Environmental Earth Sciences (2020) 79:148
https://doi.org/10.1007/s12665-020-8891-y

ORIGINAL ARTICLE

Effect of the soil biochar aging on the sorption and desorption of Pb^{2+} under competition of Zn^{2+} in a sandy calcareous soil

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Received: 3 May 2019 / Accepted: 28 February 2020 / Published online: 12 March 2020

Abstract

Applying biochar in contaminated soils can reduce the availability of the heavy metals (HMs). However, there is no sufficient evidence regarding the effects of biochar on adsorption and desorption of lead (Pb^{2+}) in the calcareous soils. Thus, the present study aimed to investigate the effect of walnut leaf biochars (1% w/w) produced at different temperatures (200 (B200), 400 (B400), and 600 (B600) °C) on the adsorption and desorption properties of Pb^{2+} in individual (Pb) and competitive (Pb + Zn) systems at two incubation times (30 and 90 days). The results showed that the maximum adsorption capacity of Pb^{2+} (qm) in the soils treated with B400 and B600 was higher than the soils treated with feedstock and B200. In the presence of Zn^{2+}, the qm value decreased. Strength of adsorption (K_L) increased with an increment in the pyrolysis temperature, and was lower in competitive system than the individual system in all treatments. Also, the KL was lower in competitive system than the individual system in all treatments. Strength of Pb^{2+} adsorption in soils treated with B400 and B600 enhanced at 90 days compared to 30 days of incubation. Lead desorbed in 10 mM $CaCl_2$ (< 1% of adsorbed Pb^{2+}) and DTPA–TEA solutions decreased with an increase in the pyrolysis temperature. The results revealed that walnut leaf biochars produced at high temperatures had a higher capacity and strength of Pb^{2+} adsorption, while lowering Pb^{2+} desorption compared with other biochars. The results of this study revealed that walnut leaf biochars produced at high temperatures could adsorb and immobilize Pb^{2+} in sandy calcareous soils at either individual or competitive systems.

Keywords Immobilization · Biochar aging · Competitive system · Adsorption capacity

Introduction

The toxicity of HMs and their accumulation due to the presence in food chain is one of the most important environmental and health problems in modern societies. The biological degradation of the HMs is not possible in the soil, making them one of the most dangerous environmental pollutants, and their removal is considered a serious problem (Tembo et al. 2006; Rashed 2010). Lead is also one of the HMs with no biological function and the potential to produce the toxicity for plants and other organisms. According to the total Pb concentration in soil reported by World Health Organization (WHO), the amount of 35 mg kg^{-1} is taken into account as the toxic concentration of Pb (Tembo et al. 2006). The availability and harmful effects of the HMs for animals, plants, and microorganisms, their movement towards the underground water, as well as the effects of these metals on the soil chemistry processes depend on the reactions occurring between these ions and the soil particles (Murali and Aylmore 1983). In other words, three processes can control the fate and availability of the HMs in soil. These processes include (1) the removal of the HMs from the soil solution due to the adsorption on the surface of soil particles, (2) desorption of metals from particles to solution, and (3) the dissolution and precipitation of the HMs in the solid soil phase. The nature of the adsorption and desorption processes influence the solubility and availability of the HMs in the soil (Sparks 2003). Adsorption characteristics are influenced by the solubility and availability of HMs, pH, redox, soil organic matter, iron oxides, and calcium carbonate (Antoniadis et al. 2007).

Desorption of the HMs in the soil is subject to three factors, soil properties, HMs properties, and extraction conditions. The soil properties influencing the adsorption and mobility of the HMs include soil pH, texture, cation

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exchange capacity (CEC), organic matter (OC), Fe and Ca, and pollutants presence in the soil. HMs properties and extraction conditions include the type and concentration of the HMs, distribution of the HMs in soil components and availability of the HMs, solution pH, electrolyte presence, solution ratio, and duration of contact (Zhu et al. 2010).

In the contaminated soils, the HMs usually exist at the same time and compete with each other for adsorbing sites. Therefore, selective adsorption and competition of the HMs by soil have high importance in determining the toxicity potential of these metals in soil, mobility, and their fate (Serrano et al. 2005; Jalali and Moharammi 2007). Individual adsorption of the HMs in soils and different minerals has been widely investigated (Trivedi and Axe 2001; Vasudevan et al. 2002). Also, numerous studies have also been done to understand the competitive adsorption of the HMs in the pure minerals, organic compost, and acidic soils (Zhu et al. 2012; Zhang et al. 2012; Sheikhhosseini et al. 2013). The results of these studies showed that the main factor influencing the competitive adsorption is not only the concentration of the HMs in the soil, but also related to the soil properties, HMs type, and environmental factors (Veeresh et al. 2003).

Due to the spread of the soil contaminated with the HMs, there is a need to develop the soil remediation techniques. These techniques must be cost-effective and reduce the pollution without influencing the soil fertility. For this purpose, studies have been done to reduce the mobility and availability of the HMs (Zhang et al. 2012; Xu et al. 2013; Melo et al. 2016). Nowadays, the biochar is widely used for remediation of the polluted soils (Zhang et al. 2013; Melo et al. 2013, 2016). Biochar is an organic matter obtained from the pyrolysis of the organic compounds. Pyrolysis of the organic matter such as plant residue, forest, and livestock manure causes the release of the volatile C compounds, fixed C compounds, and ash containing a significant amount of Ca and K (Ahmad et al. 2014). Pyrolysis temperature plays an important role in Pb sorption capacity of biochars (Ahmad et al. 2012; Ding et al. 2014). The biochar has a high surface charge, high specific area, and high stability against degradation, and thus has significant power on ion adsorption compared with other organic matter. The biochar may influence the toxicity, mobility, and fate of the various HMs in the soil as a result of improving the adsorption capacity of the soil (Ahmad et al. 2014). Several studies have shown that applying the biochar (produced from different materials or temperatures) in the contaminated soils can significantly reduce the availability of the HMs through the cationic exchange, precipitation, and pH increase (in acid soils) (Zhang et al. 2012; Xu et al. 2013; Melo et al. 2016). However, there is limited evidence regarding the effects of biochar on the mobility of the HMs in calcareous soils. Moreover, Zn is present in many soils of Pb mines (Dayani and Mohammadi 2010); therefore, it is necessary to consider the effect of Zn on the mobility of Pb in the soils.

