 to 4000 cm⁻¹. Results of investigation were compared the literature data.

Keywords: adsorption, Fe (III), maleic anhydride styrene copolymer, 2-amino-4,6-disulphoacid, preconcentration.

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Original article

Исследование адсорбции Fe (III) на модифицированном адсорбенте: изотермы и кинетика адсорбции

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Аннотация. Работа посвящена адсорбции ионов трехвалентного железа (III) сополимером малеинового ангидрида и стирола (MASC), модифицированным 2-амино-4,6-дисульфокислотой. В ходе исследований изучали влияние pH, времени достижения максимальной адсорбционной способности, ионной силы и исходной концентрации ионов трехвалентного железа (III). Результаты показали, что максимальная адсорбционная емкость сорбента по ионам трехвалентного железа составляет 373.31 мг/г при pH=5. Равновесные концентрации ионов трехвалентного железа (III) в растворе устанавливали с помощью 2,2'-(1,1'-бифенил)-4,4'-диилбис(диазен-2,1-диил)бис(бензол-1,3,5-триол) C₁₈H₁₈O₆N₄ (R) в качестве реагента. Оптическую плотность растворов измеряли на КФК-2 с l=1 см при λ_{max}=490 нм. Закон Бера выполнялся в диапазоне 0.056-0.12 мг/г. Были изучены различные изотермы адсорбции и кинетические модели. Было установлено, что модель Ленгмюра лучше всего объясняет текущий процесс адсорбции и псевдо-кинетическая модель второго порядка лучше всего соответствует полученным результатам. Структуру адсорбента исследовали методом ИК-спектроскопии на Фурье-спектрометре

Varian 3600 в области от 400 до 4000 см⁻¹. Результаты исследования сравнивали с литературными данными.

Ключевые слова: адсорбция, Fe (III), сополимер малеинового ангидрида и стирола, 2-амино-4,6-дисульфокислота, концентрирование.

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Introduction

Iron is one of the main contaminants, especially in cities where iron and steel are manufactured. It's found in chemical wastewater, metallurgical, machine-building, metalworking, petrochemical, textile, chemical-pharmaceutical and other industries. Water with iron concentration of more than 1 mg/dm³ becomes brown. Toxicity of iron leads to various health problems as well. In connection with the growth of industrial production, there is an increase in the consumption of natural water and, as a result, an increase in generated wastewater. Insufficiently treated wastewater is the main source of pollution and clogging of natural reservoirs, lead to significant physicochemical changes in the properties and composition of water, making it unsuitable for household and household use. The most common toxic impurities of wastewater are heavy metals. Sources of these impurities are waters of textile, leather, electroplating, chemical, and machine-building enterprises, as well as ore and mine enterprises production [4,5]. The removal of heavy metal ions from different objects is an important task nowadays [10-12,13-15].

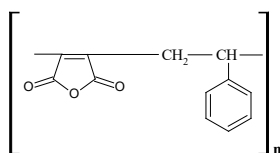
The goal of the present work is studying the adsorption of Fe (III) ions from its aque-

ous solutions by a polymeric chelate-forming adsorbent based on a copolymer of styrene with maleic anhydride and 2-amino-4,6-disulphoacid [2,6-8]. Different adsorption characteristics, i.e., the influence of pH, time, ionic strength, initial concentration of the metal ions, were studied during this work. During investigations the desorption process was also studied and the optimum eluent was established. Several adsorption isotherm and kinetic models were also investigated [9].

Experimental part

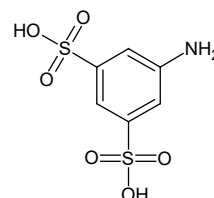
Preparation of solutions. A 5·10⁻³ mol/dm³ concentrated solution of Fe (III) ions was prepared by using a 10⁻¹ M FeCl₃ solution, obtained by dissolving reduced iron in HCl and HNO₃, was used for analysis. The equilibrium concentrations of Fe (III) ions in the solution were determined by using 2,2'-(1,1'-biphenyl)-4,4'-diilbis(diazene-2,1-diyl)bis(benzene-1,3,5-triol) C₁₈H₁₈O₆N₄ (R) [3]. Absorbances of the solutions were measured on KFK-2 and the optimal pH=5 and λ=490 nm were established.

