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Original Research

Cd^{+2} SORPTION FROM AQUEOUS SOLUTION USING ROSEMARY PLANT: PERFORMANCE AND ISOTHERM STUDY

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Abstract: The purpose of this study was to evaluate the efficacy of cadmium ion removal from an aqueous solution using a low-cost natural adsorbent. Rosemary plant was used in a batch system, and to understand this process more, several factors were investigated as follows: pH(1-10), contact time (0-180 min), initial metal ions concentration (10-250 mg/l), amount of adsorbent material (0.25-5 g), and agitation speed (100-4 rpm). All experiments were conducted at room temperature. Whereas, the results showed the best value of removal was 83% for cadmium onto rosemary plant, under optimum operating conditions: pH 5, the adsorbent material dosage 2 g/100 ml, contact time 60 min, the metal concentration 10 mg/L, and agitation speed 250 rpm. The Freundlich adsorption isotherm model fits the equilibrium adsorption data for Cd^{+2} better than the Langmuir model. This study found that the rosemary plant as a cost-effective and locally adsorbent for removing Cd^{+2} from polluted water is efficient. Thus, it is possible to use the rosemary plant as a low-cost material to be used to adsorb heavy metals from wastewater.

Keywords: *Rosemary plant; adsorption; cadmium; isotherms*

1. Introduction

Because of the increase in human population, the effects of global warming, and the impacts of industrialization, water supplies are becoming increasingly scarce. The main reason for severe problems in these areas is water pollution, which is created by the discharge of industrial pollutants into natural environments untreated or just partially treated [1].

The presence of hazardous contaminants in the environment over acceptable limits is a serious public health issue; this is because exposure to heavy metals including cadmium, lead, and mercury causing cholesterol levels in the blood to increase, which raises the risk of cardiovascular disease and stroke [2].

As a result, governments have imposed environmental regulations on wastewater quality, industry effluents should be treated to eliminate metals before being released [3].

Heavy industries including electroplating, battery manufacturers, metal polishing, and chemical industries are the main sources of heavy metals [4]. The World Health Organization (WHO) has identified a number of different heavy metals as being a significant risk to human health, and cadmium is one of those metals. The World Health Organization (WHO) has set a cadmium limit of 3.0 parts per billion in drinking water. [5].

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The removal of heavy metals from wastewater can be accomplished utilizing different treatment methods, such as membrane filtration, precipitation, ion exchange, adsorption, and co-precipitation/adsorption. Adsorption has been shown to be a more effectively method for metals ions removal from industrial effluents, and activated carbon was already commonly deployed as an adsorbent [6]. Activated carbon continues to be a costly material despite the widespread application it sees in businesses dealing with water and wastewater treatment. Natural adsorbents material can be used instead of conventional adsorbents have gained increasing interest in recent years. Adsorbents can be made from a variety of low-cost, readily available, and ecologically benign sources, such as agricultural waste or naturally occurring materials that are readily available in large numbers. There is a potential for inexpensive sorbents made from natural resources like rosemary plants or waste products from industrial or agricultural operations [7].

This study aims to investigate the efficiency of rosemary for wastewater contaminated with Cadmium as well as investigate isotherms and kinetics models of the adsorption process.

2. Materials and Methods

2.1. Adsorbate

Wastewater contains Cadmium sulfate hydrate ($3CdSO_4.8H_2O$) was employed in these experiments. Cadmium atoms have a molecular weight of 112.41 gram/mole, while the salt of 3CdSO4.8H2O utilized has a molecular weight of 769.51 g/mole. Dissolving 6.84 grams of $3CdSO_4.8H_2O$ in 1 liter of filtered water provided the required concentration of Cd^{+2} (1000 mg/l), which used later to prepare any wanted concentration of Cd^{+2}. The pH of the solution was modified by adding 0.1 M NAOH or 0.1 M HCL and was detected using a pH meter (pH Level Tester, china). The equation (1) was utilized to compute the mass of a heavy metal salt added to water under the assumption of complete dissolution:

$$W = V \times Ci \times \frac{M.wt}{At.Wt} \quad (1)$$

Whereas:

W: The weight of $3CdSO_4.8H_2O$ (mg).

V: The volume of solution (L) .

Ci: Ions concentration of cadmium (mg/L).

M.wt: Metal salt $3CdSO_4.8H_2O$'s molecular weight (gram/mole)

At.wt: Cd^{+2}ions' atomic weight (gram/mole).

