



## Simultaneous removal of copper and lead ions from a binary solution by sono-sorption process

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### ABSTRACT

The aim of this work is to compare the simultaneous sorption of copper and lead ions from a binary aqueous solution in the presence and in the absence of ultrasound. The experiments under sonication were carried out by 20-kHz apparatus. Results indicated that the removal of copper and lead ions from a binary aqueous solution was greater in the presence of ultrasound than in control method. The removal of these ions was examined by varying experimental conditions such as the amount of sorbent, contact time, and temperature. In addition, the competitive sorption of ions was considered with different concentrations of each ion under the constant total concentration. The Langmuir isotherm model fits adequately the experimental data. In point of kinetics, the second-order kinetic model describes the sorption process for both ions. It was found that more than 90% of the lead and 60% of the copper ions was removed in less than 2 min from the solution under sonication. The thermodynamic parameters such as  $\Delta G^\circ$ ,  $\Delta H^\circ$ , and  $\Delta S^\circ$  were determined for the sorption of  $\text{Cu}^{2+}$  and  $\text{Pb}^{2+}$  from the temperature dependence of the sorption process.

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### 1. Introduction

The presence of metal ions such as copper and lead in aquatic environment is of great concern due to their toxicity to many life forms. The increased use of these ions in various industries such as mining, ore processing, smelting and metal plating has resulted in  $\text{Cu}^{2+}$  and  $\text{Pb}^{2+}$  finding their way to natural bodies of water [1]. Lead is a very toxic element which can damage the nervous system, kidneys and reproductive system, specially in children. Copper in high doses can cause toxic effect like other heavy metals, but it is essential in trace amount for the body. The EPA requires lead and copper in drinking water not to exceed 0.015 and 1.3  $\text{mg L}^{-1}$ , respectively [2]. So, the elimination of these metals is important to protect health. Sorption process has been used for the removal of ions, colors, bacteria, and organic compounds from industrial process or waste effluents. Activated carbons are the most widely used sorbents because they have excellent sorption capabilities for the aforementioned pollutants [3]. However, the use of granular or powdered activated carbons is often suffered from the relatively slow sorption rate due to their micro-porous structures and long diffusion path. This has led to the possible use of kinetically improved but more expensive alternative [4]. A wide variety of materials such as rice husk [5,6], fly ash [7], pine bark [8], saw-

dust [9], animal bone [10], nut walnut shells [11], tree barks [12], lignin, chitin, chitosan, peat moss, modified wool, and modified cotton [13] are examples of low-cost materials used in the removal of ions. Most of these sorbents contain functional groups including proteins, polysaccharides and cellulose as major constituents. Metal ion sorption can occur through functional group mentioned [14].

Ultrasound is the wave at a frequency above 16 kHz. When ultrasound is irradiated through a liquid, an alternating adiabatic compression and rarefaction cycle of the medium occur [15,16]. The so-called cavitation is the formation, growth, and sudden collapse of the micro-bubbles in liquids which are formed in the rarefaction cycle of the ultrasonic wave when a large negative pressure is applied to a liquid. The critical size of the bubble depends on the liquid and the sound frequency, at 20 kHz, for example, it is roughly 100–170  $\mu\text{m}$  [15,16]. Ultrasonic vibration reduces the thickness of liquid films attached to the solid phase and enhances mass transfer. Therefore, the diffusion of dissolved species in liquids through porous media will be enhanced by ultrasound [17,18]. In the past decade, several researchers have studied the role of ultrasound on the adsorption and/or desorption processes [19–26]. For example, Rege et al. [20] reported that the desorption rate of activated carbons significantly increase with ultrasound and the ultrasonic desorption rate is favored by decreasing the temperature, aerated liquid medium, and increase ultrasonic intensity. For the sorption in a batch system, Schueller and Yang [21] found that ultrasound act like a mixer, improving the mass transfer coefficients

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through cavitation and acoustic streaming. It is well understood that ultrasonic wave have a greater efficiency for interface mixing than control agitation [27,28]. This behavior could be the reason for the enhancement of the sorption kinetic process [22]. Acoustic cavitation provides unusual conditions in the sorption process which are different from those of control method for the removing of pollutants [29–34].

