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STUDY ON NI(II) ION ADSORPTION FROM AQUEOUS SOLUTIONS BY A NOVEL MAGNETIC SORBENT: ADSORPTION ISOTHERMS

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The contamination of water sources with heavy metal ions, particularly nickel (Ni(II)), poses a serious environmental and health hazard. This study investigates the adsorption behavior of a newly synthesized magnetic adsorbent for the removal of Ni(II) ions from aqueous solutions. Various parameters influencing the adsorption process were examined, including pH, contact time and initial metal ion concentration. The results indicated that the highest adsorption capacity was achieved at pH 6. To describe the adsorption process, various isotherm models, including Langmuir, Freundlich, Temkin, and Redlich–Patterson, were investigated. The study confirmed that the results were found to be consistent with the Langmuir model. The maximum adsorption capacity for Ni^{2+} *was determined to be 145,6 mg/g. The thermodynamic parameters of adsorption were also studied, and the study confirmed that adsorption is an endothermic process.The results suggest that the new magnetic adsorbent is a promising material for Ni(II) ion removal from wastewater.*

Key words: *magnetic adsorbent, Ni (II), adsorption isotherms, wastewater treatment, adsorption capacity*

INTRODUCTION

Heavy metal pollution is a major environmental concern due to its toxicity to living organisms and its non-biodegradable nature [1, 2]. Industries such as metal plating, leather processing, textiles, pesticide and fertilizer production, battery manufacturing, paints and pigments, electronics, and chemical industries generate wastewater containing various heavy metals while also discharging harmful effluents into the environment. The excessive use of raw materials contributes to resource depletion, while wastewater contamination disrupts ecosystems, leading to serious ecological challenges. Toxic heavy metals commonly found in industrial wastewater include lead (Pb), mercury (Hg), copper (Cu), cadmium (Cd), nickel (Ni), and chromium (Cr) [3, 4]. Addressing the removal and recovery of these metals is crucial, not only for environmental protection but also from an economic perspective [5].

Nickel is a silver-white metallic pollutant that is strong, malleable, and one of the most abundant elements in the Earth's crust. Various methods are used for the removal and recovery of nickel(II) from industrial wastewater, including precipitation, oxidation, reduction, ion exchange, filtration, electrochemical treatment, membrane technologies, reverse osmosis, and solvent extraction. Each of these techniques has its own benefits and limitations. However, most require significant capital investment and are not practical for small-scale industries [6]. Adsorption has gained recognition as a highly effective, efficient, and cost-effective approach for water purification and analytical separation [7]. Recently, there has been a growing focus on adsorbents derived from naturally occurring support materials. These materials offer several advantages, such as widespread availability, affordability, and the potential for chemical modification to enhance metal-binding properties. Examples of such natural materials include chitosan [8], clays [9], and cellulose [10].

Cellulose-based materials are considered sustainable due to their abundance, renewability, biodegradability, and excellent properties, making them a viable alternative to conventional materials for environmental protection. As the most abundant biopolymer on Earth, cellulose is found in plant cell walls and is both renewable and biodegradable. It is also produced by bacteria, algae, and fungi [11]. Compared to synthetic materials derived from fossil fuels, cellulose materials have a lower carbon footprint [12]. Additionally, cellulose possesses high strength, flexibility, ease of modification, and biocompatibility [13, 14]. These characteristics make it suitable for various applications, including water treatment [15, 16, 17, 18], air purification [19], seawater desalination [20], and uses in packaging, textiles, electronics, biosensors, and biomedical fields [21, 22].

In recent years, notable advancements have been achieved in removing nickel ions from water through adsorption. However, there remains a need for developing new sorbents that can efficiently separate nickel from solutions across a wide concentration range while ensuring high effectiveness within a short period.

The aim of the present work is to investigate the adsorption capacity of Ni(II) ions from aqueous solutions using a magnetic sorbent obtained by modifying a cellulose/Fe_3O_4/SiO_2 nanocomposite with 4-aminoantipyrine. Various factors affecting adsorption, including pH, initial metal concentration, contact time, and the desorption process, were examined. Additionally, a series of isotherm and kinetic studies were conducted during the course of this research.

2. MATERIAL AND METHODS

2.1. Materials and apparatus

All materials utilized in this study were obtained from Merck and Aldrich and were used without further purification. Ferrous chloride tetrahydrate ($FeCl_2$•$4H_2O$), cellulose, methanol, ethanol, and ammonia solution (28 wt%) were sourced from Merck (Germany). Tetraethyl orthosilicate (TEOS) was provided by Sigma Aldrich (United States).

