

New Adsorbent from Red Beet Roots (CRBR) for Removal of Lead

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THE MAIN purpose of this work is to remove lead ions from aqueous solution by using carbon of red beet roots (CRBR). The influence of contact time, initial concentration, temperature, pH of solution and the dose of adsorbent were studied. It was found that the maximum removal percentage of Pb(II) has reached 99.99% at room temperature by applying the following conditions: adsorbent loading weight 0.1 g, contact time 24 hrs, and 9.993 m.mol/L concentration of lead ions. The adsorption process of Pb(II) on CRBR was of chemical nature and followed the kinetic formula of pseudo second –order reaction. Results of FTIR spectroscopy and scanning electron microscopy made it possible to propose mechanism for the adsorption process. The recovery of the adsorbed Pb(II) ions on CRBR surface was done by using 1 N acetic acid solution resulting a recovery of 87.466%.

Keywords: Red beet roots, Removal of lead, Adsorption.

Introduction

Nowadays, there has been a great concern about the raises of water pollution on the human and environmental, that can be caused by industrials effluents and wastes generated by activities of modern life [1]. Among the several types of water contaminats, heavy metals are one among the foremost necessary environmental issues that has got to be resolved, and established ways to get rid of them. [2]. Many conventional methods exist for the removal of heavy metals from wastewater [3]. These methods include chemical precipitation, coagulation, solvent extraction, electrolytic process, membrane separation, and ion exchange [4]. These methods have several disadvantages such as producing a large amount of toxic sludge and high costs. Adsorption using low cost adsorbents is the alternative process for removal of heavy metals from aqueous solutions [1].

Among the different heavy metals lead is one of the common and most toxic pollutants because it accumulates within the brain, bones, kidneys, muscles, and causes a series of disorders like anemia, uropathy and neurologic epidemic disorders that result in death [2]. In drinking water maximum allowable limit of total Pb of 50 μgL^{-1} considered safe by the World Health Organization,

whereas less than 15 μgL^{-1} is adopted by the United States Environmental Protection Agency [5]. Adsorption of lead was studied by using adsorbents of agricultural origin such as tea leaves [2], the waste pulp of sugar beet[4], rice husk [6], sago waste [7], tea waste [8], date tree leaves [9], tridax procbens plant leaves [3], rice husks, maiz cobs and sawdust [10].

Natural materials were also used for removing the lead ions from wastewater, where the removal of lead ions was studied by using the American bentonite [11], the result of this study has shown that bentonite was more effective natural material for removing the lead ions from aqueous solution than activated carbon [11].

Recently different adsorbents have been used for removal of lead ions such as carbon of sewage sludge[12], activated lawsonia inermis carbon[13], rice straw[14], and livestock biomasses[15].

This work focuses on the adsorption of lead ions from aqueous solution by using carbon of red beet roots (CRBR) without modification, The reason for using red beet roots is its availability in local area in large quantities with low cost and for being highly effective material for removing heavy metals.

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Materials and Methods

Preparation of adsorbent (CRBR)

A sample of dried red beet roots was inserted in a cylinder and calcinated for 4 hrs at 400 °C followed with further calcination for 4 hrs at 600°C, then it was cooled to reach the room temperature. The carbon was characterized by using PC X Ray Fluorescence (X-MET 5100-Oxford instrument), JASCO- FT-IR 4200 device, Scanning Electronic Microscopy (SEM, VEGA\\ XMU) coupled with Energy Dispersive X-ray Analysis (EDAX-AMETEX).

Preparation of lead solution

A stock solution (2070.5 mg/l or 9.993 m.mol/L) of Pb(II)ions was prepared by dissolving an accurately weighed analytical grade lead nitrate(RIEDEL-DE-HAEN AGSEELZE-HANNOVER, in deionized water (pH=5.27). Studied solutions of different concentrations, as required, were prepared from the stock by dilution process. Lead ions concentration was measured by using an atomic absorption device (Aurora-A11200 instrument).

Procedure of adsorption process

5 ml of lead nitrate solution were powered into each of glass tubes containing 0.1g of CRBR to study the impacts of contact time, pH solution, initial concentration of lead and change of temperature during the adsorption process.

Results and Discussion

Adsorption characterization

Morphological studies

The surface of CRBR was analyzed by scanning electronic microscopy coupled with

energy dispersive X-ray analysis. The results of SEM for CRBR before and after adsorption of lead ions are shown in Fig. 1a and 1b.

The SEM image (Fig. 1a) shows the irregular texture of pores on the surface of adsorbent. The diameters of some pores were between (8.46 - 23.98) μ m. Figure 1b shows the surface of CRBR after adsorbing lead (II) ions. Deposits of adsorbent-adsorbate complex are observed on the surface of the adsorbent following the interaction with Pb(II) ions. EDXA analysis of some spots on the surface of CRBR before and after adsorption was carried out and listed in Tables 1 and 2 respectively.

