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SORPTION OF Ca^{2+} and Mg^{2+} ions OF SULPHOCATIONITE OBTAINED BY S MODIFICATION OF POLYVINYL CHLORIDE USING CaS_x SOLUTION

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ARTICLE INFO	ABSTRACT
Article history: Received: 2025-11-09 Received in revised form :2025-11-16 Accepted :2025-12-18 Available online: 2026-06-26	<i>In this work, $-\text{SO}_3\text{H}$ groups in the material obtained from polyvinyl chloride (PVC-SC), a new ion-exchange resin for purifying water from metal ions, were proven and their physicochemical parameters were determined. Kinetics and isotherms of sorption of Ca^{2+} and Mg^{2+} ions from artificial solutions to PVC-SC were studied. As a result, the adsorption of calcium II and magnesium II ions from aqueous solutions onto the new sulfocationite have consistent with the Langmuir isotherm. This indicated that new sulfocationite chemically adsorbed calcium II and magnesium II ions from artificial solutions.</i>
Keywords: Sulphur, sorption, cationite, calcium, magnesium, isotherm.	

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1. INTRODUCTION

Many countries are facing environmental problems due to improper disposal of industrial waste. Pollutants formed in water bodies have a harmful effect on the environment even at low concentrations. Recently, there are some methods of water purification, and ion exchange resins are widely used to remove Ca^{2+} and Mg^{2+} ions, which cause water hardness [1]. The most common reason for the purification of water from contaminants by the adsorption method is its technological simplicity, ease of process, repeated recycling of ion exchange material by recycling, and cost-effectiveness [2],[3].

Water hardness is caused by certain metals, especially calcium and magnesium ions. The sorption of Ca^{2+} and Mg^{2+} ions for water purification in the food industry from existing ion exchange resins on a weak acidic resin PC200FD was studied [4]. Adsorption of calcium to an aqueous solution of Purolite C100E synthetic ion exchange resin was studied using Langmuir and Freundlich isotherm models [5]. The authors [6] determined the structure of ion exchange resin by modifying the cellulose with amines by IR spectroscopy. The absorption rate and equilibrium of calcium and magnesium ions from artificial solutions to the made cation exchange materials were researched, with the heigh sorption value for the Langmuir isotherm model being 54.1 mg/g for calcium II and 42.6 mg/g for magniseum II. New cation exchange resin made by modification of PVC has been researched in water treatment for industrial use [7]. Some kinetic models of sorption from aqueous solutions of Ca^{2+} and Mg^{2+} ions to PVC-based

sulfonation exchange resin were studied and the amount of sorption was calculated [8]. Researchers are working on creating economically viable adsorbents for industrial wastewater treatment. One of the most important means of studying the mechanism of ion exchange of ionite is the organization of the adsorption isotherm. In recent years, the isotherm models of Langmuir, Freundlich, Temkin, Dubinin-Radushkevich have been analyzed in the study of ion sorption from solutions [9]. From the above literature, it is clear that ion exchange resins were obtained by modifying natural and synthetic polymers, and the adsorption of various metals was studied using isothermal models.

In this paper, the absorption isotherms of Ca^{2+} and Mg^{2+} ions for polyvinyl chloride-based ion exchange materials were researched.

2. Methods and materials of research

Analytical pure substances were used in the preparation, oxidation of calcium polysulfide solution and the sorption of calcium and magnesium ions to ion exchange resins for the modification of sulphur in polyvinyl chloride plastic. Analytical pure substances were used in the made, oxidation of CaS_x solution and the sorption of calcium and magnesium ions to ion exchange resins for the modification of sulfa-groups in polyvinyl chloride plastificat.

2.1. Synthesis of a cation exchange resin on the basis of PVC

2.1.1. Process of obtaining a solution of CaS_x

As a result of many experiments, the optimal conditions for obtaining a solution of CaS_x are the reaction in a flask with a mass ratio $\text{CaO}:\text{S}:\text{H}_2\text{O}$ of 1.0: 3.0: 16.8, respectively, at a temperature of 358-368 K for 70-90 minutes [10]. Additional substances such as H_2S , $\text{Ca}(\text{SH})_2$, CaS_2O_3 can be formed during the reaction. The result of the reaction is a dark red solution, which is filtered off. The mass fraction of CaS_x in the solution was found to be 28-34%. The residue can be used for re-synthesis or buried in the ground as fertilizer. A longer reaction time may lead to an increase in thiosulfate ions, but this has a positive effect on the production of cation exchange resins with sulfo groups from PVC. This is because thiosulfate ions or ion radicals, such as sulfide groups, are more likely to oxidize to sulphur groups during the oxidation process after being bonded to a PVC chain.

