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REGRESSION ANALYSIS OF ZINC AND CADMIUM ION EXTRACTION FROM AQUEOUS SOLUTIONS USING A LIGNIN-BASED SULPHUR-CONTAINING SORBENT

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ABSTRACT. *The article briefly discusses aspects of the pollution of wastewater with zinc and cadmium compounds. Among the methods developed for treating such wastewater, adsorption technologies, which require the availability of effective, accessible and budget-friendly sorbents, are dominant. The most appropriate way of manufacturing such sorbents is from waste products generated by other industries. Given the fact that lignin comprises a large-tonnage waste of wood chemistry, organochlorine, epichlorohydrin and sodium polysulphide production, the possibilities of using sulphur-containing sorbents obtained from lignin for the extraction of zinc and cadmium compounds from aqueous solutions are considered. Experimental data on the pH effect on the extraction of the studied ions and their adsorption kinetics are obtained. The dependence of the adsorption value on the initial concentration of ions is constructed in the form of adsorption isotherms. Due to the complex coordination mechanism of sorption of Zn^{2+} and Cd^{2+} ions by sulphur-containing sorbents, thermodynamic and kinetic dependencies can be seen to deviate from the predications of classical laws. In this regard, the method of regression analysis is used to process the experimental data, with the obtained nonlinear equations of regression satisfactorily describing the observed regularities.*
Keywords: *water purification, wastewater, zinc, cadmium, adsorption, the sulphur-containing sorbent, lignin*

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РЕГРЕССИОННЫЙ АНАЛИЗ ЗАКОНОМЕРНОСТЕЙ ИЗВЛЕЧЕНИЯ ИОНОВ ЦИНКА И КАДМИЯ ИЗ ВОДНЫХ РАСТВОРОВ СЕРОСОДЕРЖАЩИМ СОРБЕНТОМ НА ОСНОВЕ ЛИГНИНА

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РЕЗЮМЕ. *В статье кратко рассмотрены проблемы, связанные с загрязнением сточных вод соединениями цинка и кадмия. Среди разработанных методов очистки таких сточных вод важное место принадлежит адсорбционным технологиям. Для их реализации требуется наличие эффективных, доступных и дешевых сорбентов, которые целесообразнее всего получать с использованием отходов других производств. Рассмотрены возможности применения для извлечения соединений цинка и кадмия из водных растворов серосодержащих сорбентов, получаемых из лигнина – многотоннажного отхода лесохимии, хлорорганических отходов производства эпихлоргидрина и полисульфида*

натрия. Экспериментально получены данные по влиянию величины pH на извлечение исследуемых ионов; начальной концентрации ионов на показатель адсорбции (построение изотерм адсорбции) и по кинетике адсорбции. Учитывая комплексно-координационный механизм сорбции ионов Zn^{2+} и Cd^{2+} серосодержащими сорбентами, в соответствие с которым термодинамические и кинетические зависимости могут отклоняться от классических закономерностей, для обработки экспериментальных данных использован метод регрессионного анализа. Получены нелинейные уравнения регрессии, которые удовлетворительно описывают наблюдаемые закономерности.
Ключевые слова: *водоочистка, сточные воды, цинк, кадмий, адсорбция, серосодержащий сорбент, лигнин.*

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INTRODUCTION

While drinking water quality standards are generally becoming more stringent, the environmental burden of wastewater discharge into surface water reservoirs is constantly increasing. Under current conditions, one of the most significant environmental factors is the pollution of wastewater with heavy metals[1] as a result of the increase in metal consumption on the part of major part of industries [1]. Among the heavy metals, zinc and cadmium compounds are a particular case in point. Firstly, these metals are the primary pollutants generated by the electroplating industry[2]. Secondly, they are widely used in many other areas, such as the production of chemical current sources, pigments, catalysts, alloys and lubricant additives[3]. Thus, the development of methods for removing zinc and cadmium compounds from wastewater becomes one of the priorities of modern technological civilisation.

