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Removal of Pb(II), Zn(II), Cu(II) and Cd(II) from aqueous solutions by adsorption onto olive branches activated carbon: Equilibrium and thermodynamic studies

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ARTICLE INFO

Article type:
Research article
Article history:
Received November 2018
Accepted February 2019
January 2020 Issue
Keywords:
Olive branch
Heavy metals
Biosorption
Equilibrium
Thermodynamics

ABSTRACT

The equilibrium and thermodynamics of the biosorption of Pb(II), Zn(II), Cu(II), and Cd(II) onto activated carbon prepared from olive branches were studied under different parameters of pH, initial concentration, and temperature. The batch biosorption procedure was used to find the optimum conditions. The biosorption of each metal ion was found to be pH-dependent. The maximum metal ion biosorption was achieved at pH value 5 for Pb, Cu, and Cd ions and at pH 3 for Zn ions. The extent of the metal ion biosorption increased with temperature (indicating the endothermic character) and initial metal ion concentration. The experimental data of metal ion biosorption were analyzed by Freundlich and Langmuir isotherm models. For all metal ions, the Freundlich isotherm model gave a better fit with higher correlation (R^2) to equilibrium data than Langmuir model. The adsorption capacity values were 41.32, 34.97, 43.10, and 38.17 (mg/g) for Pb(II), Zn(II), Cu(II) and Cd(II), respectively. Thermodynamically parameters, like Gibbs free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) were calculated. The biosorption of each metal ion was non-spontaneous and the order of non-spontaneity of the biosorption process being Zn(II) > Cu(II) > Cd(II) > Pb(II). Likewise, change in entropy was noticed for each metal ion and the order of disorder was Pb(II) > Cd(II) > Cu(II) > Zn(II).

Capsule Summary: Activated carbon prepared from olive branches is considered to be a low-cost, high efficient, environmental friendly biosorbent and have removed more than 90% metal ions under optimized conditions.

Cite This Article As: A. M. Alkherraz, A. K. Ali, and K. M. Elsherif. Removal of Pb(II), Zn(II), Cu(II) and Cd(II) from aqueous solutions by adsorption onto olive branches activated carbon: Equilibrium and thermodynamic studies. Chemistry International 6(1) (2020) 11-20. https://doi.org/10.5281/zenodo.2579465

INTRODUCTION

Recently, due to fast industrialization, environmental contamination is being noticed around the world. The rate of waste and synthetic chemicals disposed into the aquatic environment is raised day by day (Alqadami et al., 2017). Contaminations by heavy metals have remarkably extended due to industrial flux of smelting, battery manufacturing, mining, tanneries, metallurgical, municipal waste generation, pesticide and fertilizers applications (da Silva et al. 2016; Han, 2016; Pugazhendhi et al. 2018). In the globe, heavy metals are utilized in various industrial sectors due to which metals which are extremely toxic pollutant to the environment are released as waste (Bhateria and Dhaka, 2017). The prevalent heavy metal contaminants specifically, lead, cadmium, copper, arsenic, mercury, nickel, etc., are non-biodegradable, considerably toxic to humans and also leads to ecological imbalances (Benvenuti et al. 2017; Cardoso et al. 2017). These metals can reach the human body and results in brain and bone damage, nervous system damage, neurological disorders and even cancer (Rao and Khatoon, 2017). Various methods are used to remove heavy metals from industrial waste water, such as: solvent extraction, biological treatment, ionic exchange, adsorption on activated carbon, reverse osmosis, electrolytic methods and membrane filtration (Dursun et al. 2005; Elsherif et al., 2013a; 2013b; 2014a; 2014b; 2014c; Elsherif and Yaghi, 2016; 2017a; 2017b). Among these methods, the adsorption method is the simplest, cheapest, and fastest one for heavy metals removal and also applicable for lower concentration levels (Rodrigues et al., 2013; do Nascimento et al., 2019; Jaafari and Yaghmaeian, 2019; Londono-Zuluaga et al., 2019; Massoud et al., 2019; Moreira et al., 2019; Peng et al., 2019; Yin et al., 2019). Activated carbon is considered to be an effective method for heavy metals removal from polluted waste waters due to its higher surface area, microporosity, and high adsorption capacity (Hameed, 2008; Rodrigues et al. 2011). Activated carbon obtained from biosorbents has the desired surface area and pore volume and accordingly, it has emerged as a potential alternative for activated carbon production (de Sousa Ribeiro et al., 2017).

