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ABSTRACT

In the present study, Batch adsorption experiments of hazardous cadmium, Cd(II), ions onto low-cost Dodonaeae Viscose Legs (DVL) and processes were conducted with respect to contact time, solution pH, adsorbent dosage, initial Cd concentration and temperature. The DVLs were used without chemical or physical activation processes. Sorption isotherm, kinetic and thermodynamic models were used to describe the equilibrium stage and their constants were determined. The results showed that the adsorption capacity of DVLs for Cd(II) ions was found to be 25.29 mg/g at solution pH 5.26, 50 min and 30°C. The mean free energy was found to be 1.82E-02 using Dubinin-Radushkevich (DRK) isotherm, which indicates that the adsorption of Cd(II) ions onto DVL surface is physical adsorption. The activation energy (Ea) was 3.06 kJ/mole, which confirms that Cd(II)-DVL adsorption process is physical sorption. Pseudo-first-order, pseudo-second-order, Elovich, intra-particle and extra-particle diffusion models were used to describe the adsorption kinetics. The results showed strong correspondence to a pseudo-second-order kinetics. Thermodynamic parameters suggested that the adsorption of Cd(II) ions onto DVL surface is an exothermic process.

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INTRODUCTION

Over the past few decades, one of the most important problem facing bio systems is environmental pollution with non-degradable, non-destroyable and toxic heavy metals due to their bioaccumulation. Massive amount of industrial waste materials containing lead, copper, cadmium and chromium, etc has reached ground waters causing a serious problem to humanity at high concentrations. Cadmium (Cd) is reported to be extremely toxic heavy metal and can accumulate in human body especially in kidneys leading to disorder of the kidneys and osteomalacia. However, Cd has been included in the red and black list of priority of pollutants by Department of Environment, UK and Dangerous Substance Directive in European Economic Community, while it is classified as group B1 carcinogen by Environment Protection Agency, (EPA). Cadmium is reported to affect calcium metabolism, develop and skeletal calcification. Also, the permissible limit for Cd in drinking water by World Health Organization, WHO, is 0.005 mg/L [1].

Over the years, many technologies have been used and developed to remove heavy metals from industrial wastewaters including coagulation/flocculation process, member filtration, oxidation process, activated carbon adsorption, reverse osmosis ion exchange, solvent extraction, etc [2, 3]. However, these technologies need further costly processes and require skilled personal, which led to ineffective or expensive process due to heavy metal concentration in contaminated waters ranged between 1 to 100 mg/L. One of the most economically favorable and technically easy for water treatment processes is adsorption using non-conventional and low cost agricultural by-products. Numerous studies have been reported on effective to use by-products plants in order to remove heavy metals from aqueous solution with chemical or physical surface activation. In this research, the adsorption processes of Cd(II) ions onto inactivated or unmodified Dodonaeae Viscose leg (DVL) surface from aqueous solutions. Isotherm, kinetic and thermodynamic models were investigated.

MATERIAL AND METHOD

Dodonaeae Viscose leg charcoal

Dodonaeae Viscose, Sapindaceae legs (DVLs) were collected from Faculty of Science garden at University of Sebha, Sebha, Libya and used without any further treatment such as physical or chemical processes. They were cut into small pieces, washed for 1 h to remove impurities, air dried at 105°C for 24 h, then grounded, sieved through a 75µm mesh and stored in desiccators for further use.
Preparation of solutions
All reagents used in this study were analytical grades purchased and used without further purification. Stock solution of Cd(II) ions with concentration of 1000 mg/L was prepared by dissolving cadmium acetate (Cd(CH₃COO)₂.2H₂O) in deionized water. Several dilutions were made in order to get the desired Cd(II) concentrations for each sorption experiment.

Batch adsorption procedure
The adsorption experiments were performed on a mechanical shaker equipped with thermostatic equipment to set the temperature. In order to investigate the effect of adsorbent dose, 10, 30, 50, 70, 90 mg of DVL was added to 100 ml of 200 mg/L Cd(II) ions and kept for 120 min at 30°C. In order to investigate the effect of pH values, 10 mg of DVL powder was added to 100 ml of 200 mg/L Cd(II) ions and pH values were adjusted to 2.0, 3.0, 4.0, 5.0, 6.0, and 7.0, and kept for 120 min at 30°C. In order to investigate the effect of time contact, 100 mg of DVL powder was added to 100 ml of 200 mg/L Cd(II) ions and then agitated for 120 min at 30, 40 and 50°C. In order to investigate the effect of Cd(II) initial concentrations, 100 mg of DVL powder was added to 100 ml of 100, 200, 300, 400, 500 mg/L Cd(II) ions and attired for 120 min at 30, 40 and 50°C. All solutions were stirred at 300 rpm, filtered through wathman filter paper 112v. Cd(II) concentration before and after adsorption process was determined by titration method using 0.0005M EDTA, zinc buffer solution. All the experiments were repeated for triplicate values.