Currently, a large number of methods for extracting zinc and cadmium compounds from wastewater are known [2]. However, some of them require additional consumption of chemical reagents, while others are associated with increased power consumption. Moreover, many of them are ineffective or remain in the development stage. Often, when the application of a single method appears to be insufficient, a combination of several methods is used [3, 4]. Usually, reagent-based or electrochemical methods are provided to extract high concentrations of zinc and cadmium from wastewater [3, 5]. However, the degree of purification that meets sanitary standards for wastewater discharges into water bodies cannot be achieved by these methods. For relatively low (but excessive with respect to the allowable standards) concentrations of contaminating heavy metals, the most acceptable method of purification is adsorption [6–8]. However, the wide expansion of sorption technology in wastewater treatment is hampered by the absence of cheap and accessible high efficiency sorbents [9]. Ion exchange sorbents (ion exchange resins) possess high efficiency and selectivity [10]. Nevertheless, they are expensive and generally not produced by Russian industrial sectors. Expensive synthetic polymeric chelating sor-bents, which absorb metals by means of a complex coordination mechanism, are used only in analytical chemistry [11, 12]. For these reasons, the use of production waste is a promising approach for creating low-cost, highly-effective sorbents. Works [13–15] provide us with examples of sorbent production from wood industry [13, 14], drilling [15] by-products and others. For example, lignin represents a large-tonnage waste of the wood chemical industry, the recycling of which is an important environmental task [16]. A sulphur-containing sorbent based on lignin and organochlorine wastes from the production of epichlorohydrin and sodium polysulfide, which effectively extracts heavy metal ions (including zinc and cadmium) from aqueous solutions, has already been elaborated by the present authors (the procedure of elaboration is described in detail in [17]). This paper presents the formation scheme of a sorbent polymer molecule; the sorption activity is equal to

[1] Sotnikova E.V., Dmitrienko V.P. Technospheric toxicology. Textbook. SPb.: “Lan” publishing house, 2013. 400 p. [Sotnikova E.V., Dmitrienko V.P. Tekhnosfernaya toksikologiya. Uchebnoe posobie. St. Petersburg: Lan Publ., 2013, 400 p.]

[2] Volosatov V.A. Handbook of electrochemical and electrophysical processing methods. L.: Mechanical Engineering, 1988. 719 p. 3. [Volosatov V.A. Spravochnik po elektrokhimicheskim i elektrofizicheskim metodam obrabotki]. Leningrad: Mashinostroenie Publ., 1988, 719 p.

[3] Rosin I.V., Tomina L.D. General and inorganic chemistry. Modern course. Study guide for bachelors and specialists. Moscow: Publishing house Yurayt, 2012. 1338 p. [Rosin I.V., Tomina L.D. Obshchaya i neorganicheskaya khimiya. Sovremennyi kurs. Uchebnoe posobie dlya bakalavrov i spetsialistov. Moscow: Yurait Publ., 2012, 1338 p.]

423 mg/g and 445 mg/g for zinc and cadmium, respectively.

The present work is aimed at obtaining experimental data and regression dependencies of the sorption process for the metals under study. Namely, the effect of pH on the sorption value and the dependence of the sorption value on the initial metal concentration in solution are considered. In addition, the sorption kinetics of zinc ions is examined by means of a regression study.

Based on the data obtained by regression analysis, mathematical models of the sorption process are constructed with the possibility of description and development of the optimal conditions for the functioning of individual devices [21]. Conventional approaches to the mathematical models of adsorption processes are used for sorbents that provide adsorption isotherms of the "classical" type[4] [7] and do not contain extrema. However, the sorption of metal ions on the sorbent under study proceeds via a complex coordination mechanism [17], which causes an «anomalous» type of isotherms and kinetic curves with clearly revealed extremes [19, 20]. It is in such cases that the development of regression models is most appropriate.