2.1.2. Modification of sulphur to PVC and extraction of cation exchange resins

Various experiments have been performed to modify sulphur to PVC [11]. Granular plastic PVC (grade I-40-13A) was extracted in ethyl acetate for 2 h. The goal is to increase the porosity of plastic by producing a plasticizer (dibutyl phthalate) from PVC. PVC is filtered and poured into a vessel, a boiling solution of CaS_x is poured over it, the vessel is hermetically sealed and heated at 423 K for 4 hours. In the next step, the $-\text{S}_x-$, $-\text{SH}$ groups of the sulfur-modified polymer are reacted with a concentrated solution of HNO_3 in a water bath for 3-4 hours and are oxidized to the $-\text{SO}_3\text{H}$ group.

The gained ion exchange resin was washed and the NaOH static exchange capacity (SEC) was determined, SEC value of 3.5 mg-eq/g.

2.2. Study of adsorbent properties

The physicochemical properties of synthesized sulpho-cation exchange resins were studied and element analysis and FTIR spectroscopy. The amount of elements in polyvinyl chloride-based resin was studied by Euro EA Elemental Analyzer. The spectra corresponding to the vibration of the functional groups in the obtained material were analyzed in an IRTracer-100 IR-Fourier

spectrophotometer. The study of the physicochemical parameters of the obtained cation exchanging material showed that it meets the requirements of GOST 20298-74 for use in the separation of ions available on an industrial scale. The ion exchange resin, synthesized from PVC-based plastic, has been officially registered by the Uzbekistan Institute of Standards, operating under the Agency for Technical Regulation of Uzbekistan (UZSTANDART), under the designation Ts 02072392-00:2020.

Experiments of sorption

2.3.1 Preparation and sorption conditions of metal solutions

CaCl_2 and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ were used to research the adsorption of Ca^{2+} and Mg^{2+} ions that cause water hardness to polyvinyl chloride-based ion exchanger materials. Aqueous solutions of calcium and magnesium ions with concentration 0.1, 0.075, 0.05 and 0.025 mol/l were made. The adsorption process was researched in solutions at temperatures difference from 293 to 313 K. The adsorption process was studied in solutions with a temperature difference from 293 to 313 K. For this, the static ion exchange capacity SEC of 3.5 mg-eq/g cationite, 2 g/l in size, was studied in 100 ml flasks at a speed of 100 rpm for different time (2-12 h.) intervals.

. Initial and equilibrium concentrations of metal solutions were determined by titration with EDTA solution. The mass of absorbed metal in ionite was calculated as follows.

$$Q_e = \frac{C_0 - C_e}{m} \quad (1)$$

where Q_e is the amount of ion absorption in mg/g, C_0 is the concentration of the ion in the initial solution in mg/l, C_e is the concentration after sorption in mg/l, V is the volume of the solution containing the ion in l; m is the dry mass of the ion exchange resin in g.

2.3.2. Isothermal models

In the analysis of equilibrium processes, one of the most important tools is the adsorption isotherm. Therefore, in this work, the adsorption mechanism was analyzed using the isotherm models of Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich (D-R), which are widely used in liquid-solid systems.

3. Results and discussion

Characterization of a Cation Exchange Resin Synthesized from Polyvinyl Chloride

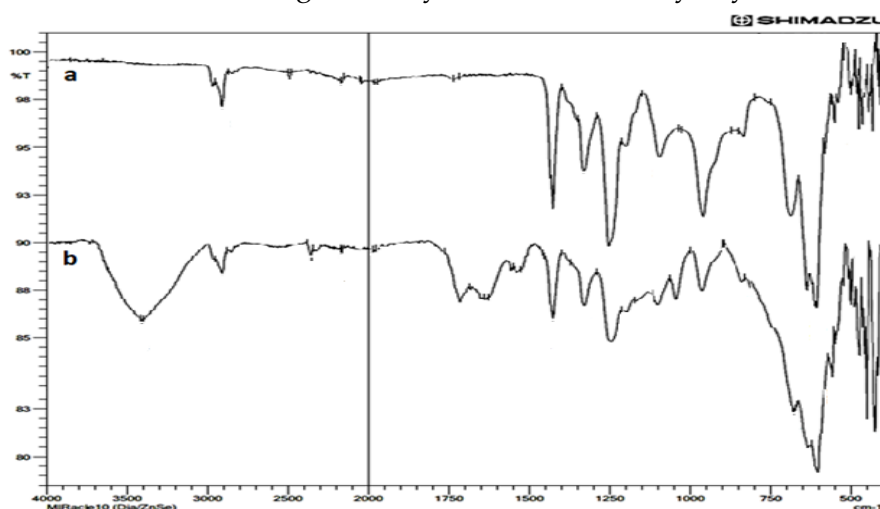


Fig 1. The functional groups in the polymer were studied by elemental analysis and IR spectroscopy of the cation exchanging composition obtained by oxidation of a polymer gained by modifying CaS_n to PVC.