Calculations
The percent removal, and adsorption capacity, mg Cd(II)/g DVL was determined using the following equations:

\[ \text{Removal \%} = \left( \frac{C_w - C_e}{C_w} \right) \times 100 \]  
\[ q_e = \left( \frac{C_w - C_e}{w} \right) v \]  
\[ q_t = \left( \frac{C_w - C_t}{w} \right) v \]

Where: \( C_0 \) and \( C_e \) =Cd(II) initial, at time t (min) and equilibrium (\( q_e \)) of Cd(II) onto DVL surface; \( v \) =total volume; \( w \) =weight of DVLs.

The best-fit for kinetic models used in this study was analyzed by the square sum of errors (SSE) values using the following equation:

\[ SSE = \sum \frac{(q_{\text{exp}} - q_{\text{cal}})^2}{q_{\text{equi}}} \]

The lowest value of SSE is the best model for the particular system.

RESULTS AND DISCUSSION
Characterization of DVL surface
In order to identify the characteristic functional groups on DVL surface, FT-IR technique was used in the range 400-4000 cm⁻¹. The FT-IR spectrum of DVL before and after adsorption of Cd(II) ions is shown in Figure 1.

Effect of pH
Figure 3 shows the effect of pH on the adsorption of Cd(II)-DVL in the range of 2.21-6.28 for average initial Cd(II) concentration of 255.6 mg/L, 100 mg of DVL, and stirring speed of 300 rpm at 303K. The results indicate that Cd(II) ions...
adsorption onto the surface of DVL was strongly pH-dependent and increased with an increase of pH until the pH reached 5.4, which called the sorption edge [4]. The highest removal efficiency in the Cd(II) adsorption onto the surface of DVL was obtained at pH 5.4, which recorded as the optimum pH value [4]. Therefore, the further adsorption experiments were set at pH 5.4 in order to ensure reach the optimum Cd(II)-DVL adsorption.

**Effect of adsorbent dose**
Figure 4 shows the effect of adsorbent dose on adsorption Cd(II) ions onto the surface of DVL surface with no change in other parameters such as pH of 5.4, Cd concentration of 200 mg/L, time of 60 min and temperature of 303K. It was noted that the percentage of removal was increased with an increase of DVL dosage, which could be due to either the more adsorption sites or an increase in the total surface area of DVL [1, 5]. Therefore, 100 mg of DVL were used for the further adsorption experiments in order to ensure reach the optimum Cd(II) removal.

**Initial concentration effect**
In order to investigate the optimum concentration of Cd(II), the adsorption batch of Cd(II) ions onto DVL surface was studied under varied Cd(II) concentrations ranging between 100 to 500 mg/L at pH 5.4, time of 60 min, temperature of 303, 313 and 323K. From Figure 5, the adsorption capacities were increased with increasing the Cd(II) concentration and no saturation stage was observed. This indicates that more active sites on the surface of DVL was available to remove Cd(II) ions from solution. Also, the adsorption capacities were increased with increasing the solution temperature probably due to increase the number of active sites for Cd(II) ions on the surface of DVL with increasing temperature [6].

**Adsorption isotherm models**
Different adsorption isothermal models were used to analyze the sorption equilibrium of Cd(II)-DVL in terms of Freundlich, Langmuir, Temkin, DR isotherms. **Freundlich isotherm**
The Freundlich isotherm model is given by in nonlinear and linear Equations (4) and (5), respectively:

$$
q_e = k_F C_e^{1/n}
$$

$$
\log q_e = \log k_F + \frac{1}{n} \log C_e
$$

Where: $K_F$ =adsorption capacity, $(mg/g)(L/g)^{1/n}$, and $n$= biosorption intensity are Freundlich constants, which can be determined by plotting the $q_e$ versus $C_e$. The Freundlich constants are also listed in **TABLE 1**. However, the $1/n$ values

![Figure 2](image2.png) **Figure 2.** Effect of contact time on the adsorption of Cd(II) ions onto DVL surface at different temperatures

![Figure 3](image3.png) **Figure 3.** Effect of pH on the adsorption of Cd(II) ions onto DVL

![Figure 4](image4.png) **Figure 4.** Effect of adsorbent dose on the adsorption of Cd(II) ions onto DVL surface

![Figure 5](image5.png) **Figure 5.** Effect of Cd concentration on the adsorption of Cd(II)-DVL
Langmuir isotherm

