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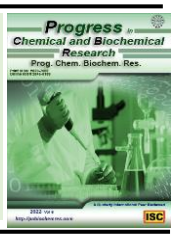

Original Research Article

Thermodynamic and kinetic Study of Adsorption of Cobalt II Using Adsorbent of Magnesium Oxide Nano-Particles Deposited on Chitosan

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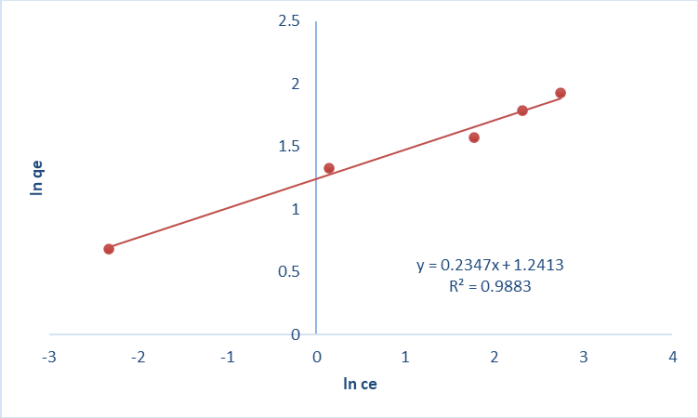
ABSTRACT

The present study is on MgO nanoparticles deposited on chitosan, which is investigated for the removal of the heavy metal cobalt II using an atomic absorption spectrometer at various concentrations. The laboratory study further investigates the removal of heavy metals such as cobalt (II) by the chitosan adsorbent in the batch adsorption test system. In experiments related to this research, some parameters such as contact time, the initial dye concentration, the adsorbent amount, temperature and pH are investigated and calculated, and finally kinetic equations including (the first and the second degree) and thermodynamic equations including obedience absorption equations are studied. Likewise, Friendlich and Langmuir thermodynamic variables including ΔS, ΔG, and ΔH were examined and calculated. In this study, the effect of magnesium oxide (MgO) magnetic nanoparticles in the removal of heavy metal contaminants such as cobalt (II) is tried in vitro.

GRAPHICAL ABSTRACT



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Introduction

Adsorption process is one of the most common methods due to its flexibility in designing the process and producing water without pollution, and also it is suitable for reuse [1-3]. By choosing a suitable adsorbent, it can be considered a reliable method for removing certain types of contaminants. Some toxic compounds of metals such as cobalt (II) have been used in many chemical and metallurgical industries and they are in the effluent stream of these industries. These toxic compounds will have the adverse effects on the environment and their health [4, 5]. One of the common methods in the field of removal of metals such as cobalt (II) is using heavy metal adsorption process on adsorbent solid particles. Some metals, such as iron, zinc, and copper are found naturally in the human body and are good for health in small amounts, as well. It should be noted that when toxic toxins enter the body, the body is not able to break them down and is not excreted, however it is deposited and accumulated in tissues such as fat, muscles, bones, and joints, and this causes disease. There will be numerous side effects in the body [6-11]. As mentioned, natural hydrogels and polymers are very suitable for the recovery and removal of metal cations due to their properties such as unique structure, reasonable price, being in the form of various adsorbents, easy to use, reusability, and high chemical, and mechanical resistance [12-14]. The crisis of water shortage in the country is one of the most significant issues due to the increasing demand for consumption in the industrial, agricultural, and domestic sectors. The increased consumption along with the recent droughts has severely reduced water resources, and as a result, the water supply from the unconventional sources such as wastewater, polluted surface, groundwater sources, and saline water has been considered [15-17]. The productive effluents in various urban, industrial, and agricultural sectors are considered as a sustainable source for water crisis management as a strategic solution [18-20].

Materials and Methods

In this study, the removal of cobalt metal from water by a magnesium nan-oxide oxide adsorbent deposited on chitosan was investigated. First, the synthesized hydrogel nanocomposite was prepared in solid form, and finally the tests were performed to analyze the adsorption process under the discontinuous conditions and the parameters of contact time, adsorbent amount, initial contaminant concentration, pH, and temperature were analyzed.

Synthesis of magnesium oxide/chitosan hydrogel nanocomposites

To prepare a magnesium oxide/chitosan hydrogel nanocomposite adsorbent, a 2% w/v solution of chitosan in 0.1 M acetic acid was initially prepared as a solvent. Then, after dispersing in an ultrasonic bath, magnesium oxide nanoparticles of 5% w/v gently were added to magnesium oxide nanoparticles and after that to the chitosan solution and it was placed on a magnetic stirrer for 30 minutes at 2000 rpm, and then it was ultrasonized again. Then after, it was placed on a magnetic stirrer for 2 hours, and finally the resulting solution was dried in a glass jar at room temperature and the resulting film was used for absorption.

Solubilization

To prepare the solution containing cobalt (II), the standard solution of 1000 ppm cobalt of atomic absorption device was used and to adjust the pH of the environment, rick acid chloride and 0.05 M of sodium hydroxide were utilized. First, a solution of 1000 ppm of cobalt (II) nitrate salt was made as a standard solution, and then from this solution, using deionized water and $N_1V_1=N_2V_2$ bond, 500,250, and 100 ppm solutions were prepared and from 100 ppm solution to

make final concentrations of 15, 20, and 25 ppm, 5, 10 were used.

The calibration curve determination

To determine the calibration curve, different concentrations of the standard cobalt solution with concentrations of 5, 10, 15, 20, and 25 ppm were prepared at ambient temperature, and then their adsorption was read by an atomic absorption device, and finally the calibration curve was drawn.

