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# Adsorption of heavy metal ions on olive leaves: Equilibrium and kinetic studies

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#### Abstract

In this study the adsorption of  $Cu^{2+}$ ,  $Pb^{2+}$  and  $Cr^{3+}$  ions from aqueous solution using olive leaves ash were investigated. The equilibrium adsorption level was determined as a function of the solution pH, temperature, contact time and adsorbent doses. Adsorption isotherms of heavy metal ions on adsorbents were determined and correlated with common isotherm equations such as Langmuir, Freundlich and Tempkin models. These studies were showed that the Langmuir isotherm model was fitted well with adsorption data. Kinetic studies were carried out on various kinetic models such as pseudo-first order, pseudo-second order and intra particle diffusion model. The pseudo-second order kinetic model was fitted very well with experimental data. Base on thermodynamic studies, this process was both endothermic and spontaneous, enthalpy and entropy were negative.

Keywords: Heavy Metal ions, Adsorption, Isotherm, Kinetic

### 1. Introduction

Heavy metals are the main hazardous nondegradable substances, persistence and can be accumulate in the environment elements such as food chain, thus may pose a significant danger to human health [1-5]. Copper is essential to all living organisms as a trace dietary mineral because it is a key constituent of the respiratory enzyme complex cytochrome oxidase. The main areas where copper is found in vertebrate animals are liver, muscle and bone. However, the excessive ingestion of copper brings about serious toxicological concerns, such as vomiting, cramps, convulsions, or even death. Chromium affects human physiology, accumulates in the food chain and causes severe health problems ranging from simple skin irritation to lung carcinoma [6]. Lead, at certain contact degrees, is a poisonous substance to animals, including humans. It damages the nervous system and causes brain disorders. Excessive lead also causes blood disorders in mammals. Like the element mercury, another heavy metal, lead is a neurotoxin that accumulates both in soft tissues and the bones. Hence, it is essential to removal Cu<sup>2+</sup>, Pb<sup>2+</sup> and Cr<sup>3+</sup>

ions from industrial wastewaters before transport and cycling into the nature environment [7].

There are various methods for removing heavy metals including chemical precipitation, membrane filtration, ion exchange, liquid extraction or electrodialysis [8]. However, these methods are not widely used due to their high cost and low feasibility for small-scale industries [9]. The widespread industrial use of low-cost adsorbents for wastewater treatment is strongly recommended at present, due to their local availability, technical feasibility, engineering applicability and cost effectiveness [10, 11]. Most agriculture wastes or byproducts are considered to be low value products. A large quantity of materials such as, rice straw [12], grape stalk wastes [13], maize cob [14], activated carbon [15, 16], banana peels[17], rice husks [18], chitosan [19], resin [20], dry plants[21], azolla filiculoides [22], nanoparticles [23], agricultural waste [24], oryza sativa husk [25] and sawdust [26, 27] has been investigated as adsorbents for removal of heavy metals from aqueous solutions.

The aim of the present investigation is to study the adsorption and removal of  $Cu^{2+}$ ,  $Pb^{2+}$  and  $Cr^{3+}$ 

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ions from aqueous solutions by olive leaves ash (OLA). Kinetic data and sorption equilibrium isotherms are carried out in batch process. The effect of different experimental conditions such as contact time, adsorbent mass, temperature and pH on the removal kinetics is studied. Equilibrium isotherm and kinetic data are analyzed and modeled using different models. The obtained results may contribute to a better understanding of the sorption phenomena at the liquid/solid interface.

### 2. Experimental

#### 2.1. Chemicals and methods

All reagents were of analytical grade and were used without further purification. All solutions for the metal sorption experiments were prepared using their nitrate salts purchased from Merck. Heavy metals analysis was carried out by using Shimadzu AA-680/G atomic absorption spectrophotometer.