EXPERIMENTAL PART

The method of obtaining the sorbent and experimental studies on the extraction of heavy metals from aqueous solutions are described in detail in [17, 20]. The concentration of metals in solution before and after sorption was determined with a ZOMZ KFK-3 spectrophotometer using the dithizone method [21]. Here, the adsorption value A, mg/g is calculated using the formula:

$$A = \frac{V(C_0 - C_k)}{m},$$

where V is the volume of the solution under study, ml; C_0 and C_k are the initial and final concentrations of zinc or cadmium ions in the solution, mg/ml; m is the mass of the sorbent used in the experiment, g. At least three experiments were carried out to determine the sorption value for every pH and concentration of Zn and Cd in the solution. Each point on the graph corresponds to an arithmetic mean of three experiments, provided that the experimental results deviate by no more than 10%. In cases where the specified deviation was exceeded, additional experiments were carried out.

The concentration of H^+ ions in the solution (pH) was estimated using a pH-meter (pH 410) and adjusted by the addition of a 0,1 N solution of HCl or NaOH.

Statistical processing of experimental data is performed using Statgraphics Plus software. The type of the regression model is chosen according to the highest value of the coefficient of determination (R^2, %), which shows what percentage of experimental data is described by this regression. The corrected coefficient of determination is also calculated (R^2_c, %) in order to assess the strength of relationship between the dependent and independent variable. Additionally, the values of mean squared σ and absolute Δ errors as well as Durbin-Watson criterion (DW) are obtained due to their function as indicators of the absence of autocorrelation in experimental data.

DISCUSSION OF THE RESULTS

Figure 1 demonstrates the effect of pH on the extraction of Zn (Fig. 1, *a*) and Cd (Fig. 1, *b*) from aqueous solution. The points correspond to experimental data, with the regression line reflecting the dependence of the pH effect described by model (1) for Zn, and model (2) for Cd. The goodness of fit of the models are presented in Table.

$$A_{Zn} = 70{,}643 - 0{,}539 \times pH. \quad (1)$$

$$A_{Cd} = 83{,}571 + 3{,}048 \times pH - 1{,}583 \times pH^2 + 0{,}083 \times pH^3. \quad (2)$$

The goodness of fit of the obtained regressions to the experimental data can be inferred from Fig. 2, where the comparison of experimental data A_e with the adsorption values A_p calculated according to model (1) for Zn (Fig. 2, *a*) and model (2) for Cd (Fig. 2, *b*) is presented.

Considering the acid-base character of the studied cations [4], the pH effect on amphoteric zinc was studied up to pH = 5, and cadmium up to pH = 7, bearing in mind that above these values a precipitation of zinc and cadmium hydroxides, respectively, is possible. As a result, the most efficient extraction of zinc and cadmium cations is exhibited in sufficiently acidic media at pH 0,5–3,0 for Zn, and 1–5 for Cd.

The obtained dependences (1) and (2) correspond well to the known behaviour of Zn and Cd ions in aqueous solutions and make it possible to determine the adsorption value at any pH within the specified limits.

Fig. 3 shows the approximations of the thermodynamic data for the adsorption of Zn (Fig. 3, *a*) and Cd (Fig. 3, *b*) ions. The adsorption time comprises 3 hours. The values of pH are equal to 1,0 and 2,0 for Zn and Cd, respectively.

As can be seen from Fig. 3, the adsorption isotherms of Zn^{2+} and Cd^{2+} cations at 20 °C do not contain any extrema. This corresponds to the coordination of ions at the most active sorbent centres that, in turn, correlate well with the formation of hexa-coordinated complexes [17, 20]. In general, for both cations, the value of adsorption is higher than the same at higher temperature values (with the exception for Zn at 60 °C, see Fig. 7, *b*).