Table 1. Absorption values for different initial concentrations of cobalt (II)

Cobalt Initial conc (ppm)	0	5	10	15	20	25
Absorption	0.0	0.07	0.125	0.190	0.250	0.309

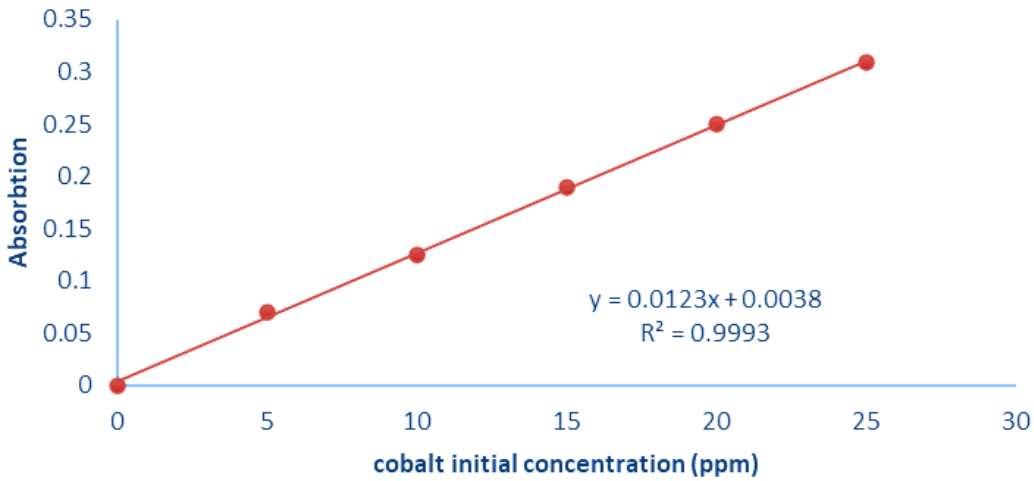

Figure 1. Adsorption calibration curve in terms of different initial concentrations of cobalt (II)

The investigation and optimization of parameters affect the adsorption of cobalt (II) ions using magnesium oxide/chitosan hydrogel nanocomposite.

Investigating the effect of pH

20 mL of cobalt (II) solution with a concentration of 20 ppm was added at different pH values to 0.03 g of magnesium oxide/chitosan hydrogel nanocomposite adsorbent at room temperature for one hour on a magnetic stirrer at a constant speed of 400 rpm. The solutions were then passed through a filter paper, and finally their adsorption was measured by an atomic absorption apparatus. The results revealed that the highest percentage of divalent cobalt ion removal occurred with the adsorption of magnesium oxide/chitosan hydrogel nanocomposite at pH=5. Therefore, pH=5 was chosen as the optimal pH.

Investigating the effect of the adsorbent amount

20 mL of cobalt (II) solution with a concentration of 20 ppm and pH=5 was added to different amounts of magnesium oxide/chitosan hydrogel nanocomposite adsorbent in grams and at room temperature for one hour on a magnetic stirrer with a constant speed of 400 rpm per minute. The solutions were then passed through a filter paper, and finally their absorption was measured by an atomic absorption apparatus. The results indicated that the highest removal percentage of divalent cobalt ions occurred with the amount of 0.1 g from the adsorbent of magnesium oxide/chitosan hydrogel nanocomposite.

Investigating the effect of time

20 mL of cobalt (II) solution with a concentration of 20 ppm and pH=5 was added to 0.1 g of the adsorbent nanocomposite of magnesium

oxide/chitosan hydrogel, at room temperature and in different times per minute on a magnetic stirrer with a constant speed of 400 rpm. The solutions were then passed through a filter paper, and finally their adsorption was measured by an atomic absorption apparatus. The results revealed that the highest removal percentage of divalent cobalt ions by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite occurred in 50 minutes.

Investigating the effect of temperature

20 mL of cobalt (II) solution with a concentration of 20 ppm and pH=5 was added to 0.1 g of the adsorbent of magnesium oxide/chitosan hydrogel nanocomposite and it was placed on a magnetic stirrer at a constant speed of 400 rpm for 50 minutes at different temperatures. The solutions were then passed through a filter paper and their adsorption was measured by an atomic absorption spectrometer. The results indicated that the highest percentage of removal of divalent cobalt ions by magnesium oxide/chitosan hydrogel nanocomposite adsorbent occurred at 298 Kelvin.

Investigating the effect of valence cobalt (II) concentration

20 mL of cobalt (II) solution with different concentrations and pH=5 was added to 0.1 g of adsorbent nanocomposite of magnesium oxide/chitosan hydrogel and at 298 K for 50 minutes on a magnetic stirrer with a constant speed of 400 rpm per minute. The solutions were then passed through a filter paper, and finally their absorption was measured by an atomic absorption apparatus.

Test Results and Calculations

Determination of adsorbent profile

FT-IR adsorbent of magnesium oxide hydrogel nanocomposite/chitosan

The presence of a strong peak in the 687 cm^{-1} region is related to the Mg-O bond.