In our previous work, we used olive leaves, coffee, tea, and orange peels powders as biosorbents for removing heavy metals from aqueous solutions (Elsherif et al., 2017a; 2017b; 2018a; 2018b; 2019). In the present study, factors affecting the biosorption efficiency of Pb(II), Cd(II), Zn(II), and Cu(II) onto activated carbon prepared from olive branches have been studied. Equilibrium studies using various models have been also investigated. Also, thermodynamic parameters of the system have also been determined.

MATERIAL AND METHODS

Chemical and reagents

Lead(II) Nitrate, ACS Reagent, ≥99%, from Honeywell Fluka, Zinc(II) Nitrate Hexahydrate, 98%, Reagent grade, from Honeywell, Copper(II) Nitrate Hemi(Pentahydrate), 98.0 to 102.0%, ACS Reagent, from Honeywell Fluka, Cadmium(II) nitrate tetrahydrate, 98%, ACS Reagent, from Merck, were used to prepare 1000 mg/L stock solutions of metal ions and using double distilled water. Desired test solutions of metal ions were prepared using appropriate subsequent dilutions of the stock solution. The range of concentrations of metal ions prepared from standard solution varies between 50 and 750 mg/L. Before mixing the adsorbent, the pH of each test solution was adjusted to the required value with 0.1 M NaOH or 0.1 M HCl. Sodium hydroxide, BioXtra, ≥98% (acidimetric), pellets (anhydrous), from Merck and hydrochloric acid, ACS reagent, 37%, from Merck were used to prepare these solutions.

Preparation of activated carbon

Olive branches were collected from Misurata city in Libya. It was cleaned, dried, ground, activated with H_3PO_4, and then carbonized using the procedure reported by Isah et al. (Isah et al, 2015). The product was washed and filtered several times with distilled water and then subsequently dried in an oven at 70°C for 24 h.

Analysis

The concentrations of Pb (II), Cd (II), Zn (II), and Cu (II) ions in the solutions before and after equilibrium were determined by atomic absorption machine Shimadzu AA7000. The pH of the solution was measured with pH Meter 3505 from JENWAY.

Biosorption experiments

Batch biosorption experiments were conducted by mixing biosorbent with metal ion solutions with desired concentration in 250 mL conical flasks. The conical flasks were stoppered during the equilibration period and placed on a temperature controlled shaker at a speed 150 r/min. The amount of biosorption was calculated based (Eq. 1) on the difference between the initial (Co, mg/L) and final concentration (Ce, mg/L).

$$Q_e = \frac{C_o - C_e}{M} . V \quad (1)$$

Where, Q_e is the metal uptake capacity (mg/g), C_o is the initial concentration, C_e is the final concentration, V the volume of the metal solution in the conical flask (L) and M is the dry mass of biosorbent (g). The biosorption efficiency % E was calculated as shown in Eq. 2.

$$\% R = \frac{C_o - C_e}{C_o} . 100 \quad (2)$$

Effect of concentration

The influence of initial metal ion concentration ranging from 50 to 1000 ppm was investigated by contacting a fixed biosorbent mass 1.0 g with 50 mL of aqueous solutions at 303 K for 24 hours.

Effect of pH

Metal ion solutions (50 ml) were taken in four separate conical flasks. The pH of solutions was maintained at pH 2, pH 3, pH 5, pH 7, and pH 10. Olive branches activated carbon 1.0 g was added to each flask and the solutions were stirred for 24 hours at 303 K. After the adsorption test each solution was filtered and the metal ion concentration was checked by AAS.