As shown in Table 2 lead and nitrogen elements appeared on the surface of CRBR after the adsorption process.

Figure 2 shows the EDXA analysis for CRBR after adsorbing lead ions on it. The appearance of Pb and nitrogen on the surface of CRBR after the adsorption corresponds to values 2.34 and 10.58 KeV for Pb [16] and 0.392 KeV [17] for nitrogen indicating the presence of both ions (lead and nitrate)on the surface of CRBR.

XRF Analysis of CRBR

Chemical composition of CRBR before adsorption was studied by using PC X Ray Fluorescence (X-MET 5100-Oxford instrument). The results of XRF analysis are listed in Table 3.

Infrared Spectroscopic Studies

The FT-IR spectra of CRBR before and after adsorption of lead ions were taken by using jasco FT-IR -4200 device. Figure 3a shows the FT-IR spectra of CRBR before adsorption of Pb(II) ions. Figure 3b demonstrates the FT-IR spectra of

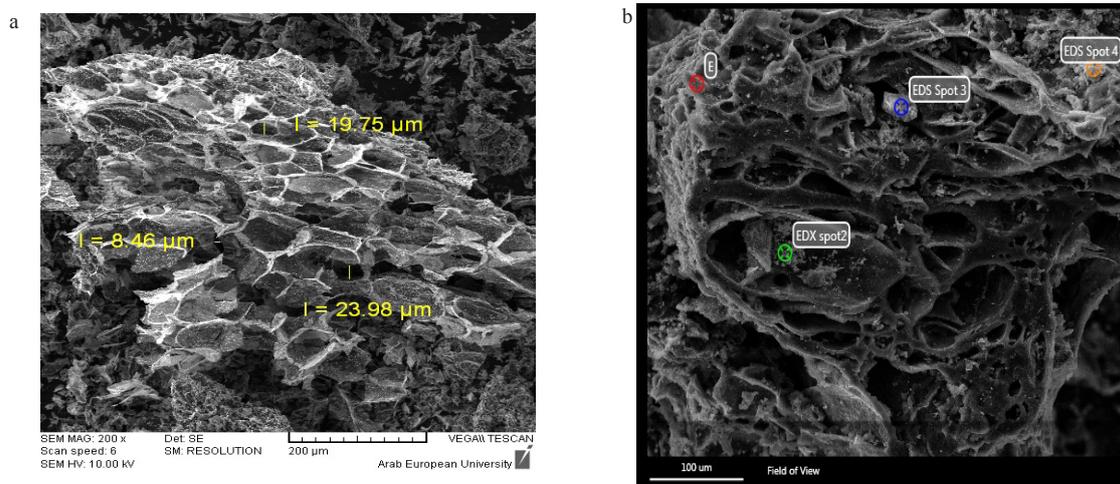


Fig. 1. SEM image of CRBR (a) before adsorption (b) after adsorption.

TABLE 1. EDXA analysis of some spots on the surface of CRBR before adsorption of lead ions

Spot 4 (wt %)	Spot 3 (wt %)	Spot 2 (wt %)	Spot 1 (wt %)	Full area (wt %)	Element
54.74	48.57	49.65	46.71	54.67	C
26.88	25.19	16.82	23.81	25.87	O
7.51	9.05	9.45	9.18	7.42	Na
0.38	0.35	0.36	0.46	0.38	Mg
0.02	0	0.05	-	0.06	Si
0.64	0.94	0.86	1.42	0.7	P
0.11	0.15	0.23	0.25	0.14	S
3.79	5.15	10.64	5.6	4.1	Cl
5.51	9.65	11.65	10.9	6.17	K
0.41	0.95	0.29	1.68	0.5	Ca

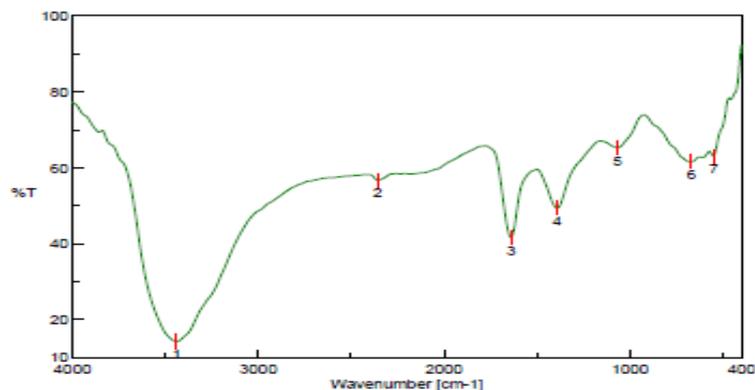
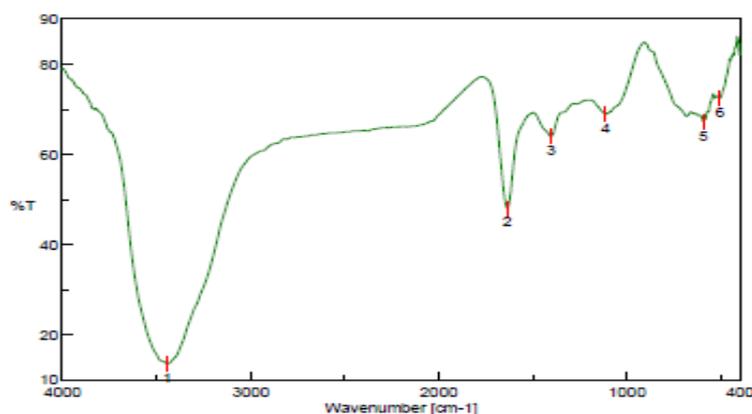
TABLE 2. EDXA analysis of some spots on the surface of CRBR after adsorption of lead ions