[4] Voyutsky S.S. The course of colloid chemistry. Moscow: Chemistry, 1975. 512 pp. [Voyutskii S.S. Kurs kolloidnoi khimii. Moscow: Khimiya Publ., 1975, 512 p.]

At 40 °C, the adsorption isotherm is described by regression (5) and (6) for zinc and cadmium ions, respectively. The goodness of fit of the models are presented in Table

$$A_{40} = 0{,}030 \times C_0 + 0{,}34 \times 10^{-4} \times C_0^2. \quad (5)$$

$$A_{40} = 0{,}222 \times C_0 - 0{,}0009 \times C_0^2 + 0{,}1 \times 10^{-5} \times$$

$$\times C_0^3 - 1{,}054 \times 10^{-9} \times C_0^4 + 2{,}738 \times 10^{-13} \times C_0^5. \quad (6)$$

For 40 °C, the shape of the adsorption isotherm for cadmium ions varies significantly. As can be seen from Figure 5b, extrema appear on the isotherm, which are caused by the desorption on the less active centres of the sorbent and their subsequent coordination on the more active centres.

The regressions (7) and (8) describe the adsorption isotherms at a temperature of 60 °C for Zn and Cd ions, respectively (see Fig. 7), with the goodness of fit of the models presented in Table.

$$A_{60} = 0{,}008 \times C_0 + 0{,}8 \times 10^{-4} \times C_0^2. \quad (7)$$

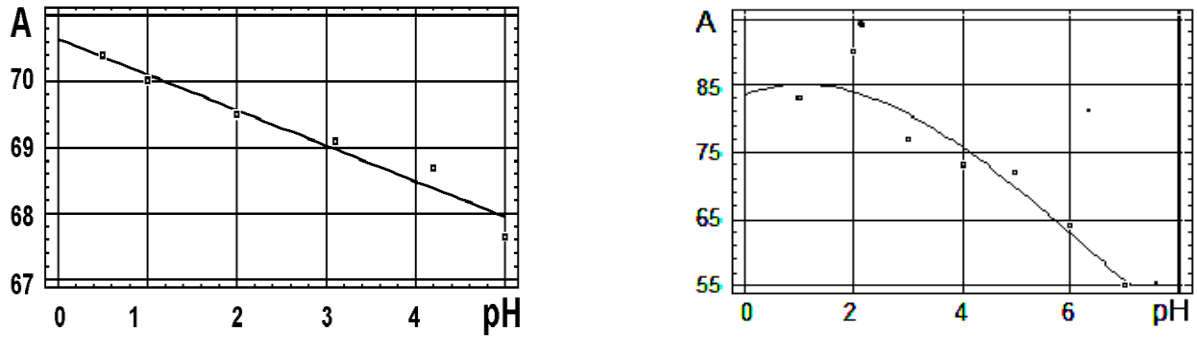
$$A_{60} = 0{,}1089 \text{ x } C_0 - 0{,}00013 \times C_0^2. \quad (8)$$

A comparison of experimental data and the same calculated by equations (5) and (6) are presented in Figure 6.

Fig. 8 depicts the comparison of experimental data and that calculated by equations (7) and (8).

The adsorption isotherm for Zn^{2+} at 60 °C shows slightly larger values of sorption than at 20 and 40 °C (see Fig. 7, *a*). Such a tendency is likely to correspond to the participation of lignin fragments in the process of sorption as an accompaniment of the coordination on sulphur atoms. Because Zn^{2+} cations are referred to stronger acids than cadmium cations[3], they are capable of coordinating residues of lignin at the oxygen positions (rigid basic centres) that obtain a higher reactivity with the temperature increased.