Effect of biosorbent particle size

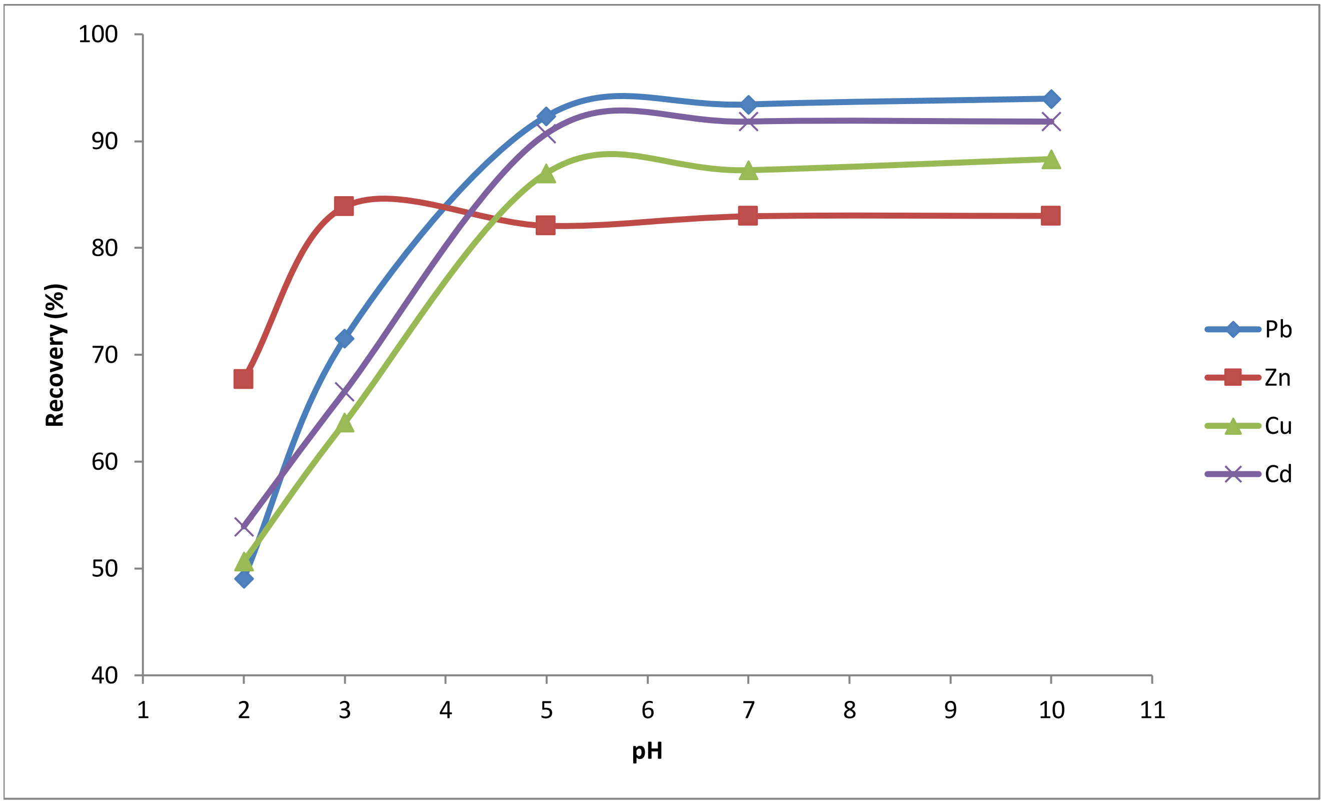

Fig. 1: Effect of pH on Pb, Zn, Cu and Cd biosorption

Adsorption experiments with activated carbon prepared from Olive branches of different particle size were studied. For that purpose, the particle size of 125, 500, 800 µm have been used. Also, three concentrations, 1000, 500, 250 ppm, of the elements were tested with each particle size. The adsorbent dose and solution volume were 1.0 g and 50 ml, respectively.

Effect of temperature

The effect of temperature on the adsorption of Pb, Zn, Cu, and Cd was studied by contacting 1.0 g of adsorbent with 50 mL of metal solution of 100 ppm initial concentration at different temperatures (303, 313, and 323 K).

RESULTS AND DISCUSSION

Effect of pH

The influence of hydrogen ion concentration is one of the most important parameter which has to be investigated in the removal of heavy metals. pH affects the solubility of metal ions and also the functionalization of the microbial adsorbent surface. Figure 1 displays that at extremely acidic solutions (pH < 2.0), the removal of metal ions was minimal. As the pH value was raised from 2.0 to 5.0, there was an increase in metal ion uptake and the maximum removal of Pb, Cu, and Cd was achieved at pH 5.0 and for Zn at pH of 3.0. When pH was further increased, the metal ions uptake remained mostly unchanged. However, the speciation of the metal ions is considerably altered with change in pH which in turn impacts the removal capacity. From the results, it is shown that biosorption of metal ions is pH dependent and affects the removal capacity of the biosorbent. At lower pH values, functional groups such as carboxylate on biosorbent wall will be protonated and the surface will be positively charged and consequently the H^+ ions will compete with metal ions, thereby leading to minimal biosorption of metal ions. This low biosorption is also due to the repulsion between positively charged H^+ ions and metal ions. As the pH raises, repulsions between the H^+ ions and metal ions decreases which leads to more metal uptake at the biosorption site. And also, as pH raises, the number of negatively charged sites increases resulting in gradual increase in the biosorption of metal ions.