Spot 4 (wt %)	Spot 3 (wt %)	Spot 2 (wt %)	Spot 1 (wt %)	Full area (wt %)	element
35.49	14.28	34.09	55.45	46.56	C
18.79	0.57	2.26	7.54	23.29	N
26.05	34.92	4.18	24.35	19.69	O
2.64	3.07	0.05	3.46	1.38	Na
0.46	0.06	0.03	1.06	0.22	Mg
-	11.55	-	0.37	-	Al
-	20.79	-	0.03	-	Si
0.49	-	3.38	1.19	0.89	P
-	-	-	0.01	-	S
-	-	43.12	-	-	Cl
15.2	0.09	1.73	4.31	6.2	Pb
0.57	6.95	6.55	1.59	1.29	K
0.29	5.72	4.61	0.64	0.48	Ca
	2.01	-	-	-	Fe

**Fig. 2. Chemical composition of CRBR after adsorption by using EDXA analysis**

TABLE 3. Elemental structure of CRBR by using XRF

Elements	Concentration
Non	main elements of concentration > 10
K	Main elements of concentration more than 1% and less than 10%
Ca, Cl, Mn, Fe, Sr, P, S	Elements of concentrations from 1% to 100µg/g
Cr, Cu, Ni, Br, Zn, Rb, Pb, Ti	Elements of concentrations of (1- 100)µg/g
Non	Trace elements of less than 1 µg/g

**Fig. 3a. The FT-IR spectra of CRBR before adsorption of lead ions.****Fig. 3b. The FT-IR spectra of CRBR after adsorption of lead ions.**

CRBR after adsorption of Pb(II) ions.

The main wave numbers of the peaks appearing in Fig. 3a and 3b are listed in Table 4.

Effects of different experimental conditions

Effect of contact time

Effect of contact time was studied in the range of (0.5-48) hrs by using a sample of 0.1 g of CRBR and 5 ml of lead nitrate with 9.993 m.mol/L concentration of solution. The experimental results are presented in Fig. 4.

It was found from Fig. 4 that the removal percentage of lead ions increased with increasing

the contact time and the equilibrium was attained at about (24) hrs, this result is in accordance with the references [3,10].

Effect of adsorbent loading weight

The effect of adsorbent loading weight is presented in Fig. 5. The dependence of Pb(II) ions adsorption on the dose of adsorbent was studied by varying the mass of adsorbent in the range of 0.05 to 0.2 g, whereas other parameters (concentration, contact time) were kept constant at room temperature. It can be inferred that the percentage removal of lead ions increases with increasing the dose of adsorbent from 0.05 g to 0.1 g. However,

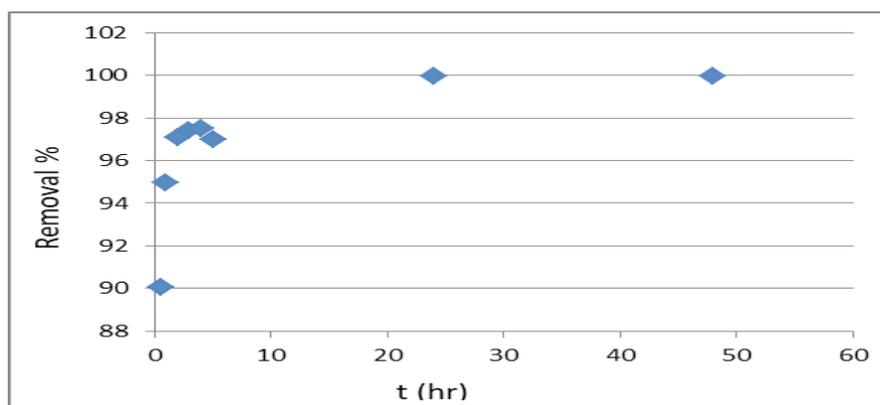


Fig. 4. Effect of contact time on Pb(II) ions adsorption by CRBR, $C_0=9.993\text{m.mol/L}$, $m=0.1\text{ g}$

