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Sorption Behavior of Lead (Pb) onto Agricultural Soils of Khyber Pakhtunkhwa, Pakistan: Potentials for Leaching

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Abstract

Lead (Pb) contamination in soil and subsequent transport in groundwater poses severe threats to the food safety and human health. In current study, the effects of soil organic matter on sorption behavior of Pb onto six agricultural soils were investigated by batch sorption experiments and microscopic characterization. Results indicated that Pb sorption onto agricultural soils was dominated by the soil organic matter content and soil texture. The decrease of organic matter content reduced the sorption capacity of Pb onto agricultural soils. Based on soil texture, the Pb sorption was highest in clay soil and lowest in silt type of soil. The overall Pb sorption was in the order of clay > clay loam > silty clay loam \approx loam > silt loam > silt. The sorption isotherms of measured aqueous and soil phase Pb concentrations were fit well with the linear sorption model. The organic carbon normalized partition coefficients ($\text{Log } K_{OC}$) ranged from 2.90 to 2.99. Linear partition coefficient (K_d) values were positively correlated with the soil properties, such as clay ($R^2=0.90$), OC ($R^2=0.94$) and pH ($R^2 = 0.45$); however, weak correlation was found between K_d and soil sand contents ($R^2 = 0.12$). The leachability model showed potential risk of Pb leaching from silt soil with lowest organic matter content. The findings are of significant importance for understanding potential threats of Pb to the soil ecosystem, groundwater, plants, and humans.

Keywords: Lead contamination; Agricultural soils; Sorption; Leaching; Health risks

Introduction

Lead (Pb) is one of the most prevalent, persistent, and potentially toxic heavy metals found in soil (Kabata-Pendias 2000, Yeboah et al. 2019). Besides the natural sources, input from the anthropogenic sources such as, rapid industrialization, economic growth, urbanization, population expansion (Muhammad et al. 2021b), agricultural fertilizers, pesticides, wastewater irrigation (Khan et al. 2021, Muhammad et al. 2020), mining operations and vehicle emissions enrich the soil Pb concentrations (Sisman et al. 2002).

The elevated Pb concentrations in soil may pose serious threats to plants, animals and humans due to its persistent nature and toxicity (Hussain et al. 2020, Muhammad et al. 2021a, Muhammad & Nafees 2018, Muhammad et al. 2021b). The Pb percolates through the soil horizons and contaminates the groundwater reservoirs (Luo et al. 2012). Edible crops and vegetables also uptake Pb from soil, and enter human body through food chain, thus causing potential health risks (Muhammad et al. 2020). The bioavailability and fate of potentially toxic metals (PTEs) in soil are largely controlled by (i) sorption process (removal of the concerned PTE(s) from soil solution by surface attachment on soil particles), (ii) desorption process (release of the concerned PTEs(s) from soil particles to soil solution), and (iii) precipitation process (Cerqueira et al. 2011, Moreno et al. 2006, Shaheen 2009). Empirical laboratory investigations of sorption-desorption provide information on the interaction of PTE(s) in soil, predicting the environmental hazards and development of policies to regulate the use of PTEs (Pb) containing products and disposal in soil (Sastre et al. 2007, Shaheen 2009).

Sorption is an important natural process that takes place at the sediment-water interface (Zhang et al. 2017). Heavy metals that are released into aquifers by a variety of natural and anthropogenic sources, interact with various geosorbents through sorptive interactions (Yeboah et al. 2019). As the sorption is a surface phenomenon, which strongly depends on surface properties, *i.e.* soil texture, and the most finer particle (clay fraction) is the primary responsible source for sorption of heavy metals (Ahmed et al. 2019).