In this study, the effects of biochar produced at different temperatures were investigated on mobility (adsorption and desorption) of Pb^{2+} during incubation period. According to the mentioned explanations, the following hypotheses are put forth: (1) desorption and adsorption of Pb^{2+} is influenced by the presence of Zn^{2+}, (2) biochar produced at different temperatures influences the desorption and adsorption of Pb^{2+}, and (3) mobility of Pb^{2+} varies during the incubation times.

Materials and methods

Properties of the studied soil

The soil sample was taken from 0 to 30 cm depth from surface of the calcareous soils with semi-arid climate in Central Iran. This soil was classified as Typic Calcixerepts according to the USDA Soil Taxonomy (Soil Survey Staff 1990). The selected sandy soil was air-dried and ground to pass through 2-mm sieve. At the beginning of study, soil subsamples were used to determine some chemical and physical properties. The values of these measurements were as follows: clay 170 g kg^{-1}, silt 300 g kg^{-1}, sand 530 g kg^{-1}, carbonate calcium equivalent (CCE) 150 g kg^{-1}, electrical conductivity (EC, soil:water = 1:2) 0.28 dS m^{-1}, pH (soil:water = 1:2) 7.9, organic carbon (OC) 8.6 g kg^{-1}, available Pb 0.01 mg kg^{-1}, and available Zn 0.36 mg kg^{-1}. The predominant clay minerals in the soil of the present study were micas, smectites, and chlorite, with fewer quantities of kaolinite and vermiculites (Hosseinpur et al. 2012).

Biochar production and properties

The biochar was produced from walnut leaves (*Juglans regia* L.). Walnut leaves (WL) were oven-dried at 60 °C for 24 h and were placed in the cylindrical pyrolyzers of 608 cm^3 inside a muffle furnace, and were sealed and warmed up at 200 (B200), 400 (B400), and 600 (B600) °C at a rate of 10 °C/min, using a batch pyrolysis facility (Khadem and Raiesi 2017). The slow pyrolysis process was applied, the favorable temperature was kept for 2 h, and the temperature of the pyrolyzed feedstock gradually came down to the room temperature. The biochar samples were ground and passed through 1-mm sieve. Some chemical properties of biochars are presented in Table 1.

To study the effect of applying 1% (weight of amendment/weight of soil) amendment on the individual and competitive adsorption and desorption of Pb^{2+} in the sandy calcareous soils during incubation, a completely randomized design with a 5 × 2 factorial treatment combination was used in

Table 1 Properties of used biochars in this study

Properties	Feedstock	B200	B400	B600
pH	4.8	5.2	9.3	10.4
EC (dS m^{-1})	3.11	4.42	5.63	8.06
P (mg kg^{-1})	380	419	833	1092
CEC ($cmol_+$ kg^{-1})	30.5	28.5	22.9	19.9
Surface area (m^2 g^{-1})	2.2	3.3	5.4	9.2
Pseudo-total Zn (mg kg^{-1})	14.4	27.6	44.9	46.8
Pseudo-total Pb (mg kg^{-1})	4.2	4.7	9.8	11.4
Yield (%)	-	89	47	36

pH-H_2O (1:10), EC (1:10); B0 is feedstock; B200, B400, and B600 are biochars produced at 200, 400, and 600 °C, respectively

three replicates with the following factors: (1) amendments (control, WL, B200, B400, and B600), (2) adsorption system (individual and competitive) during incubation time (30 and 90 days). Therefore, 300 g of soil was put in each jar and mixed with the amendments, then incubated for 90 days at 25 ± 2 °C (Mirzaei Aminiyan et al. 2014; Abbruzzini et al. 2017). To determine the adsorption and desorption of Pb^{2+}, the soils were sampled on 30 and 90 days after incubation. The moisture of all soils was kept at field capacity by weighing during incubation period.

Characteristics of the individual and competitive adsorption

The competitive adsorption of Pb^{2+} in the presence of Zn^{2+} was carried out in the amended soils using batch method (Antoniadis et al. 2007; Jalali and Moharammi 2007). In individual system, subsample (2 g) of the amended soils was placed into 50 ml centrifuge tubes, and 20 ml of equilibrating solution (0.05, 0.1, 0.2, 0.4, 0.5, 1, 2, and 3 mM) of Pb^{2+} (as $Pb(NO_3)_2$) containing 10 mM $CaCl_2$ as the background electrolyte was added to each tube. In competitive system, the amount of Pb^{2+} and Zn^{2+} (as Zn $(NO_3)_2$) was similar to the individual system (mole ratio $Pb^{2+}/Zn^{2+} = 1$ to 1). The suspensions were shaken at 200 rpm for 2 h and then kept still for 24 h at a constant temperature of 25 ± 2 °C. At the end of the adsorption period, the suspensions were centrifuged at 4000 revolutions min^{-1} for 3 min, and the supernatants were filtrated to measure Pb^{2+} concentrations. Then, concentration of Pb^{2+} was measured using an atomic absorption spectrophotometer (AAS model G.B.C 932, G.B.C. Melbourne, Australia).

The concentration of adsorbed Pb^{2+} (q, mg kg^{-1}) was calculated as

$$q = \frac{(C_i - C_e) \times V}{W} \quad (1)$$

where C_i and C_e represent the initial and equilibrium concentrations (mg l^{-1}) of Pb^{2+} in the solution, respectively. The v/w represents the volume of the solution (l) to mass of the sorbent (kg), respectively (Echeverria et al. 1998; Jalali and Moharammi 2007; Mohan et al. 2007).

Adsorption isotherms were obtained by plotting the amount of adsorbed Pb^{2+} on the amended soils versus the concentration of Pb^{2+} in equilibrium solutions. Then, the linear Langmuir (2), and Freundlich (3) (Echeverria et al. 1998; Sparks 2003; Jalali and Moharammi 2007; Hararah et al. 2012; Yang et al. 2019) were used to describe the distribution of Pb^{2+} between solution and solid phases of the soils.