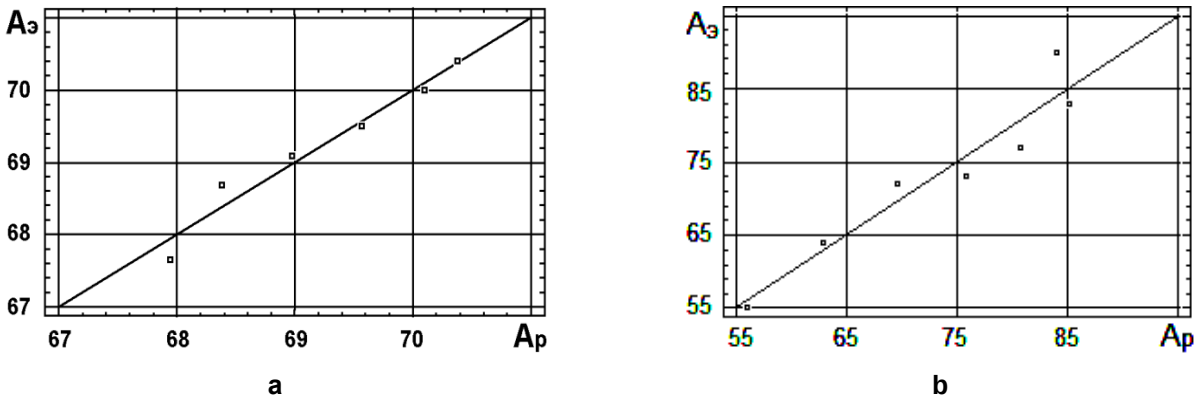
For Cd^{2+} cations, at this temperature, both a decrease in the adsorption value and its extremum dependence on the initial concentration are observed (Fig. 7, *b*). This corresponds to the "softer" nature of Cd ions and their lack of coordination in the oxygen positions of lignin.



a b

Fig. 1. pH dependence of Zn (a) and Cd (b) extraction in aqueous solution

Рис. 1. Зависимость извлечения Zn (a) и Cd (b) от pH водного раствора



a b

Fig. 2. Comparison of calculated values A_c with experimental data A_e for Zn^{2+} (a) and Cd^{2+} (b) ions

Рис. 2. Сопоставление расчетных значений A_p с данными $A_э$: Zn^{2+} (a), Cd^{2+} (b)

The regression dependence of the final concentration of zinc ions C_k, mg/l in solution on the time of adsorption t, min (kinetic curve pattern) with initial concentration C_0 = 1 g/l, *m* = 0,242 g, *V* = 24 ml and 20 °C are described by equation (9) with the goodness of fit listed in the table and presented in Fig. 9, *a*. In Fig. 9, *b*, the comparison of the final concentration of zinc ions C_{kc} calculated by the formula (9) with experimental values C_{ke} is presented.

$$C_k = 981{,}725 - 31{,}645 \times t^{0,9} + 1{,}087 \times t^{1,5}. \quad (9)$$

As can be seen from Fig. 9, *a*, for the first 30 minutes, zinc concentration in the solution decreases by more than 2 times when using a sorbent. However, after 90-100 minutes, zinc concentration in the solution increases again, and then begins to gradually decrease. This may possibly be determined by the course of desorption at the less active centres of the sorbent that are occupied in the first minutes of the process. The obtained regression dependence (9) provides the determination of the adsorption rate at any given time. The value of this rate can, in turn, be used in technological calculations.

The extraction of Cd^{2+} ions at 20 °C, which proceeds very rapidly (≈10 min), is presented in Fig. 10.

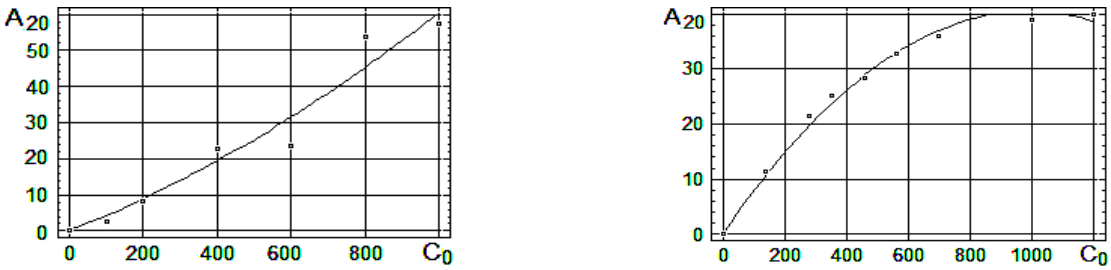


Fig. 3. Adsorption isotherms at 20 °C for zinc (a) and cadmium ions (b)