2.2. Rosemary preparation

Rosemary plant was utilized as an adsorbent, the rosemary (leaves) were rinsed many times with pure water to remove all remaining impurities and dust then put in an oven for 10 hours to dry at 105 C˚. Then material was powdered and sieved by a 600 µm particle size sieve. Fourier transform infrared spectrophotometer (FTIR) analysis was tested for adsorbent material by [FT-IR spectrometer in environmental Service Center, University of Baghdad].

3. Experimental works

Cadmium's samples were obtained by dissolving a predetermined amount of cadmium sulfate hydrate $3CdSO_4.8H_2O$ in 1 liter of pure water and used as a stock solution then diluted to the appropriate concentration. A 100 ml sample of cadmium ions with intended concentration was added to each 250 ml flask with a wanted amount of rosemary and was mixed at 200 rpm in the shaker (Heidolph Animax 2010, Germany) at 25ºC for a specific contact time span, then a filter paper (qualitative filter paper) was utilized to filter the solution. An Atomic adsorption spectrophotometer AA-7000

(Shimadzu, Japan) was utilized to determine the residual concentration of Cd^{+2} ions. The following formula was used to determine the percentage of Cd^{+2} ions absorbed (R):

$$R\% = \frac{Co-Ce}{Co} \times 100 \quad (2)$$

Where:

C_o: Cd^{+2}ions initial concentrations (mg/L).

Ce: Cd^{+2}ions final concentrations (mg/L).

4. Results and discussion

4.1. Surface area analysis

The Brunauer-Emmett-Teller (BET) method was used to compute the specific surface area of the Rosemary. BET experiments have been conducted at the Center of Petroleum Research and Development/Catalysts Department (Ministry of oil/Iraq). According to BET analysis the surface area of Rosemary was equals 0.505 m^2/g.

4.2. FT-IR Results

An FT-IR study in the solid form was performed on the biomass grown in a KBr disk to identify which functional groups had been in command of metal uptake. Before and after the bio sorption procedure, adsorbent solid specimens were analyzed by FT-IR spectroscopy [8, 9 and 10]. As displayed Fig. 1, the spectra exhibited a number of peaks before and after adsorption process as follow the hydroxyl groups have increased from (3415 cm-1 to 3419.79 cm-1), carboxyl groups have changed from (2829 cm-1 to 2860.43 cm-1) , aromatic groups have transmitted from (815cm-1 to 827.46cm-1), alcohol groups from (1431 cm-1 to 1450.47 cm-1), and nitro aliphatic groups from (1319 cm-1 to 1321.24 cm-1)

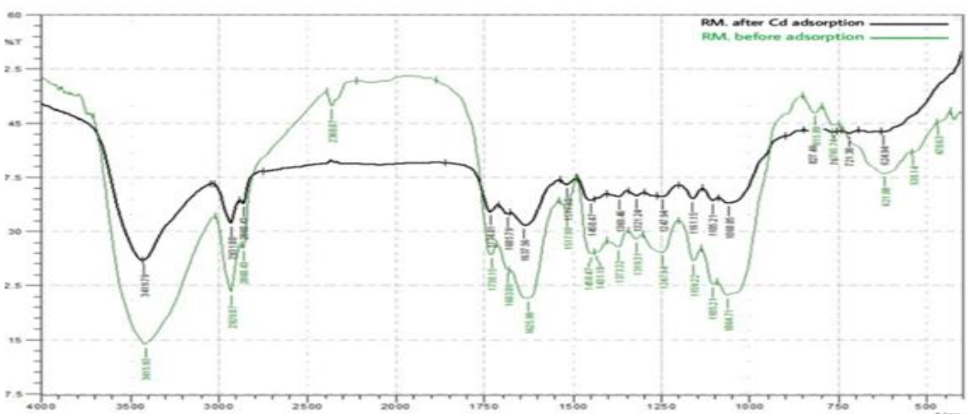

Figure.1. FT-IR spectra of the rosemary plant, before and after Cd^{+2}adsorption.

4.3 FESEM analyses

Using a Field Emission Scanning Electron Microscope (FESEM) (VEGA II SBH, Czech), we were able to observe and describe the micro structures on the surface of the adsorbents. Figure.2 shows that the adsorbent material's surface is rough and features irregularly sized cavities, both of which help in the adsorption of Cadmium ions. As cadmium ions agglomerate in these holes and on the surface of the adsorbent material, they form masses and smooth structures, as seen in Figure.3.