$$\frac{C_e}{q} = \frac{1}{K_L\,qm} + \frac{1}{qm}C_e \quad (2)$$

$$\log q = \log K_f \times n \log C_e \quad (3)$$

where q is the amount of adsorbed Pb^{2+} at equilibrium (mg kg^{-1}); C_e is the concentration of Pb^{2+} in solution at equilibrium (mg l^{-1}); and qm is the maximum adsorption capacity of Pb^{2+} on soil (mg kg^{-1}) (Echeverria et al. 1998; Sui and Thompson 2000; Jalali and Moharammi 2007; Hararah et al. 2012; Yang et al. 2019). The K_L is the Langmuir constant (l mg^{-1}), which increases exponentially with the energy of sorption (energy constant related to the strength of adsorption), K_f is the Freundlich constant (l kg^{-1}) reflecting the adsorption capacity or distribution coefficient, and n is an empirical constant (unit less) (Vega et al. 2006; Jalali and Moharammi 2007; Mohan et al. 2007; Hararah et al. 2012; Yang et al. 2019). Maximum buffering capacity (MBC) (4) indicates the ability of the soil to resist change of Pb^{2+} concentration in soil solution (Sui and Thompson 2000; Yang et al. 2019). The MBC is an integrated parameter that combines the qm and K_L of Langmuir isotherm (Yang et al. 2019). Yang et al. (2019) reported that higher amount of MBC reveals more elements will be adsorbed onto soil.

$$MBC = qm \times K_L \quad (4)$$

Desorption of Pb^{2+}

To determine the desorption of adsorbed Pb^{2+}, 20 ml of 10 mM $CaCl_2$ or DTPA–TEA (0.005 M DTPA + 0.1 M triethanolamine + 0.01 M $CaCl_2$, pH 7.3) was added to the residual soils in centrifuge tubes from the adsorption study, and their contents were shaken for 24 and 2 h, at a constant temperature of 25 ± 2 °C (Wang and Harrel 2005; Hararah et al. 2012). At the end of desorption period, the suspensions were centrifuged at 4000 r min^{-1} for 3 min, and the supernatants were filtrated to measure Pb^{2+} concentrations. Then, the concentration of Pb^{2+} was measured using AAS (above model). Percentage of the desorbed Pb^{2+} was calculated as

$$\text{Desorbed Pb}^{2+}(\%) = \frac{\text{Pb}_{\text{de}}}{\text{Pb}_{\text{ad}}} \times 100 \tag{5}$$

where Pb_{de} is the concentration of desorbed Pb^{2+} (mg kg^{-1}) and Pb_{ad} the concentration of adsorbed Pb^{2+} (mg kg^{-1}).

Statistical analysis

Linear regression analysis was used to fit the Langmuir and Freundlich isotherms to the adsorption data using the Excel software (Jalali and Moharammi 2007; Remenyi et al. 2009). The goodness of fit of these models to the data was investigated based on the coefficients of determination (R^2).

A repeated measures ANOVA was performed to analyze the effects of the independent factors (treatments) and incubation times on the coefficients of isotherms. Before the ANOVA analysis, Mauchly's sphericity test was performed. When the test value was significant at 5%, the degree of freedom (df) was multiplied by the epsilon coefficient (Huynh–Feldt correction). The means of the treatments were separated at 5% of significance level using the least significant difference (LSD). Simple linear regression was performed between all the attributes. The statistical analyses of the data were carried out using STATISTICA 8 (StatSoft, Inc. 2007; Moghimi et al. 2018).

Results and discussion

Adsorption characteristics

The relation between the equilibrated concentration of Pb^{2+} in solution (Ceq) and the amount of adsorbed Pb^{2+} (q) onto the treated soils is shown in Fig. 1. All relations between Ceq and q are L shape (Sposito 1989; Limousin et al. 2007; Park et al. 2016). This figure showed that concentration of the adsorbed Pb^{2+} onto the soils treated with biochars was higher than the soil treated with the feedstock and control soil. The amount of adsorbed Pb^{2+} was also found to be increased in the soil treated with B600 compared with the soils treated with B400 and B200. Applying the biochar to

