

Study of the kinetics and isotherm adsorption of Pb(II) and Cd(II) ions from aqueous solution by *Eucalyptus Gomphocephala* roots

Ragwan Mohammed Ghait*, Saada Salem Abdulhafeed

Faculty of Science, Sirte University, Sirte, Libya

E-mail:ragwan3000@su.edu.ly

*Corresponding author: ragwan3000@sci.misuratau.edu.ly

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Abstract

This study is concerned about the adsorption of lead (II) and cadmium (II) ions from their aqueous solutions using *Eucalyptus* roots as adsorbent material, High percentage of ion removal equal to 93.5% and 96.6%, Pb(II) and Cd(II) with the adsorption capacity of 45.9mg/g and 47.6mg/g for both metal ions (respectively) at pH of 5-7, was reported. The system reached the equilibrium state approximately after 30min, and the adsorption percentage of these metals increased with increasing the concentration and the amount of adsorbent until reaching the equilibrium state. The adsorption process followed pseudo-second-order model and corresponded to the Freundlich isotherm correlation coefficient, which means that the adsorption is multi-layered and heterogeneous. The adsorption capacity of the studied sorbent towards both metal ions was increased with the increase in temperature, indicating that the adsorption process is endothermic. The thermodynamic constants were also calculated. Negative and positive values of Gibbs free energy and entropy were reported. These values indicated that the applied system is spontaneous and random. Aim of research is to prove that *Eucalyptus* roots are an one of the low cost materials are capable of adsorbing pollutants such as heavy metals from aqueous media.

Keywords: Adsorption, Heavy metals, Lead, Cadmium, Carbonized *Eucalyptus* roots

Introduction:

Recently, environmental pollution has been observed all over the world due to industrial activities. The rate of waste and chemicals that are disposed of in the aquatic environment is increasing day by day. The pollution occur in its various forms, whether it is air, water, or soil pollution due to the presence of some harmful organic and inorganic substances, or due to the increase in the proportions of some basic components in the environment from the natural ratios or their deficiency. This happens as a result of human intervention or by some natural phenomena [1,2].

Common water pollutants include non-biodegradable chemicals such as pesticides, industrial dyes, and heavy metals (such as lead, cadmium, chromium, copper, mercury, and zinc) [3,4]. Most of the heavy metals are highly toxic and non-degradable, and caused a serious concern, especially if they exceed the permissible limits. They can reach the human body and lead to brain and bone damage, neurological disorders, and even cancer [5,6].

Various methods were used to remove heavy metals from industrial wastewater. The most important of which are chemical precipitation, electrolysis, extraction, ion exchange, osmosis, filtration, and reverse osmosis. Despite the importance of these techniques, they are limited in use because of their high cost. The adsorption technology is one of the important methods of treatment, because it has a low cost and provides many natural resources that can be used as adsorbent surfaces [7].

Many researchers have recently tended to develop new adsorbent materials using some materials of natural origin. The adsorption method on porous surfaces is one of the common methods for purifying polluted water. Banana peel [8], mango peel [9], orange peel, citrus peel [10], jackfruit peel [11], ponkan peel [12], and pomegranate peel [13]. *Eucalyptus gomphocephala* plant was used as an adsorbent after the preparation of activated carbon from it, and study its ability to adsorb lead and cadmium ions from aqueous solutions [7,6].

2. Materials and method

Preparation of standard solutions

The stock solution containing 1000mg/L of lead and cadmium were prepared by dissolving 1.598 g lead nitrate, from CC; and 1.791 g cadmium chloride, from AG; in 1000mL water.

Preparation of activated carbon

In this study, eucalyptus roots of the cultivar were used, which were collected from eucalyptus trees located at the headquarters of the University of Sirte.