Some studies were carried out worldwide on the interactions of Pb in soils (Ahmed et al. 2019, Vytopilova et al. 2015, Yeboah et al. 2019, Zhang et al. 2017), but no study has been conducted in Pakistan, nor in the study area, that assessed the interactions' (sorption-desorption) of Pb onto different textures, organic carbon (OC) contents, organic matter (OM) contents and pH, agricultural and non-agricultural soils. Therefore, the present study was designed with the purpose to: (i) investigate the sorption-desorption behavior of Pb in six different agricultural soils of Khyber Pakhtunkhwa province, Pakistan, (ii) assess the role of soil properties (texture, OC, and pH) in Pb retention, and (iii) identify the leaching potential of Pb from the selected agricultural soils.

Materials and methods

Study area

This study was conducted in five districts of Khyber Pakhtunkhwa province, Pakistan including Peshawar, Charsadda, Swabi, Lower Dir and Upper Dir (Fig. 1). The land area of Peshawar, Charsadda and Swabi is largely plain

and fertile, and the agricultural fields are mostly irrigated with municipal wastewater. Conversely, the land area in Lower Dir and Upper Dir districts is largely mountainous and the agricultural fields are irrigated with fresh water of river and natural streams. These 5 districts are the most productive districts of Khyber Pakhtunkhwa province, so these were selected for the conducting this study.

Soil sampling and preparation

Soil samples were collected from the selected districts, *i.e.* Peshawar, Charsadda (I & II), Swabi, Lower Dir and Upper Dir (Fig. 1). The soil samples were collected at 0-100 cm depth using stainless steel auger from the agricultural fields in the selected districts (Fig. 1). Composite samples were collected from the agricultural fields of the concerned districts. Briefly, two soil samples were collected from less than one acre and four samples were collected from more than one acre agriculture fields and thoroughly mixed to become one composite sample (Muhammad & Nafees 2018). All the collected samples were stored in Kraft paper, sealed and labelled (Yang et al. 2011). The samples were homogenized, air dried, and sieved through a 2-mm mesh.

Soil analysis for physicochemical properties

The prepared soil samples were analyzed for basic physico-chemical properties such as, soil texture, pH, OM, and OC were determined using standard techniques (Estefan 2013). Soil pH was measured using 1:5 soil-water extract with a calibrated pH meter pH (pH meter, Seven2Go™), The OM contents in the soil samples were determined through loss on ignition technique as described by Khan et al. (2016). Briefly, take 10 g of the air dried and ground sample in crucible and keep at 110 ± 5 °C for 2 h, cold and weighted. Then the samples were heated in muffle furnace at 550 ± 5 °C for 4 h, cold and weighted again. The percent variance was counted as OM contents. Soil texture was calculated through pipette method. Briefly, a mixture of 20 g soil, 5 mL sodium hexa-metaphosphate ($\text{Na}_6\text{P}_6\text{O}_{18}$) and small amount of DI water was prepared and shaken for 5 minutes, poured in graduated cylinder and diluted up to 500 ml and mixed. Then took 1st aliquot at 10 cm top at 48 S interval and 2nd aliquot at 5 cm top at 40 minutes interval; dried overnight at 105 ± 5 °C in oven. The reading of the dried samples were noted and put in the pre-defined formula (Klute 1986). Textural class was assigned to the soil samples using the textural triangle of the United States Department of Agriculture.

Preparation of Pb solution

Stock solution (1000 mg L^{-1}) was prepared as $\text{Pb}(\text{NO}_3)_2$ in 0.01 M background electrolyte solution of calcium nitrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$. A series of Pb dilution solutions (5, 10, 25, 45, 60 mg L^{-1}) were prepared from the stock solution by diluting a known amount from the stock solution of desired concentration. The 0.01 M $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ was selected to eliminate the nonspecific adsorption of Pb as the low affinity sorption sites are saturated with Ca ions (Gray et al. 1998).