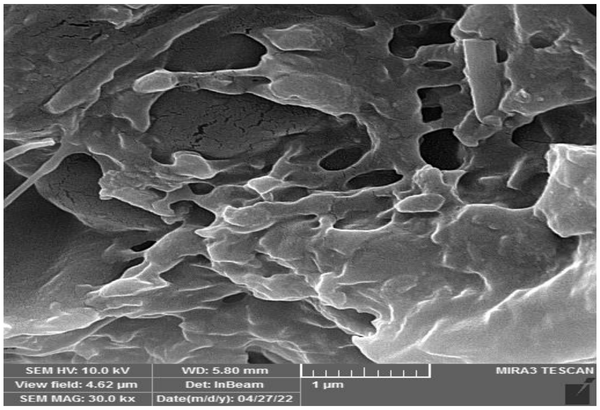

Figure 2. FESEM analyses for Rosemary before Cd^{+2}sorption

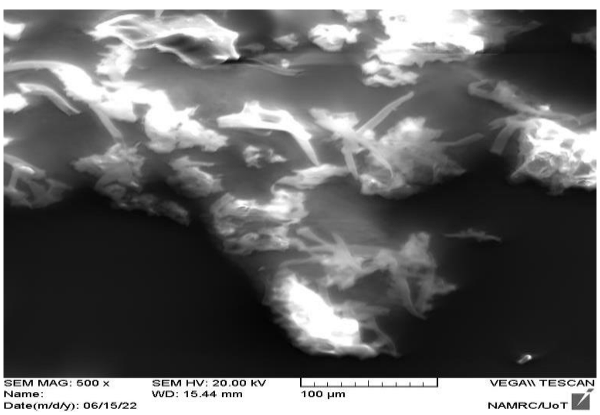

Figure 3. FESEM analyses for Rosemary after Cd^{+2}sorption

4.4. Effect of pH

During adsorption investigations; the pH values of the solutions were tested because pH is an important operational parameter. Batch system experiments were carried out using a solution with initial pH varying between 1 and 10, at a constant concentration of 40 mg/l, rosemary dosage 1 gm/100 ml; 2 hrs. contact time, and 200 rpm mixing speed. The initial pH value of the prepared samples was adjusted using the required volumes of 0.1 M NaOH and/or HCL. Fig.4. shows the removal efficiencies percentage at various values of pH. It could be observed from the figure that the efficiency of Cd^{+2} removal using rosemary is clearly affected by solution pH value, the adsorption of cadmium increases with increasing pH at the acidic range until it reaches the best value pH= 5 where the maximum removal efficiency equals to 73%. At low pH values, the lowest adsorption was found. When the pH was low (less than 4), there was an inordinate amount of protonation on the active surface sites of the adsorbent, and this excessive protonation frequently impeded the creation of bonds between positively charged metal ions and the active sites. An increase in the number of metal ions adsorbed is typically observed at intermediate pH values (between 4 and 6), when coupled H+ is transferred from the active sites. Because of the area covered by negatively charged surfaces grows as pH rises, the removal efficiency decreases at extremely high pH levels [11].

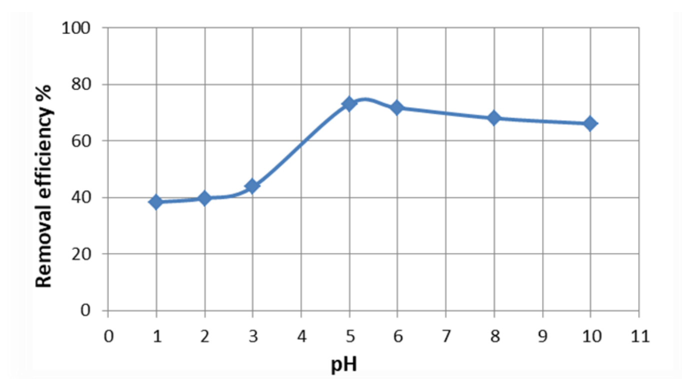

Figure.4 Impact of pH on Cd+2 removal efficiency

4.5. Effect of Adsorbent Dosage

The amount of adsorbent used has an impact the active site's availability for Cd^{+2} ion binding in the solution [12, 13]. In the current study, experiments of adsorption were carried out by adding different amounts of the adsorbent ranging from 0.25 to 5 g/100 ml to investigate the impact of sorbent dosage on the Cd^{+2} removal percent and detect the optimum dosage of adsorbent material at pH = 5, constant metal concentration = 40 mg/l, 2 hours contact time and 200 rpm agitation speed.