Рис. 3. Изотермы адсорбции при температуре 20 °С ионов цинка (a) и кадмия (b)

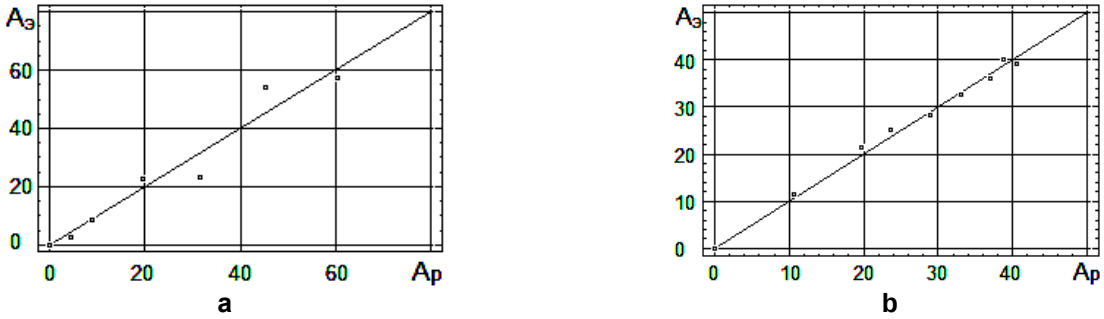


Fig. 4. Comparison of calculated values A_c with experimental data A_e at 20 °C for Zn (a) and Cd (b) ions.

Рис. 4. Сравнение расчетных значений Ар с экспериментальными Аэ при 20 °С: a – для ионов цинка, b – для ионов кадмия.

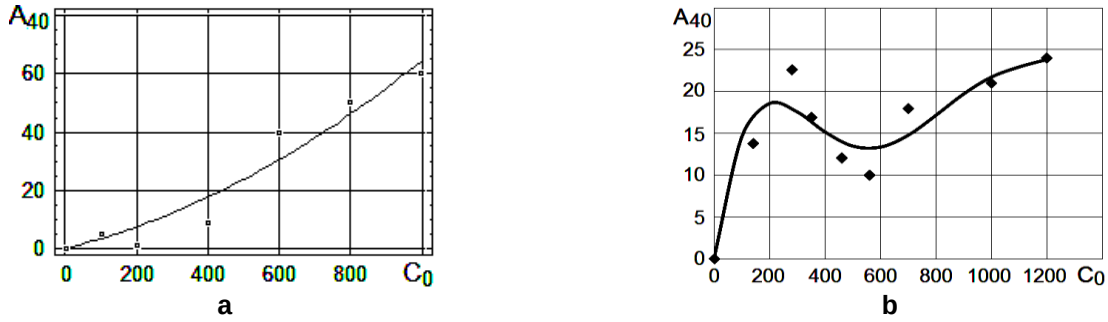


Fig. 5. Adsorption isotherms at a temperature of 40 °C for zinc (a) and cadmium (b) ions

Рис. 5. Изотермы адсорбции при температуре 40 °С ионов цинка (a) и кадмия (b)

Fig. 6. Comparison of calculated values A_c with experimental data A_e at 40 °C for zinc (a) and cadmium (b) ions

Рис. 6. Сравнение расчетных значений Ар с экспериментальными Аэ при 40 °С: a – для ионов цинка, b – для ионов кадмия