The Langmuir isotherm is commonly used to describe quantitatively the formation of a monolayer adsorption at specific homogenous sites on outer surface of DVLs with no further adsorption takes place. The Langmuir equations in linear are expressed as follows:

\[ \frac{1}{q_e} = \frac{1}{K_Lq_mC_f} + \frac{1}{q_m} \]  

(7)

Where: \( q_m \)= monolayer sorption capacity (mg/g) and \( K_L \)=Langmuir equilibrium constant (L/mg). A straight line can be obtained by plotting \( 1/q_e \) versus \( 1/C_f \) with its intercept/slop of \( K_L \) and \( 1/intercept \) of \( q_m \). Figures are not shown here and the Langmuir constants are listed in Table 1. The \( q_m \) values are decreased with increasing temperature solution form 303 to 313 K indicating that the Cd(II) ions is not favorable adsorbed by DVL surface at low temperature and this is confirmed that the adsorption process is exothermic system. Also, \( K_L \) values are decreased with increasing temperature from 303 to 313 K, which indicating less adsorption of Cd(II) onto DVL surface with increasing temperature and the bond strength between Cd(II) and DVL decreased with increasing temperature. Also, this indicates that the adsorption process is exothermic

On the other hand, the constant separation factor, \( R_L \), is used to indicate the shape of isotherm by the Langmuir model:

\[ R_L = \frac{1}{1 + bC_f} \]  

(8)

The value of \( R_L \) can be unfavorable (\( R_L > 1 \)), favorable (0<\( R_L <1 \)) and irreversible (\( R_L = 1 \)). It was found that the \( R_L \) values are in the range of 0-1, which confirmed that the Cd(II)-DVL adsorption is more favorable under these conditions using Langmuir isotherm and at higher temperature. Also, \( R_L \) values are greater than 0 but less than 1, confirming that Langmuir isotherm is favorable which prove by \( R^2 \) values. Moreover, the Cd(II)-DVL adsorption data was fitted well with Langmuir than Freundlich isotherm due to Langmuir data has higher \( R^2 \) values. The results from Freundlich and Langmuir data imply that the desorption of Cd(II) onto DVL surface shows a complex mechanism involving a multilayer surface condition (Figure 6).

Surface Coverage

The behavior of adsorption can be also understood by using surface coverage related with Langmuir equation type:

\[ K_L C_f = \frac{\theta}{(1-\theta)} \]  

(9)

where: \( K_L \) is Langmuir constant, \( C_f \) is the initial Cd concentrations and \( \theta \) is the surface coverage. Figure 7 shows the relationship between \( \theta \) versus initial Cd concentrations

Temkin isotherm

Temkin isotherm is usually used for adsorbent-adsorbate interactions and to study the heterogeneous surface energy systems (non-uniform distribution of sorption heat assuming the heat of the adsorption in layer decreases linearly with coverage. Temkin constants can be obtained from the liner plot (\( q_e \) versus ln \( C_f \)) [2, 5, 7].

\[ q_e = \frac{RT}{b} \ln A + \frac{RT}{b} \ln C_f \]  

(10)

Where: \( \frac{RT}{b} = B \) constant related to heat of sorption (\( \Delta H_{ad} \), J/mol); \( A \), slope = Temkin isotherm equilibrium binding constant (L/g); \( b \), intercept = Temkin isotherm constant related to heat sorption as adsorption intensity (J/mol); \( R \) = universal gas constant (8.314 J/mol/K); \( T \) = Temperature at 303, 313 and 323 K. Temkin constants are listed in Table 1 and figures are not shown in here. However, the heat of Cd(II) adsorption, \( b \), is

![Figure 6. Plot of separation factor versus initial Cd(II) concentration](image)

![Figure 7. Relationship between surface coverage and initial Cd concentrations](image)

### Table 1. Isotherm constants

<table>
<thead>
<tr>
<th>( T )</th>
<th>Freundlich Constants</th>
<th>Langmuir Constants</th>
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<td></td>
<td>( K_F ) ( L/mg )</td>
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<tr>
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Table of Isotherm constants

<table>
<thead>
<tr>
<th>( T )</th>
<th>( K ) ( L/mg )</th>
<th>( q_m ) ( mg/g )</th>
<th>( B )</th>
<th>( R^2 )</th>
<th>( D ) ( J/mol )</th>
<th>( E )</th>
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directly related to Cd(II) coverage onto the surface of DVL. It was found that the adsorption heat was decreased with increasing the system temperature from 14,2E-02, 5,53E-02 and 3,69E-02 J/mole for 303, 313 and 323K, respectively, as listed in TABLE 1 which indicates that the heat of adsorption is exothermic. However, the values of b are lower than 8 J/mol which indicating the adsorption process is a physical adsorption process. R² is found to be the poor fit of all experimental data and the fitting is increased with increasing the solution temperature.