The optical densities of the solutions were measured with a KFK-3 spectrophotometer using 1 cm thick quartz cuvettes. The pH values of the solutions were measured using a pH-meter equipped with a pH-121 standard solution with a glass electrode. Distilled water was obtained through a-10 device. The sorbent was dried in a Zymark Turbo Vap LV drying oven.

2.2. Preparation of solution

Nickel standard solution (1x10^{-2})

All the reagents used are of high purity. The 10^{-2} M solution of nickel was obtained by dissolving 0.2628 g of $NiSO_4$x $6H_2O$ in 100 ml of distilled water. Different concentrations of Ni(II) solution was prepared by diluting 10^{-2} stock solution.

Buffer Solution

The buffer solutions (pH 3–8) used in the experiment were prepared by mixing appropriate amounts of 0.1 M CH_3COOH and $NH_3\cdot H_2O$, while HCl was used to achieve a pH of 2.

2.3. Synthesis of magnetic adsorbents

The magnetic cellulose-based nanocomposite functionalized with 4-aminoantipyrine was synthesized following a previously reported multi-step procedure with minor modifications [23]. Firstly, Cellulose/Fe_3O_4 was prepared via chemical co-precipitation. $FeCl_2\cdot 4H_2O$ was added to a cellulose dispersion pretreated by ultrasonication and nitrogen purging. Under stirring at 90°C, ammonia solution (28%) was added to adjust the pH to 11–12, leading to the formation of a black precipitate. The product was magnetically separated, washed, and dried at 60°C.

Secondly, the composite was coated with SiO_2 using the modified Stöber method. The Cellulose/Fe_3O_4 material was dispersed in ethanol, ultrasonicated, and mixed with ammonia. TEOS (350 μL) was added dropwise, and the mixture was stirred for 4 hours. The product was collected, washed, and dried.

Finally, 4-aminoantipyrine was immobilized via impregnation by mixing the Cellulose/Fe_3O_4/SiO_2 nanocomposite with the organic compound (9:1 ratio) in acetone at room temperature for 8 hours. The resulting material was washed and dried at 40°C.

2.4. Sorption experiments

Sorption studies of Ni(II) ions were conducted at room temperature. In each experiment, 2 ml of metal ion solution was added to 50 ml conical flasks, followed by the addition of 30 mg of sorbent and adjustment to the appropriate pH. The pH levels of the solutions were monitored using a pH-121 pH meter. The resulting mixtures were left to stand for 24 hours, after which the liquid phase was separated from the solid phase using filter paper.

Subsequently, 1 ml of the sample was taken from each flask and diluted with a buffer solution at pH 5. The final concentration of Ni(II) ions was determined using the reagent 2,2′,2″,2‴-{(1,1-Dioxo-2,1λ6-benzoxathiole-3,3(1H)-diyl)bis[(6-hydroxy-5-methyl-3,1-phenylene)methylenenitrilo]}tetraacetic acid d (R). The measurements were carried out with a KFK-3 photoelectrocolorimeter at a wavelength of λ = 590 nm. The removal of metal ions from the solution, as well as the metal uptake in the solid phase or surface loading q_e (mg·g^{-1}), were calculated using the equations provided below:

$$R, \% = \frac{C_0 - C_e}{C_e} x100 \qquad (1),$$

where R is a percentage of metal ion removal.

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

where C_0 is the initial metal ion concentration (mg L^{-1}), C_e is the equilibrium metal ion con- centration (mg L^{-1}), V is the volume of the solution (L), and m is the mass of adsorbent (mg).

2.5. Desorption process

The preparation of adsorbents is typically a complex process that requires significant time and energy. Additionally, the substances used for synthesis are often costly and hard to obtain. Therefore, investigating the reusability of the sorbent is crucial. In this study, we examined the effect of different acids and their concentrations on the desorption process.

Desorption experiments were conducted using 0.5 mol·L^{-1} solutions of various acids, including HNO_3, HCl, H_2SO_4, and CH_3COOH, all at the same concentration. For each experiment, 30 mg of sorbent was placed into four separate flasks, to which 2 ml of 10^{-2} M Ni(II) solution and 18 ml of pH 6 were added and kept for 24 hours. Afterward, the solid phase was separated from the liquid phase by filtration.