#### 2.2. Characterization of OLA

In order to observe the surface morphology of the OLA, scanning electron microscopic (SEM) analysis was employed in this study. SEM images were recorded using AIS2100C SEI. Figure 1 shows a highly porous morphology of the OLA with pores of different sizes and shapes. The image also reveals that the external surface is full of cavities which suggest that OLA exhibits a high surface area and irregular in shape.



Figure 1. Scanning electron microscopic photograph of OLA

#### 2.3. Batch adsorption studies

The adsorption experiments were carried out in a series of 100 mL Erlenmeyer flasks of 100 mg  $L^{-1}$  metal ions solution at pH=5 containing 0.065 g OLA and If necessary, an appropriate volume of HNO<sub>3</sub> or NaOH solutions was used to adjust the pH of the solution. The solutions were shaken (150 rpm) at 25°C for 6 min. Then solid/liquid phases were separated by centrifuging and then filtration by Whatman filter paper. The removal percentage was calculated as:

$$\% \text{ Removal} = \frac{C_0 - C_e}{C_0} \times 100 \tag{1}$$

Where  $C_0$  and  $C_e$  are the initial and the final concentration of Cr(III), Cu(II) and Pb(II) ions in solution phase, respectively. All the adsorption experiments were carried out in triplicate and the average of at least three values is used everywhere in the text.

### 3. Results and discussion

#### 3.1. Effect of contact time

The effect of time on the removal of metal ions by OLA was studied. Figure 2 shows the removal of metal ions with contact time. It is clear that the removal efficiency of three ion reached a maximum value after 6 min and then no further significant increase was observed for contact time of up to 10 min. This may be due to the fact that initially all adsorbent sites were vacant and the solute concentration gradient was high. Therefore based on these results, a contact time of 6 min was selected in subsequent isotherm studies.



Figure 2. Effect of contact time on adsorption of  $Cr^{3+}$ ,  $Cu^{2+}$  and  $Pb^{2+}$  on Conditions: metal ions concentrations 100 mg  $L^{-1}$ , temp. 298 K and pH 5.

### 3.2. Effect of pH

The pH of the solution has a significant impact on the uptake of heavy metals, since it determines the surface charge of the adsorbent, the degree of ionization and speciation of the adsorbate. In order to establish the effect of pH on the adsorption of chromium (III), copper(II) and lead(II) ions, the batch equilibrium studies at different pH values were carried out in the range of 2–8. The pH of the metal ions solution was adjusted to different values by adding the required amount of dilute NaOH or HNO3 solutions. Figure 3 shows that the maximum percent removal of three ions on the adsorbents was observed at pH = 5.

Therefore pH=5 was considered as optimum condition and was used for further study for the mixture of heavy metals.



Figure 3. Effect of pH on adsorption of  $Cr^{3+}$ ,  $Cu^{2+}$  and  $Pb^{2+}$  onto OLA. Conditions:metal ions concentrations 100 mg  $L^{-1}$ , temp. 298 K

### 3.3. Effect of adsorbent dose

The effect of adsorbent dose on the adsorption of heavy metals was studied at contact time of 6 min for initial heavy metals concentration of 100 mg  $L^{-1}$ . The maximum adsorption of heavy metals was obtained for the adsorbent dose of 0.065 g  $L^{-1}$ (Figure 4). However, it is observed that after a dosage of 0.065 g  $L^{-1}$ , there was no significant change in percentage adsorption of heavy metals. It may be due to the overlapping of active sites at a higher dosage. There is a decrease in the effective surface area resulting in the conglomeration of exchanger particles. Therefore 0.065 g  $L^{-1}$  was considered as an optimum dose.



Figure 4. Effect of adsorbent dose on adsorption of  $Cr^{3+}$ ,  $Cu^{2+}$  and  $Pb^{2+}$  onto OLA. Conditions: soln concentration. 100 mg  $L^{-1}$ , temp. 298 K, pH 5.0

### 3.4. Effect of temperature

The adsorption of metal ions were carried out at six different temperatures 278, 288, 298, 308, 323 and 338 K using OLA as an adsorbent. The experimental results showed that the adsorption capacity decreases with increase of solution temperature. This indicates that the adsorption of metal ions on the adsorbent is an **e**xothermic process (Figure 5).