**Dubinin-Radushkevich (D-R) isotherm**

The apparent energy of Cd(II) adsorption onto DVL surface can be determined using D-R isotherm from the following the linear equation:

\[
\ln q_e = \ln q_m - \beta e^2
\]  
(11)

Where: \(\beta\) is the activity coefficient constant related to sorption energy; \(e\) is Polanyi potential. The D-R constants are determined from the slope and intercept of the plot (ln \(q_e\) versus \(e^2\)) to give \(\beta\) (mol²/J²) and \(q_m\) (mg/g) as listed in TABLE 1. The mean free energy (E) of sorption per molecule of the sorbate when it is transferred to the surface of the solid from infinity in the solution can be calculated by the following equation:

\[
E = \frac{1}{\sqrt{2\beta}}
\]  
(12)

The E values were <8 at all temperature studied that reveals the sorption process follows physical adsorption as shown in TABLE 1.

**Adsorption dynamic study**

To study the speed and reasonable length of adsorption time, various kinetic models were used in this work for the experimental adsorption data of Cd(II) ions onto DVL surface. Lagergren pseudo-first order and pseudo-second order were conducted to describe experimental data using the following equations:

\[
\ln(q_e - q_t) = \ln q_e - K_1t
\]  
(13)

\[
t = \frac{1}{K_2} + \frac{t}{q_2}
\]  
(14)

Where: \(q_e\) is the adsorption equilibrium capacity (mg/g), \(q_t\) is the amount of Cd(II) adsorbed (mg/g) at time \(t\), \(K_1\) is the rate constant of pseudo-first-order (1/min), \(q_2\) is the maximum adsorption capacity (mg/g), \(k_2\) is the rate constant of pseudo-second-order (g/mg.min), which were obtained from slope and intercept of pseudo-first-order and pseudo-second-order plots between \(\log(q_e - q_t)\) versus \(t\) and \((t/q_t)\) versus \(t\), respectively [8]. All kinetic parameters for two models are shown in Figure 8 and presented in Error! Reference source not found.. As can be seen, the calculated \(q_e\) values were decreased with increasing the solution temperature, therefore, the adsorption process is confirmed to be exothermic system. From the values of R², the experimental data were followed pseudo-second-order kinetics comparatively better than pseudo-first-order. Also, the pseudo-second-order kinetic has lower SSE value (5.46) than pseudo-first-order kinetic (102.2). Therefore, pseudo-second-order is the best model to describe the Cd(II)-DVL adsorption process.

The kinetic data were studied using Elovich, intra-particle and extra-particle diffusion models. However, Elovich equation is generally expressed as follows:

\[
q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln(t)
\]  
(13)

Where: a plot of \(q_t\) versus \(ln(t)\) gives linear relationship with a slope of \((1/\beta)\) and intercept of \((1/\beta)ln(\alpha\beta)\). The low values of \(R^2\) were noted, which ascertained the unsuitability of Elovich adsorption model for the adsorption process of Cd(II)-DVL (Figure 8).

For porous adsorption process, the pore (intra-particle) diffusion model can be obtained by Morris-Weber equation:

\[
q_t = k_{ip}t^{0.5} + C
\]  
(14)

\[
\log q_t = \log k_{ip} + a \log t
\]  
(15)

Where: \(k_{ip}\) is the rate of intra-particle diffusion (mg/g.min¹), which gives the idea about the thickness of boundary layer by plotting \(q_t\) versus \(t^{0.5}\) (see Figure 9). The plot was linear over the range of adsorption but not pass the origin. Also, the data exhibited multi-linear plots, which indicated that two steps influence the Cd(II) adsorption process onto surface of DVL. From Error! Reference source not found., the \(k_{ip}\) was increased at high temperature, which indicated that the greater boundary layer effect. Therefore, the small value of intraparticle diffusion rate constant has less significant effect about the diffusion mechanism of Cd(II) ions uptake on the surface of DVL. Therefore, intraparticle diffusion process was not only the rate-controlling step.