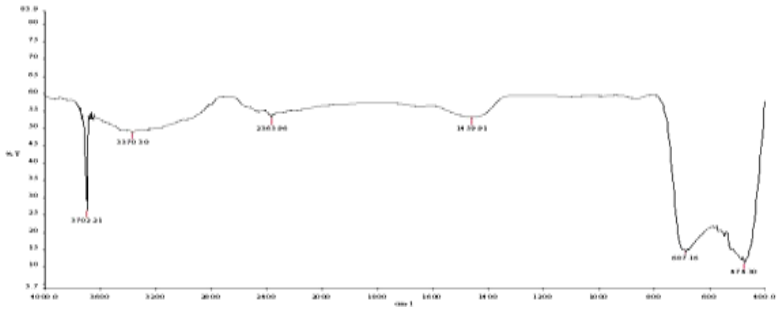

Figure 2. Infrared spectrum of magnesium oxide nanoparticles

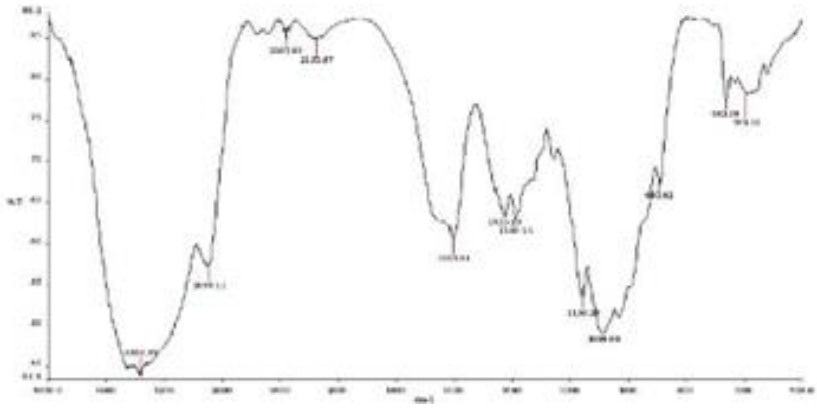

Figure 3. Infrared spectrum of chitosan composition

The chitosan spectrum which is depicted in Figure 3 consists of an absorption band in the 3369 cm^{-1} region corresponding to the peak of a combination of NH_2 and OH vibratory tensile groups. The absorption peaks in 2894 cm^{-1} region are related to the asymmetric tensile stress CH_2 and CH_3 and the absorption peaks from 1500 cm^{-1} to 1600 cm^{-1} belong to the carbonyl group attached to the amide and absorption bands from 1020 cm^{-1} to 1150 cm^{-1} can be applied to the tensile C-O-C, as attributed to be asymmetric.

In the infrared spectrum belonging to the magnesium oxide/chitosan hydrogel composite as displayed in Figure 4, compared to the chitosan infrared spectrum, the absorption band is 3369 cm^{-1} as wider and shifts to the left to 3550 cm^{-1}, as being displaced indicating a strong interaction between these groups and magnesium oxide, and further the absorption peak in the 650 cm^{-1} region is related to the amide group bond and the tensile state of magnesium oxide.

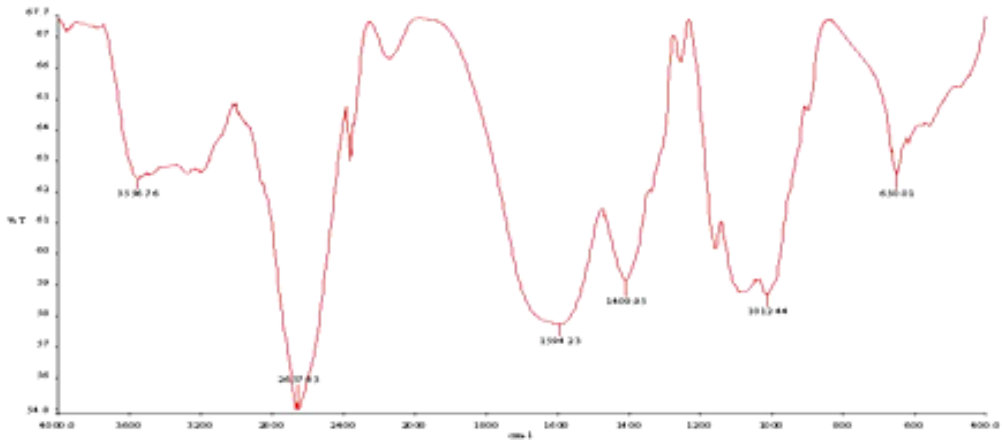

Figure 4. Infrared spectrum of magnesium oxide/chitosan hydrogel nanocomposite

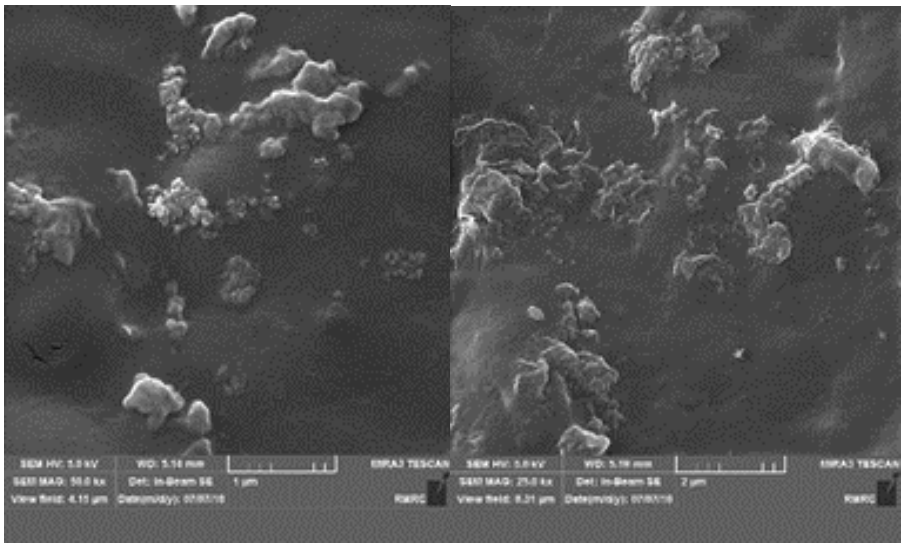

Figure 5. SEM spectrum image of magnesium oxide/chitosan hydrogel nanocomposite adsorbent

Figure 6. SEM spectrum image of adsorbent nanocomposite of magnesium oxide/ chitosan hydrogel

Results of the XRD spectrum of magnesium oxide nanoparticles

Using the Debbie-Scherer relationship, the results of the XRD spectrum can be used to calculate the size of the crystals.