Subsequently, 20 ml of 0.5 M HNO_3, HCl, H_2SO_4, and CH_3COOH solutions were added to the filtered solutions. The mixtures were then tightly sealed and was kept for 24 hours. After this period, the liquid was again separated from the solid phase. To determine the final concentration of Ni(II) ions, 1 ml of the homogeneous solution was taken, followed by the addition of 1-2 ml of 2 mol/L KOH solution. The solution was then diluted with a pH 6.0 buffer solution, and the final concentration of Ni (II) ions was measured using the KFK-3 device.

3. RESULTS AND DISCUSSION

3.1. Effect of pH on the sorption of Ni (II)

The pH of the solution is a crucial factor influencing the sorption capacity of Ni(II) because it affects the stability of nickel in the solution and can alter its chemical forms. For instance, at different pH values, nickel can exist in forms such as Ni^{2+}, $Ni(OH)^+$, $Ni(OH\ )_2$, or $Ni(OH)^{3-}$ or [24]. Additionally, the pH can modify the electrical properties of the sorbent surface. Above pH 8, Ni^{2+}

becomes the dominant form of nickel. To avoid the formation of hydroxides during sorption, the initial solution pH was selected within the range of 2.0 to 8.0.

For the experiment, 30 mg of sorbent was weighed and placed into separate containers, followed by the addition of 2 ml of 10^{-2} M metal ion solution and 18 ml of the appropriate pH buffer. The mixture was kept for 24 hours.

After this period, the solution was filtered using filter paper to separate the solid particles, and the concentration was measured at λ = 590 nm using a KFK-3 spectrophotometer. The effect of solution pH on nickel sorption is shown in the figure below (Figure 1).

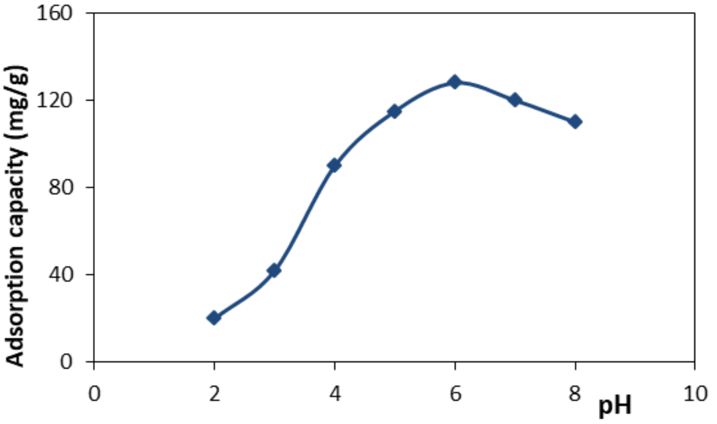

Fig.1. Effect of pH on the sorption of nickel

As a result of the measurement, it was found that the capture of Ni (II) ions with the highest percentage from the solution was at pH 6. Therefore, this value of pH was used in all subsequent experiments.

3.2. Effect of initial concentration of Ni(II) ions on adsorption capacity

The initial concentration of the metal ion plays a crucial role in the adsorption process, as it is one of the key factors influencing the movement of metal ions toward the adsorbent surface [25].

In this study, the effect of the metal ion concentration on the adsorption capacity of the synthesized sorbent was investigated. A concentration range of Ni(II) ions from 10 mg/L to 200 mg/L was used. For the experiment, 30 mg of sorbent was weighed, and the appropriate volume of metal ion solution with pH 6.0 was added and left for 24 h. After this period, the optical densities of the homogeneous solutions were measured using a KFK-3 spectrophotometer at λ = 590 nm with a pH 6.0 buffer solution. The results of the experiment are shown in the figure below (Figure 2).

3.3. Effect of contact time

In order to study the dependence of sorption on time, 30 mg of sorbent was taken and 2 ml of 10^{-2} M metal solution was added to it, and then 18 ml of pH 6 was added and the rate of sorption of metal ions in the solution was studied for 0-240 minutes. The adsorption of Ni(II) ions reached equilibrium at 120 min with 95% Ni(II) removal. The first 30 minutes accounted for 80% of total uptake, followed by slower intraparticle diffusion.

3.4. Desorption process

The desorption process was investigated to evaluate the reusability of the sorbent. In this study, desorption was carried out using different inorganic acids of equal concentration, specifically 0.5 mol/L solutions of HNO_3, HCl, H_2SO_4, and CH_3COOH. The results indicated that the highest desorption capacity for Ni(II) ions was achieved with a 0.5 mol/L HNO_3 solution.