The sorption process is an easy method for the removal of pollutants and finding a cheap sorbent with high capacity of removal is very important. In addition, the diffusion of species has a critical role in the sorption process. Therefore, combination of the two mentioned points was considered in this study. The corm of Saffron was used as a natural sorbent for the simultaneous removal of copper and lead ions from an aqueous binary solution in the presence and absence of ultrasound.

## 2. Materials and methods

### 2.1. Materials

All chemicals and reagents used for experiments and analyses were of analytical grades. Stock solution of  $1000 \text{ mg L}^{-1}$  of  $\text{Cu}^{2+}$  and  $\text{Pb}^{2+}$  were prepared from  $\text{CuCl}_2$  and  $\text{Pb}(\text{NO}_3)_2$  in de-ionized water. This solution was diluted as required to obtain the standard solutions.

Saffron is a plant which grows in north-east of Iran. We used corm of saffron as a natural sorbent for the simultaneous removal of copper and lead ions from a binary solution. It was washed with distilled water to remove soluble impurities. Then, the sample was dried in an electric oven for 24 h at  $200^\circ\text{C}$ .

### 2.2. Apparatus

The ultrasonic irradiation was carried out with equipment operating at 20 kHz (Branson Digital sonifier, Model W-450D). The acoustic power was set at 25% position ( $33 \text{ W/cm}^2$ ) and the tip diameter of horn was 1.1 cm. The reactor was a double cylindrical jacket and the temperature was controlled by circulating water through the cylindrical jacket. Experiments without ultrasound (control method) were performed in a batch reactor with stirring at 300 rpm.

Atomic absorption spectrophotometer (Varian, spectra-110-220/880 Australia Pty Ltd.) equipped with a Zeeman atomizer was used to determine the concentration of the ions in the solution.

### 2.3. Procedure

Single ( $200 \text{ mg L}^{-1}$ ) and binary uptake of  $\text{Cu}^{2+}$  and  $\text{Pb}^{2+}$  ions ( $100 \text{ mg L}^{-1}$ , with respect to each ion) by the sorbent from aqueous solutions were studied by both methods at constant temperature ( $30^\circ\text{C}$ ). A known quantity of 0.2 g sorbent was added into 50 mL aqueous solution and the contact time of 10 min was selected for single and binary solutions. Following the sorption process, the aqueous phase was separated from the sorbent and the final concentration of the metal ions in the solutions was determined by AAS.

The uptake of the metal ions in single and binary solutions was calculated by the difference in their initial and final concentrations. Each experiment was repeated two times and the results given were the average values. The obtained data were used to calculate the equilibrium metal uptake capacity for each ion according to the Eq. (1).

$$q_e = \frac{V(C_0 - C_e)}{m} \quad (1)$$

where  $q_e$  ( $\text{mg g}^{-1}$ ) is the amount of sorption of ion in the sorbent phase at equilibrium,  $C_0$  and  $C_e$  the initial and equilibrium concen-

trations of metal ion ( $\text{mg L}^{-1}$ ) in the aqueous solution,  $V$  the volume of solution (L), and  $m$  is the weight of sorbent (g) in the mixture.

## 3. Results and discussion

### 3.1. Amount of sorbent

The amount of sorbent was varied in the range of 0.1–0.4 g for the simultaneous removal of  $\text{Pb}^{2+}$  and  $\text{Cu}^{2+}$  from the aqueous solution by the sorption process, in the presence and in the absence of ultrasound. In these series of experiments the concentration of  $\text{Pb}^{2+}$  and  $\text{Cu}^{2+}$  in binary solution were fixed at  $100 \text{ mg L}^{-1}$  for each ion, and the contact time was 10 min. As it was shown in Fig. 1, by increasing the mass of sorbent the amount of the removal was also increased which is due to the increase of the sorption sites. In both control and sonicated methods the removal of lead ion was faster than copper ion. This means that the sorbent used is more favorable for the lead ion than copper ion. For both ions, the removal was higher in the presence of ultrasound. This is due to the cavitation process which increased the diffusion process by the micro-jet and streaming produced in the collapse of the cavity. According to Fig. 1, the proper amount of sorbent for this removal was about 0.2 g.