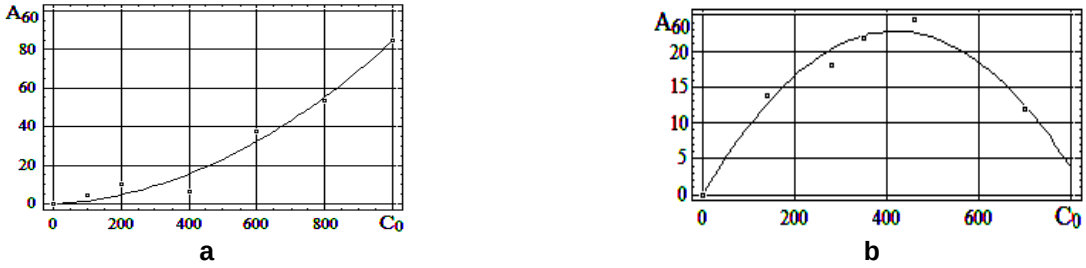


Fig. 7. Adsorption isotherms at 60 °C for zinc (a) and cadmium (b) ions

Рис. 7. Изотермы адсорбции при температуре 60 °С ионов цинка (a) и кадмия (b)

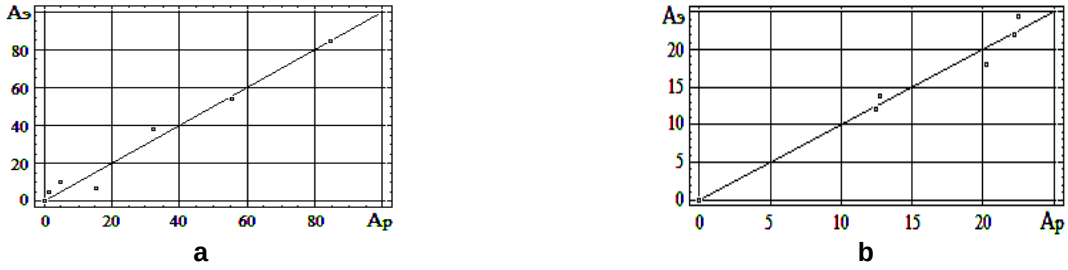


Fig. 8. Comparison of calculated values A_c with experimental data A_e at 60 °C for (a) zinc and (b) cadmium ions

Рис. 8. Сравнение расчетных значений Ар с экспериментальными Аэ при 60 °С: a – для ионов цинка, b – для ионов кадмия

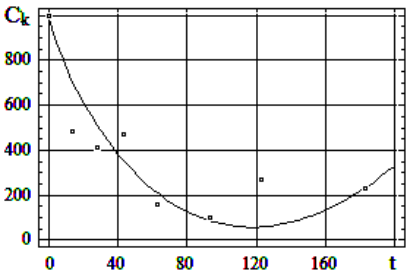


Fig. 9 a. Dependence of the final concentration of zinc ions C_k on the adsorption time t

Рис. 9 a. Зависимость C_k ионов цинка от времени адсорбции t

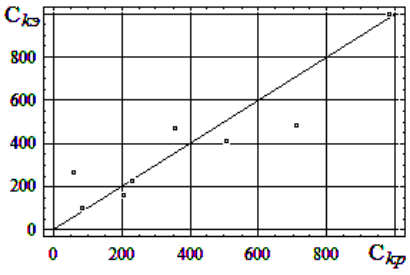


Fig. 9 b. Comparison of calculated final concentration of zinc ions C_{kc} with experimental data C_{ke}