Debbie-Scherer relationship $D= K\lambda/\beta\cos\theta$

In the above relation, k as Scherer constant, λ as the wavelength of the device, β as the peak width at half-height, θ as Bragg diffraction angle, and D as the particle size are produced, the obtained amount of particle size as observed from the SEM spectrum is reported to be between 40 and 60 nm.

Results of the effect of pH on the amount of adsorbent

To investigate the effect of pH on cobalt (II) pollutant, different acidic, alkaline, and neutral environments were investigated and the results of this study were presented in the following table. The following diagram indicates the pH effect on the removal percentage of cobalt metal ions.

Table 2. The results of calculations related to the adsorption of cobalt (II) ions at different pH values

pH	Adsorption of equilibrium solution	Equilibrium solution concentration (ppm)	Delete percentage
3	0/105	8/23	58/8
4	0/098	7/66	61/7
5	0/082	6/36	68/2
6	0/118	9/28	53/6
7	0/135	10/66	46/7

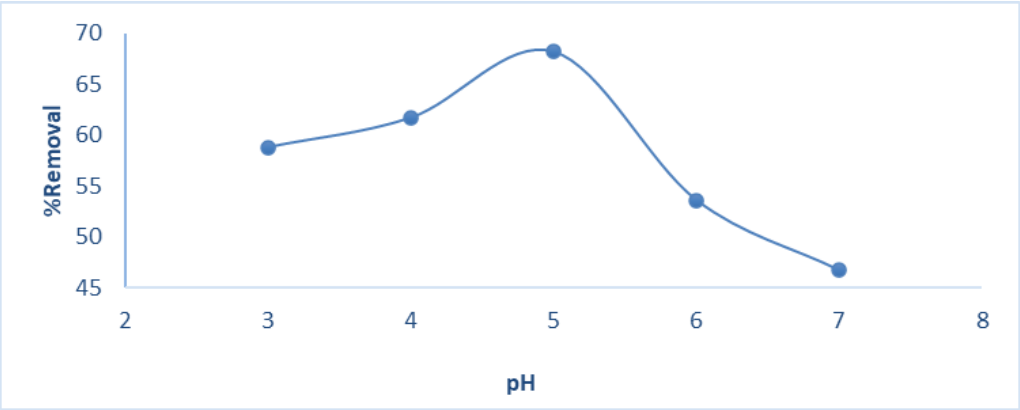

Figure 7. The effect of pH on the adsorption rate of cobalt (II) ion by the adsorbent on magnesium oxide/chitosan hydrogel nanocomposites

According to the pH chart, the optimum for the experiments is 5. As the pH decreases, the removal percentage decreases, which can be due to the competition of the ambient H^+ with CO^{+2}. Because the addition of ric acid chloride increases the H^+ amount in the environment, which H^+ neutralizes the negative charge of the adsorbent and reduces the tendency of CO^{+2} to be placed on the adsorbent and reduces adsorption. By adding sodium to the cobalt solution, the amount of OH^- ions increases and prevents CO^{+2} from settling on the adsorbent and decreases the adsorption.

Results of the effect of the adsorbent amount on the amount of adsorption

According to the obtained data from the experiments, the percentage of cobalt (II) ion removal was calculated by different amounts of adsorbent. The graph of the effect of the adsorbent amount on the removal of cobalt ions by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite is as follows.

Table 3. The results of calculations related to the adsorption of cobalt (II) with different amounts of adsorbent

Delete percentage	Equilibrium solution concentration (ppb)	Adsorption of equilibrium solution	Absorbent amount (g)
67	6/60	0/085	0/03
78	4/40	0/058	0/05
87/3	2/53	0/035	0/07
94/2	1/15	0/018	0/1
95	0/99	0/016	0/12

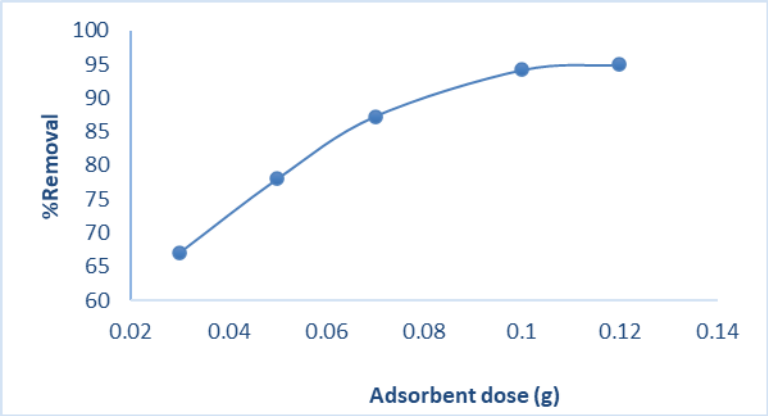

Figure 8. The effect of adsorbent on adsorption of cobalt (II) ion by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite

Obviously, the optimal amount of adsorbent is 0.1 g. The number of adsorbent increases initially with enhancing the amount of adsorbent up to 0.1 g due to the increase of active sites, but gradually increasing the amount of adsorbent has no effect on the adsorption process.

Results of the effect of contact time on the amount of adsorbent

According to the experimental data, the adsorption capacity of cobalt (II) ion was calculated by the adsorbent of magnesium oxide/chitosan hydrogel nanocomposite at times of 10, 20, 30, 40, 50, and 60 minutes.