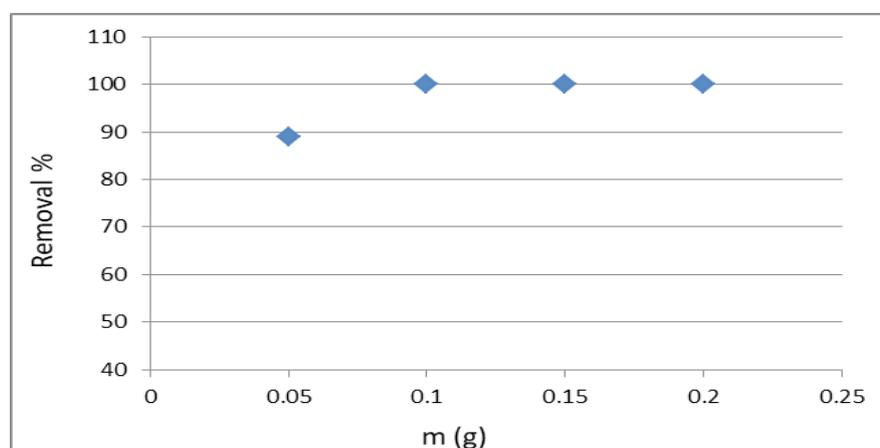


Fig. 5. Effect of adsorbent dose on removal of Pb(II) ions, $C_0=9.993\text{ m.mol/L}$, $t=48\text{ hrs}$.

when the dose of adsorbent was increased from 0.1 to 0.2 g no significant increases in removal percentage were observed. This suggests that after a certain dose of adsorbent, the maximum adsorption is reached and hence the amount of ions bound to the adsorbent and the amount of free ions in the bulk remains constant even with further increasing of the dose of adsorbent. The increasing of Pb(II) ions removal percentage with the increasing of the dose of adsorbent is as a result of the availability of the active sites or surface area at higher doses of the adsorbent [3,10].

Effect of initial concentration

In order to find the effect of Pb(II) ions concentration in aqueous solution on the adsorption process, tests were made at room temperature in the range of (4.997-9.993)m.mol/L of Pb(II) ions within a fixed time 2 hrs, The mass

of CRBR was 0.05g and the volume of lead solution was 5 ml. Figure 6 shows the adsorbed lead amount as a function of equilibrium lead concentration.

It is noticed from Fig. 6 that the amount of adsorbed lead increases with increasing equilibrium lead concentration

Effect of the change of temperature

Tests for adsorption of Pb(II) ions in aqueous solution by CRBR were made at different temperature (20-50) $^{\circ}\text{C}$ when contact time, the dose of adsorbent and concentration of Pb(II) ions solution were 2 hrs, 0.1 g, 9.993 m.mol/L respectively. Results of these tests are shown in Fig. 7.

It is clear from Fig. 7 that the percentage removal of Pb(II) ions is almost constant with

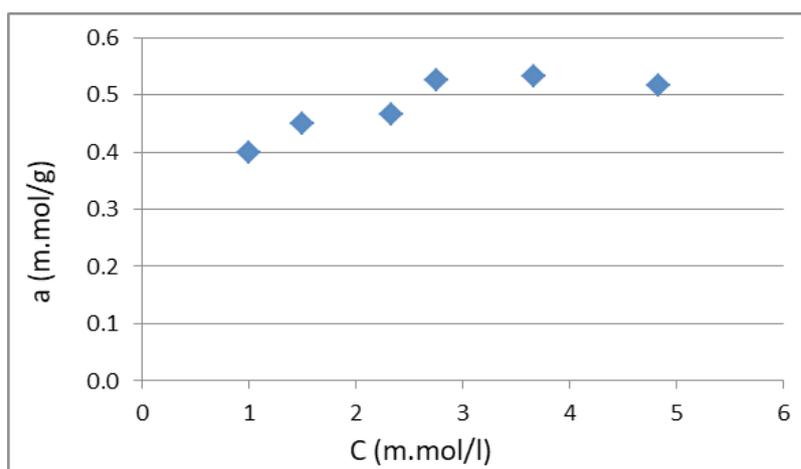


Fig. 6. Adsorbed lead amount as a function of equilibrium lead concentration $m=0.05$ g, $t=2$ hrs, $C_0=4.997-9.993$ m.mol/L