3.1.1. IR spectroscopy of a cation exchange resin based on PVC

IR spectrum analysis was performed to determine the structure of the cation exchange resin oxidized with HNO_3 of the sulfurized polyvinyl chloride material modified with calcium polysulfide. 1. IR spectroscopy of PVC without polyvinyl chloride plasticizer (a) and PVC-based cation exchange resin (b).

A comparative analysis of the infrared spectroscopy data reveals the presence of sulfur groups in the cation exchange resin. In the IR spectrum shown in Figure 1-(b), distinct absorption peaks associated with the monovalent $-\text{SO}_3\text{H}$ bonds are observed at 1714 cm^{-1} and 1210 cm^{-1} , which are indicative of the functional groups present in the resin, as well as the valence oscillations of the $\text{S}=\text{O}$ bond in the $1220\text{--}1080\text{ cm}^{-1}$ area, around 3400 cm^{-1} characterizing the $-\text{OH}$ groups wide absorption areas have emerged.

This observation suggests that the sulfide, polysulfide, and hydrosulfide groups in the sulfurized polymer underwent oxidation, transforming into sulfur-containing functional groups. As shown in Figure 1 (a, b), the IR spectra exhibit distinct absorption bands at 700 cm^{-1} and 605 cm^{-1} , which are characteristic of $\text{C}-\text{Cl}$ bond stretching vibrations. This indicates that some of the chloride groups in the PVC chain are replaced by polysulfide, hydrosulfide groups. In particular, absorption peaks were detected in the regions of 2966 cm^{-1} , 2922 cm^{-1} and 2862 cm^{-1} , which are characteristic of the stretching vibrations of the $\text{C}-\text{C}$, $\text{C}-\text{H}$ and CH_2 groups in the PVC macromolecule. Additionally, deformation vibrations corresponding to the methylene groups were observed at 1420 cm^{-1} , which provides additional evidence of the structural properties of the polymer [7], [11]. This means that the polymer base chain is preserved during the stages of obtaining a sulpho-cation exchange material by modifying polyvinyl chloride.

3.1.2. Elemental analysis of the PVX-SC

The results of quantitative analysis of elements in the composition of cation exchange materials based on PVC, PVC-S, and PVC are presented in Table 1 below.

Table 1. The content of elements in the composition of PVC, PVC-S, and PVC-SC

Sample	PVX			PVX-S			PVX- SO_3H		
Element	C	H	S	C	H	S	C	H	S
Quantity(m,%)	36,4	5,2	0	29,2	4,2	14,3	22,4	4,1	10,4

It can be seen from the table above that the sulfur content of sulfur-modified PVC is 14.3%. This is because the chlorine atoms in the polymer chain may have been partially replaced by sulfur. The oxidation product of the sulfur-containing polymer to convert it to a sulpfocationite showed that it contained 10.4% sulfur, which may be related to the oxygen content of the sulpfogroups.

The presented physicochemical analyses confirm the formation of ion-exchange polymer materials with sulfate groups of sulfur-modified PVC using CaS_x and its oxidation with HNO_3 . Modern studies, in particular, using various isotherm models, were used to determine the mechanism of absorption of calcium II and magnesium II ions from artificial solutions by the new PVC-SC ion exchanger.