From fig.5, it is clear the relationship between the dose of the adsorbent material and the efficiency of the removal, It can be seen that adsorption increases with increasing adsorbent dose up to 2 g/100ml, with negligible adsorption for the remaining dosages. Due to increased surface area, ion exchange site capability, and active site number, the sorption capacity of the adsorbent increases with increasing dosage [14, 15, 16 and 17]. Rosemary is optimal at 2g/100ml, with maximum removal of Cd^{+2} of 82 percent. Uncomplete accumulation of adsorbed material leads in decrease Cd^{+2} uptake surface area and as well as decreasing Cd removal effectiveness. Subsequence experiments employed an adsorbent dosage of 2g/100ml.

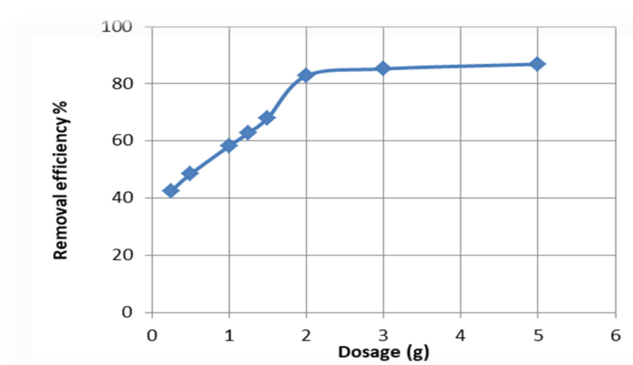

Figure.5 Impact of sorbent dosage on Cd+2 removal%.

4.6. Effect of contact time

The effect of contact time on rosemary's ability to absorb Cd^{+2} was investigated at pH=5, adsorbent dosage = 2g/100 ml, metal concentration = 40 mg/l, and agitation speed = 200 rpm and during a period from 5 to180 minutes so as to determine the optimal time and to investigate the kinetics of the adsorption process. As shown in Fig.6, there was quick adsorption of Cd^{+2} by rosemary at first 60 minutes up to 84% removal efficiency, and then a progressive decrease in the adsorption process to reach an equilibrium state after one hour. This could be because of the large numbers of active sites on the adsorbent's surface; however, increased contact time had no impact on removal efficiency, and the sorption rate slowed as a result of site saturation. [18, 16 and 13]. According to these results, the optimal value of the contact time is 60 minutes.

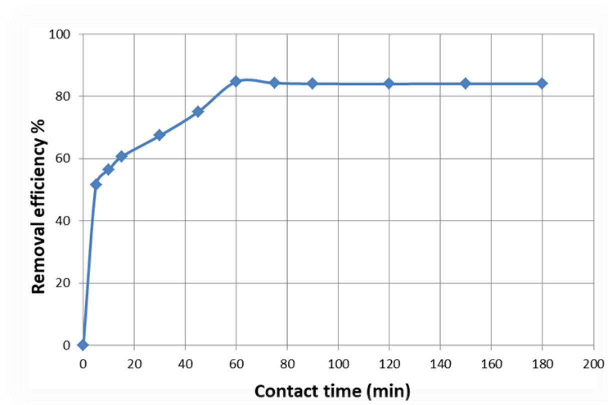

Figure.6 contact time impact on Cd+2 removal percent.

4.7. Effect of Cd^{+2} initial concentration

because water and wastewater pollution can be a result of different metal ions concentrations, in sorption experiments, determining the initial concentration of metal ions is important. Determining the effect is required for a detailed sorption analysis.

The influence of cadmium ions concentration present in aqueous solution on the removal efficiency for rosemary was studied with a 2 g/100 ml dose of adsorbent, pH value of 5, 200 rpm agitation speed, and 60 min contact time. Initial Cd^{+2} ions concentrations evaluated ranged from 10 to 250 mg/l and their impacts on the efficiency of removal were plotted as shown in figure 7 .

This figure shows that as the cadmium concentration increased, the percentage removal started going down gradually. At 10 mg/l, the best percentage removal was 91%, because there weren't enough active sites to absorb the higher concentration of cadmium. As cadmium accumulated in the water, the percentage removal dropped. [18, 12].