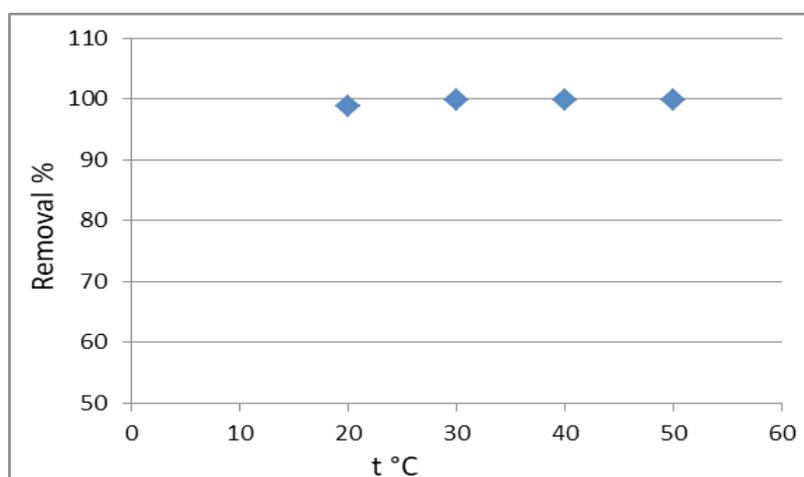


Fig. 7. Effect of temperature on Removal of lead ions, $t=2$ hrs, $m=0.1$ gr, $C_0=9.993$ m.mol/L.

the increasing of temperature. This indicates that the adsorption of Pb(II) ions on CRBR was chemisorption.

Effect of pH

The pH in solution is one of the most important parameters of adsorption of heavy metals. The adsorption of lead ions by CRBR at different pH values is presented in Fig. 8. The effect of pH was studied at room temperature by varying the pH of metallic solution from (2.1-5.3).

Removing of lead at lower pH was not observed. This result can be explained on the bases that at lower pH values, the H^+ ions were high and competed with the metal cation in the adsorption sites of the system [10]. By increasing

pH, the negative charge density on the adsorbent increases due to deprotonation of metal binding sites, thus increasing metal adsorption. The maximum amount of removing Pb(II) ions was at $pH = 4.7$, whereas at pH higher than 5.3, lead ions were precipitated due to the formation of lead hydroxide [3].

Adsorption isotherms

The experimental data, for removing the lead ions by CRBR at different concentrations of lead solution, were analyzed by the well-known models given by Langmuir, Freundlich and Dubinin-Radushkevich models.

The Langmuir isotherm takes an assumption that the adsorption occurs at specific homogeneous

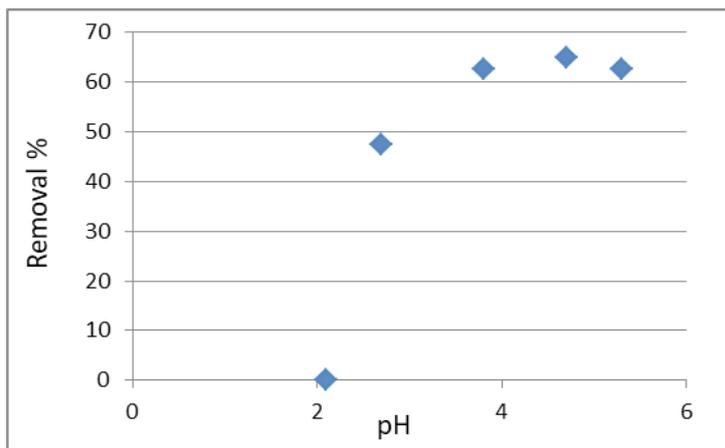


Fig. 8. Effect of pH on removal of lead ions, t=2 hrs, m=0.01 gr, C₀=5 m.mol/L

sites within the adsorbent [3]. The general form of Langmuir equation is given as (1):

$$a_{\epsilon} = \frac{a_m K_L C_{\epsilon}}{1 + K_L C_{\epsilon}} \quad (1)$$

Where:

C: The equilibrium metal ions concentration in solution (m.mol/L).

a, a_m: The amount of metal ions adsorbed on the carbon and the maximum metal ions uptake per unit mass of adsorbent, respectively (m.mol/g).

K_L: Langmuir constant (L/m.mol).

The linear form of isotherm equation can be written as (2):

$$\frac{1}{a_{\epsilon}} = \frac{1}{a_m} + \frac{1}{a_m \cdot K_L \cdot C_{\epsilon}} \quad (2)$$

The parameters a_m and K_L can be determined from Fig. 9 which shows the plot of (1/a) versus (1/C)

The Freundlich expression is an empirical equation derived for the heterogeneous surfaces [3]. The general form of Freundlich equation is (3):

$$a_{\epsilon} = K_F \cdot C_{\epsilon}^{1/n} \quad (3)$$

Where:

K_F: Freundlich constant (lit)^{1/n}/g (m.mol)^{1/n-1}

n: The adsorption intensity.

C: The equilibrium metal concentration in solution

(m.mol/L).

a: The amount of metal ions adsorbed per mass unite of the carbon (m.mol/g).

The linearized form of this model is given by equation (4):

$$\text{Log } a_{\epsilon} = \text{log } K_F + \frac{1}{n} \text{log } C_{\epsilon} \quad (4)$$

Where the intercept log K_F is a measure of adsorption capacity, and the slope 1/n is the intensity of adsorption [3]. The Freundlich adsorption models are presented in Fig. 10.