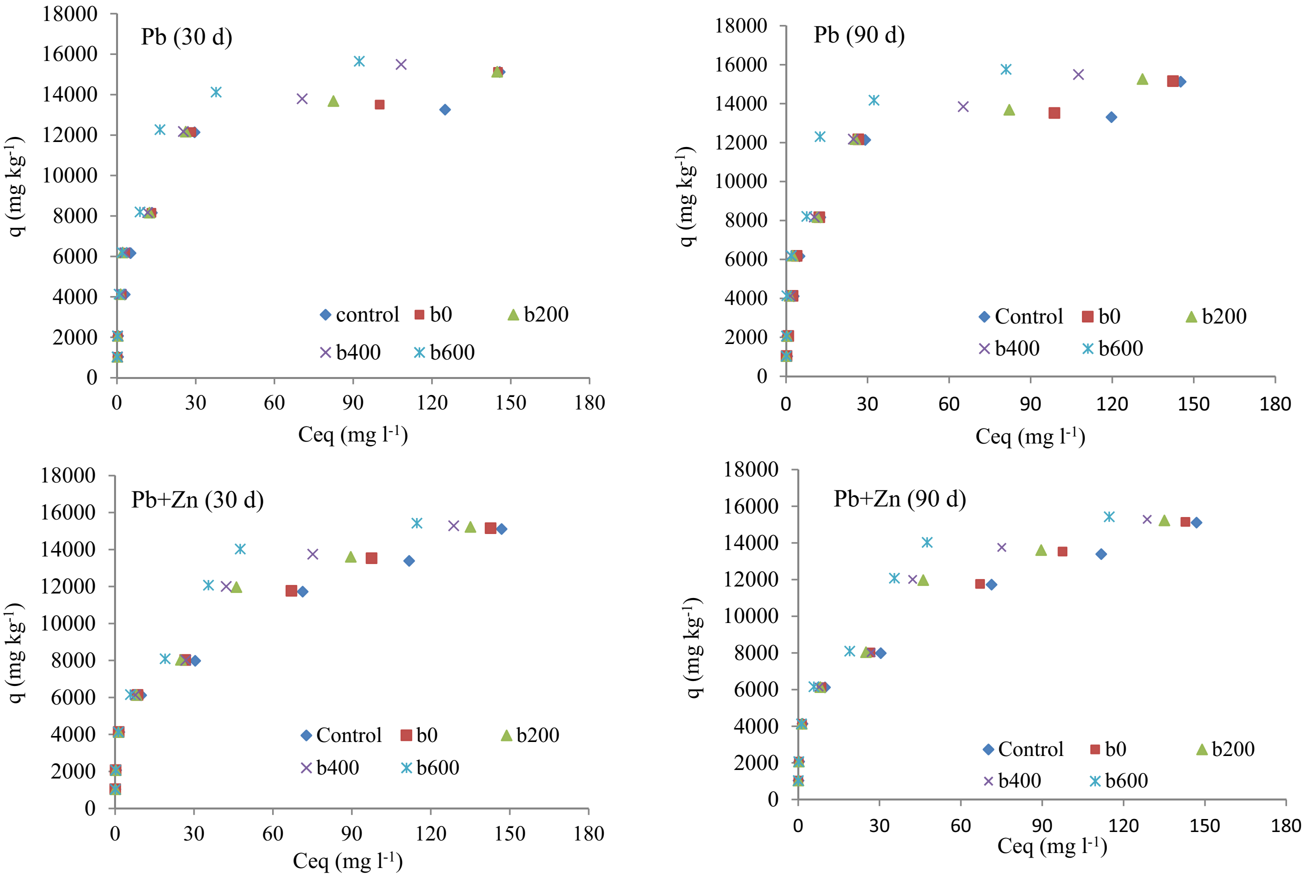

Fig. 1 Relation between equilibrated concentration of Pb in solution and amount of adsorbed Pb onto soils. B0 is feedstock; b200, b400, and b600 are biochars produced at 200, 400, and 600 °C, respectively

the soil enhanced the concentration of equilibrium Pb^{2+} in the soil solution through the increase in the pyrolysis temperature.

Coefficients of determination (R^2) for two models are shown in Table 2. The results of this table revealed that Langmuir and Freundlich isotherms could describe the adsorption of Pb^{2+} onto all the studied soils. However, the Langmuir model ($R^2 = 0.983$–0.995) fitted slightly better than the Freundlich model ($R^2 = 0.915$–0.977). Trakal et al. (2011), Ding et al. (2014), and Wang et al. (2015) obtained a similar result for Pb^{2+} adsorption by different biochars.

Isotherm coefficients are useful parameters to compare the capacity of different soils regarding the adsorption of the HMs under the same experimental conditions (including HMs concentration, equilibrium time, temperature, etc.) (Jalali and Moharrami 2007; Trakal et al. 2011). Summary of analysis of variance (ANOVA) results for isotherm models is shown in Table 3. ANOVA showed a significant interaction effect of the biochar, adsorption, and time ($p < 0.01$) on all coefficients of isotherms except qm in the Langmuir model. Main effects (biochar, adsorption, and time) were only significant on qm.

Table 2 Coefficient of determination (R^2) of two models applied to describe Pb^{2+} adsorption onto different soils

Adsorption	Biochar	Langmuir		Freundlich	
		Time (day)		Time (day)	
		30 days	90 days	30 days	90 days
Individual	Control	0.992	0.992	0.915	0.916
	B0	0.994	0.994	0.922	0.931
	B200	0.995	0.994	0.922	0.935
	B400	0.992	0.993	0.950	0.933
	B600	0.994	0.993	0.957	0.954
Competitive	Control	0.983	0.985	0.970	0.975
	B0	0.985	0.989	0.977	0.966
	B200	0.986	0.981	0.976	0.961
	B400	0.987	0.991	0.975	0.967
	B600	0.990	0.981	0.976	0.977

R^2 is significant at 0.01. B0 is feedstock; B200, B400, and B600 are biochars produced at 200, 400, and 600 °C, respectively

The qm (maximum adsorption capacity of Pb^{2+}) augmented in all the amended soils compared with control (Fig. 2A). The qm value in the soils treated with B400 (443–627 mg kg^{-1}) and B600 (814–998 mg kg^{-1}) increased ($p < 0.05$) compared with the soils treated with the feedstock and B200. Therefore, the adsorption ability of Pb^{2+} enhanced with the increase in the pyrolysis temperature. Moreover, maximum adsorption capacity of Pb^{2+} in the soil treated with B200 did not improve ($p > 0.05$) compared with the soil treated with the feedstock (Fig. 2A). The qm value was higher in the individual system (93 mg kg^{-1}) than the competitive system (Fig. 2A). Therefore, it can be concluded that the maximum adsorption capacity of Pb^{2+} in the soil will decrease in the presence of Zn. In agreement with this result, Echeverria et al. (1998) demonstrated that the qm value was greater in the individual than competitive systems. The averaged qm value increased (85 mg kg^{-1}) on 90 days compared with 30 days of the incubation. There is limited evidence regarding the effects of biochar on Pb^{2+} adsorption in calcareous soils. Trakal et al. (2011) reported that the qm value increased in an acidic soil treated with 1% willow biochar (41.4 mg kg^{-1}) compared with control (39.4 mg kg^{-1}). In some studies, Pb^{2+} adsorption by biochars was determined in aqueous solutions (Mohan et al. 2007; Lu et al. 2012). Mohan et al. (2007) reported that adsorption capacity of Pb^{2+} from solution by biochars produced at

Table 3 Summary of analysis of variance (ANOVA) results (mean square values) for isotherm models

SOV	df	MS				
		Langmuir			Freundlich	
		q_m	K_L	MBC	K_f	n
Between-subject effects						
Biochar (B)	4	1,883,584**	2.14×10^{-2}**	6,330,641**	2,435,614**	1.34×10^{-3}**
Adsorption (A)	1	131,087**	1.16×10^{-1}**	28,919,188**	519,783**	3.00×10^{-2}**
B × A	4	7097 ns	8.85×10^{-3}**	2,405,790**	60,285**	9.94×10^{-4}**
Error	20	7440	8.00×10^{-5}	19,877	6190	4.70×10^{-5}
Within-subjects effects						
Time (T)	1	110,626**	4.90×10^{-4}**	174,359**	228,052**	3.20×10^{-5} ns
T × B	4	2822 ns	1.24×10^{-3}**	314,959**	325,841**	4.73×10^{-4}**
T × A	1	206 ns	1.58×10^{-3}**	416,772**	84,530**	1.82×10^{-4}*
T × B × A	4	4794 ns	1.03×10^{-3}**	263,348**	119,746**	3.49×10^{-4}**
Error	20	2786	2.00×10^{-5}	5225	2598	2.00×10^{-5}