### 3.2. Contact time

The removal of  $\text{Cu}^{2+}$  and  $\text{Pb}^{2+}$  from single and binary solutions as a function of time are presented in Figs. 2 and 3, respectively. It can be seen that the adsorbed amount of single and binary metal ions increased from 30 s to 2 min and then reached to the maximum and constant removal. Therefore, 2 min were selected as a suitable contact time for both metal ions in the single and binary solutions.

The removal of lead and copper ions was higher in the presence of ultrasound than its absence. In sonication, the lead ion almost removed near completion in 30 s. This short time of the removal indicates that a high interaction of sorbent with pollutant exists especially in the presence of ultrasound. It is assumed that the shear forces generated in the medium due to the cavitation process may be the reason for the higher removal rate.

In control method, for a single component the removal was about 27% for copper ion and 70% for lead ion and in average it comes to about 48% which is lower than 64% removal for two components solution. In the case of sonication, for a single component the removal was about 34% for copper ion and about 86% for lead ion and in average it comes to about 60% which is also

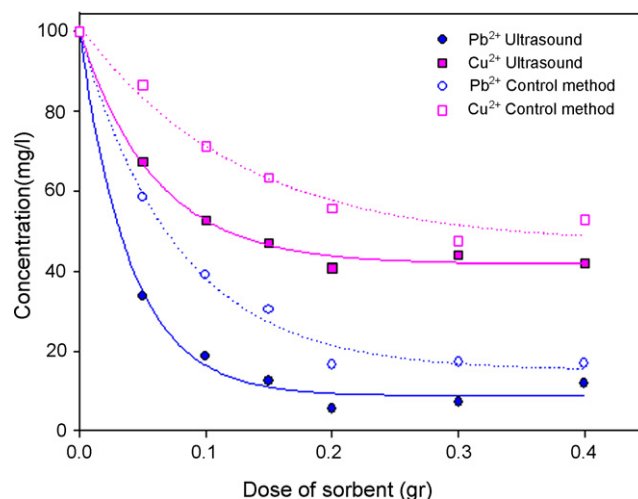
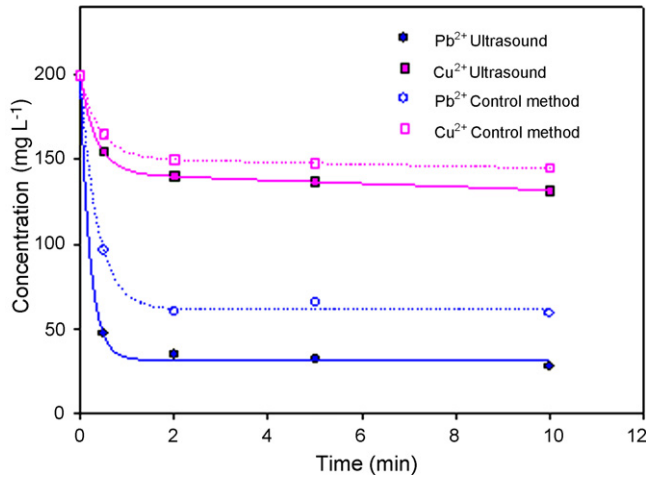


Fig. 1. Effect of amount of sorbent on the simultaneous removal of lead and copper ions (stirring speed: 300 rpm; initial concentration:  $100 \text{ mg L}^{-1}$ ; temperature:  $30^\circ\text{C}$ ; time: 10 min).



**Fig. 2.** Effect of contact time on the sorption of lead and copper ions separately (stirring speed: 300 rpm; amount of sorbent: 0.2 g; initial concentration: 200 mg L<sup>-1</sup>; temperature: 30 °C).

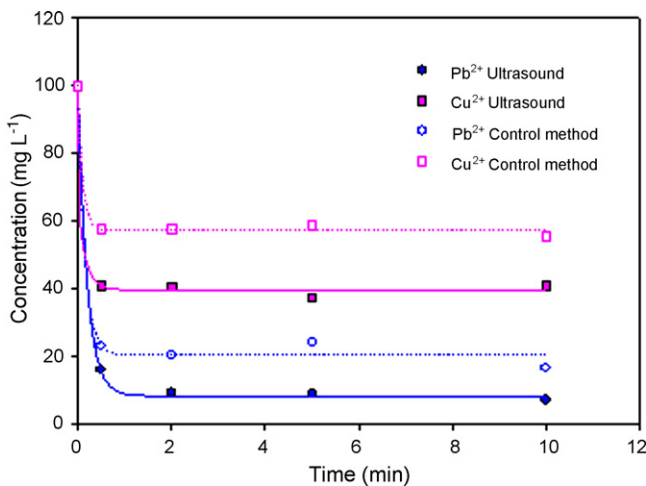
lower than 76% removal for two components solution. Therefore, in two-component system, the species with higher removal efficiency (lead ion) facilitates the removal of species with lower efficiency (copper ion).