Dubinin-Radushkevich isotherm is generally applied to express the adsorption mechanism with a Gaussian energy distribution onto heterogeneous surfaces. This model has often successfully fitted high solute activities and intermediate range of concentration [22]

$$\ln a_{\epsilon} = \ln a_s - k_{ad} \epsilon^2 \quad (5)$$

Where a_s is the theoretical saturation capacity (mol/g), ε: Polanyi potential, which is equal to RT ln(1+1/C_ε), R(J/mol.K) is the universal gas constant, T (K) is the absolute temperature, and K_{ad} is a constant related to the mean free energy of adsorption per mole of adsorbate (mol²/KJ²),

$$E = \frac{1}{\sqrt{2 K_{ad}}} \quad (6)$$

Where k_{ad} is denoted as the isotherm constant and a_s, K_{ad} can be determined by plotting ln a_ε versus ε² (Fig. 11).

Dada et al [22] assumed that if the magnitude

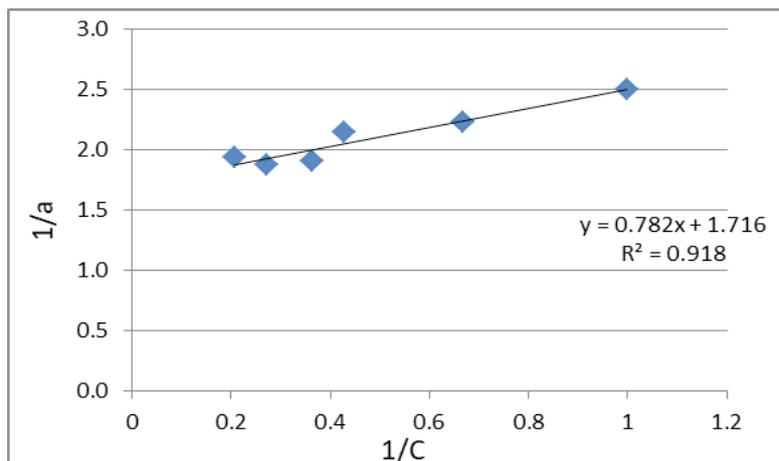


Fig. 9. Application of langmuir isotherms for Pb(II)ions adsorption, m=0.05 g, t=2 hrs.

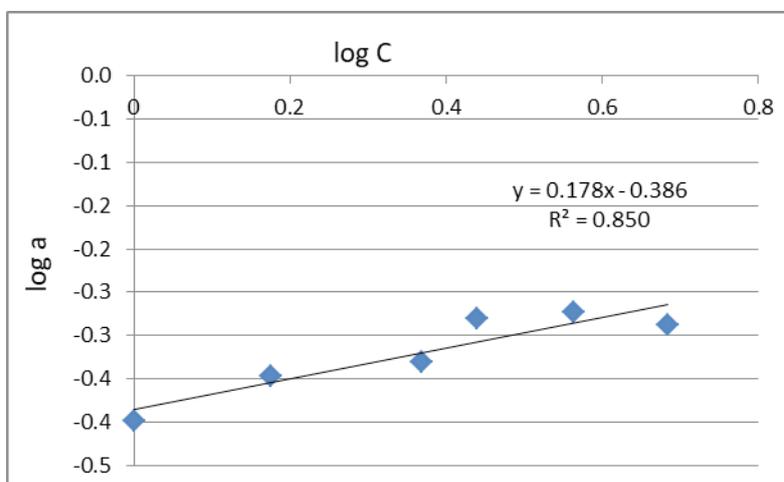


Fig. 10. Application of freundlich isotherms for Pb(II)ions adsorption, m=0.05 g, t=2 hrs.

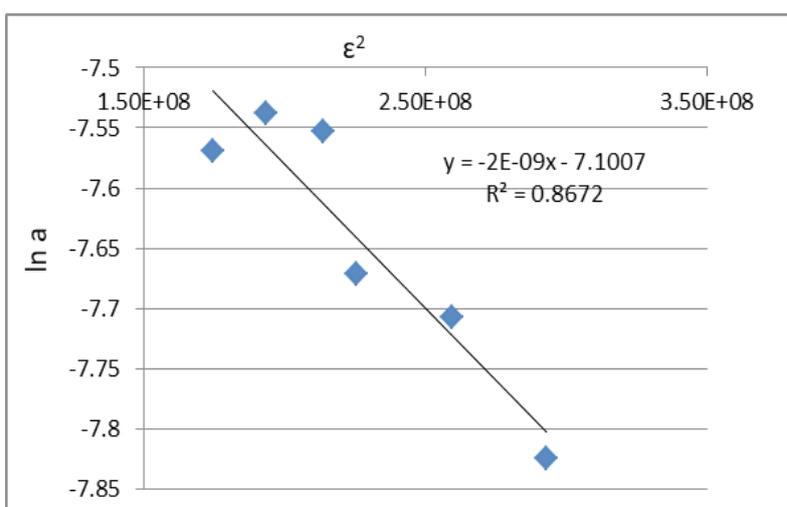


Fig. 11. Application dubinin-Radushkevich isotherms for Pb(II)ions adsorption, m=0.05 g, t=2 hrs.