Table 4. The results of adsorption values of cobalt (II) ions at different times by adsorbent

Time (min)	Adsorption of equilibrium solution	Equilibrium solution concentration (ppm)	Delete percentage
10	0/148	11/72	41/4
20	0/102	7/98	60/1
30	0/076	5/87	70/6
40	0/042	3/1	84/5
50	0/017	1/07	94/6
60	0/015	0/91	95/4

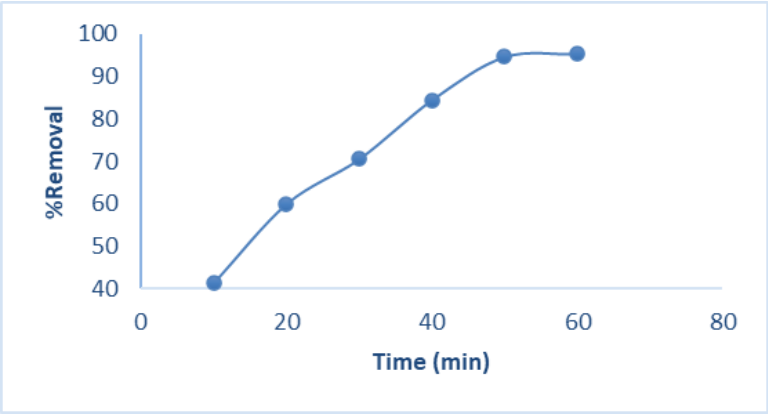

Figure 9. The effect of contact time on the adsorption rate of cobalt (II) ion by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite

As can be seen from the diagram, the slope of the curve is high in the early times, due to the fact that in the initial moments, the concentration of cobalt ions in the environment is high and the adsorbent has many empty sites, i.e. the concentration gradient is at its maximum value. Concentration affects the mass transfer rate, and therefore ions are rapidly absorbed by the adsorbent. Through the passage of time, the solution's concentration decreases, while the concentration of the adsorbent surface increases. Therefore, the time delta shrinks, and as a result, the mass transfer rate decreases until the mass transfer no longer takes place. Because the adsorbent is completely saturated with cobalt, then the system reaches equilibrium, which is called the equilibrium time. According to the diagram, the optimal contact time is 50 minutes, and after this time, the adsorbent surface is saturated with cobalt and no adsorption takes place. In other words, after reaching the optimal contact time, the absorption and repulsion speeds are equal and the system becomes balanced and the absorption capacity is completed, as well.

The results of the studied temperature effect

According to the obtained data from the experiments, the percentage of cobalt (II) ion removal by the adsorbent was measured and calculated at different temperatures. The graph of the temperature effect on the removal of cobalt by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite is as follows.

Table 5. The results of calculations related to the removal of cobalt (II) ions at different temperatures

Temperature (K)	Adsorption of equilibrium solution	Equilibrium solution concentration (ppm)	Delete percentage
298	0/019	1/23	93/8
308	0/045	3/35	83/2
318	0/076	5/87	70/6
328	0/098	7/66	61/7
338	0/117	9/20	54

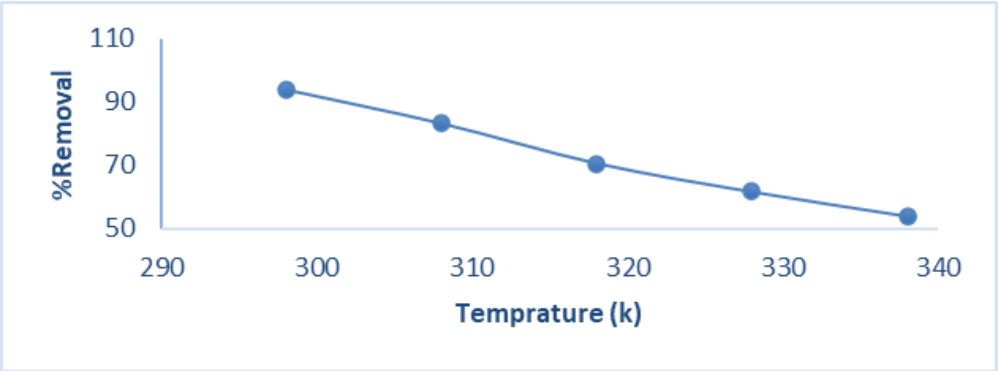

Figure 10. The effect of temperature effect on cobalt ion removal by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite

Based on the above diagram, it is visible that the adsorption percentage decreases with increasing temperature, which may indicate the physical nature of the adsorption. Indeed, increasing the temperature enhances the entropy and weakens the bonds between the adsorbent and adsorbent species, as well.

The results of the effect of initial concentration on the amount of adsorption

According to the obtained data from the experiments, the percentage of cobalt (II) ion removal by the adsorbent was measured and calculated at different concentrations of the contaminant. The studies reveal that with increasing cobalt concentration, the removal rate decreases. The graph of the concentration effect on cobalt removal by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite is as follows.

Table 6. The results of Cobalt (II) ion adsorption values in different concentrations of cobalt (II) nitrate

Initial concentration (ppm)	Adsorption of equilibrium solution	Equilibrium solution concentration (ppm)	Delete percentage
10	0/005	0/097	99
20	0/018	1/15	94/2
30	0/076	5/87	80/4
40	0/129	10/18	74/5
50	0/196	15/62	68