Adsorption Isotherms

3.2.1. Langmuir Isotherm Model

According to Langmuir's theory, adsorption occurs exclusively at specific active sites on the surface of the adsorbent. In this model, the binding energy of each adsorption site is constant,

and the adsorbate molecules form a monolayer without any interaction between the adsorbed molecules. The Langmuir equation can be expressed in the following forms (2,3):

$$Q_e = \frac{Q_{\max} \cdot (K_L \cdot C_e)}{1 + K_L \cdot C_e} \quad (2)$$

$$\frac{C_e}{Q_e} = \frac{1}{Q_{\max} \cdot K_L} + \frac{1}{Q_{\max}} \cdot C_e \quad (3)$$

Where: $Q_{\max}$ is the maximum amount of metal ions adsorbed on the ion exchanger (mg/g), K_L is the equilibrium constant in the Langmuir isotherm in equation (l/mg). To calculate the Langmuir constant, the linear equation of the Langmuir equation (3) above is used, that is, a graph is formed between C_e/Q_e and C_e and it is found from the tangent of the angle. The mechanism of interaction between the ion exchanger and metal ions in the solution can be understood by using the separation coefficient (R_L) to understand the Langmuir adsorption properties (4).

$$R_L = \frac{1}{1 + K_L C_0} \quad (4)$$

In this equation, when $0 < R_L < 1$, the sorption mechanism is considered favorable, while $R_L > 1$ indicates an unfavorable sorption mechanism, $R_L = 1$ indicates a linear sorption isotherm, and $R_L = 0$ indicates an irreversible adsorption [12].

Based on the results obtained in this study, a C_e/Q_e dependence graph of C_e was constructed in Figure 2 (a) to calculate the Langmuir isotherm constants. The Langmuir constants obtained using this graph is given in Table 2 below. These constants show that the maximum adsorption value ($Q_{\max}$) for Ca^{2+} and Mg^{2+} was determined to be 55.3 mg/g and 37.5 mg/g, respectively. The K_L values for Ca^{2+} and Mg^{2+} were 0.00162 and 0.00116, respectively, and the partition coefficients (R_L) were 0.134 and 0.264, respectively, indicating the favorable nature of the sorption mechanism for these metal ions on PVC-SC. The understanding that the sorption isotherms for the new PVC-SC resins for calcium II and magnesium II ions fit the Langmuir model is confirmed by the high R^2 values of 0.995 and 0.991, respectively.

3.2.2. Freundlich isotherm model

The Freundlich isotherm model used for the analysis of the adsorption process in heterogeneous systems is expressed as follows (5, 6). [13]:

$$Q_e = K_F \cdot C_e^{1/n} \quad (5)$$

$$\log Q_e = \log K_F + \frac{1}{n} \cdot \log C_e \quad (6)$$

In the equations 5 and 6, the values of the Freundlich constant K_F (mg g^{-1} (l g^{-1})ⁿ) and the sorption intensity $1/n$ ($1 < n < 10$ represent the sorption ease) are found from the intercept plotted against $\log Q_e$ and $\log C_e$. Based on the data in Figure 2 (b), the Freundlich parameters were calculated. For the sorption of calcium and magnesium ions with the Freundlich constants shown in Table 2 below, the K_F values are 8.15 and 1.14, respectively, and the sorption intensity (n) is 4.56 and 2.43, respectively, which means that the sorption process is favorable.

3.2.3. Temkin isotherm model.

This isotherm allows us to consider theories about the sorbent-sorbate interaction, binding energy. During the sorption process, the sorption of ions decreases linearly under the influence of temperature. To find the magnitudes of the Temkin isotherm, equation (7) is linearly fitted to equation (8) and plotted on a graph of $\ln C_e$ versus Q_e .

$$Q_e = \frac{RT}{b} \cdot \ln(K_T C_e) \quad (7)$$

$$Q_e = \frac{RT}{b_T} \cdot \ln K_T + \frac{RT}{b_T} \cdot \ln C_e \quad (8)$$

$$B = RT/b_T$$

$$Q_e = B \ln K_T + B \ln C_e$$

Here, B - Temkin constant J mol⁻¹, K_T – Temkin equilibrium constant l g⁻¹, R – 8,314 J mol⁻¹·K⁻¹, T-temperature K. The value of B is proportional to the adsorbate adsorption rate. K_T value represents the binding strength of adsorbate with sorbent [14]. In this work, the relationship between Q_e and lnC_e in Figure 2(b) is plotted to find the Temkin constants and calculated from the cross section of this curve. The K_T Temkin constants in Table 2 were found to be 0.0364 at sorption Ca²⁺ and 0.0137 L g⁻¹ (-73.2) at sorption Mg²⁺ and values R² (0.977 and 0.986), respectively. The sorption heat parameter (B) for Ca²⁺ and Mg²⁺ was found to be 9.7 and 8.6 J mol⁻¹, respectively.