Effect of biosorbent particle size

The effect of particle size as presented in Figure 2 showed that the metal ions uptake decreased as the particle size increases. The effect of particle size of activated carbon prepared from Olive branches was examined for three metal ion concentrations which were 1000, 500, and 250 ppm.

Table 1: Langmuir and Freundlich isotherms parameters

Ion metal	Pb	Zn	Cu	Cd
		Freundlich		
R^2	0.992	0.993	0.985	0.998
K_F	1.31	1.23	1.15	1.39
n	1.70	1.73	1.53	1.85
		Langmuir		
R^2	0.979	0.926	0.995	0.945
K_L (L/mg)	0.013	0.007	0.009	0.012
Q_m (mg/g)	41.32	34.97	43.10	38.17

Table 2: Thermodynamic parameters for the biosorption of metal ions onto Olive branches activated carbon

Metal ion	T, K	ΔG°, kJ/mol	ΔH°, kJ/mol	ΔS°, J/mol.K	R^2
	303	1.29			
Pb	313	0.80	16.25	49.36	0.995
	323	0.31			
	303	3.37			
Zn	313	3.17	9.38	19.84	0.871
	323	2.97			
	303	2.9			
Cu	313	2.68	9.43	21.56	0.954
	323	2.47			
	303	1.85			
Cd	313	1.48	12.85	36.31	0.967
	323	1.12			

Effect of metal ion concentration (Biosorption Isotherms)

The influence of initial metal concentration on biosorption is ordinarily investigated to find out the dependent of the initial metal ions concentration in aqueous phase and the metal biosorption capacity of biosorbent. The uptake of metal ions is dependent on initial concentration and it increases with increasing in initial concentration as shown in Figure 3. This change in biosorption at different concentrations of metal ions is due to the enhancement in the motivating gradient force. At higher concentrations, there were a greater driving force to transport the metals. But at extremely high concentration of metals, the binding sites will be saturated. It is substantial to find out the maximum biosorption capacity of biosorbent, so as to conduct the experiment at the highest possible initial metal concentration. Hence the initial concentration of metal ions supplies a driving force and also reduces the mass transfer resistance for allowing metal biosorption.
However, the equilibrium data obtained was analyzed using Langmuir and Freundlich adsorption isotherm model equations (Elsherif et al, 2018a; 2018b) and the plots are presented in Figures 4 and 5. The Eqs. 3-4 are expressing the Langmuir and Freundlich isotherms, respectively.

$$\frac{C_e}{Q_e} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m} \quad (3)$$

$$Log Q_e = Log K_F + \frac{1}{n} Log C_e \quad (4)$$

Where, Q_e represents metal uptake capacity in mg/g, Q_m represents maximum metal uptake capacity in mg/g, C_e represents equilibrium concentration in mg/L; K_L and K_F are the constants related to its intensity and n is a measure of feasibility of the adsorbents. The results obtained from the modeling showed that the removal of Pb, Zn, Cu, and Cd ions using activated carbon prepared from Olive branches followed Freundlich adsorption equation with higher correlation coefficients (R^2) of about 0.99 for all metal ions. The implication of Langmuir correlation revealed that the Cu(II) ion has a maximum adsorption capacity while the Zn(II) ion has the lowest. Generally, the result showed that the interaction between the metal ions and the surface of the Olive branches activated carbon was indicative of a physical type of adsorption. The parameters for these models are presented in Table 1.

Effect of temperature (Thermodynamic parameters)

Temperature is a more significant consideration which affects the biosorption of heavy metals. The biosorption of metal ions onto olive branch activated carbon increased with increase in temperature from 303 to 323 K (Fig. 6).