The bark (outer husk) of the roots was firstly removed. The roots were then washed with tap water several times and dried at room temperature to remove moisture. Roots were cut into smaller pieces and then ground with an electric grinder to a fine powder, sieved to obtain granule size, and then placed in the crucibles for combustion under 500°C for 3h. The adsorbent was obtained from the roots of carbonized Eucalyptus (CER) [14].

Preparation of reagent

A 0.001M of the reagent (1,5-diphenylcarbazide) was prepared by dissolving 0.250g of the salt (ACS-Sigma-Aldrich) in 50mL ethanol. The reagent was used to estimate the concentration of Pb(II) and Cd(II) ions after adsorption, forming a pink complex with Pb(II) and a reddish-pink complex with Cd(II) in an alkaline medium using drops of ammonia. The absorbance of both complexes was then measured spectrophotometrically at λ_{max} of 530nm and 540nm for both lead and cadmium, respectively [15].

Adsorption experiments

The adsorption was carried out by mixing 0.1g of adsorbent with the ion solution at a concentration of 100mg/L in a closed 50mL glass beaker at pH=7.0. The flask was placed on a vibrator at 150rpm for 90min. Filtered with ALTAY filter paper (12.5cm) to obtain a clear solution and the absorbance of the solution was then measured to determine the residual concentration (at equilibrium). The

effect of several factors on the adsorption of Pb(II) and Cd(II) ions was studied. The adsorption capacity (q_e) was calculated, and the ion removal rate (%R) was calculated to determine the effect of these factors according to the following equations:/

$$q_e = \frac{C_o - C_e}{M} \times V \dots\dots\dots(1)$$

$$\% R = \frac{C_o - C_e}{C_o} \times 100 \dots\dots\dots(2)$$

Where, C_o , and C_e are the initial and equilibrium concentration (mg/L) of the metal ion, respectively. V is the volume of the metal solution (L), and M the weight of the adsorbent (g) used.

Effect of initial concentration

The effect of metal ion (Pb(II) and Cd(II)) concentration on the adsorption of was studied. Dilute solutions of different concentrations (25, 50, 100, 200 and 300 mg/L) of metal ions were prepared from the stock solution and mixed (each) with 0.1g of adsorbent in total volume of 50mL. Solutions were placed on a shaker for 90min, filtered and metal ion concentration was determined as mentioned earlier.

Effect of pH

The pH in the samples was set at 3.0, 5.0, 7.0, 9.0 and 11.0, using 0.1M HCl and 0.1M NaOH. The pH was then measured using a pH meter. A 0.1g of adsorbent was added to 50ml of ion solutions (100mg/L) at fixed conditions of other factors. Then, the metal ion concentration was then determined.

Effect of contact time

To each sample solution (50mL) contains 100mg/L of ion and 0.1g of adsorbent was added and shaken at different times (1, 5, 10, 20, 30, 40, 60, 70, 100 and 120 min) at 298K at 150rpm. Then filtration and absorbance measured.

Effect of adsorbent dose

The effect of the amount of the adsorbent used was also studied. Different amounts of adsorbent (0.1, 0.3, 0.5, 0.7 and 1g) were added to 50mL of ion solutions (100mg/L and

pH=7.0) at room temperature and then placed on a shaker for a 90min at 150rpm. Then filtered and treated as previously mentioned.

Effect of temperature

A 0.1g of adsorbent was added to each sample (50mL, 100mg/L) and the equilibration was conducted at different temperatures (298, 303, 323 and 328K). Sample were placed in a water supplied with a shaker, and shacked for 90min, and then filtered. The absorbance was measured to estimate the residual concentration.

3. Results and discussion

Effect of pH

The effect of pH on removing ions was studied using eucalyptus carbonized roots as shown in Figure 1. At low pH values, the adsorption of the pb(II) ion decreases and increasing at pH 5, while the Cd(II) adsorption is increases slowly, because the adsorbing surface carries a negative charge, so competition occurs between hydrogen ions H⁺ and metal ions M⁺². The surface tends to adsorb hydrogen ions due to its small size and high velocity.