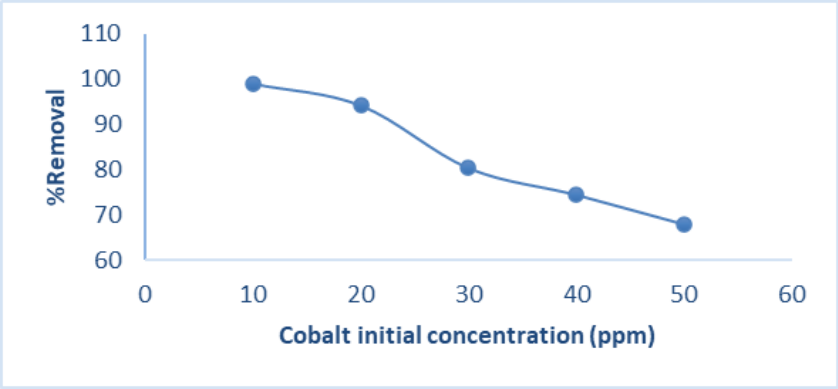

Figure 11. The effect of the initial cobalt concentration on the removal of cobalt ion by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite

Investigating adsorption isotherms and analysis of isothermal data

Using the obtained adsorption data from the adsorption curves, the relationship between the analyte values adsorbed by the adsorbent and the amount remaining in the solution at equilibrium can provide useful information about the mechanism, the surface properties, and the adsorbent affinity for the adsorbent. These equilibrium relationships are expressed by absorption isotherms. Absorption isotherms are scientifically and theoretically very prominent. Adsorption isotherms are also used to understand the type of interaction between the adsorbent and the adsorbent. Isothermal data can be adapted to different isothermal models to find a suitable model to study the process.

Investigating cobalt (II) adsorption isotherms on magnesium oxide/chitosan hydrogel nanocomposite composition

The values of adsorbed cobalt on 0.1 g of adsorbent in different initial concentrations of cobalt (II) nitrate solution according to Tables (5-6) and in the optimal conditions of the other variables are investigated and adsorption isotherm diagrams such as Langmuir, Friendlich, and Tamkin on the data were applied and the

results of this study revealed that the adsorption of cobalt (II) ion on the adsorbent follows the Friendlich adsorption isotherm.

Table 7. The effect of the initial concentrations on the adsorption percentage of cobalt (II) ion by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite to investigate adsorption isotherms at pH=5, adsorbent dose of 0.1 g, contact time 50 minutes, and temperature 298 K

C0	Adsorption	C_e	%Removal	q_e	C_e/q_e	Ln C_e	Ln q_e
10	0/005	0/097	99	1/980	0/049	-2/33	0/683
20	0/018	1/15	94/2	3/77	0/350	0/139	1/327
30	0/076	5/87	80/4	4/83	1/215	1/769	1/575
40	0/129	10/18	74/5	5/96	1/71	2/320	1/785
50	0/196	15/62	68	6/87	2/27	2/748	1/927

Investigating Langmuir Adsorption Temperature

Using the data of the table, Langmuir isotherm equation was solved, the Ce/qe curve was plotted in terms of Ce, and the line equation was obtained. Thus, the Langmuir isotherm constants were calculated. The values obtained from the dimensionless parameter of separation factor (RL) at different initial concentrations indicate the desirable adsorption of divalent cobalt ions on the adsorbent of magnesium oxide/chitosan hydrogel nanocomposite.

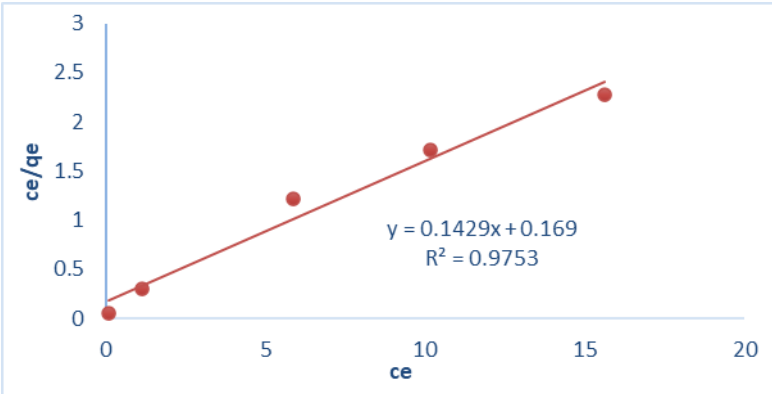

Figure 12. Langmuir adsorption isotherm for different concentrations of divalent cobalt with different initial concentrations by magnesium oxide/chitosan hydrogel nanocomposite adsorbent

Table 8. Langmuir surface adsorption isotherm constants

q_m (mg g^{-1})	K_L (L mg^{-1})	R^2
6/997	0/846	0/9753

Table 9. Values of the dissociation factor related to the Langmuir isotherm

C0(mg g^{-1})	R_L
10	0/084
20	0/055
30	0/037
40	0/023
50	0/023

Investigating Friendlich surface adsorption isotherm

The values obtained from the nf parameter, which is related to the adsorption intensity, indicate that the adsorption process occurred physically and optimally.

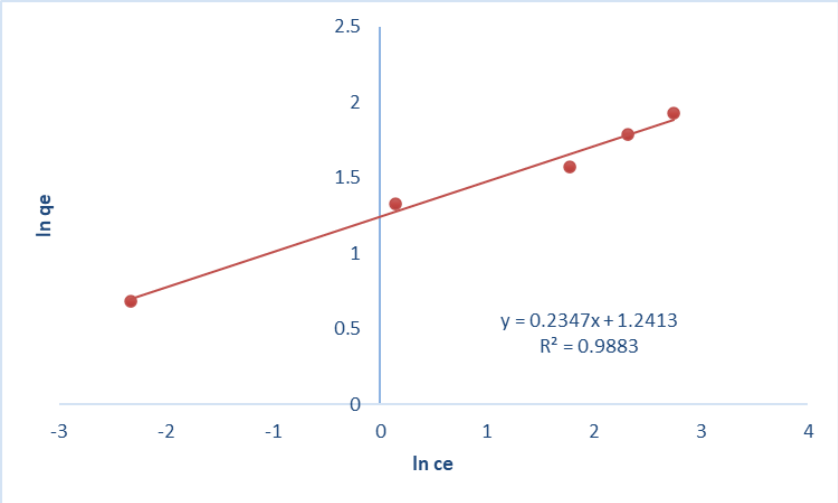

Figure 13. Friendlich isotherm for adsorption of divalent cobalt ions with different initial concentrations by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite under the optimal conditions