Figure 5. Effect of temperature on adsorption of  $Cr^{3+}$ ,  $Cu^{2+}$  and  $Pb^{2+}$ onto OLA Conditions: adsorbent dose 65 mg  $L^{-1}$ , soln concentration 100 mg  $L^{-1}$ , temp. 298 K and pH 5.5

### 3.5. Construction of isotherms and model fitting

Sorption isotherms were constructed by plotting the amount of metal sorbed (mg/g) against the equilibrium concentration of metal in solution (mg/L). Four models have been adopted in this paper, namely, the Freundlich [28], Langmuir [29], Tempkin [30] and Dubinin–Radushkevich (D–R)[31] equilibrium isotherm models.

### 3.6. Freundlich isotherm

The Freundlich isotherm is applicable to both monolayer (chemisorption) and multilayer adsorption (physisorption) and is based on the assumption that the adsorbate adsorbs on to the heterogeneous surface of an adsorbent. The linear form of Freundlich equation is expressed as:

$$\log q_e = \log K_F + \log C_e \tag{2}$$

where  $K_F$  and n are Freundlich isotherm constants related to adsorption capacity and adsorption intensity, respectively and  $C_e$  is the equilibrium concentration (mg L<sup>-1</sup>).

#### 3.7. Langmuir isotherm

The Langmuir isotherm assumes monolayer adsorption on a uniform surface with a finite number of adsorption sites. Once a site is filled, no further sorption can take place at that site. As such the surface will eventually reach a saturation point where the maximum adsorption of the surface will be achieved. The linear form of the Langmuir isotherm model is described as:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \tag{3}$$

where  $K_L$  is the Langmuir constant related to the energy of adsorption and qm is the maximum adsorption capacity (mg g<sup>-1</sup>).

The essential characteristics of Langmuir can be expressed in terms of a dimensionless equilibrium parameter, RL, which describes the type of isotherm and is defined by:

$$R_{L} = \frac{1}{1 + (b \times C_{0})}$$
(4)

Where b (l/mg) is the Langmuir constant and C<sub>0</sub>

(mg/L) is the initial concentration of Cr(III), Cu(II) and Pb(II) ions in solution. The parameter  $R_L$ shows the shape of isotherm according to Table 1. The values of  $R_L$  for different Cr(III), Cu(II) and Pb(II) initial concentrations are listed in Table 2. As it is clear from Table 1, all  $R_L$  values range between 0 and 1, indicating the favorable adsorption of Cr(III),Cu(II) and Pb(II) ions on to OLA (Figure 6).



Figure 6. Langmuir plots for the adsorption of  $Cr^{3+}$ ,  $Cu^{2+}$  and  $Pb^{2+}$  onto OLA .Conditions: metal ions concentration 100 mg  $L^{-1}$ , temp. 298 K and pH 5.5

Table 1. Effect of separation factor, RL on isotherm shape

RL value	Isotherm
RL>1	Unfavorable
RL = 1	Linear
0 < RL<1	Favorable
RL=0	Irreversible

Table 2. RL values based on Langmuir quation for Cr(III),Cu(II) and Pb(II) ions adsorbed on OLA  $% \mathcal{A}$ 

Ion	R <sub>L</sub> value
Cr <sup>3+</sup>	0.0182
$Cu^{2+}$	0.0127
$Pb^{2+}$	0.0434

The Tempkin isotherm model assumes that the adsorption energy decreases linearly with the surface coverage due toadsorbent–adsorbate interactions. The linear form of Tempkin isotherm model is given by the equation:

$$q_{e} = \frac{RT}{b} \ln K_{T} + \frac{RT}{b} \ln C_{e}$$
 (5)

where b is the Tempkin constant related to the heat of sorption(J  $mol^{-1}$ ) and KT is the Tempkin isotherm constant (L g<sup>-1</sup>).