**Significant at 1%. *Significant at 5%. *ns* non-significant

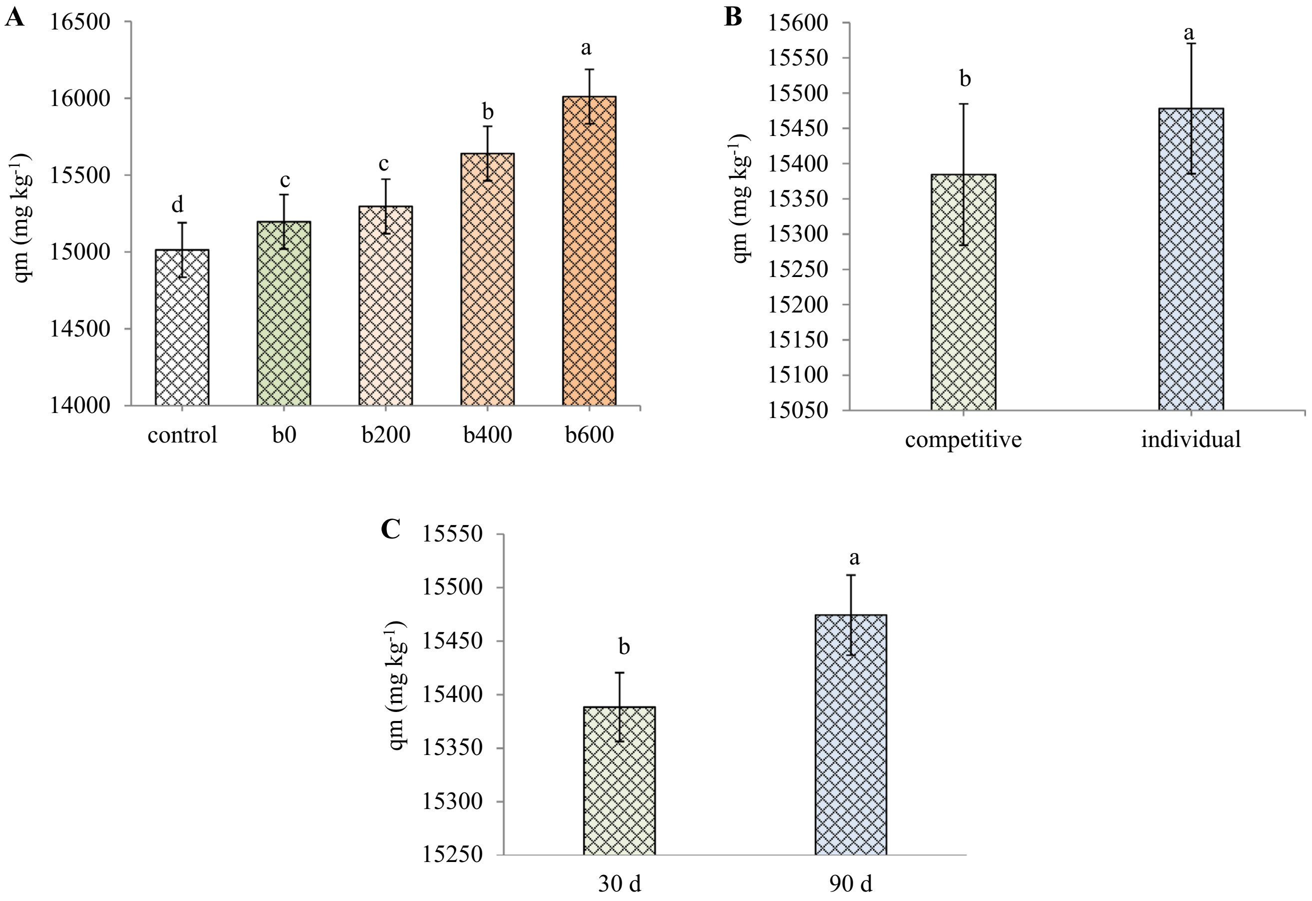

Fig. 2 Effect of biochar (**A**) and/or in competitive or individual system (**B**) at two incubation times (**C**) on the maximum adsorption capacity of Pb^{2+} on soils. b0 is feedstock; b200, b400, and b600 are biochars produced at 200, 400, and 600 °C, respectively. Values are mean and bars indicate SE

400–450 °C ranged from 2620 to 1310 mg kg^{-1}. Also, Lu et al. (2012) reported that adsorption capacity of Pb^{2+} from aqueous solutions by sludge biochar (produced at 550 °C) was 30,900 mg kg^{-1}. Therefore, the qm value in our study was lower than these studies. It might be related to the experimental condition (such as absorbent, soil type, concentration of HMs, equilibrium time, etc.).

The interaction effects of the biochar, adsorption type, and time on K_L (strength of adsorption or energy of sorption) are presented in Table 4. The results showed that energy of sorption was lower ($p < 0.05$) in the competitive adsorption than the individual adsorption of Pb^{2+} in all treatments. Park et al. (2016) obtained a similar result for Pb^{2+} adsorption by different biochars. They reported that the strength of adsorption of HMs from solution by pepper stem biochar ranged from 0.0481 to 0.1267 in individual system and from 0.0413 to 0.0685 in competitive system. In both of the individual and competitive systems, K_L increased with the increase in the pyrolysis temperature. This coefficient was higher in the soil treated with B600 than other soils. In agreement with this result, Wang et al. (2015) investigated the effects of biochars produced from peanut shell and Chinese medicine material residues at 300 to 600 °C on Pb^{2+} adsorption from solution. Their results showed that the K_L value increased with the increase in the pyrolysis temperature. Ding et al. (2014) reported that the strengths of adsorption of Pb^{2+} from solution by bagasse biochars produced at low temperatures (0.051–0.055) were lower than biochar produced at high temperatures (0.11–0.18). In the competitive system, the K_L value was not different in the soils treated with B0, B200, and B400 at both incubation times. It might be attributed to surface functional groups, pore size distribution, and concentration of PO_4^{3-} and CO_3^{2-} in these amendments (Cheng and Lehmann 2009; Ding et al. 2014; Cao et al. 2017). However, this result deserves further study. Strength of Pb^{2+} adsorption enhanced on 90 days compared with 30 days of incubation in the soils treated with B400 and B600. Cheng and Lehmann (2009) and Cao et al. (2017) reported that acidic surface functional groups (carboxylic and phenolic

Table 4 The interactions effects of biochar, adsorption, and time on K_L and MBC of Langmuir