The Boyd kinetics model was used to study film particle diffusion and expressed as follows:

\[
Bt = -0.4977 - \ln(1 - \frac{q_t}{q_e})
\]  
(16)

Where: \(q_t/q_e\) is the fraction of solute adsorbed at any time \(t\). The plot of B, versus \(t\) (min) was used to distinguish the kinetic data between particle and film diffusion. However, none of the intraparticle and film diffusion linear plots pass through zero suggested that these two diffusion models are not rate determining steps.
### TABLE 2. Efluent quality and removal percentage of jar test experiment

<table>
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**T** intra-particle diffusion

**Elovich**

### Adsorption thermodynamics

The change in standard free energy ($\Delta G^\circ$), enthalpy ($\Delta H^\circ$) and entropy ($\Delta S^\circ$) of Cd(II)-DVL adsorption were calculated using the thermodynamic equilibrium constant ($K_c$) value from the following equation:

$$K_c = C_{Ae} / C_{Se}$$  \hspace{1cm} (17)

Where: $C_{Ae}$ (mg/L) is the equilibrium Cd(II) concentration adsorbed on the surface of DVL and $C_{Se}$ (mg/L) is the equilibrium Cd(II) concentration in solution after 60 min stirring [2,9]. Then, the following thermodynamic equation is used to calculate the change in standard free energy ($\Delta G^\circ$), enthalpy ($\Delta H^\circ$) and entropy ($\Delta S^\circ$):

$$\Delta G^\circ = -RT \ln K_c$$  \hspace{1cm} (18)

Where: R= universal gas constant (8.314 J/mol/K and T=absolute temperature (K). Van’t Hoff plot was estimated thermodynamic parameters from slope and intercept of ln $K_c$ versus 1/T plot by following equation:

$$\ln K_c = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R}$$  \hspace{1cm} (19)

All calculated thermodynamic parameters are listed in TABLE 2. The values of $\Delta G^\circ$ were negative at three temperature studied, which indicate the spontaneous. The activation energy of Cd(II) ions onto DVL surface was also studied and calculated from Arrhenius equation:

$$\ln k_2 = \ln k_0 - \frac{E_a}{RT}$$  \hspace{1cm} (20)

Where $k_2$ is the rate constant of pseudo second order type II, $k_0$ is the factor of temperature and $E_a$ is the activation energy. However, the magnitude of $E_a$ was found to be 41.3 kJ/mol, which confirmed that the adsorption process was physical adsorption.

### CONCLUSIONS

The findings in this research revealed that DVL powder, without surface modifications by physical or chemical activator, can be used as green adsorbent for Cd(II) ions from aqueous solutions at low temperature. The equilibrium data was described by Langmuir isotherm. The kinetic of the adsorption process was followed pseudo-second-order model. The biosorption mechanism of Cd(II)-DVL is physico-sorption process. The negative values of $\Delta H^\circ$ and $\Delta S^\circ$ showed that the Cd(II) adsorption process onto ACROL is exothermic in nature.
TABLE 2. Thermodynamic parameters for sorption of Cd(II) on DVL

<table>
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<tr>
<th>Initial [Cd(II)] (mg/L)</th>
<th>$\Delta G^\circ$ (kJ/mol)</th>
<th>$\Delta H^\circ$ (kJ/mol)</th>
<th>$\Delta S^\circ$ (kJ/mol)</th>
<th>$E_a$ (kJ/mol)</th>
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REFERENCES


Persian Abstract

چیکده در مطالعه حاضر، آزمایش های جذب دسته آی از کادمیوم نشریات، و فرآیندها با توجه به Dodonaeae Legs ویسکوز (DVL) در مواد فیزیکی (DVL) بر روی کم هزینه کادمیوم (II) و به مساحت ضروری این مواد در محلول pH زمان نمایش جاذبی، مقدار اولیه، مقدار اولیه قد و دما انگیز مدل DVL جذب دو مدل دو DVL به فرمول جذب دو DVL (Sorption) مدلی گرم به مدل در محلول 50 دقیقه و دو 30 شیء گرادیا یافتن شد. متوسط آزمایش اثر (Ea) برای دو DVL جذب فیزیکی دارد. از نظر دو DVL جذب فیزیکی (Cd (II) بر سطح دو DVL جذب فیزیکی 25.29 (DRK) به دست آمد که دانه دو DVL جذب فیزیکی (Cd (II) بر سطح دو DVL جذب فیزیکی 25.29 (DRK) به دست آمد که دانه دو DVL جذب فیزیکی 25.29 (DRK) به دست آمد که دانه دو DVL جذب فیزیکی 25.29 (DRK) به دست آمد که دانه DVL جذب فیزیکی (Cd (II) بر روی سطح DVL یک فرآیند اکسترمیمیک است. DVL جذب فیزیکی (Cd (II) بر روی سطح DVL یک فرآیند اکسترمیمیک است. DVL جذب فیزیکی (Cd (II) بر روی سطح DVL یک فرآیند اکسترمیمیک است.