Based on 0.1 N solutions of CH₃COOH and NH₄OH buffer solutions from 1-5 were prepared. 2 mol/dm³ KCl solution was used for studying of ionic strength effect. For desorption studies 2 mol/dm³ KOH solution was used. Adsorbent synthesis was carried



Scheme 1. Molecular structure of a copolymer of styrene with maleic anhydride

Схема 1. Молекулярная структура сополимера стирола с малеиновым ангидридом

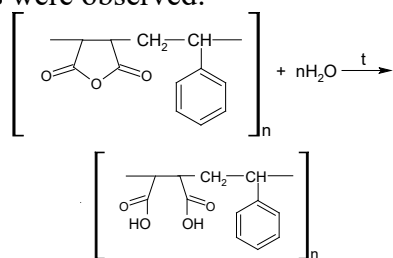


Scheme 2. Molecular structure of 2-amino-4,6-disulphoacid

Схема 2. Молекулярная структура 2-амино-4,6-дисульфокислота

out on the basis of styrene maleic anhydride copolymer and 2-amino-4,6-disulphoacid.

Adsorbent synthesis. The adsorbent was synthesized by a known technique [1]. As the polymer matrix for the synthesis of the adsorbent, styrene maleic anhydride copolymer and 2-amino-4,6-disulphoacid were used. For that purpose, 3 g of copolymer was added to the round bottom flask. Subsequently, the appropriate amount of 2-amino-4,6-disulphoacid was weighed, dissolved in water and added to the original contents of the flask. The synthesis was carried out in the presence of formalin, as a cross-linking agent. The reaction was carried out at a temperature of 60-70°C, for 30-40 minutes. During the reaction, the following transformations were observed:



Reaction is carried out in an aqueous medium. As a result the anhydride groups of the polymer undergo hydrolysis.

Due to the mutual influence of formaldehyde and amine, an unstable carbonylamine is formed. Carbonylamine interacts with the carboxyl groups of the macromolecule and thus the amine is introduced into the macromolecule.

After resulting adsorbent is passed through a filter paper, washed, dried, ground, and used for further research.

Preconcentration. All studies were carried out at room temperature. For each experiment, 2 cm³ of a solution of metal ions with a known concentration (5·10⁻³ mol/dm³) was added to 50 cm³ conical flasks. 30 mg of adsorbent and corresponding pH were added to each of them. An Ionomer-I30 pH meter was used in order to control the pH of the solutions. The ready solutions were kept for 24 hours. Then the contents of the flasks were passed through a filter paper so that the liquid phase was separated from the solid phase.

Afterwards, 1 ml samples were taken from each flask, diluted with a buffer solution pH 5, and final concentrations of Fe (III) ions were measured with the reagent (R) on the photoelectrocolorimeter KFK-2 at λ=490 nm.

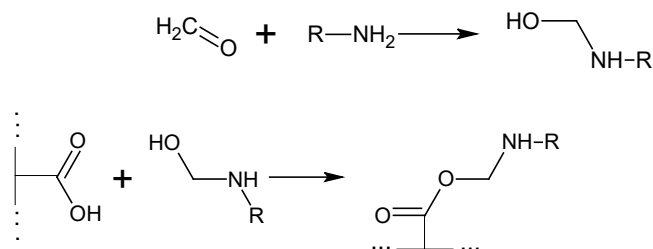
The degree of metal ion extraction was calculated by the following formulas:

$$R, \% = \frac{C_0 - C_e}{C_e} \cdot 100, \quad (1)$$

$$q_e = \frac{(C_0 - C_e)V}{m}, \quad (2)$$

where C₀ is the initial concentration of the metal ion (mol/dm³), C_e is the equilibrium concentration of the metal ion (mol/dm³), V is the volume of the solution (dm³), and m is the adsorbent mass (mg).

The desorption process. For desorption studies different inorganic acids of the same concentration were used, mainly: 0.5 mol/dm³ solutions of HNO₃, HCl, H₂SO₄, and CH₃COOH acids. Experiment was carried out in the following way: 30 mg of adsorbent were weighed into 4 flasks, 2 cm³ of a 5·10⁻³ mol/dm³ Fe (III) solution and 18 cm³ of pH 5.0 were added and left for 24 hours.