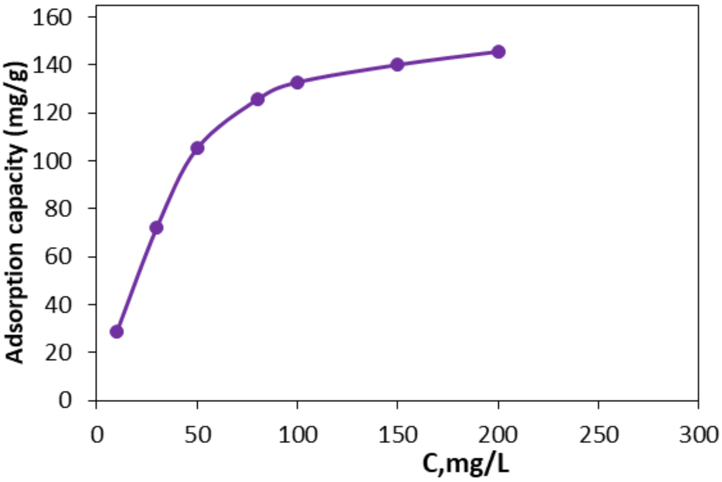

Fig.2. Effect of initial concentration adsorption capacity

3.5. Adsorption isotherm

To gain a better understanding of the adsorption process, it is essential to analyze the corresponding adsorption equilibrium. Adsorption equilibrium provides insights into the interaction between the adsorbate and the adsorbent. In this study, the equilibrium data were evaluated using the Langmuir and Freundlich isotherm models.

3.5.1.Langmuir isotherm

The Langmuir adsorption isotherm applies to the process where a solute is adsorbed from a solution as a single layer on a surface with a limited number of identical binding sites. These sites have consistent adsorption energies, and the adsorbed molecules do not move laterally along the surface [26]. The nonlinear equation of the Langmuir isotherm model can be expressed as [27].

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

where C_e and q_e are the equilibrium liquid-phase concentrations of adsorbate (mg/L) and the amount of adsorbate adsorbed onto the adsorbent (mg/g), respectively. Q_0 is the maximum adsorption capacity reflected on a complete monolayer (mg/g); K_L is the adsorption equilibrium constant (L/mg) that is related to the apparent energy of sorption.

Linear plot of dependence of $1/q_e$ versus $1/C_e$ is built. The values of the q_{max} and K_L were calculated from the slope and the intercept of the plot, respectively.

The key features of Langmuir isotherms can be expressed using the separation factor (R_L), which is determined by the following equation [28].

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

where C_0 is the initial concentration of the adsorbate (mg/L) and K_L is the Langmuir adsorption constant (L/mg). The value of the separation factor (R_L) indicates the nature of the adsorption process.

3.5.2. Freundlich isotherm

The Freundlich isotherm is an empirical equation that describes adsorption on a heterogeneous surface, assuming that adsorption takes place at sites with different adsorption energies. This isotherm is typically expressed by Eq. (5) [29].

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

where K_F (mg/g) and n are the Freundlich constants' characteristics of the system, representing adsorption capacity and adsorption intensity, respectively. The parameter n - is an empirical factor that varies depending on the degree of surface heterogeneity and is associated with the distribution of bound ions on the sorbent surface. Generally, when $n > 1$, it indicates favorable adsorption of the adsorbate onto the adsorbent, with higher n values corresponding to stronger adsorption intensity [30]. Notably, in all the examined temperatures and concentrations, the n value was significantly greater than unity.

3.5.3. Temkin Isotherm

The Temkin isotherm can be used to study the effect of indirect adsorbent±adsorbate interactions on the adsorption, and it suggests that the heat of adsorption of all the molecules in the layer would decrease linearly with the coverage due to these interactions. This model also assumes that adsorption is characterized by a uniform distribution of binding energies upto some maximum binding energy [31,32]. The linear form of Temkin isotherm is expressed as follows:

$$q_e = B \ln K_t + B \ln C_e \qquad (6)$$

where B=RT/b, K_t - is the equilibrium binding constant (L/mg), and B - is related to the heat of adsorption. A plot of q_e versus $\ln C_e$ enables the determination of the isotherm constants.