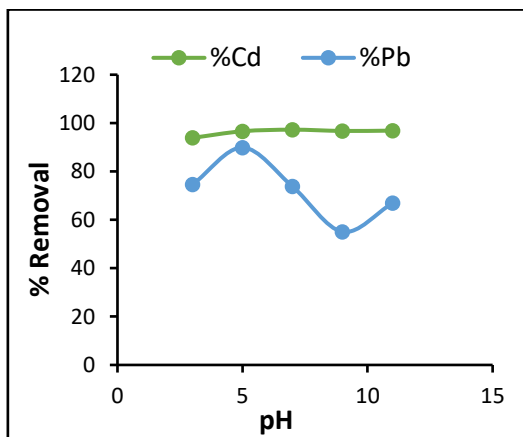


Figure 1: Effect of pH on the adsorption Pb(II) and Cd(II).

At pH values higher than 7.0, a decrease in the concentration of Pb(II) and Cd(II) ions occurs. This decrease is not due to the adsorption process, but rather due to the formation of insoluble hydroxides such as Pb(OH)₂ and Cd(OH)₂. Therefore, it can be concluded that the highest removal percentage

was in the range of pH = 5.0 and pH = 7.0, for both metals, respectively. These results agree with previous studies. [16-18].

Effect of initial concentration

Results of this study showed that (% R) and adsorption capacity (q_e) increased with the increase of metal ion concentration in solution, as shown in Figure 2.

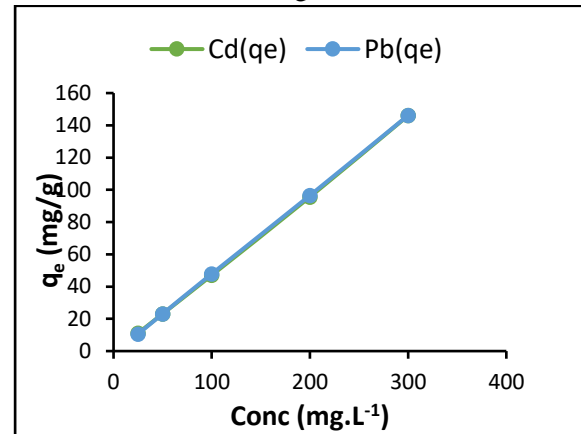


Figure 2: Effect of concentration on the adsorption Pb(II) and Cd(II).

This increase in adsorption with the increase in concentration indicates the presence of a large number of eligible active sites on the surface of the adsorbent material, and therefore, the removal of metal ions from the solution continues to fill all the active sites on the surface of the adsorbent material [19].

Effect of contact time

Through the results of this study, it was observed that the (% R) and the adsorption capacity increased at each time (qt) until they reached the equilibrium state as shown in Figure 3. In the first minutes (5-20 min) the adsorption process is rapid until it reaches equilibrium and the active sites become saturated and High percentage of ion removal equal to 93.5% and 96.6%, Pb(II) and Cd(II) with the adsorption capacity of 45.9mg/g and 47.6mg/g for both metal ions (respectively) at pH of 5-7 was reported.

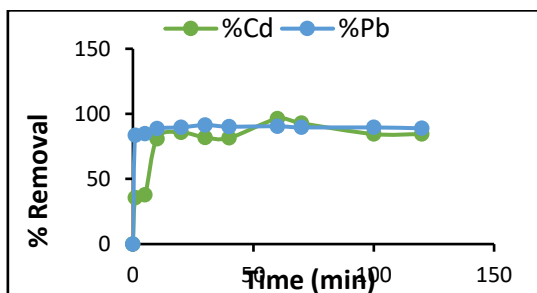


Figure 3: Effect of contact time on the adsorption of Pb(II) and Cd(II).

We conclude that the best equilibrium time for the two elements was 30 min for lead and 60 min for cadmium. These results agree with other results observed in previous studies [16,17,19].