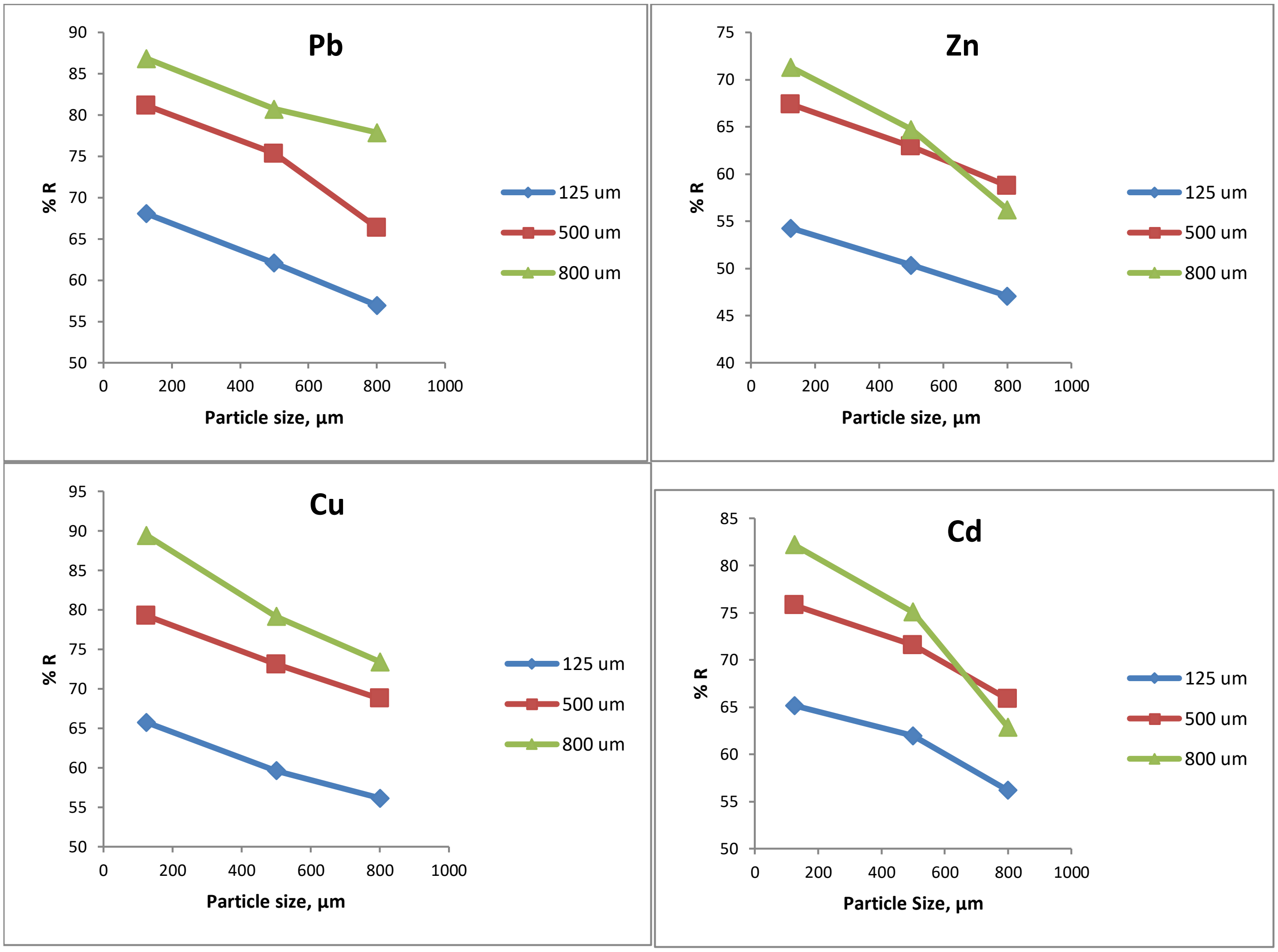

Fig. 2: Effect of particle size on Pb, Zn, Cu, and Cd biosorption (R = recovery)

Increasing in adsorption at higher temperature values is due to the strengthening of the interaction between the metal ion and active sites. Moreover, higher temperatures will also motivate more mobility in the particles and reduces the liquid viscosity. Conventionally, the adsorption process takes place in two accompanying processes, which will be a fast diffusion and slow complexation. The increase in temperature will not only speeds up the diffusion rate of metal ions from solution to the surface of biosorbent, but also accelerates the complexation of metal ions with the functional groups of the biosorbent (Ding et al., 2019; Gupta et al., 2019; Hadiani et al., 2019; Noormohamadi et al., 2019; Que et al., 2019; Rai et al., 2019; Sedlakova-Kadukova et al., 2019; Wu et al., 2019; Xu et al., 2019). In order to describe thermodynamic behavior of the biosorption of metal ions onto olive branches activated carbon, thermodynamic parameters including the change in free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) were calculated using relation shown in Eq. 5 (Farhan et al., 2012).