3.3. Sorption isotherm

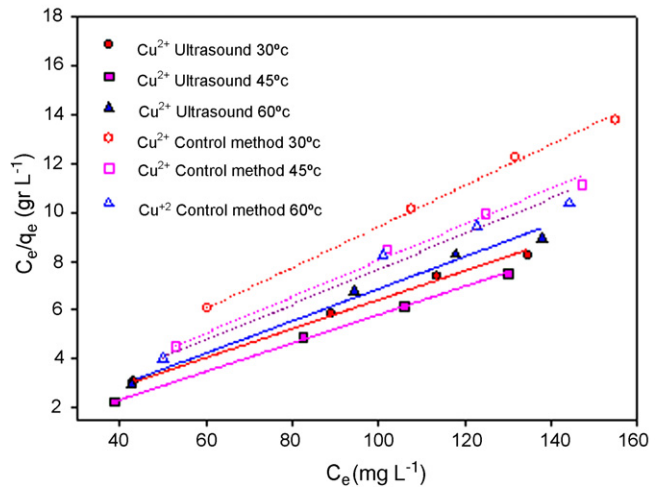
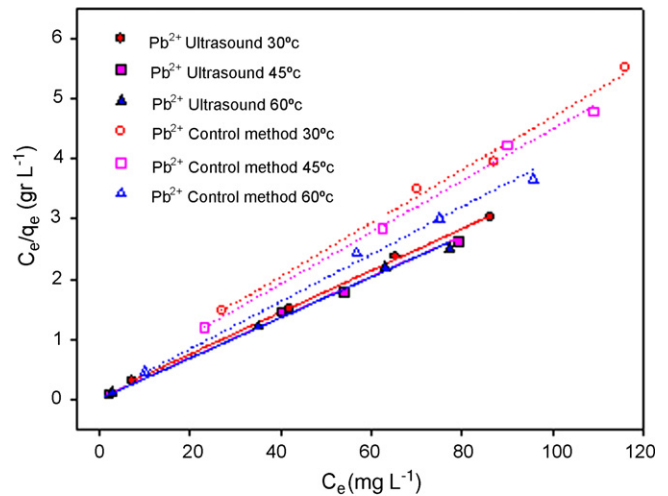
The experimental data were applied to different sorption models such as Langmuir, Freundlich, and Temkin. The Langmuir model was fitted properly with high-correlation coefficients (*R*<sup>2</sup>) in the applied range of initial concentration (100, 150, 175, 200 mg L<sup>-1</sup>) in comparison with other sorption models. The linear form of the Langmuir model is represented by Eq. (2).

$$\frac{C_e}{q_e} = \left(\frac{1}{q_m b}\right) + \left(\frac{1}{q_m}\right) C_e \quad (2)$$

where *q<sub>e</sub>* is the amount of solute adsorbed per unit weight of sorbent at equilibrium (mg g<sup>-1</sup>), *C<sub>e</sub>* the equilibrium concentration of solute in the bulk solution (mg L<sup>-1</sup>), *q<sub>m</sub>* corresponds to complete coverage of available sites or the maximum amount of metal ions adsorbed per unit weight of sorbent (mg g<sup>-1</sup>), and *b* (L mg<sup>-1</sup>) is the equilibrium adsorption constant.



**Fig. 3.** Effect of contact time on the binary sorption of lead and copper ions (stirring speed: 300 rpm; amount of sorbent: 0.2 g; initial concentration: 100 mg L<sup>-1</sup>; temperature: 30 °C).



**Fig. 4.** (a) Langmuir adsorption isotherms for lead in the presence and absence of ultrasound. (b) Langmuir adsorption isotherms for copper in the presence and absence of ultrasound.