Scheme 3. The mechanism of the adsorption process
 Схема 3. Механизм процесса адсорбции

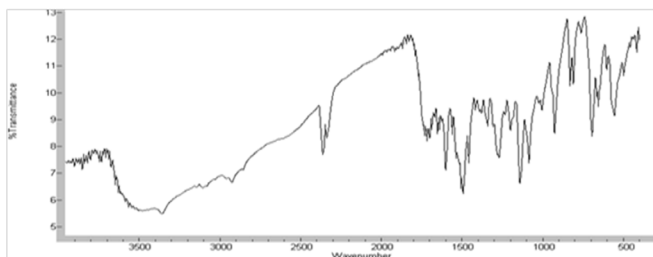


Fig. 1. IR spectra of the adsorbent
 Рис. 1. ИК спектры адсорбента

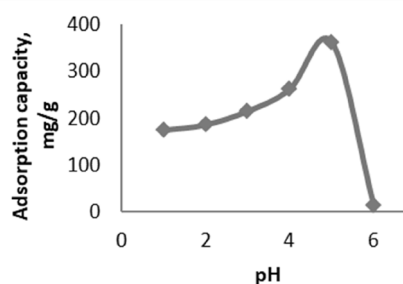


Fig. 2. Effect of pH on the adsorption capacity: $m_{\text{sorb}}=30 \text{ mg}$, $V_{\text{gen}}=20 \text{ cm}^3$, $C_{\text{Me}}=5 \cdot 10^{-3} \text{ mol/dm}^3$
 Рис. 2. Влияние pH на адсорбционную емкость

Thereafter, the solid part of the solution was separated from the liquid by filtration. To the filtered part 20 cm^3 of 0.5 mol/dm^3 solutions of HNO_3 , HCl , H_2SO_4 , and CH_3COOH acids was added.

Tightly closed and left for 24 hours. At the end of the day, the liquid portion of the solution was again separated from the solid. 1 cm^3 was then taken from the homogeneous solution, $1\text{-}2 \text{ cm}^3$ of a 2 mol/dm^3 solution of KOH was added, diluted with a buffer solution of pH 5.0, and final concentrations of Fe (III) ions were determined with reagent R on KFK-2 [5].

Equipment. Photoelectrocalorimeter KFK-2 in a cuvette with a layer thickness $l=1 \text{ cm}$ was used for absorption studies. pH values were measured on the pH meter Ionomer I-130. The IR spectrum of the adsorbent was taken on a Varian 3600 Fourier spectrometer in the region from 400 to 4000 cm^{-1} .

Results and discussions

Adsorbent structure. Results of IR spectroscopy are given in Figure 1.

In the IR spectrum of the sorbent $3668\text{-}3111 \text{ cm}^{-1}$ corresponds to valence oscillations of the $-\text{OH}$ group in the carboxyl group, as well as valence oscillations of the $-\text{NH}$ group (3361 cm^{-1}), $1733\text{-}1745 \text{ cm}^{-1}$ (valence oscillations of the $-\text{C}=\text{O}$ group in the carboxyl group carboxyl group) are observed.

Absorption bands are observed at frequencies of $1585\text{-}1565 \text{ cm}^{-1}$ (valence oscillations of C-N and deformation vibrations of N-H), $1605\text{-}1457 \text{ cm}^{-1}$ (valence oscillations in the benzene ring), $730\text{-}695 \text{ cm}^{-1}$ (deformation vibrations in the benzene ring).

Effect of pH on the degree of extraction of Fe (III). pH effect is one the essential factors during adsorption. To study this effect to 30 mg of adsorbent 2 cm^3 of a $5 \cdot 10^{-3} \text{ mol/dm}^3$ metal ion solution and 18 cm^3 of an appropriate pH of 1 to 5 were added. The contents of the flask were kept for 24 hours.

After a day, the solid part of the solution was separated from the liquid and the absorbances of solutions were measured on a KFK-2 photoelectrocalorimeter at pH 5.0 and $\lambda_{\text{max}} 490 \text{ nm}$. The results have shown that the greatest metal recovery is observed at a pH 5.0. Further adsorption experiments were carried out at pH 5.0. The resulting data is illustrated in Figure 2.

The effect of contact time. The effect of time required for reaching of maximum adsorption capacity is shown in Figure 3. For that purpose, the time was varied in the range from 0 to 240 minutes. The equilibrium concentrations of metal ions in the sample were determined at the appropriate time intervals at KFK-2 and $\lambda=490 \text{ nm}$.

As can be seen from Figure 3, the degree of adsorption changes slightly. This shows the quick reaching of complete adsorption equilibrium.