3.5.4. Redlich–Peterson Isotherm

The Redlich–Peterson equation [33] is widely used as a compromise between the Langmuir and Freundlich isotherm systems. This model has three parameters and incorporates the advantageous significance of both models. The Redlich–Peterson model can be represented as follows:

$$q_e = \frac{K_{RP} C_e}{1 + (\alpha C_e)^{\beta}} \qquad (7)$$

where K_{RP} (l/g) and α $(l/mg)^{\beta}$ are Redlich–Peterson isotherm constants and β is the exponent that lies between 0 and 1. The Redlich–Peterson isotherm model has two limiting cases, when β = 1, the Langmuir equation results, whereas when β = 0, the Redlich–Peterson isotherm equation transforms to Henry's law equation.

Table 1.

Constants and coefficients of adsorption isotherm models for the Ni^{2+}

Model	Parameter			
Langmuir isotherm	q_{max} ($mg \cdot g^{-1}$)	K_L ($L \cdot mg^{-1}$)		R^2
	145,6	0,042		0,992
Freundlich isotherm	K_F ($mg^{1-n} \cdot L^n \cdot g^{-1}$)	n		R^2
	12,3	2,1		0,945
Temkin isotherm	B ($kJ \cdot mol^{-1}$)	K_t ($L \cdot mg^{-1}$)		R^2
	85,2	1,45		0,928
Redlich-Patterson isotherm	K_{RP} ($L \cdot mg^{-1}$)	RPα ($L \cdot mg^{-1}$)	Rβ	R^2
	18,6	0,11	0,89	0,965

Table 1 shows the adsorption isotherm constants, reflecting surface properties and adsorption affinity. The maximum adsorption capacity (q_{max}) and Langmuir constant (K_L) were determined through nonlinear fitting. With an R^2 value of 0.992, the Langmuir model showed an excellent fit, likely due to the homogeneous distribution of adsorption sites, as assumed by this model. The q_{max} for Ni was 145.6. While the Langmuir model effectively described the adsorption data, other models were also evaluated. The Freundlich isotherm indicated that metal ion concentration on the adsorbent increased with solution concentration, with an R^2 of 0.945 for Ni, lower than that of the

Langmuir model. The Freundlich constant (K_F) was 12.3, and the power (n) was within the optimal range (0–10). The Temkin model, considering adsorption energy distribution, showed that lower BT values indicated weaker bonds between the adsorbate and adsorbent. The Redlich–Peterson model, combining Langmuir and Freundlich models, also exhibited an R^2 of 0.965.Thus, the Langmuir isotherm best described the adsorption data, confirming a uniform distribution of active sites with consistent adsorption energy.

3.6. Thermodynamic parameters

Thermodynamic parameters such as Gibbs free energy change (ΔG), enthalpy change (ΔH), and entropy change (ΔS) are commonly used to analyze adsorbate-adsorbent systems. A negative ΔG value signifies that the adsorption process is spontaneous. The ΔH parameter helps determine the adsorption nature-positive ΔH suggests an endothermic reaction, while negative ΔH indicates an exothermic process [26]. A positive ΔS reflects increased disorder of adsorbate molecules on the adsorbent surface compared to their state in solution. The relationship between the adsorption free energy (ΔG) and the Langmuir equilibrium constant can be expressed as follows:

$$\Delta G = -RT \ln K_L \qquad (8)$$

where K_L is the equilibrium constant, R is the Universal gas constant (8.314 J/mol·K), T is the Temperature (K).

Enthalpy and entropy changes are also related to the Langmuir equilibrium constant by the following expression:

$$\ln K_L = \frac{\Delta S}{} - \frac{\Delta H}{} \qquad (9)$$

Table 2.

Thermodynamic parameters for the adsorption of nickel(II)

T (K)	ΔG	ΔH(kJ/mol	ΔS
298	-12,3	+25,6	+0,130
308	-13,1		
318	-14,0		

Table 2 shows the calculated values of the thermodynamic parameters for the adsorption of nickel on magnetic adsorbent. The values of ΔG suggest the spontaneous nature of the adsorption process and give the information about the type of adsorption. The increase in ΔG with increasing temperature showed that adsorption was favourable at high temperatures. The positive value of ΔH suggests that the interaction of nickel (II) ions adsorbed by magnetic adsorbent is an endothermic process, which supported by the increasing adsorption of nickel (II) ions with the increase in temperature. The positive value of ΔS indicates that there is an increase in the randomness in the system solid/solution interface during the adsorption process.