Fig. 4a and b confirm that the sorption of Pb<sup>2+</sup> and Cu<sup>2+</sup> from the binary solution by the sorbent at different temperatures in the presence and absence of ultrasound was obeyed the Langmuir model. The sorption capacity of metal ions was found to be greater in the presence of ultrasound than control method. This behavior could be explained by the acoustic cavitation (formation, growth, and collapse of the cavity) which produced during the sonication. Tables 1a and 1b summarize the Langmuir constants for the uptake of both ions in the presence and in the absence of ultrasound at different temperatures. The *R<sub>SQR</sub>* values indicate that the Langmuir model is a proper model for this sorption. The value of *q<sub>m</sub>* which represents the monolayer saturation at equilibrium increased with increasing the temperature in control method. It may be attributed to the activation of the sorbent surface. In sonicated method, the

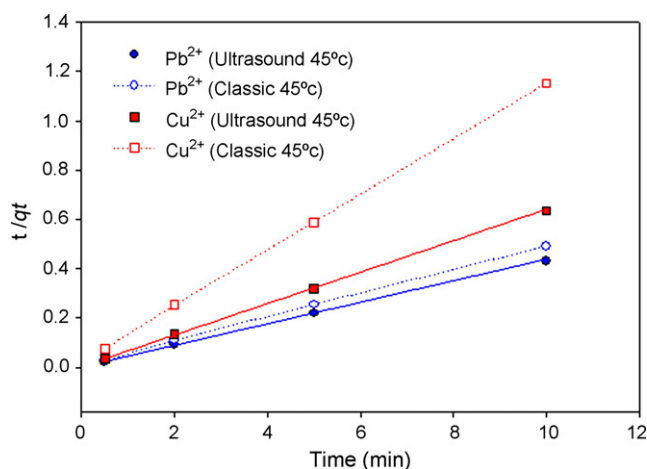
**Table 1a**  
Langmuir constants for the uptake of lead ion in the presence and absence of ultrasound

<i>t</i> (°C)	Sonicated method			Control method		
	<i>q<sub>m</sub></i> (mg g <sup>-1</sup> )	<i>b</i> (L mg <sup>-1</sup> )	<i>R<sub>SQR</sub></i>	<i>q<sub>m</sub></i> (mg g <sup>-1</sup> )	<i>b</i> (L mg <sup>-1</sup> )	<i>R<sub>SQR</sub></i>
30	28.9	0.45	0.999	22.4	0.17	0.996
45	30.6	0.63	0.998	23.3	0.22	0.998
60	30.4	0.7	0.998	26.4	0.30	0.997

**Table 1b**

Langmuir constants for the uptake of copper ion in the presence and absence of ultrasound

$t$ (°C)	Sonicated method			Control method		
	$q_m$ (mg g <sup>-1</sup> )	$b$ (L mg <sup>-1</sup> )	$R_{sqr}$	$q_m$ (mg g <sup>-1</sup> )	$b$ (L mg <sup>-1</sup> )	$R_{sqr}$
30	17.2	0.10	0.997	12.1	0.07	0.999
45	18.7	0.12	0.999	14.0	0.08	0.996
60	15.6	0.14	0.992	14.5	0.09	0.999

**Fig. 5.** Pseudo-second-order kinetic model for the removal of ions (initial concentration for each one: 100 mg L<sup>-1</sup>).

temperature behavior was different and it was more effective at 45 °C. This is due to the cavitation process which is more effective at this temperature. The value of  $b$  represents the affinity of the sorbate to the sorbent. This affinity was different for the two selected ions and affected by the sonication.

### 3.4. Sorption kinetics

The sorption data was analysed in terms of pseudo-first-order and pseudo-second-order kinetic models. The correlation coefficients for the second-order kinetic model were higher than first-order for both ions in the presence and absence of ultrasound. This indicates that this model is proper for the removal of these ions from solution. When the second-order kinetics is applicable, the plot of  $t/q$  versus time (Eq. (3)) should be linear.

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{t}{q_e} \quad (3)$$

From the slope and the intercept of the plot (Fig. 5), the rate constant of sorption ( $k$ ) and the amount of adsorbed species at equilibrium ( $q_e$ ) can be determined. Table 2 summarizes the results and shows a good agreement between experimental and calculated values of  $q_e$ .