Table 10. Friendlich surface adsorption isotherm constants

n_f	K_f(mg g^{-1})	R^2
4/26	3/460	0/9883

Investigating isothermal isotherm ion adsorption isotherm

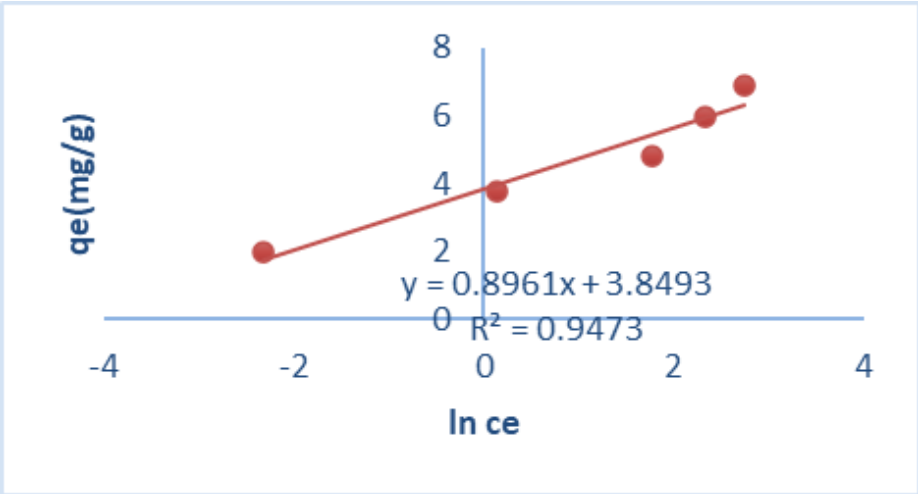

Figure 14. Tamkin isotherm for adsorption of divalent cobalt ions with different initial concentrations by adsorbent of magnesium oxide/chitosan hydrogel nanocomposite under the optimal conditions

Table 11. Absorption isotherm constants of obedience

B (J mole^{-1})	K_T (L mg^{-1})	R^2
0.8961	73.33	0.9473

Adsorption kinetics

Adsorption is a multi-step process involving the transfer of adsorbed molecules from the dissolved phase to the adsorbent surface, and then the penetration of soluble particles into the internal pores of the adsorbent. Nowadays, researchers seek to develop nanotechnology and the application of nanotechnology in human life. In this regard, the construction of efficient, recyclable, and easily prepared adsorbents which can perform the adsorption process with high accuracy and precision in the shortest possible time, and it has been a part of nanotechnology studies. To achieve this goal, the kinetic studies are necessary.

Investigating cobalt (II) adsorption kinetics on magnesium oxide/chitosan hydrogel nanocomposite

Investigating the quasi-first order kinetic model

To investigate the adsorption kinetics of cobalt (II) on the nanocomposite of magnesium oxide/chitosan hydrogel, a series of experiments were performed at different times at a concentration of 20 mg/l of cobalt (II) nitrate solution and under the optimal conditions of the other variables. Different kinetic models were used on experimental data to evaluate the speed of the adsorption process and the potential of the velocity-determining step. In this regard, to investigate the adsorption rate and calculate the relevant constants, the quasi-first-order and the second-order quasi-second-order kinetic models were used. The correlation coefficient revealed that the adsorption of cobalt (II) follows the quasi-second order kinetics.

Table 12. The effect of contact time on the adsorption percentage of cobalt (II) by magnesium oxide/chitosan hydrogel nanocomposite to investigate the adsorption kinetics at pH=5, adsorbent dose of 0.1 g, 298 K temperature and concentration of 20 mg/l cobalt

Time (min)	Adsorption	C_e (mg L^{-1})	Adsorption	q_t (mg g^{-1})	q_e-q_t	Log (q_e-q_t)	t/q_t
10	0/148	11/72	41/4	1/65	3/18	0/502	6/06
20	0/102	7/98	60/1	2/40	2/43	0/385	8/33
30	0/076	5/87	70/6	2/82	2/01	0/303	10/63
40	0/042	3/1	84/5	3/38	1/45	0/161	11/83
50	0/017	1/07	94/6	3/78	1/05	0/021	13/22
60	0/015	0/91	95/4	3/82	1/01	0/004	15/70

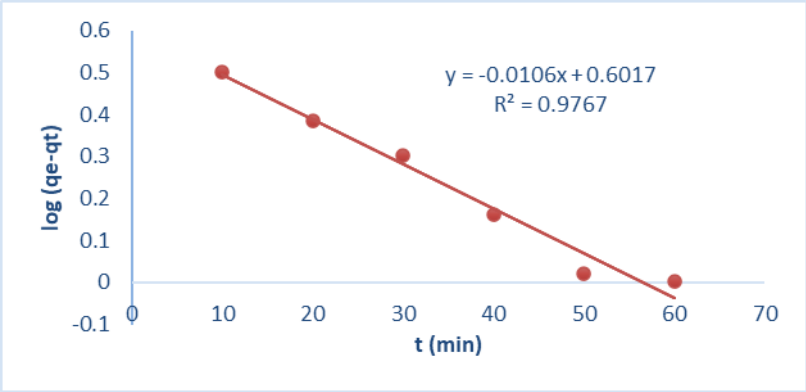

Figure 15. Kinetics of the quasi-first-order adsorption of cobalt (II) by magnesium oxide/chitosan hydrogel nanocomposite adsorbent