### 3.8. Dubinin–Radushkevich (D–R) isotherm

The D–R isotherm model is a semi-empirical equation where adsorption follows a pore filling mechanism. It assumes that the adsorption has a multilayer character, involves van der Waals forces and is applicable for physical adsorption processes. The linear form of D–R isotherm model is expressed as:

$$lnq_e = lnq_d - \beta \epsilon^2$$
(6)

where  $q_d$  is the D–R constant (mg g<sup>-1</sup>),  $\beta$  is the constant related to free energy and  $\epsilon$  is the Polanyi potential which is defined as:

$$\varepsilon = \operatorname{RT} \ln \left[ 1 + \frac{1}{C_e} \right] \tag{7}$$

The correlation coefficients, R2, showed that the Langmuir isotherm model, an indication of an adsorptions mechanism, fits better the experimental data than the other isotherms model (Table 3).

### 3.9. Kinetic study

The dynamics of the adsorption process in terms of the order and the rate constant can be evaluated using the kinetic adsorption data. The process of Pb(II), Cu(II) and Cr(III) ions removal from an aqueous phase by adsorbent can be explained by using kinetic models and examining the rate-controlling mechanism of the adsorption process such as chemical reaction, diffusion control and mass transfer. The kinetic parameters are useful in predicting the adsorption rate which can be used as important information in designing and modeling of the adsorption operation. The kinetics of removal of metal ions is explicitly explained in the literature using Intra-particle diffusion, pseudo-first-order and pseudo-second-order kinetic models.

#### 3.10. Intra-particle diffusion model

In order to investigate the mechanism of the metals adsorption onto OLA, intra-particle diffusion based mechanism is studied. The most commonly used technique for identifying the mechanism involved in the adsorption process is, fitting an intra-particle diffusion plot. It is an empirically found functional relationship, common to the most adsorption processes, where uptake varies almost proportionally with t<sup>0.5</sup> rather than with the contact time t. According to the theory proposed by Weber

and Morris.

$$q_t = k_{\rm pi} t^{0.5} + C_i \tag{8}$$

Where $k_{pi}$ (mg g <sup>-1</sup> min <sup>-0.5</sup> ), the rate parameter	of
stage i, is obtained from the slope of the straight lin	ne
of $q_t$ versus t 0.5 whereas $C_i$ is the intercept of the	he

plot that gives an idea about the thickness of the boundary layer.

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Metal ions	Langmuir isotherm			Freundlich isotherm			Tempkin isotherm		D–R isotherm			
	q <sub>max</sub> (mgg <sup>-1</sup> )	K <sub>L</sub>	$\mathbb{R}^2$	1/n	$\mathbf{K}_{\mathrm{f}}$	$\mathbb{R}^2$	A <sub>T</sub>	$B_{T}$	$\mathbb{R}^2$	K <sub>D-R</sub> (mol <sup>2</sup> KJ <sup>-2</sup> )	$Q_{m}$	R <sup>2</sup>
Cr <sup>3+</sup>	142.85	0.538	0.995	0.235	46.618	0.791	653162	13.63	0.831	2×10 <sup>-6</sup>	132.29	0.686
$Cu^{2+}$	62.5	0.772	0.995	0.023	499.696	0.670	281.95	50.80	0.997	5×10-6	69.61	0.982
$Pb^{2+}$	90.909	0.22	0.997	0.307	1733.67	0.976	1136.8	33.03	0.947	6×10 <sup>-6</sup>	101.08	0.774

#### 3.11. Pseudo-first-order model

The sorption kinetics may be described by the pseudo-first-order Lagergren rate model. The equation is as follows:

$$\operatorname{Ln}(q_1 - q_t) = \ln q_1 - k_1 t \tag{9}$$

where  $q_1$  and  $q_t$  are the amounts of metals adsorbed on the sorbent(mg g<sup>-1</sup>) at equilibrium and at time t, respectively, and k1 is the rate constant of the first-order adsorption (min<sup>-1</sup>). The straight line plots of  $\ln(q_1-q_t)$  against t were used to determine the rate constant, k1and correlation coefficient, R<sub>2</sub> values of the metals were calculated from these plots (Figure. 7).