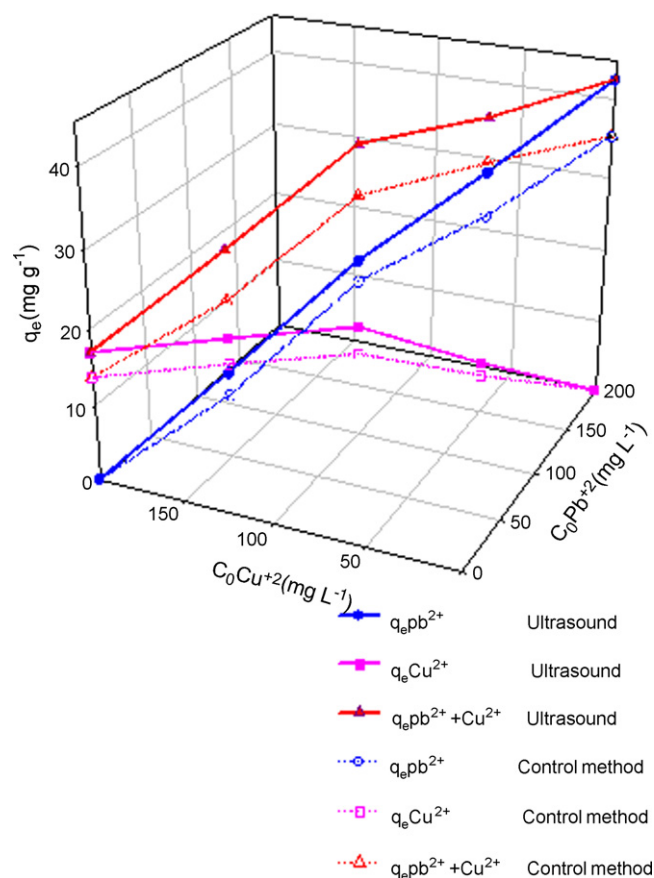
**Table 2**

Kinetic parameters of sorption in the presence and absence of ultrasound

	Sonicated method		Control method	
	Pb <sup>2+</sup>	Cu <sup>2+</sup>	Pb <sup>2+</sup>	Cu <sup>2+</sup>
$q_{e(\text{exp})}$ (mg g <sup>-1</sup> )	22.17	15.13	18.90	7.89
$q_{e(\text{cal})}$ (mg g <sup>-1</sup> )	22.88	15.70	19.80	8.70
% Dev	3.20	2.61	4.76	10.26
$k$ (g mg <sup>-1</sup> min <sup>-1</sup> )	0.95	1.40	0.75	0.68
$R_{sqr}$	0.99	0.99	0.99	0.99

### 3.5. Competitive sorption in the total constant concentration

The object of this part is to study the effect of two metal ions coexistence on the total adsorption capacity of sorbent. The experiments were carried out in keeping the total concentration constant (200 mg L<sup>-1</sup>) and changed each metal ion concentrations independently. The results were shown in Fig. 6. As it is shown in this figure, the values of sorption ( $q_e$ ) obtained for each metal ion from the binary component system were less than those for the single component solutions. However, the total sorption in the binary system was higher than that of copper ion solution but less than that of lead ion solution as a single component system. The presence of one of the metal ion interfered with the uptake of another one in the system. It is also indicated that the sorbent has a relatively stronger affinity for Pb<sup>2+</sup> than Cu<sup>2+</sup> and this change of affinity in the sorption could be attributed to the properties of ions itself and the difference in their interaction with the sorbent. According to Fig. 6, ultrasound has improved the sorption of each ion in the binary system with respect to the control method. This enhancement was higher for the copper ion than lead ion. The overall uptake of ions was also higher than the control method. This behavior could be attributed to the cavitation process which influences the surfaces of the sorbent and also the mass transfer which is important in heterogeneous solutions. Table 3 summarizes the percent of removal of ions in the presence and absence of ultrasound with change of the initial concentrations of ions under the constant of overall concentration. This table also shows that the best efficiency for the removal of both ions was achieved in the presence and in the absence of ultrasound when the concentration of both ions was the same.

**Fig. 6.** Effect of composition at fixed total initial concentration on the sorption capacity of each ions (stirring speed: 300 rpm; amount of sorbent: 0.2 g; initial concentration: 200 mg L<sup>-1</sup>; temperature: 30 °C; time: 10 min).