Batch sorption and desorption experiments

Batch sorption experiments were performed to quantify Pb sorption onto the soil samples following the method adopted by Yang et al. (2006) and (Dutta & Singh 2011, Dutta et al. 2011). Briefly, soil samples (1 g) in triplicates were taken in 50 ml polypropylene centrifuge tubes, added 20 ml of the Pb solution with different concentrations (5, 10, 25, 45, 60 mg L⁻¹) and equilibrated on an end-over-end rotary shaker (40 rpm) for 24 hrs at room temperature (24 ± 2 °C). The equilibrated solutions were centrifuged at 3500 rpm for 10 min. Finally, the supernatants were filtered through filter paper (Whatman No. 41) and stored in refrigerator for the analysis of Pb concentrations through ICP-OES (PerkinElmer Optima 8300 ICP-OES) following standard working procedures. Similarly, desorption experiments were performed using the method adopted by Cerqueira et al. (2011) and Yang et al. (2006). Briefly, 20 ml of 0.01 M Ca(NO₃)₂·4H₂O solution was added to the soil residues in centrifuge tubes following sorption. The suspensions were equilibrated on a rotary shaker for 24 hrs at room temperature, centrifuged at 3500 rpm for 10 min and the supernatants were filtered into 50 ml polypropylene centrifuge tubes. Desorption process was repeated thrice and stored in refrigerator for the analysis of Pb concentrations through ICP-OES (PerkinElmer Optima 8300 ICP-OES) following standard working procedures.

Sorption Model

The quantity of Pb sorbed onto the soil samples was calculated from the difference between the applied initial concentration (C_i) and the equilibrium Pb concentration (C_w) by mass balance equation, as given in Eq. (1).

$$C_s = (C_i - C_w) \times \frac{V}{W} \quad (1)$$

Where, C_s is the sorbed concentration of Pb per unit mass of soil (mg kg⁻¹), C_i and C_w are the Pb initial and aqueous equilibrium concentrations (mg L⁻¹) respectively, V is the volume of solution used (L) and W is the air-dried mass of the soil sample (Kg) (Echeverria et al. 1998, Hooda & Alloway 1998). The sorption isotherms were developed by plotting the amount of Pb sorbed (C_s) against equilibrium solution concentrations (C_w) and used to compare the sorption capacity of the different soils (Cerqueira et al. 2011, Yang et al. 2006). Whereas the amount of Pb retained by the soil samples was determined from the quantity that had been sorbed in sorption experiments, and the total recovered Pb concentration in equilibrium desorption solution (Yu et al. 2002).

Partition Coefficients

Linear partition coefficient (K_d) is the ratio of the sorbed Pb in the soil sample to the dissolved Pb in equilibrium solution (Lee et al. 1998, Li et al. 2013, Yang et al. 2006). K_d values were calculated using Eq. (2).

$$K_d = \frac{[C]_s}{[C]_w} \quad (2)$$

Where $[C]_s$ is the sorbed Pb concentration (mg Kg^{-1}) in soil, $[C]_w$ is the Pb concentration in solution (mg L^{-1}) at equilibrium. The soil organic carbon (OC) normalized partition coefficient values (${}_{10}\log K_{OC}$) were calculated from Eq. (3).

$$K_{OC} = \frac{K_d}{f_{OC}} \quad (3)$$

Where, f_{OC} is the fraction of soil OC and K_d (L/Kg) is the linear partition coefficient.

Soil Leaching Model

Leaching potential of the selected soils was determined by using transport model CXTFIT 2.1 and the STANMOD software package was used to simulate transport (Simunek et al. 1999). Influent or solute concentration was assumed to be dimensionless while calculating Breakthrough Curves (BTCs). Retardation factor (R) for the soil samples were calculated from Eq. (4).

$$R = 1 + \frac{\rho}{\theta} \times K_d \quad (4)$$

Where " ρ " denotes bulk density, " θ " represents porosity, and K_d (LKg^{-1}) is the distribution coefficients obtained from the batch sorption analysis. Dispersion coefficient " D " ($\text{cm}^2\text{day}^{-1}$) was calculated by the following equation;

$$D = \alpha_L v \quad (5)$$

Where α_L = the longitudinal dispersivity of the porous media in the direction of transport, and " v " denotes to average linear velocity.