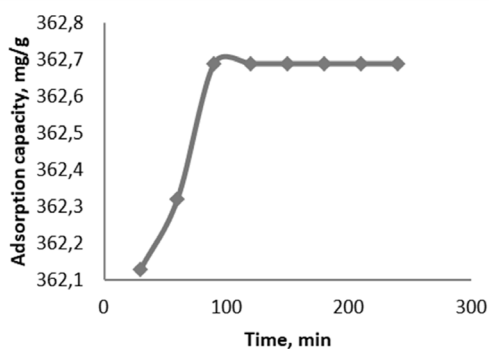


Fig. 3. Effect of time on the adsorption capacity: $m_{\text{sorb}}=30$ mg, $V_{\text{gen}}=20$ cm³, $C_{\text{Me}}=5 \cdot 10^{-3}$ mol/dm³

Рис. 3. Влияние времени на адсорбционную способность

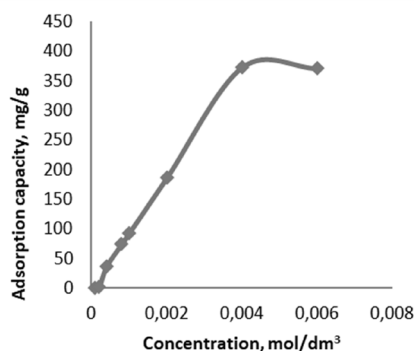


Fig. 4. Effect of initial metal ion concentration on the adsorption capacity:

$m_{\text{sorb}}=30$ mg, $V_{\text{gen}}=20$ cm³, $C_{\text{Me}}=5 \cdot 10^{-3}$ mol/dm³

Рис. 4. Влияние начальной концентрации ионов металла на адсорбционную емкость

Effect of ionic strength. In this study, the effect of ionic strength on the degree adsorption capacity was investigated. To achieve this, a 2 mol/dm³ KCl solution was used.

It was found that in the range of $\mu=0.2-1.4$ mol/dm³, the presence of K⁺ and Cl⁻ ions have a small effect on the extraction of metal ions and practically does not affect the adsorption capacity of the adsorbent.

Effect of the initial concentration of Fe (III) ions. To study the influence of primary metal ion concentration on adsorption capacity it was varied from $0.1 \cdot 10^{-3}$ mol/dm³ to $6 \cdot 10^{-3}$ mol/dm³. The corresponding volumes of the metal ion solution and pH 5.0 were added to 30 mg of adsorbent. Absorbances of the homogeneous solutions were measured on KFK-2 at pH 5.0 and $\lambda_{\text{max}}=490$ nm. The degree of extraction was calculated according to formula (1). The results are graphically depicted in Figure 4 and Table 1.

It was found that the greatest adsorption capacity is observed at a concentration of $4 \cdot 10^{-3}$ mol/dm³ and is equal to 373.31 mg/g.

The desorption process. Desorption studies were carried out by using different inorganic acids with the same concentration, in particular 0.5 mol/dm³ solutions of HNO₃, HCl, H₂SO₄, and CH₃COOH acids. The maximum desorption ability over Fe (III) ions was shown by a 0.5 mol/dm³ solution of HNO₃.

Langmuir isotherm. Equation below is used in order to describe Langmuir isotherm model [Langmuir, 1918]:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}, \quad (3)$$

where C_e (mmol/dm³) is the concentration of adsorbate in the aqueous phase at equilibrium, q_e (mmol·g⁻¹) is the equilibrium adsorption capacity, q_m is equal to q_e for the complete monolayer, and K_L (dm³/mmol) is the Langmuir isotherm constant.

Curve of dependence of $1/q_e$ on $1/C_e$ is given in Figure 5. The values of the q_{max} and K_L were calculated from the slope and the intercept of the plot, correspondingly.

Table 1. Langmuir, Freundlich, and Dubinin-Raduskhevich isotherm parameters

Таблица 1. Параметры изотерм Ленгмюра, Фрейндлиха и Дубинина-Радускевича

Langmuir				Freudlich			Dubinin- Raduskhevich			
q_{max} , mg·g ⁻¹	K_L , dm ³ · mg ⁻¹	R_L	R^2	K_F , mg· g ⁻¹	$1/n$	R^2	q_s , mg· g ⁻¹	E , kJ· mol ⁻¹	k_{D-R} , mol ² · kJ ⁻²	R^2
7.608	4.28	0.988	0.992	14.18	1.658	0.958	291.2	2.8	$6 \cdot 10^{-8}$	0.933