$$\Delta G^O = -RT \ln K_D \qquad (5)$$

Where, R is the universal gas constant (8.314J/mol K), T (K) is the temperature and K_D is the distribution coefficient. According to thermodynamics, the Gibb's free energy change is also related to the enthalpy change (ΔH°) and entropy change (ΔS°) at constant temperature by the Van't Hoff equation (Farhan et al., 2012).

$$\Delta G^O = \Delta H^O - T\Delta S^O \qquad (6)$$

Equations (5) and (6) can be written as:

$$\ln K_D = -\frac{\Delta H^O}{RT} + \frac{\Delta S^O}{R} \qquad (7)$$

According to the equation 5, the values of enthalpy change (ΔH°) and entropy change (ΔS°) were calculated from the slope and intercept of the plot of ln K_D vs. 1/T (Fig. 7). The calculated values of thermodynamic paramours (ΔG°, ΔH°, and ΔS°) for the biosorption of metal ions onto olive branches activated carbon are reported in Table 2.

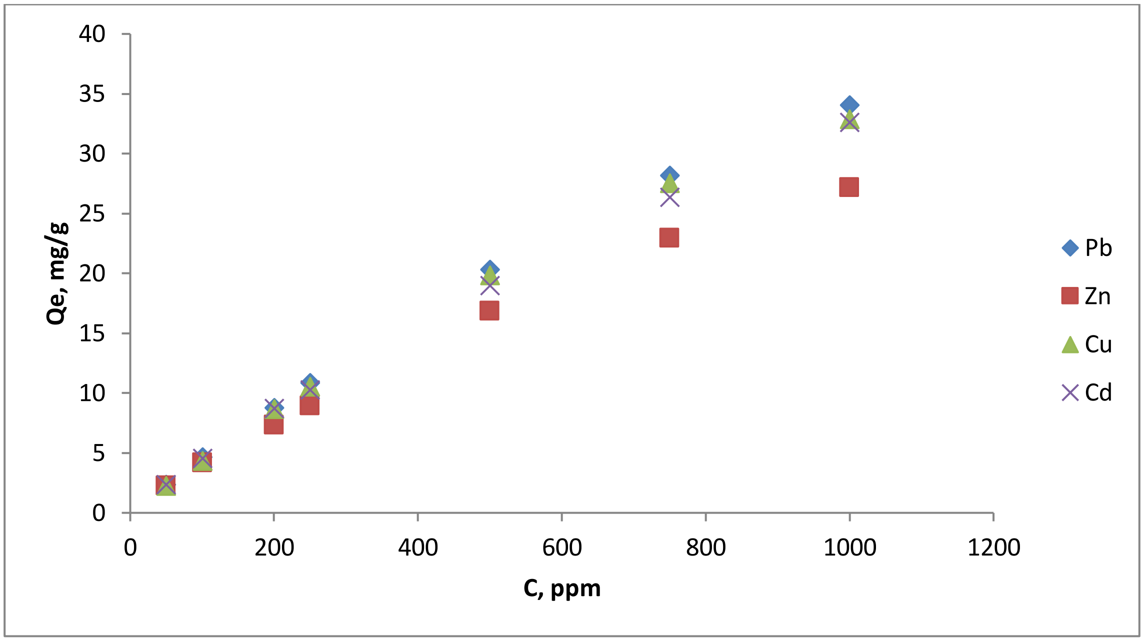

Fig. 3: Effect of initial concentration on metal ion biosorption