Effect of adsorption dose

Results obtained from Figure 4, revealed that with the increase in the amount of adsorbent, the number of sites eligible for adsorption increases. This increase however, increases the %R of the aqueous solution to a certain level, and then decreases, causing a decrease in the adsorption capacity (qe).

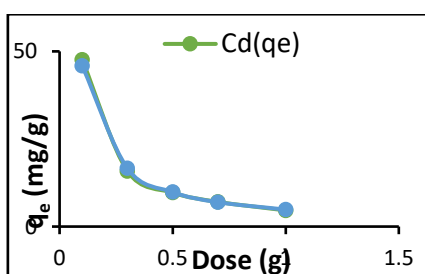


Figure 4: Effect of adsorbent dose on the adsorption Pb(II) and Cd(II).

According to previous studies [15,16,20], the calculated %R in terms of the adsorbent for each mass unit of adsorbent material (mg/g), the highest the adsorption capacity was found when 0.1g of adsorbent was used. Since, the decrease in the adsorption capacity with an increase in the amount of the adsorbent material is caused by the unsaturation of the effective adsorption sites or the reason for this change may be the result of the implicit

molecular interactions between the molecules of the adsorbent material.

Effect of temperature

It was found that the values of the adsorption capacity of the adsorbent (qe) increase with increasing temperature, which indicates that the adsorption process is endothermic. Through this, thermodynamic functions such as Gibbs free energy, enthalpy, entropy, and equilibrium constant $K_c = q_e/C_e$, can be calculated from the obtained practical data as a function of temperature using the following equations:

$$\Delta G^\circ = -RT \ln(K_c) \dots \dots \dots (3)$$

where $R = 8.314 \text{ J/mol.k}$ and T is the temperature in Kelvin (K).

The enthalpy and entropy are also calculated using the following equation:

$$\ln(K_c) = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \dots \dots \dots (4)$$

By plotting $\ln(K_c)$ against $(1/T)$ (Figure 5), the slope of the straight line will be can [16,20,21].

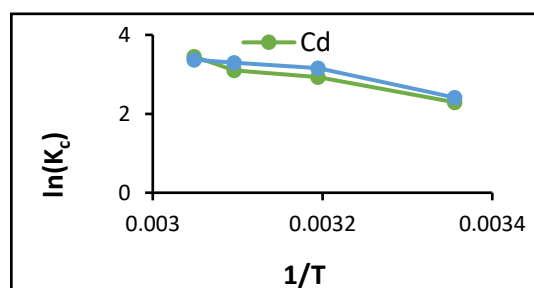


Figure 5: Plot of $\ln(K_c)$ vs. $1/T$ for Pb(II) and Cd(II) by carbonized Eucalyptus roots.

Table 1: Thermodynamic parameters

Metal ions	T (K)	ΔG° (KJ/mol)	ΔH° (J/mol)	ΔS° (J/mol.K)
Pb(II)	298	-5.978	26.02	107.99
	313	-8.222		
	323	-8.850		
	328	-9.193		

Cd(II)	298	-5.681	29.24	117.22
	313	-7.627		
	323	-8.318		
	328	-9.415		

Table 1, shows that the values of the change in Gibbs free energy are small and its sign is negative. This means that the adsorption process is spontaneous, and the change in enthalpy is less than 40 KJ/mol. The positive sign means that the adsorption process is physical and endothermic, and the change in entropy (positive sign) indicates the randomness of the system according to previous studies [16,21].

Adsorption kinetic

Pseudo-first-order kinetic model

The Lagergren pseudo-first-order rate expression is given by the equation:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \dots\dots\dots(5)$$

where q_e (mg/g) is the amount of adsorbent at equilibrium, q_t (mg/g) is the amount of adsorbent at time t and k_1 (min⁻¹) is the rate constant for the pseudo-first-order model adsorption [16,20].