of E is between 8 and 16 KJ/mol, the adsorption process proceeds by ion exchange while for $E < 8$ KJ/mol the adsorption process is of a physical nature [23], in this study the value of E in Table 3 is calculated as 15.811 KJ/mol, this may be an indication that ion exchange reaction is taking place along with the adsorption process

It was found from Fig. 9-11 that the experimental data fitted Langmuir isotherm equation more than other isotherms. Values of parameters for Langmuir, Freundlich and Dubinin-Radushkevich isotherms are written in Table 5.

Separation factor R_L can be used to predict affinity between the sorbate and sorbent in the adsorption system according to the studies [3,24]. The expression of separation factor in the dimensionless form of Langmuir isotherm is

$R_L = 1 / (1 + K_L \cdot C_0)$, where C_0 is the initial concentration of metal ion, K_L is the Langmuir constant.

The characteristics of R_L value indicates the nature of adsorption as unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$), and irreversible ($R_L = 0$), in the whole studied concentration (4.997-

9.993) m.mol/L of metal ions the separation factor R_L is between (0.0436-0.0836) and it is less than 1 indicating the favourable adsorption.

Adsorption Kinetics

The kinetics of the adsorption of lead ions was studied by applying pseudo second order model as shown in Fig. 12. Pseudo second order model expression is given in the following equation:

$$\frac{t}{a_t} = \frac{1}{k_2 a_e^2} + \frac{1}{a_e} t \quad (7)$$

Where a_e , a_e (m.mol/g) are the amount of lead ions adsorbed by 1 g of the carbon within time (hr) and at equilibrium respectively, k_2 L.(m.mol.hr)⁻¹ is the rate constant of adsorption in the pseudo second order reaction

The constants a_e and k_2 were calculated from equation of plot t/a_t versus t and Table 6 shows the related values.

It is concluded from Table 6 that the adsorption of lead ions onto CRBR fitted pseudo second order reaction and the value of (a_e) which was calculated according to pseudo second order model was close to experimental value.

TABLE 4. Some important functional groups with their wave numbers in FT-IR spectrum for CRBR before and after adsorption of Pb(II)

Wave number cm ⁻¹ after adsorption	Wave number cm ⁻¹ Before adsorption	Functional groups	References
3442	3440	OH	[19]
-	2356	C≡C	[16], [19]
1635	1639	C=C	[16], [19]
1403	1391	OH bending	[18], [20], [19]
1112	1066	C-O stretching vibration	[16], [19]
-	668 - 549	C-X or M-X	[19]
593-504	-	Pb-O	[21]

TABLE 5. Langmuir, Freundlich and Dubinin-Radushkevich parameters for adsorption of Pb(II) on CRBR.

Langmuir parameters			Freundlich parameters			Dubinin-boliani parameters			
a_m (m.mol/g)	K_L (lit/ mol)	R^2	n	K_F (lit) ^{1/n} /g (m.mol) ^{1/n-1}	R^2	E (KJ/mol)	a_s (mol/g)	K_{ad} (mol ² /J ²)	R^2
0.583	2194.373	0.918	5.618	0.411	0.85	15.811	0.000825	2E-09	0.867