Quality control and data analysis

For the consideration on the accuracy and precision of the data collected, all the apparatus and glassware for batch sorption experiments was acidified in 5% HNO_3 (V/V) and washed with distilled water prior to use. Solution matrix and blanks were analyzed with each sample batch to verify the precision and accuracy of the sample preparation and analysis. Each sample was analyzed in triplicate, and thus uncertainties of the measurement were calculated. After each batch of 10 samples, the ICP-OES was calibrated for standardization ($R^2 > 0.99$). The data was statistically analyzed through Statistix 10 (Analytical software, TN, USA), graphs were plotted through Origin Lab (version 2018), and elevations of samples locations were recorded using Global positioning system.

Results and discussion

Soil Properties

Based on the percentage (%) contribution of sand, silt and clay, soils of the districts were classified into several textural classes such as, silt clay loam (Peshawar), silt (Charsadda-I), clay (Charsadda-II), clay loam (Swabi), loam

(Lower Dir), and silt loam (Upper Dir) as shown in table 1. The textural classes were deeply related to the pH of the soils, such as alkaline pH (7.9) classified into clay class and acidic pH (5.8) classified into silt loam class. According to the pH, the soils were in the order of clay > silty clay loam > clay loam > silt > loam > silt loam for pH 7.9 > 7.8 > 7.7 > 7.4 > 6.5 > 5.8. Similarly, the OM and OC contents in the soils are linked to the pH of the soils. Increasing the pH from acidic to alkaline nature, the contents of OM and OC are increased and vice versa (Table 1).

Sorption Isotherms

The sorption isotherms obtained for the studied soils fit well with linear sorption model (Fig. 2). Generally, all the soil samples exhibited high affinity for Pb and sorption increased with increasing Pb initial applied concentrations. This can be explained as with increase in application more amount of Pb is available for sorption onto the soil sample. However, the percentage of Pb sorption decreased since the binding sites were more occupied (Dutta & Singh 2011). Highest Pb concentration was sorbed by the clay soil following the order as; clay > clay loam > silty clay loam \approx loam > silt loam > silt (Fig 2). Sorption coefficient and correlation (R^2) values obtained from sorption isotherm model fits for the various soils are given in Table 2.

The clay soil showed highest K_d values for the different Pb concentrations used followed by silty clay loam, clay loam, loam, silt loam and silt. A high consistency was found in the log K_{oc} values across all the six experimental soils (Table 2). The small K_d value for the silt soil can be attributed to the low OM and clay contents in the sample (Table 1). Previously, Sastre et al. (2007) obtained low K_d values for soils with high sand content, low CEC, and Fe_2O_3 fractions.

Correlation coefficients resulting from the linear regressions between K_d (L/kg) and various soil properties showed positive correlation between K_d and clay ($R^2 = 0.90$), K_d and OC ($R^2 = 0.94$) and K_d and pH ($R^2 = 0.45$), however weak correlation found between K_d and sand contents ($R^2 = 0.12$) (Fig. 3).

The OM and clay content contribute greater negative surface charge to the soils and therefore exhibit high metal sorption capacity and vice versa. Whereas, sandy soils have less negative surface charge and are therefore weakly correlated with metal sorption (Hooda & Alloway 1998). The lowest sorption capacity associated with sand fraction is due to the fact that soil samples with larger particle size have lower surface area for metal sorption (Jain & Ram 1997, Rybicka et al. 1995). The alkaline soils exhibit highest metal sorption followed by neutral soils, whereas acidic soils show lowest sorption (Veeresh et al. 2003).