Pseudo-second-order kinetic model

The pseudo-second-order model is given by equation:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \dots\dots\dots(6)$$

Where k_2 (g .mg⁻¹ min⁻¹) is the rate constant of the pseudo-second order kinetic model. In the pseudo-second-order model chemical sorption is the rate limiting step. In reactions involving chemisorption of adsorbate onto a solid surface without desorption of products, adsorption rate decreases with time due to an increased surface coverage [16,20].

Table 2: Kinetic parameter for the removal of Pb and Cd by carbonized Eucalyptus roots.

First Order				
Metal ions	$q_e(\text{exp})$	$q_e(\text{cale})$	k_1	R^2
Pb(II)	45.7	1.598	0.007	0.20
			6	7
Cd(II)	42.2	13.947	0.063	0.84
			5	2
Second Order				
Metal ions	$q_e(\text{exp})$	$q_e(\text{cale})$	k_2	R^2
Pb(II)	45.7	44.6	5.749	0.99
				9
Cd(II)	42.2	44.05	0.644	0.99
				2

The kinetic adsorption and correlation coefficients were calculated for both models as shown in Table (5). It was noted that the values of R^2 for the second order - false are relatively high, and (cale (q_e)) calculated by this model is close to the value which has been determined through experiments (exp (q_e)). While the value of R^2 for the first-order model-The lie is weak and unconvincing. Therefore, the adsorption process is more compatible with the pseudo-second order model, It indicates that the adsorbent and the adsorbent are the ones that control the reaction [22][23].

Adsorption isotherms

Langmuir isotherm

It is assumed that the molecules adsorb on a fixed number of well-known vacuoles on the surface of the adsorbent material. These cavities are energetically equivalent and each vacuole can carry only one adsorbent molecule. The molecules adsorbed on the surface do not interfere with each other or with other molecules present in the solutions, and thus one layer of the adsorbed molecules will form on the surface of the adsorbent material [20]. Equation 5 for this model has been applied to practical data for the adsorption of the two components. The constants (a/kL and $1/kL$) were calculated through the slope and

section of the straight lines resulting from plotting the relationship between C_e/q_e versus C_e [16]. The obtained results are shown in Figure 6.

$$C_e/q_e = 1/k_L + a/k_L(C_e) \dots\dots\dots(7)$$

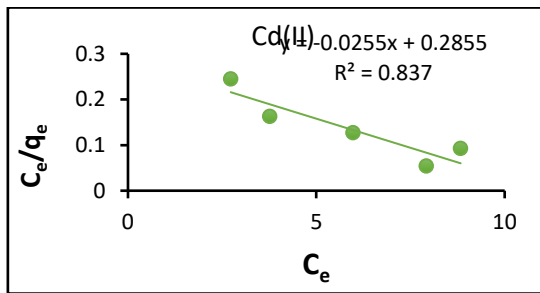
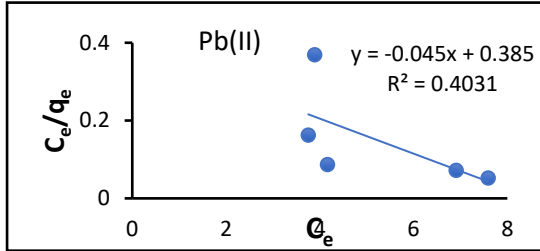


Figure 6: Langmuir adsorption isotherms of Pb(II) and Cd(II) by Carbonized Eucalyptus roots

It is assumed that the surface of the adsorbent material is heterogeneous and has a multi-layered due to the non-uniformity of the potential energy on it and that the adsorption sites have varying levels of energy [20]. The Freundlich isotherm equation (7) was applied to the practical data of the two components and the Freundlich constants (K_F, n) were calculated from the slopes of the straight lines resulting from plotting the relationship between $\log q_e$ versus $\log C_e$. The obtained results are shown in Figure 7 [16].