3.2.4 Dubinin-Radushkevich (D-R) isotherm model

In heterogeneous systems, information about the adsorption energy can be obtained in accordance with the characteristic porosity of the adsorbent. The Dubinin-Radushkevich isotherm model is commonly used to represent the adsorption mechanism using a Gaussian energy distribution. The formula of the Dubinin-Radushkevich isotherm model is expressed as follows (9).

$$Q_e = Q_{D-R} \exp(-\beta \varepsilon^2) \quad (9)$$

In this equation: Q_{D-R} – Absorbed amount of adsorbate (ions) according to D-R model (mg g⁻¹), β – constant depending on adsorption energy (mol² (kJ²)⁻¹), ε – The Dubinin-Rudushkevich isotherm constant (Polanyi potential), ie the adsorption energy (kJ/mol⁻¹), is calculated as follows. (10).

$$\varepsilon = RT \ln(1 + \frac{1}{C_e}) \quad (10)$$

To calculate the Dubinin-Radushkevich isotherm constants, the equation is reduced to a linear form (11).

$$\ln Q_e = \ln Q_{D-R} - \beta \varepsilon^2 \quad (11)$$

The total free energy of adsorption E_{ads} (kJ mol⁻¹) is found by this equation (12).

$$E_{ads} = \frac{1}{\sqrt{2\beta}} \quad (12)$$

where E_{ads} explains the nature of adsorption. They state that E_{ads}<8 kJ/mol corresponds to physical adsorption, E_{ads} =8-16 kJ/mol and E_{ads} >16 kJ/mol represent the occurrence of ion exchange diffusion and ion exchange reactions, respectively [9]. In this research work, a graph dependence ε² of lnQ_e in Figure 2 (d) was formulated to calculate the D-R isotherm magnitudes for the adsorption of calcium II and magnesium II ions into PVC-SC resin. The values of the isotherm constants found using this graph are listed in Table 2. Based on the Dubinin-Radushkevich isotherm model, the maximum sorption capacity for Ca²⁺ ions of the cation exchange resin was calculated as Q_{D-R} =46.9 mg g⁻¹ and Q_{D-R} =26.3 26.3 mg g⁻¹ for Mg²⁺ ions, and the value of R² was respectively 0.927 and 0.885. The sorption free energy E_{ads} found for calcium and magnesium ions according to the D-R isotherm theory is probably 10.5 and 8.2 kJ/mol, respectively, and the adsorption of ions into PVC-SC was accompanied by an ion exchange reaction under the influence of chemical forces with a small value.