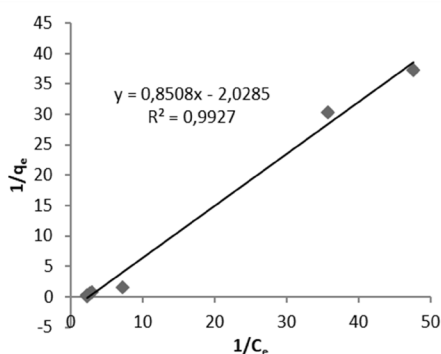


Fig.5. Langmuir isotherm model
 Рис.5. Модель изотермы Ленгмюра

Equilibrium parameter (R_L) is an important Langmuir isotherm model factor and can be calculated using the following formula:

$$R_L = \frac{1}{1 + bC_0}, \quad (4)$$

where b is the Langmuir constant ($\text{dm}^3 \cdot \text{mmol}^{-1}$), C_0 is the initial concentration of adsorbate (mmol/dm^3). The value R_L indicates the isotherm type. A value between 0 and 1 shows favorable adsorption process.

Results show that R_L lies between 0 and 1 and is equal to 0.988, which shows that adsorption is favorable under the current conditions by Langmuir model. Also, the value of the coefficient of regression $R^2=0.992$ shows that isotherm model fits good with experimental adsorption data.

Freundlich isotherm. Equation below can be used to describe the Freundlich model [Freundlich, 1906]:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e, \quad (5)$$

where C_e (mmol/dm^3) is adsorbate concentration at equilibrium, q_e ($\text{mmol} \cdot \text{g}^{-1}$) is the equilibrium adsorption capacity, K_F is the Freundlich constant and $1/n$ the heterogeneity factor.

The curve of dependence of $\ln q_e$ on $\ln C_e$ is shown in Figure 6. The values of K_F and $1/n$ were calculated from the slope and intercept of the plot, accordingly.

K_F value characterizes adsorption capacity (mg/g), n -adsorption intensity. The value of $1/n$ is a function of adsorption strength. If the value of $1/n$ is lower than 1 it means a

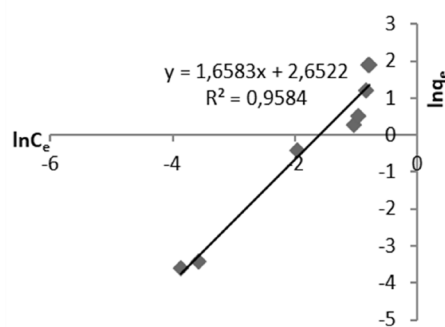


Fig. 6. Freundlich isotherm model
 Рис. 6. Модель изотермы Фрейндлиха

normal adsorption, if $1/n > 1$ it shows cooperative adsorption. In our case $1/n=1.658$, which indicates a cooperative adsorption process.

Dubinin-Raduskhevich isotherm. Adsorbent porosity can be described by Dubinin-Radushkevich (D-R) model. Following equation lies on the basis of this model [Dubinin, 1947]:

$$\ln q_e = \ln q_s - k_{D-R} \varepsilon^2, \quad (6)$$

where q_s is the theoretical saturation capacity ($\text{mmol} \cdot \text{g}^{-1}$), k_{D-R} is the D-R isotherm constant related to the free energy of adsorption, and ε is Polanyi potential that is related to the equilibrium concentration as follows:

$$\varepsilon = RT \ln \left(1 + \frac{1}{C_e} \right), \quad (7)$$

where R ($8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$) is the gas constant and T (300 K) is the absolute temperature.

Adsorption energy is found using the equation below:

$$E = \frac{1}{\sqrt{2k_{D-R}}}, \quad (8)$$

Plot of $\ln q_e$ versus ε^2 is given in Figure 7. The values of q_s and k_{D-R} are calculated from the intercept and slope.

The value of R^2 is equal to 0.933. E is equal to 3.8 kJ mol^{-1} . If E lies between 8 and 16 kJ mol^{-1} then it shows chemisorptions process, while the value of E lower than 8 kJ mol^{-1} means physical adsorption process. So, in our case, the value of E equal to 2.8 kJ mol^{-1} means that physical adsorption process is observed between S and Fe (III).