Figure 7. Pseudo-second-order model for the adsorptio n of  $Cr^{3+}$ ,  $Cu^{2+}$  and  $Pb^{2+}$  onto LA Conditions :conc = 100 mg L<sup>-1</sup>; contact time = 1-6 min.

The calculated correlation coefficients for pseudo-first-order were in the range of 0.942–0.984.

#### 3.12. Pseudo-second-order model

The pseudo-second-order rate equation shown as follows has been popularly applied to adsorption systems:

$$\frac{t}{q_t} = \frac{1}{K_2 q_2^2} + \frac{1}{q_2} t$$
(10)

where  $k_2$  is the rate constant of adsorption (g mg<sup>-1</sup> min<sup>-1</sup>), q<sub>2</sub> the amount adsorbed at equilibrium and qt is the amount adsorbed at any time. The

equilibrium adsorption amount  $(q_2)$  and the pseudo second-order rate parameters  $(k_2)$  can be calculated from the slope and intercept of plot of  $t/q_t$  versus t. The values of constants were shown in Table 4. The correlation coefficients,  $R_2$ , showed that the pseudosecond-order model, an indication of an adsorptions mechanism, fits better the experimental data than the pseudo-first-order model.

### 3.13. Study thermodynamically of adsorption on OLA

The effect of temperature, a major factor, influencing the adsorption was studied in the range of 303–363 K (Table5). The thermodynamic parameters, such as enthalpy ( $\Delta H^0$ ), entropy ( $\Delta S^0$ ) and Gibb's free energy ( $\Delta G^0$ ) were estimated using the following relation:

$$K_{c} = \frac{C_{a}}{C_{e}}$$
(11)

$$\Delta G^0 = -RT \ln K_c \tag{12}$$

Where  $K_c$  is the equilibrium constant, Ce the equilibrium concentration in solution (mg L<sup>-1</sup>) and Ca is the solid phase concentration at equilibrium (mg L<sup>-1</sup>). Standard enthalpy ( $\Delta H^0$ ) and entropy ( $\Delta S^0$ ) were determined from Van't Hoff equation.

$$\operatorname{Ln} K_{\rm c} = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}$$
(13)

 $\Delta H^0$  and  $\Delta S^0$  were obtained from slope and intercept of the plot Ln K<sub>c</sub> versus 1/T and presented in Table 4.Values of free energy changes G° are negative; confirming that adsorption onto OLA is spontaneous and thermodynamically favorable. The more negative values of  $\Delta G^0$  imply a greater driving force to the adsorption process. As the temperature increases, the  $\Delta G^0$  value decreases, indicating less driving force and hence resulting in lesser adsorption capacity at higher temperatures. The value of  $\Delta H^0$  is negative, indicating that adsorption process is exothermic in nature. The negative value of  $\Delta S0$  indicates the stability of sorption process with no structural change at solid–liquid interface.