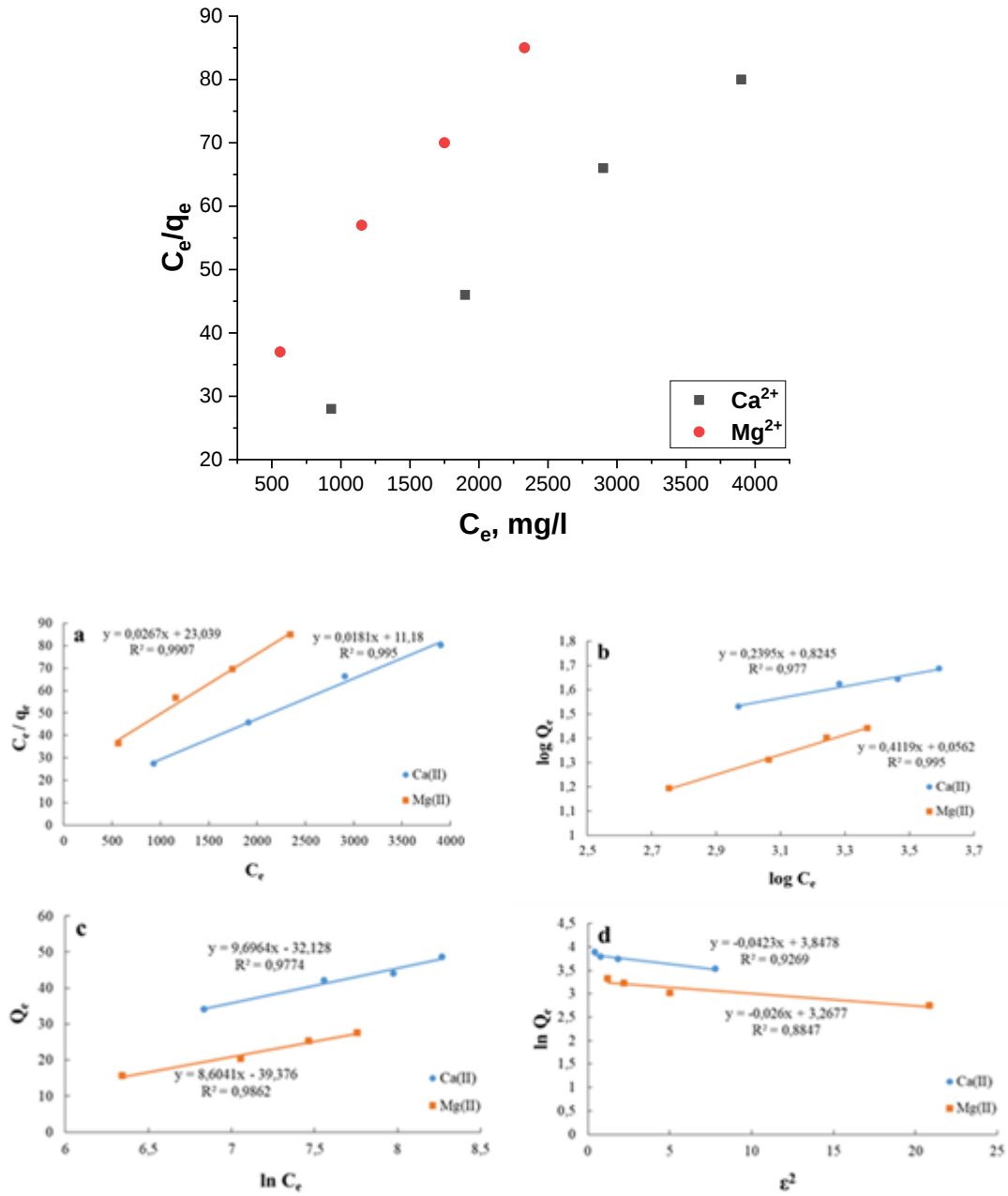


Fig 2. Expression of adsorption of Ca^{2+} and Mg^{2+} ions on PVC-based ion exchange resins in (a) Langmuir, (b) Freundlich, (c) Temkin and (d) Dubinin-Radushkevich isotherm models.

Table 2. Isothermal constants in the adsorption of metal ions on PVC-based cation exchange resins

adsorbate	Langmuir constants				Freundlich constants			Temkin constants			D-R constants		
	$Q_{\max}$	K_L	R_L	R^2	n	K_F	R^2	$-K_T$	B	R^2	Q_{D-R}	E_{ads}	R^2
Ca^{2+}	55.3	0.0016	0.134	0.995	4.56	8.15	0.977	27.7	9.7	0.977	46.9	10.1	0.927
Mg^{2+}	37.5	0.0011	0.264	0.991	2.43	1.14	0.995	73.2	8.6	0.986	26.3	8.1	0.885

4. Conclusion

- CaS_x solution was initially used to modify PVC with sulphur to produce an ion exchange resin containing sulphur groups. In the next step, a nitric acid solution was used to oxidize the hydrosulphides groups in the resulting macromolecule to sulphur groups. According to the results of elemental analysis, the cation exchange resin obtained contained 14.3% sulphur and the presence of sulphur groups was proven by IR spectroscopy.
- The sorption isotherms for Ca^{2+} and Mg^{2+} ions were successfully developed on the PVC-based cation exchange resin. As a result of studying Langmuir isotherm, Freundlich isotherm, Temkin isotherm and Dubinin-Radushkevich isotherm model, $R^2 > 0.88$ was achieved and it was shown that this sorption process is suitable for these models. According to Langmuir's monomolecular theory, the high sorption capacity Q_{max} is equal to 55.3 mg g^{-1} (Ca^{2+}) and 37.5 mg g^{-1} (Mg^{2+}) and the R_L worth (between of 0-1 at all concentrations of Ca^{2+} and Mg^{2+}) and the value n of Freundlich constant of 4.56 (Ca^{2+}) and 2.43 (Mg^{2+}) confirms that the sorption process is convenient. In the Temkin isotherm, the adsorption energy was determined to be $0.268 \text{ kJ}\cdot\text{mol}^{-1}$ and $0.303 \text{ kJ}\cdot\text{mol}^{-1}$ for Ca^{2+} and Mg^{2+} ions, respectively. The adsorption process, as modeled by the Dubinin-Radushkevich equation, suggests that the free energy (E_{ads}) for calcium and magnesium ions indicates their chemical sorption, with values of $10.5 \text{ kJ}\cdot\text{mol}^{-1}$ and $8.2 \text{ kJ}\cdot\text{mol}^{-1}$, respectively.
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