Adsorption	Biochar	K_L (l mg^{-1})		MBC (l kg^{-1})	
		Time (day)		Time (day)	
		30	90	30	90
Individual	Control	0.140 ± 0.003f	0.144 ± 0.003f	2100 ± 45f	2175 ± 43f
	B0	0.174 ± 0.006e	0.149 ± 0.010f	2643 ± 95e	2285 ± 43f
	B200	0.195 ± 0.003d	0.201 ± 0.001d	3045 ± 46d	3080 ± 5d
	B400	0.199 ± 0.003d	0.216 ± 0.004c	3063 ± 58d	3390 ± 67c
	B600	0.281 ± 0.007b	0.360 ± 0.006a	4506 ± 112b	5798 ± 56a
Competitive	Control	0.102 ± 0.001i	0.097 ± 0.003i	1523 ± 12ij	1462 ± 41j
	B0	0.118 ± 0.003 g	0.109 ± 0.002hi	1779 ± 55 g	1651 ± 36hi
	B200	0.118 ± 0.004gh	0.114 ± 0.003gh	1794 ± 64gh	1743 ± 33gh
	B400	0.120 ± 0.001gh	0.118 ± 0.002gh	1865 ± 19 g	1847 ± 22 g
	B600	0.144 ± 0.002f	0.141 ± 0.001f	2286 ± 38f	2250 ± 28f

Each data is a mean value of three replications with standard errors. Different letter(s) represent significant differences between treatments by Fisher's LSD test at $p < 0.05$. B0 is feedstock; B200, B400, and B600 are biochars produced at 200, 400, and 600 °C, respectively

structures) developed in biochar aging. Accordingly, negative charge (cation exchange capacity) of biochars could be increased during incubation period. Therefore, total adsorbed Pb^{2+} onto the soils increased as a result of the soils' biochar aging.

The interactions effects of the biochar, adsorption, and time on the MBC are shown in Table 4. Similar to qm and K_L trends, the MBC increased in the soils treated with the biochars compared with control and the soil treated with the feedstock. The MBC was lower ($p < 0.05$) in the competitive system than the individual system in all treatments. In the individual and competitive systems, the MBC increased in the soil treated with B600 compared with the soil treated with B400. In the individual system, the MBC was lower ($p < 0.05$) on 30 days of incubation than 90 days of incubation in the soils treated with B400 and B600. The MBC value did not differ in the control soil on 90 days of incubation compared with 30 days of incubation. The soils' resistance to the change of Pb^{2+} concentration in the soil solution against adding Pb^{2+} to the soil has been shown previously by the maximum buffering capacity (Sui and Thompson 2000; Yang et al. 2019). Soil with high MBC (soil treated with B600) is capable to adsorb Pb^{2+} more than the soil with low MBC. Therefore, this amendment can improve the contaminated soils in the presence of $Pb^{2+} + Zn^{2+}$ or Pb^{2+} alone.

The interaction effects of the biochar, adsorption, and time on K_f (adsorption capacity) are presented in Table 5. In the individual system, the adsorption capacity of Pb^{2+} increased on 90 days of incubation in the soils treated with

Table 5 The interactions effects of biochar, adsorption, and time on constants of Freundlich model

Adsorption	Biochar	K_f (l kg^{-1})		*n*	
		Time (day)		Time (day)	
		30	90	30	90
Individual	Control	2100 ± 9f	2175 ± 45f	0.404 ± 0.001a	0.403 ± 0.002a
	B0	2643 ± 11e	2285 ± 15f	0.378 ± 0.004bc	0.402 ± 0.008a
	B200	3045 ± 11d	3080 ± 48d	0.369 ± 0.002c	0.370 ± 0.004c
	B400	3063 ± 12d	3390 ± 17c	0.384 ± 0.005b	0.371 ± 0.005c
	B600	4506 ± 23b	5798 ± 34a	0.369 ± 0.006c	0.335 ± 0.005de
Competitive	Control	1523 ± 20ij	1462 ± 24j	0.329 ± 0.001ef	0.344 ± 0.005d
	B0	1779 ± 64 g	1651 ± 93hi	0.334 ± 0.001ef	0.335 ± 0.001de
	B200	1794 ± 16gh	1743 ± 44gh	0.333 ± 0.003ef	0.338 ± 0.001de
	B400	1865 ± 57 g	1847 ± 11 g	0.336 ± 0.004de	0.329 ± 0.002ef
	B600	2286 ± 21f	2250 ± 50f	0.331 ± 0.003ef	0.332 ± 0.001ef

Each data is a mean value of three replications with standard errors. Different letter(s) represent significant differences between treatments by Fisher's LSD test at $p < 0.05$. B0, feedstock; B200, B400, and B600 biochars produced at 200, 400, and 600 °C, respectively

B400 and B600. In the competitive system, the adsorption capacity did not change in the soils treated with the biochars ($p > 0.05$). The K_f decreased (40–61%) in the competitive system compared with the individual system. Park et al. (2016) obtained a similar result for Pb^{2+} adsorption by pepper stem biochar from solutions. The K_f value increased in the soil treated with B600 than other treatments (22–88%). The trend of K_f was similar to the qm value in the studied soils ($r = 0.89$, $p < 0.01$).

The interaction effects of the biochar, adsorption, and time on n are given in Table 5. The *n* coefficient did not change in the soils treated with the biochars.

Individual and competitive desorption

Percentages of Pb^{2+} desorbed in DTPA–TEA and 10 mM $CaCl_2$ solutions are shown in Table 6. Lead extracted by 10 mM $CaCl_2$ was found to be higher in the competitive system at two incubation times than the individual system. Also, Pb^{2+} desorbed in 10 mM $CaCl_2$ was higher on 90 days of incubation than 30 days of incubation. The results of this table showed that Pb^{2+} extracted by 10 mM $CaCl_2$ was lower than 1% of adsorbed Pb^{2+}. Therefore, concentration of the adsorbed Pb^{2+} was very little on the exchangeable sites in all treatments. The concentration of the lead extracted by 10 mM $CaCl_2$ decreased with the increase in the pyrolysis temperature.

The amount of Pb extracted by DTPA–TEA was higher than 40% of the adsorbed Pb. However, extractants are not able to extract all amounts of the adsorbed Pb^{2+}. Desorbed Pb by DTPA–TEA in the presence of Zn^{2+} was higher at two incubation times. Also, Pb^{2+} extracted by DTPA–TEA was higher (more than 20%) in the competitive system than the individual system. Moreover, the amount of Pb^{2+} extracted by this extractant decreased with the increase in the pyrolysis temperature.

The DTPA–TEA extractable Pb^{2+} is the amount of Pb^{2+} adsorbed in the labile pool of the soils, whereas the DTPA–TEA unextractable Pb is the amount of Pb^{2+} adsorbed in the nonlabile pool of the soil (Wang and Harrel 2005). Therefore, it can be said that the concentration of Pb^{2+} adsorbed in the nonlabile pool of the soil will reduce in the presence of Zn^{2+} on 90 days of incubation.