**Table 3**  
Uptake of Pb<sup>2+</sup> and Cu<sup>2+</sup> ions in single and binary solution (amount of sorbent: 0.2 g; temperature: 30 °C; time: 10 min)

Pb <sup>2+</sup> /Cu <sup>2+</sup> (mg L <sup>-1</sup> )	Sonicated method			Control method		
	Uptake % (Pb <sup>2+</sup> /Cu <sup>2+</sup> )	Adsorbed concentrations (Pb <sup>2+</sup> /Cu <sup>2+</sup> ) (mg L <sup>-1</sup> )	Total adsorbed concentrations (Pb <sup>2+</sup> + Cu <sup>2+</sup> ) (mg L <sup>-1</sup> )	Uptake % (Pb <sup>2+</sup> /Cu <sup>2+</sup> )	Adsorbed concentrations (Pb <sup>2+</sup> /Cu <sup>2+</sup> ) (mg L <sup>-1</sup> )	Total adsorbed concentrations (Pb <sup>2+</sup> + Cu <sup>2+</sup> ) (mg L <sup>-1</sup> )
200/00	86/00	172/00	172	70/00	140/00	140
150/50	82/44	123/22	145	73/28	109/14	123
100/100	94/59	94/59	153	84/44	84/44	128
50/150	92/43	46/64	110	66/24	33/36	69
00/200	00/34	00/68	68	00/28	00/55	55

**Table 4a**  
Thermodynamic parameters for the uptake of lead ion

t (°C)	Sonicated method			Control method		
	-ΔG° (kJ mol <sup>-1</sup> )	ΔH° (kJ mol <sup>-1</sup> )	ΔS° (J mol <sup>-1</sup> )	-ΔG° (kJ mol <sup>-1</sup> )	ΔH° (kJ mol <sup>-1</sup> )	ΔS° (J mol <sup>-1</sup> )
30	28.9			26.5		
45	31.1	12.7	13.7	28.4	16.1	139.0
60	33.0			30.5		

**Table 4b**  
Thermodynamic parameters for the uptake of copper ion

t (°C)	Sonicated method			Control method		
	-ΔG° (kJ mol <sup>-1</sup> )	ΔH° (kJ mol <sup>-1</sup> )	ΔS° (J mol <sup>-1</sup> )	-ΔG° (kJ mol <sup>-1</sup> )	ΔH° (kJ mol <sup>-1</sup> )	ΔS° (J mol <sup>-1</sup> )
30	22.0			21.2		
45	23.6	9.5	10.4	22.6	7.2	93.4
60	25.2			24.0		

### 3.6. Thermodynamic functions

According to the Eq. (4), it is possible to calculate the standard Gibbs free energy changes for the sorption process at different temperatures

$$\Delta G_0 = -RT \ln b \quad (4)$$

The standard enthalpy and entropy changes of sorption can be determined through the slope and intercept of the line produced by drawing  $\ln b$  versus  $1/T$  (van't Hoff equation). The amount of thermodynamic functions for each ion in the sorption process was shown in Tables 4a and 4b.

The negative values of the standard Gibbs free energy at various temperatures indicated the spontaneous nature of the sorption of Cu<sup>2+</sup> and Pb<sup>2+</sup> from the solution by the sorbent in the presence and absence of ultrasound. The positive values of the standard enthalpy indicate the endothermic nature of the sorption of Cu<sup>2+</sup> and Pb<sup>2+</sup> on the sorbent. The positive values of standard entropy changes reflect the increasing randomness at the solid/liquid interface during the sorption of metal ions on the sorbent.

## 4. Conclusion

The present study compares the results obtained for the removal of copper and lead ions from single and binary system in the presence and absence of ultrasound. Results show that the experimental data fitted very well with Langmuir model in the studied range of concentrations and temperatures in sonicated and control methods. The affinity of lead ion was higher than copper ion with respect to the sorbent and the ultrasound enhanced the removal of both ions from the solution. The effect of ultrasound was higher for the removal of lead ion in single component than copper ion with respect to the control method. In contrast, in the binary solution, the effect of ultrasound was higher for the copper ion than lead ion with respect to the control method. This means that the species with higher affinity for the removal facilitated the removal of species with lower affinity.

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