CONCLUSION

This study demonstrates the efficiency of a magnetic adsorbent for removing Ni ions from contaminated water as a wastewater treatment method. The high adsorption capacity, ease of separation, and good reusability of the magnetic adsorbent make it a promising candidate for wastewater treatment applications. The adsorption process was optimized for Ni^{2+} solutions to achieve the best results. Various isotherm models, including Freundlich, Langmuir, Temkin, and Redlich–Patterson, were analyzed to assess adsorption behavior. The strong correlation of the Langmuir model ($R^2 > 0.9$) indicated a good fit with experimental data, confirming that Ni ion adsorption aligns best with this model. The maximum adsorption capacity for Ni was 273.7. Based on the Langmuir hypothesis, the active sites on the adsorbent surface are uniformly distributed with consistent adsorption energy. Thermodynamic analysis confirmed that Ni(II) adsorption is spontaneous ($\Delta G° < 0$), endothermic ($\Delta H° > 0$), and entropy-driven ($\Delta S° > 0$).

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YENİ MAQNİT XASSƏLİ SORBENT İLƏ SULU MƏHLULLARDAN Ni (II) İONUNUN ADSORPSİYON TƏDQİQATLARI: ADSORPSİYA İZOTERMLERİ

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Su mənbələrinin ağır metal ionları, xüsusilə nikel Ni(II) ionu ilə çirklənməsi ətraf mühit və sağlamlıq üçün ciddi təhlükə yaradır. Bu tədqiqat Ni(II) ionlarının sulu məhlullardan ayrılması üçün yeni sintez edilmiş maqnetik sorbentin adsorbsiya davranışını araşdırır. Adsorbsiya prosesinə təsir edən müxtəlif parametrlər, o cümlədən pH, təmas müddəti və ilkin metal ionlarının konsentrasiyası tədqiq edilmişdir. Nəticələr göstərdi ki, ən yüksək adsorbsiya qabiliyyəti pH 6-da əldə edilib. Adsorbsiya prosesini təsvir etmək üçün müxtəlif izoterm modelləri, o cümlədən Langmuir, Freundlich, Temkin və Redlich-Patterson tədqiq edilmişdir. Tədqiqat nəticələrin Langmuir modelinə uyğun olduğunu təsdiqlədi. Ni^{2+} üçün maksimum adsorbsiya qabiliyyəti 145,6 mq/q müəyyən edilmişdir. Adsorbsiyanın termodinamik parametrləri də öyrənilmiş və tədqiqat təsdiq etmişdir ki, adsorbsiya endotermik prosesdir. Nəticələr göstərir ki, yeni maqnit adsorbent tullantı sularından Ni(II) ionlarının ayrılması üçün perspektivli materialdır.

Açar sözlər: *maqnit adsorbent, Ni (II), adsorbsiya izotermləri, çirkab suların təmizlənməsi, adsorbsiya qabiliyyəti*

ИССЛЕДОВАНИЕ АДСОРБЦИИ ИОНОВ NI(II) ИЗ ВОДНЫХ РАСТВОРОВ С ИСПОЛЬЗОВАНИЕМ НОВОГО МАГНИТНОГО СОРБЕНТА: АДСОРБЦИОННЫЕ ИЗОТЕРМЫ

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Загрязнение источников воды ионами тяжелых металлов, особенно никеля (Ni(II)), представляет серьезную опасность для окружающей среды и здоровья. В этом исследовании изучается адсорбционное поведение недавно синтезированного магнитного сорбента для удаления ионов Ni(II) из водных растворов. Были изучены различные параметры, влияющие на процесс адсорбции, включая pH, время контакта и начальную концентрацию ионов металла. Результаты показали, что самая высокая адсорбционная способность была достигнута при pH 6. Для описания процесса адсорбции были исследованы различные изотермические модели, включая Ленгмюра, Фрейндлиха, Темкина и Редлиха-Паттерсона. Исследование подтвердило, что результаты согласуются с моделью Ленгмюра. Максимальная адсорбционная способность для Ni^{2+} была определена как 145,6 мг/г. Также были изучены термодинамические параметры адсорбции, и исследование подтвердило, что адсорбция является эндотермическим процессом. Результаты показывают, что новый магнитный адсорбент является перспективным материалом для удаления ионов Ni(II) из сточных вод.

Ключевые слова: *магнитный адсорбент, Ni (II), изотермы адсорбции, очистка сточных вод, адсорбционная емкость*