It seems that the adsorption on the exchangeable sites is not the main mechanism controlling the adsorption of Pb, and the complexation and precipitation are also important mechanisms controlling Pb sorption by the soil treated with the biochar (Lu et al. 2012; Cao et al. 2009; Xu et al. 2013; Ding et al. 2014).

The results of this study indicated that the affinity of soil for sorption of Pb^{2+} is higher than Zn^{2+} in all treatments and incubation times, which has also been reported in many studies (Covelo et al. 2004; Vega et al. 2006). The tendency of the HMs cations to form the strong complexes is according to the ionic radius and the ionization potential (Misono softness parameter) (Sposito 1989). Moreover, electronegativity is an important factor in determining the ability of these cations' chemisorption. Ionic radius of Pb^{2+} (1.20 $\dot{A}$) is greater than Zn^{2+} (0.74 $\dot{A}$) and subsequently, hydrated radius of Pb^{2+} is smaller than Zn^{2+}. Also, the sequence of electronegativity is Cu^{2+} (1.9) > Pb^{2+} (1.8) > Ni^{2+} (1.8) > Cd^{2+} (1.7) > Zn^{2+} (1.6). Therefore, Pb^{2+} is a suitable metal than Zn^{2+} for electrostatic adsorption and inner-sphere surface complexation on the soils (McBride 1994). The results of Table 6 indicate the highest amount of added Pb^{2+} adsorbed as inner-sphere. Meanwhile, Fontes et al. (2000) reported that competition between Cd^{2+}, Cu^{2+}, Pb^{2+}, and Zn^{2+} had a very small effect on Pb^{2+} and Cu^{2+} adsorption on an Oxisol.

Cation exchange, complexation, and precipitation are the main mechanisms controlling Pb^{2+} sorption through the use of the biochar (Lu et al. 2012). Biochar generally has a higher surface area and greater cation exchange capacity than soil; it has been used in trials aimed at decreasing the solubility and toxicity of the HMs and organic compounds in the soils (Beesley et al. 2011). However, their sorption mechanisms depend on the characteristics of the biochar, influenced by the feedstock and pyrolysis temperature (Li et al. 2017). Melo et al. (2013) reported that sorption capacity of Zn^{2+} and Cd^{2+} onto the sugarcane straw biochar produced at 700 °C was higher (four times) than that of the sugarcane straw biochar pyrolyzed at 400 °C. Biochars pyrolyzed at high temperature have higher stable carbon compared with the biochars produced at low temperature (Melo et al. 2016). Therefore, biochars pyrolyzed at high temperature are able to keep the HMs longer than the biochars produced at low temperature (Melo et al. 2016) because these amendments are stable. Lu et al. (2012) studied the Pb sorption capacity of the sludge biochar from acid solution. They reported that 38.2 to 42.3% of the total adsorbed Pb^{2+} sorbed on the organic hydroxyl and carboxyl functional groups at different pH (pH 2 to 5). Moreover, they also argued that the coprecipitation and complexation of Pb on the mineral surfaces (57.7 to 61.8%) are the main mechanisms governing Pb^{2+} adsorption capacity using the sewage sludge biochar. In contrast, Mohan et al. (2007) investigated the potential of wood/bark biochars as adsorbents for As^{3+}, Cd^{2+}, and Pb^{2+} from water. They reported that the cation exchange was the main mechanism for Pb^{2+} adsorption using the wood/bark biochars, indicating that the trend of Pb adsorbed onto the biochar was similar to that of the desorbed cations. Meanwhile, in agreement with our findings (Table 1), Ding et al. (2014) reported that the cation exchange in hydroxyl and carboxyl functional groups played a dominant role in Pb adsorption onto the biochar produced at low temperature (250 °C). However, the cation

Table 6 Percentage of Pb desorbed in 10 mM $CaCl_2$ and DTPA–TEA solutions

Extractant	Treatment	System	Time (day)	Concentration of Pb (mM)							
				0.5	1.0	2.0	3.0	4.0	6.0	7.0	8.0
10 mM $CaCl_2$	Control	Individual	30	0.086	0.051	0.057	0.061	0.087	0.233	0.282	0.380
		Competitive		0.130	0.142	0.149	0.170	0.252	1.82	1.93	2.54
		Individual	90	0.078	0.094	0.085	0.125	0.205	0.338	0.426	0.451
		Competitive		0.029	0.030	0.166	0.179	0.431	0.724	1.39	5.87
	B0	Individual	30	0.066	0.048	0.055	0.057	0.076	0.208	0.272	0.370
		Competitive		0.124	0.130	0.112	0.109	0.200	1.79	1.91	2.48
		Individual	90	0.067	0.085	0.086	0.108	0.181	0.333	0.406	0.445
		Competitive		0.026	0.095	0.117	0.145	0.339	0.585	1.10	4.04
	B200	Individual	30	0.061	0.044	0.054	0.057	0.075	0.184	0.289	0.343
		Competitive		0.109	0.133	0.113	0.116	0.216	1.70	1.73	2.36
		Individual	90	0.048	0.076	0.071	0.101	0.133	0.303	0.370	0.414
		Competitive		0.024	0.022	0.060	0.126	0.275	0.414	0.954	2.83
	B400	Individual	30	0.054	0.041	0.051	0.049	0.069	0.176	0.248	0.283
		Competitive		0.089	0.107	0.102	0.098	0.156	1.61	1.57	1.65
		Individual	90	0.049	0.053	0.067	0.092	0.128	0.23	0.327	0.377
		Competitive		0.019	0.017	0.050	0.085	0.240	0.328	0.858	2.43
	B600	Individual	30	0.046	0.036	0.046	0.042	0.055	0.120	0.185	0.187
		Competitive		0.070	0.098	0.087	0.080	0.104	1.51	1.38	1.34
		Individual	90	0.045	0.043	0.054	0.066	0.118	0.193	0.279	0.356
		Competitive		0.014	0.015	0.043	0.068	0.140	0.173	0.664	2.033
DTPA–TEA	Control	Individual	30	62	68	73	54	81	67	65	65
		Competitive		63	67	66	59	48	37	33	30
		Individual	90	53	70	65	60	77	72	69	60
		Competitive		75	85	90	83	84	65	66	59
	B0	Individual	30	61	68	72	53	68	59	60	64
		Competitive		61	66	66	60	46	37	33	30
		Individual	90	51	63	64	59	73	71	65	60
		Competitive		82	87	92	88	86	61	65	58
	B200	Individual	30	60	67	68	52	64	59	57	59
		Competitive		61	65	66	57	45	36	33	49
		Individual	90	53	60	59	59	68	69	62	60
		Competitive		82	87	90	84	85	60	64	58
	B400	Individual	30	61	66	66	51	64	59	55	57
		Competitive		60	63	64	56	43	35	32	29
		Individual	90	46	54	55	41	63	65	62	56
		Competitive		79	84	93	95	82	62	61	57
	B600	Individual	30	58	65	63	49	62	58	52	56
		Competitive		58	60	62	54	42	33	26	24
		Individual	90	44	46	50	34	55	59	57	51
		Competitive		71	82	88	84	78	59	58	54