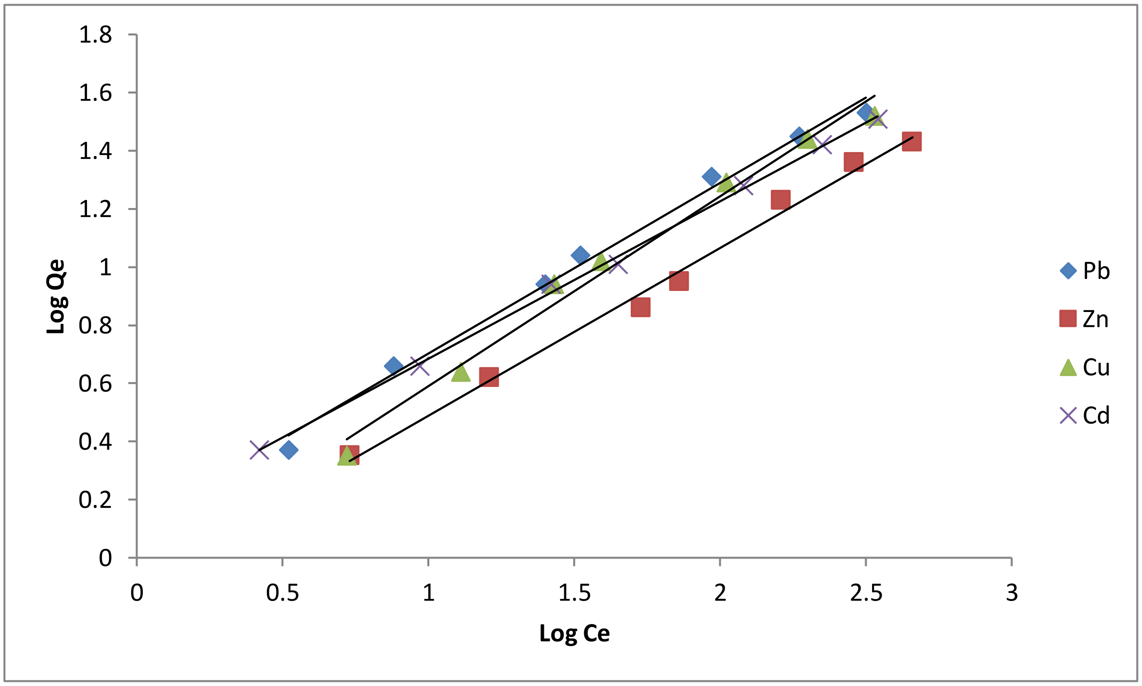

Fig. 4: Equilibrium studies of metal ions biosorption onto Olive branches activated carbon (Freundlich isotherm)

A positive value of the free energy (ΔG°) indicated the non-spontaneous nature of the biosorption process. The physical adsorption the free energy change (ΔG°) ranges from (−20 to 0) kJ/ mol and for chemical adsorption it ranges between (−80 and −400) kJ/ mol.

The ΔG° (Lohaniet al., 2008), for metal ions adsorption onto olive branches activated carbon was in the range of (0.80 to 3.37) kJ/mol and so the adsorption was more likely a physical adsorption. A positive value of ΔS^{o} as 19.84-49.36 J/mol.K showed increased randomness at solid solution interface during the adsorption of metal ions (Cataldo et al., 2018; Marciniak et al., 2019; Zhuang et al., 2019).

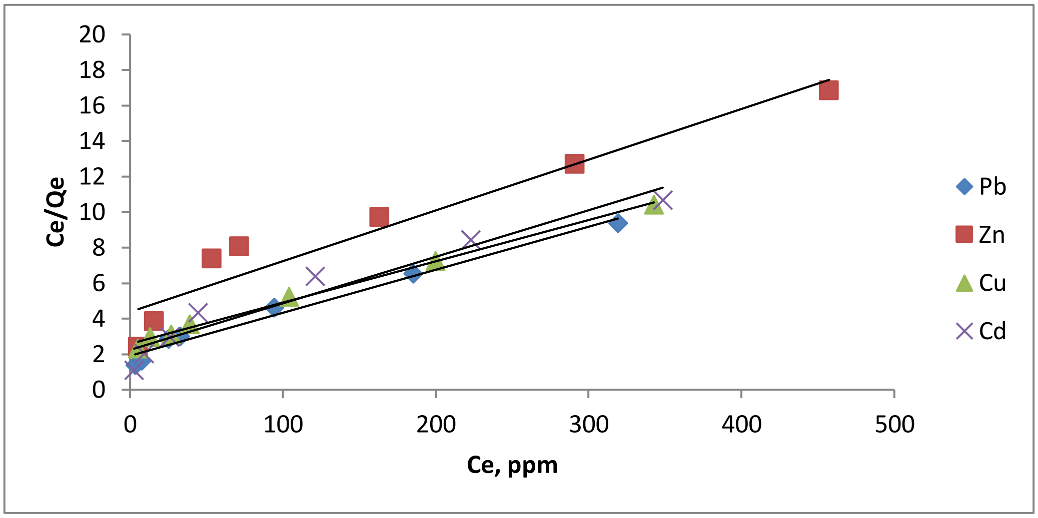

Fig. 5: Equilibrium studies of metal ions biosorption onto Olive branches activated carbon (Langmuir isotherm)