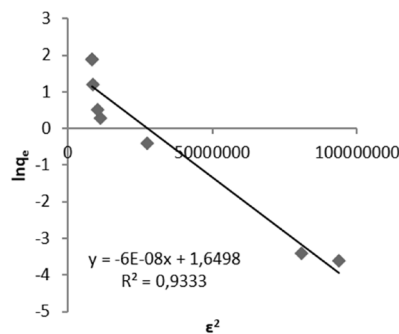


Fig. 7. Dubinin- Raduskhevich isotherm model
 Рис. 7. Модель изотермы Дубинина-Радушкевича

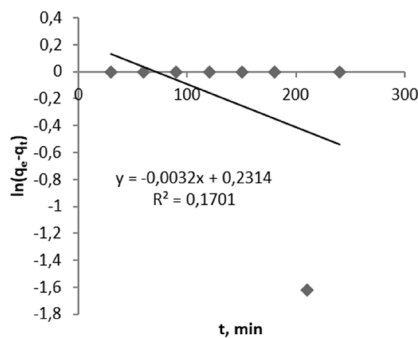


Fig. 8. Pseudo-first-order kinetic model
 Рис. 8. Кинетическая модель псевдо-первого порядка

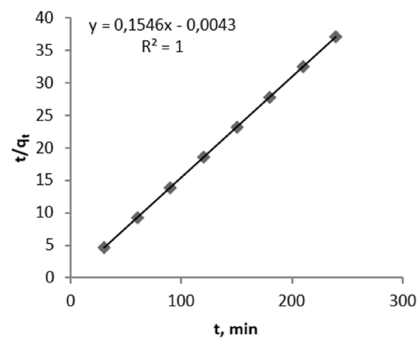


Fig. 9. Pseudo-second-order kinetic model
 Рис. 9. Кинетическая модель псевдо-второго порядка

Pseudo-first-order model. The equation below is used in order to describe adsorption process by pseudo-first-order kinetic model:

$$\ln(q_e - q_t) = \ln q_e - k_1 t, \quad (9)$$

where q_e and q_t are adsorption capacities at equilibrium and time t (min), respectively ($\text{mg} \cdot \text{g}^{-1}$) and k_1 is the rate constant of pseudo-first-order adsorption (min^{-1}). The plot of dependence of $\ln(q_e - q_t)$ versus t is shown in Figure 8.

Values of k_1 and q_e were calculated from slope and intercept of the plot of $\ln(q_e - q_t)$ versus t . The large difference between the experiment q_e value $q_{e(\text{exp})}$ and the calculated q_e value $q_{e(\text{cal})}$ shows that pseudo-first order kinetic model was poor fit for the adsorption process of adsorbent S for Fe(III).

Pseudo-second-order model. The pseudo-second-order model can also be used to describe the adsorption process. It is based on the equation below:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t, \quad (10)$$

where k_2 is the rate constant of pseudo-second-order adsorption ($\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$) and $k_2 q_e^2$ is the initial adsorption rate ($\text{mg} \cdot \text{g}^{-1} \cdot \text{min}^{-1}$). The plot of dependence of $\frac{t}{q_t}$ versus t is shown in Figure 9.

Values of k_2 and q_e were evaluated from the intercept and slope of the plot of $\frac{t}{q_t}$ versus t . Results show that values of $q_{e(\text{exp})}$ and $q_{e(\text{cal})}$ are close to each other, so that adsorption process of synthesized adsorbent S for Fe(III) can be well described by the pseudo-

Table 2. Adsorption kinetic parameters
 Таблица 2. Кинетические параметры адсорбции

Pseudo-first-order				Pseudo-second-order		
q_e , (exp), $\text{mg} \cdot \text{g}^{-1}$	k_1 , min^{-1}	q_e (cal), $\text{mg} \cdot \text{g}^{-1}$	R^2	k_2 , $\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$	q_e (cal), $\text{mg} \cdot \text{g}^{-1}$	R^2
373.33	-0.003	70	0.170	5.93	363.44	1

second order kinetic model. Results of kinetic studies are shown in Table 2.

Conclusion

The results of the investigations are quite high, in particular, when studying the effect of the initial concentration of metal ions on the adsorption capacity of the adsorbent, the maximum adsorption capacity of the adsorbent is 373.33 mg/g. The aforementioned allows us to assume the possibility of using the

synthesized adsorbent, based on a copolymer of styrene with maleic anhydride and 2-amino-4,6-disulphoacid, for extracting Fe (III) ions from various natural and industrial objects.

Конфликт интересов

Авторы заявляют, что у них нет известных финансовых конфликтов интересов или личных отношений, которые могли бы повлиять на работу, представленную в этой статье.

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