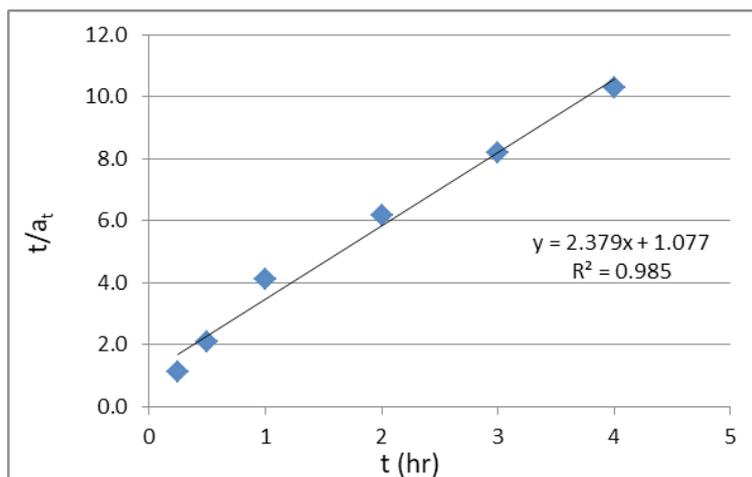


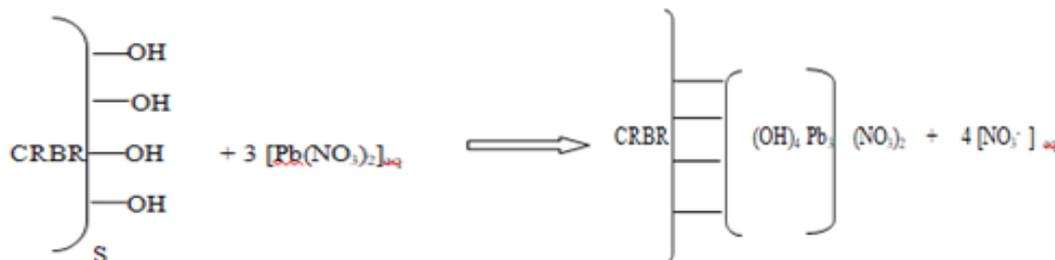
Fig. 12. Application of pseudo second order model on adsorption of lead by CRBR.

TABLE 6. The rate constant and equilibrium amount of lead (II) for pseudo second order model

R ²	k ₂ (m.mol.hr) ⁻¹ .L	a _e (m.mol/g)	a _e exp (m.mol/g)
0.985	5.264	0.420	0.500

Mechanism of adsorption of pb(II) ions on CRBR

Our experimental results may be explained by the following mechanism



Where: S is the surface of CRBR, OH are hydroxyl groups existing on the surface of CRBR. The pH of lead solution during the adsorption process was observed, where the initial concentration of lead nitrate solution had a pH =5.27 and within adsorption process the pH increased to 7-8. The mechanism of this reaction can be suggested with $[\text{Pb}_3(\text{OH})_4]^{+2}$ or $[\text{Pb}_6\text{O}(\text{OH})_6]^{+4}$ [25].

Desorption of the Pb(II) adsorbed from the surface of CRBR

The Pb(II) ions complex deposited in a layer bounded to the surface of CRBR as a result of the adsorption process. The previous material was treated with 5 ml of acetic acid solution (1

N), left for a half an hour and then filtrated for the measurement of the Pb²⁺ concentration ions by using the atomic absorption device. The percentage of recovery of Pb(II) was 87.466% of

adsorbed Pb(II) on the surface of CRBR.

Adsorption of NO₃⁻ ions by CRBR

The concentration of NO₃⁻ ions in the solution before and after adsorption of lead ions by CRBR was measured by the method mentioned in the reference [26] and it was found that the NO₃⁻ ions were adsorbed on the surface of CRBR and the percentage of NO₃⁻ adsorption increased with the increasing of contact time, temperature, and the dose of adsorbent. The maximum removal for NO₃⁻ ions was 38% at contact time 24 hrs, 0.1 g the dose of adsorbent at room temperature.

Conclusions

Single and non-modified carbon from red

beet roots has been prepared in this work for the removal of lead ions from aqueous solution.

The change of temperature had no effect on pb(II) ions adsorption.

The removal percentage of lead ions increased with increasing the dose of adsorbent and contact time.

A 87.446% recovery of the adsorbed pb(II) ions on CRBR has been reached by use of little amount with acetic acid solution 1N.

Adsorption of lead ions onto CRBR fitted the kinetic of pseudo second order reaction

A mechanism for the formation of the complex surface structure of the process has been proposed.

It can be concluded that the carbon of red beet roots was effective as for removal of lead ions from aqueous solution with a percentage of 99.99% accompanied with a partial removal of NO_3^- ions with a percentage of 38%.

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مادة مازة جديدة من جذور الشمندر الأحمر لإزالة أيون الرصاص من المحلول المائي

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يهدف هذا البحث إلى إزالة أيون الرصاص من المحلول المائي باستخدام فحم جذور الشمندر الأحمر (CRBR). حيث درس تأثير كل من زمن التماس، التركيز الابتدائي لأيون الرصاص، تأثير pH المحلول، وكمية المادة المازة. وجد أن نسبة الإزالة العظمى للرصاص وصلت إلى ٩٩,٩٩٪ باستخدام وزن ٠,١ g من المادة المازة وتركيز ٩,٩٩٣ m.mol/l لأيون الرصاص وذلك عند زمن التماس ٢٤ ساعة وبدرجة حرارة الغرفة. لوحظ أن عملية امتزاز أيون الرصاص على الفحم CRBR ذات طبيعة كيميائية وتتبع تفاعل المرتبة الثانية الكاذبة. وتمكنا من وضع آلية عملية الامتزاز بناءً على نتائج أطياف FTIR ونتائج قياسات المجهر الإلكتروني الماسح واسترجع الرصاص الممتز على الفحم CRBR باستخدام حمض الخل ١N وكانت نسبة الاسترجاع ٨٧,٤٦٦٪.