Table 13. The quasi-first order kinetic parameters

q_e (mg g^{-1})	K_1(min^{-1})	R^2
3/996	0/024	0.7679

Investigating the quasi-second order kinetic model

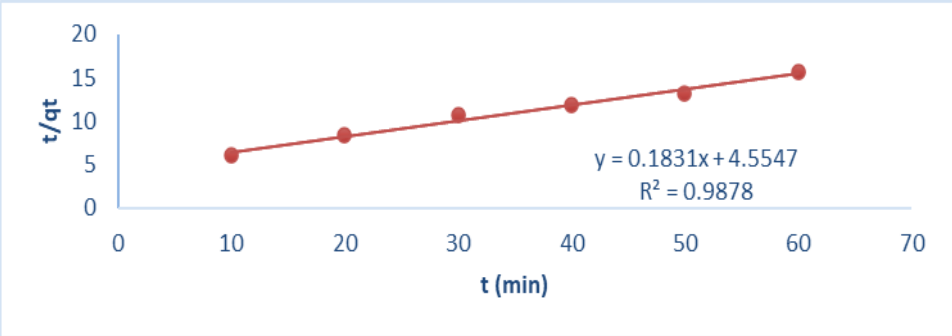

Figure 16. The quasi-second-order kinetics of cobalt (II) adsorption by magnesium oxide/chitosan hydrogel nanocomposite adsorbent

Table 14. The quasi-second order kinetic parameters

q_e(mg g^{-1})	K_2 (g $mg^{-1}min^{-1}$)	R^2
5/461	0.0073	0.9878

Adsorption thermodynamics

Calculation of thermodynamic variables

Using data and thermodynamic equation, the equilibrium constants (Kd) at different temperatures are calculated, and then using equations, drawing a linear curve lnKd in terms of 1/T, and obtaining the line equation of thermodynamic variables are extracted at different temperatures, and further the results are obtained. The negativity of the Gibbs standard free energy changes indicates that the adsorption process is spontaneous. It should be noted that the negation of standard enthalpy changes in the reaction is a sign that the

adsorption process is warm. Moreover, the positivity of standard entropy changes in the system indicates an increase in irregularities in the interface between the solid-solution adsorption processes. In other words, the positive entropy changes of the standard system indicate an increase in irregularity in the adsorbent in the pollutant adsorption process compared to the initial state before the adsorption process. Thus, the absorption has occurred.

Table 15. The equilibrium constant values for adsorption of valence cobalt (II) cobalt on magnesium oxide/chitosan hydrogel nanocomposite adsorbent

T(K)	C_e (mg L^{-1})	K_d(mL g^{-1})	LnK_d	1/T(K^{-1})
298	1/23	187/7	5/235	0/0033
308	3/35	166/5	5/115	0/0032
318	5/87	141/	4/950	0/0031
328	7/66	123/4	4/815	0/003
338	9/20	108	4/682	0/0029

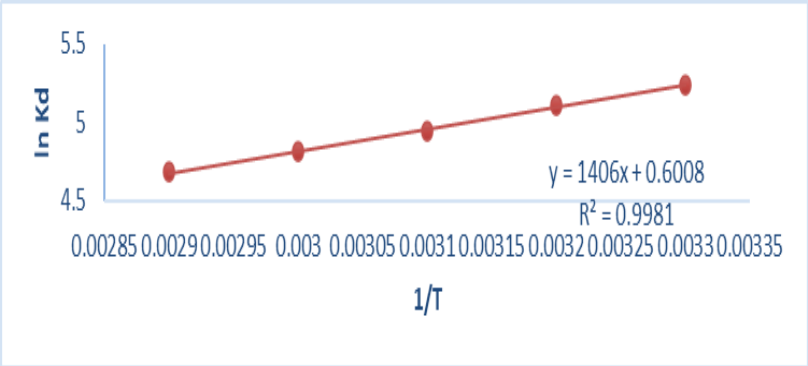

Figure 17. Vanthof diagram for derivation of thermodynamic parameters of cobalt (II) ion adsorption by magnesium oxide/chitosan hydrogel nanocomposite adsorbent

Table 16. The thermodynamic parameters of cobalt (II) adsorption by magnesium oxide/chitosan hydrogel nanocomposite adsorbent

T(K)	ΔG^0(Kj $mole^{-1}$)	H^0(KJ $mole^{-1}$)Δ	S^0(Kj $mole^{-1}$)Δ
298	-12/97		
308	-13/18		
318	-13/08	11/68-	0/005
328	-13/13		
338	-12/76		

Conclusion

1- The kinetic studies reveal that the adsorption of cobalt (II) on the adsorbent of magnesium oxide/chitosan hydrogel nanocomposite follows the quasi-second order kinetics.

2- By increasing the concentration of the solution containing divalent cobalt ions, the amount of adsorption decreases per unit mass of the adsorbent.

3- Increasing the temperature reduces the adsorption process; therefore, the adsorption

process can be considered an exothermic process.

4- The negative free energy of Gibbs standard indicates that the absorption process is spontaneous.

5. The negative enthalpy changes of the standard reaction indicate the exothermic process in the adsorption system, and also the positive entropy changes of the standard system indicate the increase of irregularity in the solid-solute interface.

6- According to the results of the adsorption isotherms, it was obvious that the Friendlich adsorption isotherm equation has a higher and more appropriate correlation coefficient than the other adsorption isotherm equations.

7- As the contact time increases until the equilibrium is reached, the absorption rate increases.

8- By calculating the nf values, it was observed that the adsorption occurs optimally and physically.

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