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \dots\dots\dots(8)$$

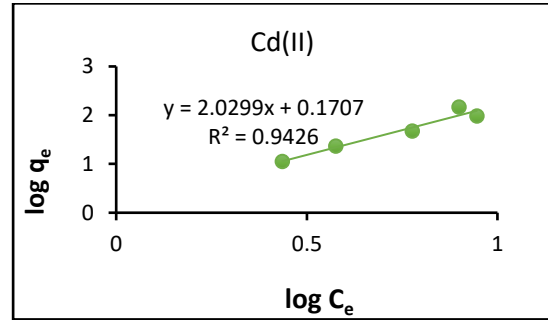
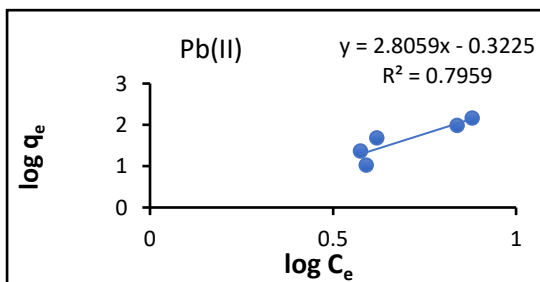
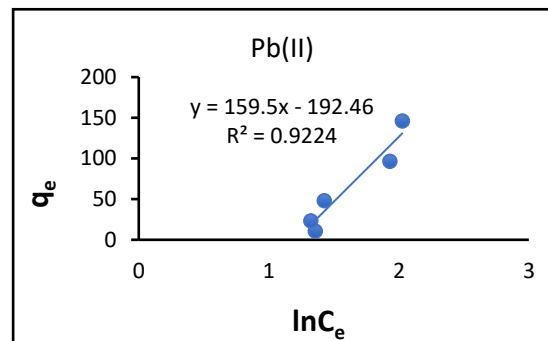


Figure 7: Freundlich adsorption isotherms of Pb(II) and Cd(II) by Carbonized Eucalyptus roots

Temkin isotherm

The basic assumption of isotherm revealed that, the adsorption energy decreases linearly with increasing surface coverage resulting from the interference between the adsorbent and the adsorbent [20]. The TTM isotherm was applied to the two components, using equation (7). Figure 8 illustrates the plotting of the relationship between q_e versus $\ln C_e$. The values of the BT constants were then calculated from the slope and section of the straight lines [16]. These results showed that the R^2 values of the Freundlich isotherm were 0.942 for Cd(II) and 0.759 for Pb(II), while the R^2 values for the Temkin isotherm were 0.744 for Cd(II) and 0.922 for Pb(II).

$$q_e = B_T \ln K_T + B_T \ln C_e \dots\dots\dots(9)$$



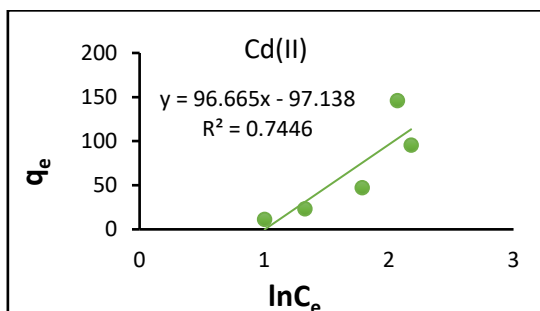


Figure 8: Temkin adsorption isotherms of Pb(II) and Cd(II) by Carbonized Eucalyptus roots.