B0 is feedstock; B200, B400, and B600 are biochars produced at 200, 400, and 600 °C, respectively

exchange was not the important mechanism for Pb sorption by the biochar produced at 500 °C, which may be attributed to the reduction of the surface functional groups (low CEC, Table 1). Ding et al. (2014) stated that the intraparticle diffusion mechanism was the predominant mechanism of Pb sorption by the biochar produced at high temperature. The results of the current study indicated that the qm value was correlated with increasing biochar surface area ($R^2 = 0.943$, $p < 0.05$, Fig. 3), suggesting that the addition of biochars produced at high temperature retain Pb^{2+} more effectively and thus would be more favorable for adsorption of Pb^{2+} than the biochars produced at low temperature. In this study,

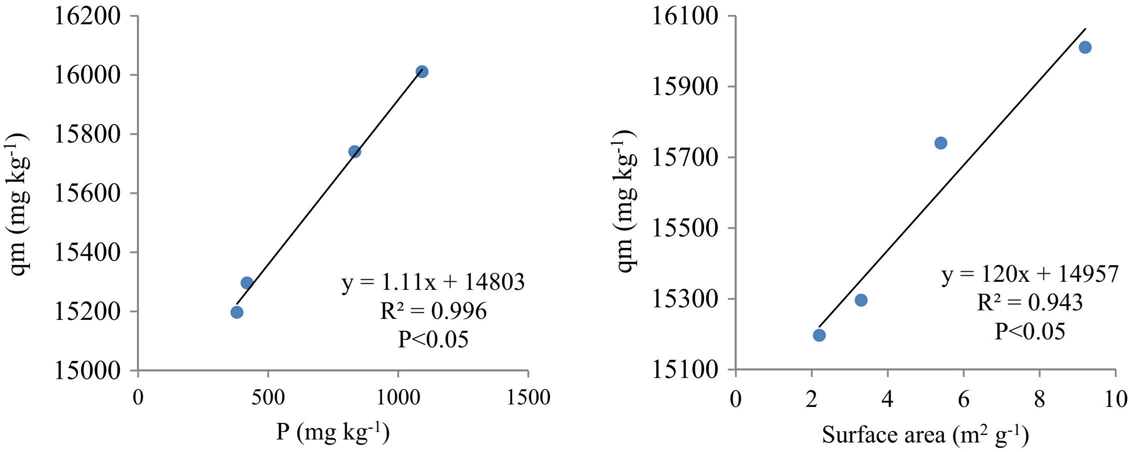

Fig. 3 Simple linear regressions between maximum adsorption capacity of Pb^{2+} on soil (qm) and P of biochar and biochar surface area when averaged across treatments

a significant relation found between the strength of adsorption of Pb^{2+} (K_L) and the surface area of biochar ($R^2=0.938$, $p<0.05$, Fig. 4) indicates that K_L increased with the increase in the surface area of biochar.

Phosphorus has been applied for Pb^{2+} immobilization by forming the low soluble Pb–P minerals (Bolan et al. 2003). Lu et al. (2012), Cao et al. (2009), and Xu et al. (2013) reported that the high concentration of PO_4^{3-} and CO_3^{2-}, and high precipitation as Pb^{2+} phosphate and Pb carbonate minerals (84 to 87%) were the main mechanisms of the dairy manure biochar. Garcia-Perez et al. (2002) reported that P was one of the macronutrient in the bagasse biochar with a concentration of 1330 mg kg^{-1}. Therefore, Pb^{2+} precipitation also occurred on the surface of biochars, especially for biochars with high P content (Ding et al. 2014). Hence, the amount of P in the biochars increased with the increase in the pyrolysis temperature (419 to 1092 mg kg^{-1}) (Table 1). This is supported by the strong correlation between the qm values and the increase in the P amount of the biochar ($R^2=0.996$, $p<0.05$, Fig. 3).

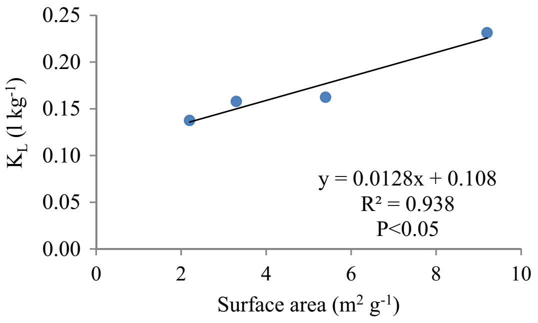

Fig. 4 Simple linear regressions between strength of adsorption (K_L) and biochar surface area when averaged across treatments

Conclusion

The results clearly revealed that the adsorption and desorption of Pb^{2+} are influenced by the presence of Zn^{2+} in the soil. Also, biochar produced at different temperatures influenced the adsorption and desorption of Pb^{2+}. In the presence of Zn^{2+}, maximum adsorption capacity and strength of adsorption of Pb^{2+} decreased. The results showed that maximum adsorption capacity and strength of adsorption of Pb^{2+} increased with the increase in the pyrolysis temperatures. Strength of Pb^{2+} adsorption enhanced on 90 days of incubation compared with 30 days of incubation in the soil treated with B400 and B600. The amount of the desorbed Pb^{2+} decreased with the increase in the pyrolysis temperature. Biochar produced at 600 °C has higher capacity and strength of adsorption of Pb^{2+}, while it had lower desorption of Pb^{2+} compared with other treatments. Also, adsorption capacity of Pb^{2+} increased as a result of biochar aging. Our findings demonstrated that walnut leaf biochars produced at high temperatures could be used for immobilization of Pb^{2+} and reduce toxicity of Pb^{2+} in sandy calcareous soils.

Acknowledgements This study is supported by funds allocated by the Vice President for research of Shahrekord University.

Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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