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Metal ions	Intra-pai	Intra-particle diffusion			Pseudo First-order			Pseudo second-order			
	$\mathbf{k}_{\mathrm{pi}}$	$C_i$	$\mathbb{R}^2$	$\mathbf{q}_1$	$K_1$	$\mathbb{R}^2$	$q_2$	K <sub>2</sub>	$\mathbb{R}^2$		
				(mg g <sup>-1</sup> )	(min <sup>-1</sup> )		(mg g <sup>-1</sup> )	(g mg <sup>-1</sup> min <sup>1</sup> )			
Cr <sup>3+</sup>	51.58	40.1	0.986	213.364	0.883	0.942	200	0.004	0.994		
$Cu^{2+}$	29.06	81.71	0.996	72.024	0.432	0.984	166.66	0.012	0.997		
$Pb^{2+}$	32.43	81.55	0.987	107.339	0.749	0.966	166.66	0.012	0.998		

Table 4. Intra-particle diffusion, Pseudo-first-order and pseudo-second-order rate constants at 250C and different time

Table5. Thermody	namic parameters	for the adsorption	of Cr(III),Cu	u(II) and Pb(II)	ions onto OLA
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Ion	Temperature (K)	∆G° (kJ/mol)	ΔH° (kJ/mol)	$\Delta S^{\circ}$ (kJ/mol)	
Cr <sup>3+</sup>	303	-7.137	-32.599	-0.0843	
	313	-6.251	-32.599	-0.0843	
	323	-5.266	-32.599	-0.0843	
	333	-4.433	-32.599	-0.0843	
	348	-3.178	-32.599	-0.0843	
	363	-2.152	-32.599	-0.0843	
Cu <sup>2+</sup>	303	-3.858	-12.171	-0.0274	
	313	-3.589	-12.171	-0.0274	
	323	-3.295	-12.171	-0.0274	
	333	-3.008	-12.171	-0.0274	
	348	-2.700	-12.171	-0.0274	
	363	-2.167	-12.171	-0.0274	
$Pb^{2+}$	303	-1.684	-9.544	-0.0257	
	313	-1.497	-9.544	-0.0257	
	323	-1.201	-9.544	-0.0257	
	333	-1.007	-9.544	-0.0257	
	348	-0.580	-9.544	-0.0257	
	363	-0.241	-9.544	-0.0257	

### 4. Conclusion

The main objectives of this study were to study the simultaneous heavy metal ions removal using olive leaves ash as an adsorbent. The results suggest that OLA is highly efficient for simultaneous adsorption of heavy metal ions from aqueous solutions. The Langmuir, Freundlich and Tempkin adsorption models were used to express the sorption phenomenon of the sorbates. The equilibrium data were well described by the Langmuir model. In addition, thermodynamic studies also showed that the negative values of free energy changes ( $\Delta$ Go) and enthalpy changes ( $\Delta$ H<sup>o</sup>) for the adsorption which indicated that the metal ions adsorption onto OLA were a spontaneous exothermic process. The adsorption process is very fast and pseudo-secondorder rate model accurately describes the kinetics of adsorption.

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# جذب یون های فلزی سنگین بر روی خاکستر برگ زیتون: مطالعه ایزوترم و سینتیک

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### چکیدہ:

در این مقاله حذف یون های مس، سرب و کروم از محلول های آبی با استفاده از جاذب خاکستر برگ زیتون مورد بررسی قرار گرفت. همچنین تاثیر پارامترهای موثر بر فرآیند حذف شامل PH، زمان تماس، مقدار جاذب و دما مورد بررسی قرار گرفت. ایزوترم های جذبی لانگمویر، فرندلیچ و تمکین مطالعه شد. با بررسی ایزوترمهای جذب، بهترین انطباق نتایج تجربی با ایزوترم لانگمویر به دست آمد. مطالعات سینتیکی انجام شده بوسیله مدل های شبه درجه اول، شبه درجه دوم و نفوذ بین ذره ای مورد بررسی قرار گرفت که از میان آنها بهترین تطبیق دادهای تجربی با مدل سینتیک شبه درجه دوم بود. بر اساس مطالعات ترمودینامیکی، فرآیند از نوع گرمازا و به صورت خود به خودی بوده و مقادیر آنتالپی منفی به دست آمد.

**کلمات کلیدی:** یونهای فلزات سنگین، جذب سطحی، ایزوترم، سینتیک

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