Table (.3) values of the Langmuir, Freundlich and Timken isotherms

	Pb(II)			Cd(II)		
	a	K L	R ²	a	kL	R ²
Langmuir	-	2.	0.	-	3.	0.
Equation	0.	59	40	0.	50	83
	11	7	3 -	08	2	7
	6			9		
Freundlich	n	Kf	R ²	n	Kf	R ²
equation	0.	0.	0.	0.4	1.4	0.9
	35	47	79	92	82	42
	6	5	5			
Timken	B	K T	R ²	B	K T	R ²
equation	15	0.	0.	96	0.	0.
	9.	29	92	.6	36	74
	5	9	2	6	6	4

From the results obtained in Table (.3) it was noted that the R² values of the Freundlich and Timken isotherms were 0.942 for cadmium and 0.759 for lead, while the R² values for the Isotherm Timken were 0.744 for cadmium and 0.922 for lead. Adsorption is multilayer and heterogeneous.

Conclusion

The main objective of the present contribution was focused on the development of removal the heavy metals (i.e. cadmium, and lead) from aqueous solution by Eucalyptus roots. The mechanism of metal adsorption by Eucalyptus is found dependent on the initial pH of the solution. Thus, at acidic pH, the competition between protons and M(II) ions decreases the adsorption performance of the adsorbent material, whereas for pH greater than 7, a saturation of the adsorption capacity is observed. The precipitation to hydroxides also accounts for this saturation, because of the low-solubility products. An increase in the metal concentration and the mass of the adsorbent favours the adsorption process. The adsorption isotherms of the two toxic metals by Eucalyptus Gomphocephala is satisfactorily described by the Freundlich and Temkin models throughout the investigated concentrations for all metals, whereas the Langmuir model does not fit suitably the experimental data for Pb(II) and Cd(II) ions. The spontaneous and endothermic adsorptions of the metals were evidenced from the negative free energy (ΔG°) and positive enthalpy (ΔH°).

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دراسة الحركية والايزوثيرم لاد مصاص أيونات Pb (II) و Cd (II) من محاليلها المائية
بواسطة جذور نبات الكافور (*Eucalyptus Gomphocephala*)

د.رجوان محمد رجوان*¹ و سعدة سالم عبدالحفيظ²

E-mail:ragwan3000@su.edu.ly

الملخص

درس هذا البحث عملية ادمصاص أيونات الرصاص (II) والكاديوم (II) من محاليلهما المائية باستخدام جذور الأوكالبتوس المتفحمة كمادة مدمصة ، حيث بينت بعض النتائج وجود نسبة عالية من الازالة لأيونات الرصاص (II) والكاديوم (II) بلغت 93.5% و 96.6% ، و سعة ادمصاص 45.9 ملجم / جم و 47.6 ملجم / جم لكلا أيونات المعدنين (على التوالي) عند درجة حموضة 5-7، و وصل النظام إلى حالة التوازن بعد 30 دقيقة تقريباً ، وزادت نسبة الامتزاز لهذه المعادن مع زيادة التركيز الابتدائي للعناصر المذكورة وكمية المادة الممتازة حتى الوصول إلى حالة التوازن. اتبعت عملية الامتزاز نموذج الدرجة الثانية الوهمية وتوافق مع معامل ارتباط isotherm Freundlich ، مما يدل أن هذا الامتزاز متعدد الطبقات وغير متجانس. ارتفعت سعة ادمصاص للمادة الماصة المدروسة تجاه كلا أيونات المعدن مع زيادة درجة الحرارة، مما يشير إلى أن عملية ادمصاص ماصة للحرارة. كما تم حساب الثوابت الديناميكية الحرارية ، حيث كانت قيم سلبية بينما ايجابية قيم الطاقة الحرة وانتروبيا جيبس ، وتدل هذه القيم إلى أن النظام المطبق عشوائي. بما يدل تحقيق الهدف من البحث هو إثبات أن جذور الأوكالبتوس هي إحدى المواد منخفضة التكلفة القادرة على امتصاص الملوثات مثل المعادن الثقيلة من الوسائط المائية.

الكلمات المفتاحية: الامتزاز ، المعادن الثقيلة ، الرصاص ، الكاديوم ، جذور الأوكالبتوس المتفحمة.