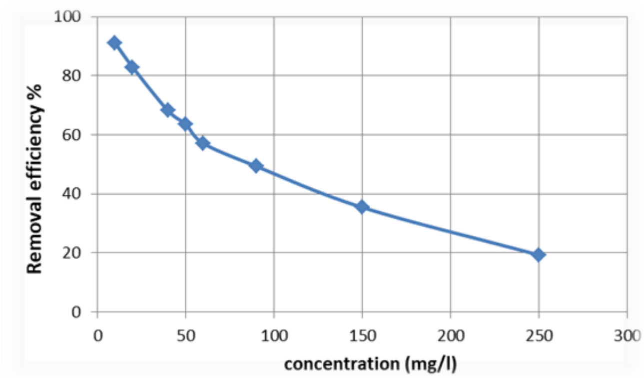

Figure.7 Cd^{+2} concentration effect on efficiency of removal.

4.8. Effect of agitation speed

To enhancing the interaction of Cd^{+2} ions in aqueous solution with the adsorbent material's binding sites, agitation speed maintains that Cd^{+2} ions are transported to the active sites [17]. The impact of agitation speed on the Cd^{+2}ion adsorption was studied utilizing the optimal removal efficiency of Cd^{+2} ions at pH 5.

Figure 8 shows how removal efficiency was affected by agitation speed at a range from 100 to 400 rpm, and it can be seen that as the agitation speed was increased from 100 to 250 rpm, the removal efficiency of metal ions increases, but further increases have few effects (i.e., above 250 rpm). The higher efficiency is due to more turbulence, which makes the film around the adsorbent particles less resistant to mass transfer. [19]. Furthermore, these results show that agitation at a speed of 250 rpm is sufficient to achieve optimum removal about 82%.

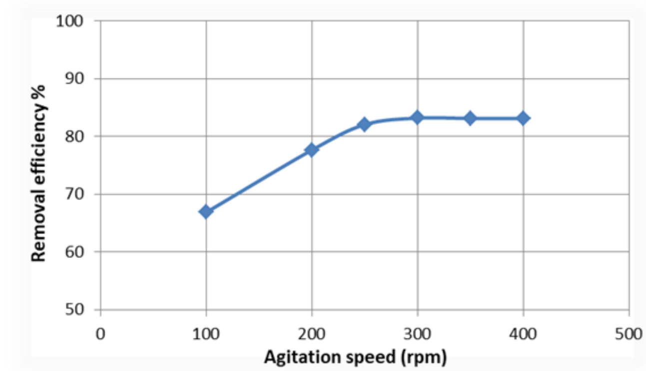

Figure.8 Effect of agitation speed on Cd^{+2} removal efficiency.

5. Adsorption Isotherm

generally, mathematical models that are termed as adsorption isotherm models have been used in describing the properties of adsorption [20]. The adsorption isotherm illustrates how adsorbate molecules distributed when the liquid and solid phases at equilibrium. [12].

In this research, using the Langmuir and Freundlich isotherm models, researcher examined the adsorption characteristics of the rosemary plant for the removing Cd^{+2}ions from an aqueous solution. The Langmuir isotherm model describes monolayer adsorption by assuming that adsorbent materials own a limited capacity, which is defined as the point at which no farther adsorption happens. The fundamental assumption of this model is that adsorption happens at particular homogenous sites inside the adsorbent. The Freundlich isotherm model can also be used to characterize the adsorbent material's adsorption behavior. Adsorption on a heterogeneous surface with interactions between adsorbate molecules is a significant use of this concept. The equation of Langmuir is as follows [21]:

$$qe = \frac{qmax\, K_L\, C_e}{1+K_L\, C_e} \qquad (3)$$

To simplify the equation, it can be written as follows:

$$\frac{C_e}{qe} = \frac{1}{qm\, k_L} + \frac{C_e}{qm} \qquad (4)$$

Whereas :

Ce: the concentration of Cd^{+2} at the point of equilibrium (mg/L).

qe: is the quantity of Cd^{+2} (mg) that is absorbed by one gram of the adsorbent when the conditions are in equilibrium (mg/g).

qmax: is the greatest theoretical adsorption capacity (mg/g).

K_L: the constant of Langmuir isotherm (L/mg).