Рис. 9 b. Сравнение расчетных C_{kp} с экспериментальными данными $C_{kэ}$

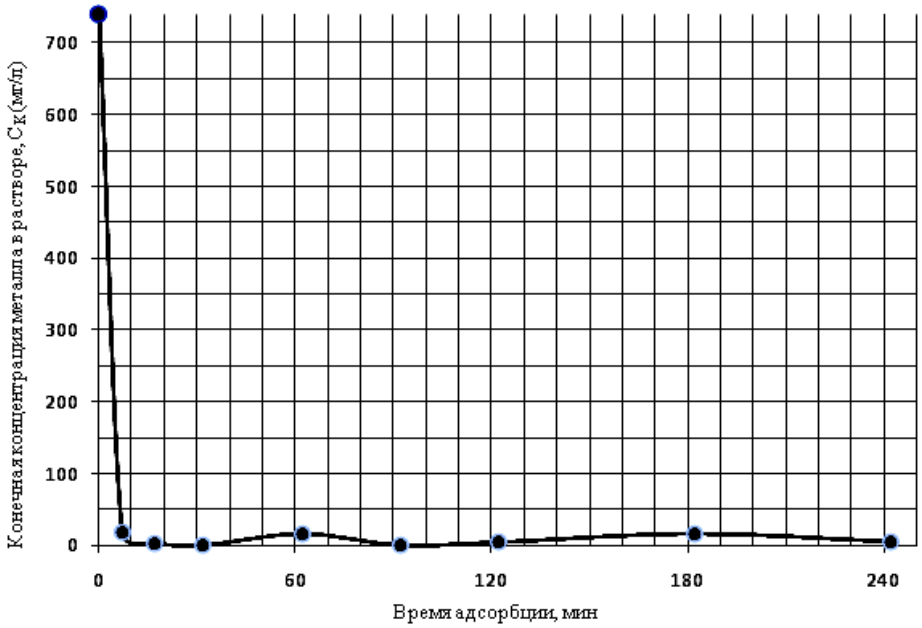


Fig. 10. Dependence of the sorption activity of the sorbent on the adsorption time during the extraction of Cd^{2+} ions (C_0= 740 mg/l, m = 0,5 g, V = 50 ml)

Рис. 10. Зависимость сорбционной активности сорбента при извлечении ионов Cd^{2+} от времени адсорбции (С0=740 мг/л, m = 0,5 г, V = 50 мл)

Table

The goodness of fit of the models

Таблица

Критерии адекватности регрессионных моделей

Formula number	R^2, %	R^2_c, %	DW	σ	Δ
(1)	95,50	94,38	2,52	0,23	0,16
(2)	91,40	82,81	2,77	4,82	2,71
(3)	95,00	94,00	3,46	5,75	3,65
(4)	99,19	99,08	1,82	1,28	0,98
(5)	94,02	92,82	2,09	6,84	4,85
(6)	91,82	83,63	2,37	2,97	1,56
(7)	97,56	97,07	3,21	5,47	3,53
(8)	97,44	96,80	2,59	1,57	0,97
(9)	95,23	92,04	1,95	92,81	92,15

CONCLUSION

The obtained data substantiate the possibility of efficient extraction of zinc and cadmium ions from aqueous solutions using a sulphur-containing lignin-based sorbent. The extraction patterns are well matched with regression models that can also be used to mathematically describe the adsorption isotherms and the kinetic curve pattern. As a result, the most effective sorption occurs at 20 °C and pH ranges of 0,5–3,0 and 1–5 for Zn and Cd, respectively. However, the possibility to increase the temperature for zinc ions is acceptable. The resulting regression equations provide the sufficient scientific background for the design of wastewater treatment plants aimed at the extraction of zinc and cadmium compounds.

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Contribution

Aslamova V.S., Chernysheva E.A., Grabelnykh V.A., Levanova E.P., Russavskaya N.V. carried out the experimental work, on the basis of the results summarized the material and wrote the manuscript. Aslamova V.S., Chernysheva E.A., Grabelnykh V.A., Levanova E.P., Russavskaya N.V. have equal author's rights and bear equal responsibility for plagiarism.

Conflict of interests

The authors declare no conflict of interests regarding the publication of this article.

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