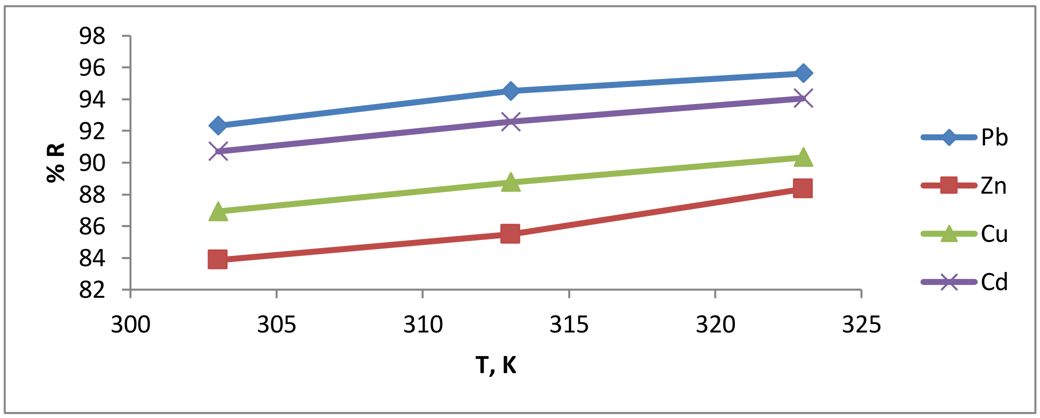

Fig. 6: Effect of temperature on biosorption efficiency of metal ions

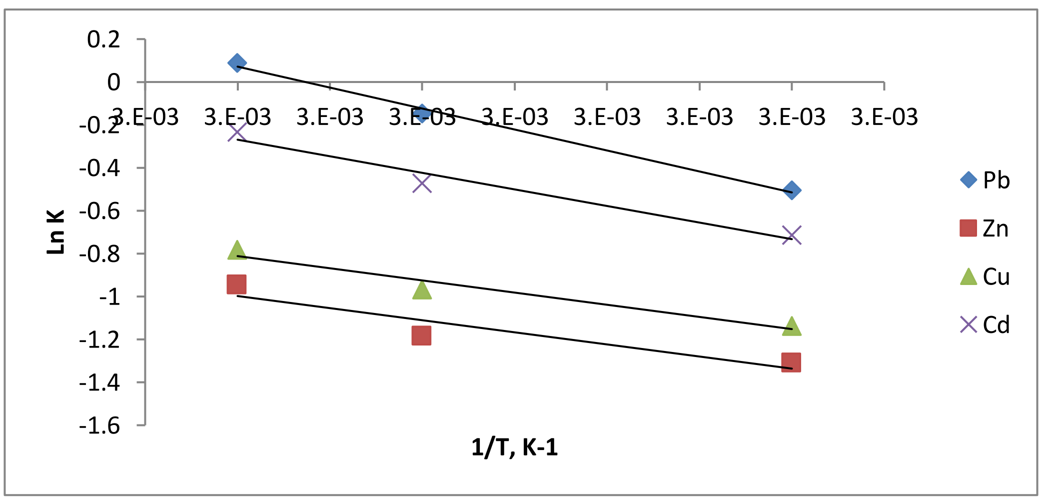

Fig. 7: Plots of In Ka against 1/T for metal ions biosorption

CONCLUSIONS

The biosorption of Pb(II), Zn(II), Cu(II) and Cd(II) by activated carbon prepared from olive branches under several circumstances was investigated. The pH has the most influence on the biosorption of these metal ions from aqueous solutions. The biosorption isotherms of the metal ions onto the olive branches activated carbon are well described by Freundlich isotherm model. The thermodynamic study demonstrated that the biosorption of all metal ions was non-spontaneous. The present study reveals that olive branches activated carbon has high potential for handling industrial waste waters containing heavy metals.

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