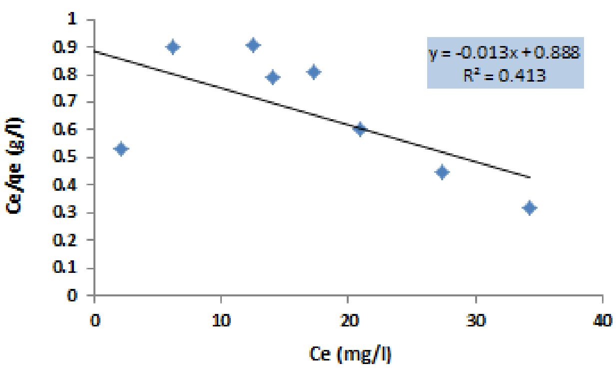

Figure.9 Langmuir adsorption models for Cd^{+2}on a rosemary plant.

The slope and intercept of a linear graph of 1/qe against 1/Ce is utilized to determine the values of qmax and KL, respectively.
Figure.9 shows how the Langmuir isotherm for Cd^{+2}adsorption appears as a line. Table.1 shows that the maximum adsorption capacity, qm is 76.923 mg/g, and the KL calculated value is 0.0146 L/mg, illustrating that the particle size of rosemary was small toward adsorption, while the Freundlich equation is utilized to study the adsorption of heterogeneous surfaces. The equation below shows how to describe the Freundlich model [22].

$$q_e = K_f C_e^{\frac{1}{n}} \qquad (5)$$

To simplify the equation, it can be written as follows:

$$\ln q_e = \ln K_f + \frac{1}{n} lnC_e \qquad (6)$$

the Freundlich adsorption constant Kf is associated with the capacity of adsorption, qe is the amount of Cd^{+2} (mg) adsorbed per gram of the adsorbent at equilibrium (mg/g), Ce: is the equilibrium concentration of Cd^{+2} (mg/L), and n is the adsorption process intensity factor. The 1/n value varies from 0 to 1, and when the ratio approaches 1, it indicates good adsorption.

The values of Kf and 1/ n can be calculated from the intercept and slope, respectively, of a linear plot of natural logarithm of qe against natural logarithm of Ce.as shown in figure.10, where Kf and n are equal to 1.0942 and 0.8718, respectively.

From Table.1 which shows the parameters of Freundlich isotherm, as can be seen that R^2 is equal to 0.8956. This means that the results are fitted with Freundlich model more than Langmuir model. As a result of that, Cd^{+2}adsorption on the rosemary plant occur on heterogeneous layer and the adsorption process is chemisorption [23].

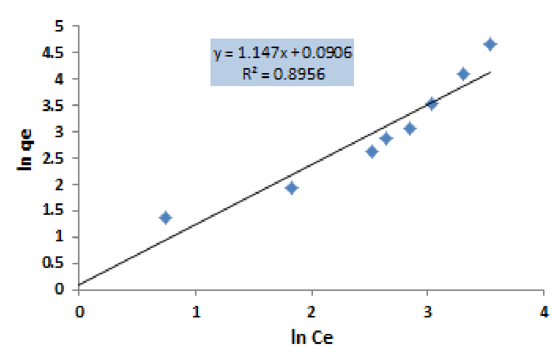

Figure.10 Freundlich adsorption models for Cd^{+2}on a rosemary plant.

Table.1 Isotherms parameters for removal of Cd^{+2} using rosemary plant.

models	Parameters		
Langmuir model	qm(mg/g)	KL(L/mg)	R^2
	76.923	0.0146	0.4139
Frendlich model	n	Kf	R^2
	0.8718	1.0942	0.8956

6. Conclusion

Cadmium ions can be sorption onto the rosemary plant in a significant amount of capacity. The parameters of pH, adsorbent dosage, contact time, adsorbate concentration, and agitation speed were some of the most important ones to take into consideration for the purposes of this investigation. The best removal percentage was equal to 82% when the pH was set to 5, when the contact time was set to 60 minutes, when the adsorbent dosage was set to 2 g/100 ml, and when the initial Cd+2 concentration was set to 10 mg/L. Additionally, according to the Langmuir model, the maximum amount of adsorption that could be achieved was 76.923 mg/g. In comparison, the R2 value for the Freundlich model was 0.8956, while the value for the Langmuir model was 0.4139. This substance has the potential to be utilized in a manner that is efficient to remove heavy metals from water that is extremely polluted. This is a potential eco-friendly substance.

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Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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