Desorption Experiment

Generally, a small percentage of the sorbed Pb concentration was desorbed with the 0.01 M electrolyte solution of Calcium nitrate (Fig. 4). However, more Pb mass was desorbed at high sorbed Pb concentrations. These results conformed to the study of Dutta and Singh (2011). The silt soil sample desorbed highest Pb mass (6.8%) followed by silt loam (6.4%), loam (5.7%), silty clay loam (5.2%), clay loam (4.0%) and clay (2.9%) (Fig. 4). The different sorption

and desorption relationships of soils suggested that various soils may have different sorption mechanisms (Yang et al. 2006).

Among the heavy metals, Pb is the most retained and highly sorbed metal in the soil matrix (Covelo et al. 2007, Pokrovsky et al. 2012, Zhao-Fu et al. 2007). It is always presumed that the clayey soils having negative charge interact with cations such as Pb, and retard its transport into the subsurface (Wa'il & Haddad 2007).

Sorption and desorption of Pb in soils is largely influenced by the soil physico-chemical properties such as the soil pH, OM and clay contents (Huang et al. 2012, Martinez-Villegas et al. 2004) (Lee et al. 1998, Sipos et al. 2005). At lower concentration in soil, Pb is strongly sorbed at inner sphere complexes of the soil components with bonding functional groups (Moreno et al. 2006, Mouni et al. 2009). However, at higher concentration Pb may also be sorbed weakly and or precipitated at non-specific or ion exchange sites (Moreno et al. 2006, Mouni et al. 2009).

Soil Leaching Model

The calculated leaching parameters for the selected soil samples are summarized in Table. 3. Break through curves (BTCs) was calculated on K_d values obtained from the batch sorption part. Plots of the curves obtained were identical and each plot arrived earlier at the point of equilibrium (Fig. 5). The identical BTC plots for the different soils may be due to the less difference in K_d values and soil properties. Short breakthrough times indicated that the sorption bed will become saturated quickly, thus indicated potential of Pb leaching. Besides, less time is available for sorption under the field conditions, which may result in high probability of leaching from the agricultural soils.

Conclusion

In-vitro sorption studies are useful to assess the metal mobility, availability, and risk from contaminated soils. The six agricultural soils used in this study sorbed different concentration of Pb depending on the soil physico-chemical properties. Highest Pb concentration was sorbed by the clay soil with high OM content. Sorption of Pb was positively correlated with the soil OM, pH, and clay contents. Whereas silt soil showed least affinity for Pb sorption. The results of sorption-desorption studies fitted well with Langmuir adsorption isotherms. The best correlation of the K_d with soil clay and OC suggest that clay and OC contents are the major Pb sorption domains in soil. However, with increasing Pb initial applied concentrations, the percentage of Pb sorption was decreased indicating that at high concentration Pb may become mobile and migrate down the soil profile. The leachability model also indicated potential risk of Pb leaching from the agricultural soils.

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Competing interests

The authors declare no competing interest in the present study.

Availability of data and materials

The datasets used and analysed during the current study are available from the corresponding author on reasonable request (if needed).

Code availability

The datasets codes and basic data used and analysed during the current study are available from the corresponding author on reasonable request.

Authors' contribution

(i) Iqbal Ahmad: Conceptualization, Methodology, Funding acquisition, Data curation, Formal analysis, and Writing original draft. (ii) Bushra Khan; Investigation, Analysis, Data curation, Review, and Editing. (iii) Nida Gul: Validation, Data curation, Review, and Editing. (iv) Muhammad Tariq Khan: Methodology, Data curation, Review, and Editing. (v) Javaid Iqbal: Visualization, Data curation, Review, and Editing. (vi) Nisar Muhammad: Conceptualization, Funding acquisition, Project administration, Resources, Supervision, Validation, Writing, Review, and Editing.

Ethical approval

This research has been obliged to research integrity, transparency and ethical values.

Consent to participate

No public community or person was involved to get some kind of data from him/her.

Consent to publish

No data has been taken from public person, nor his/her guardian that needed specific permission to publish.

Animal research

No animal was carried out under study in this research.

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Figures

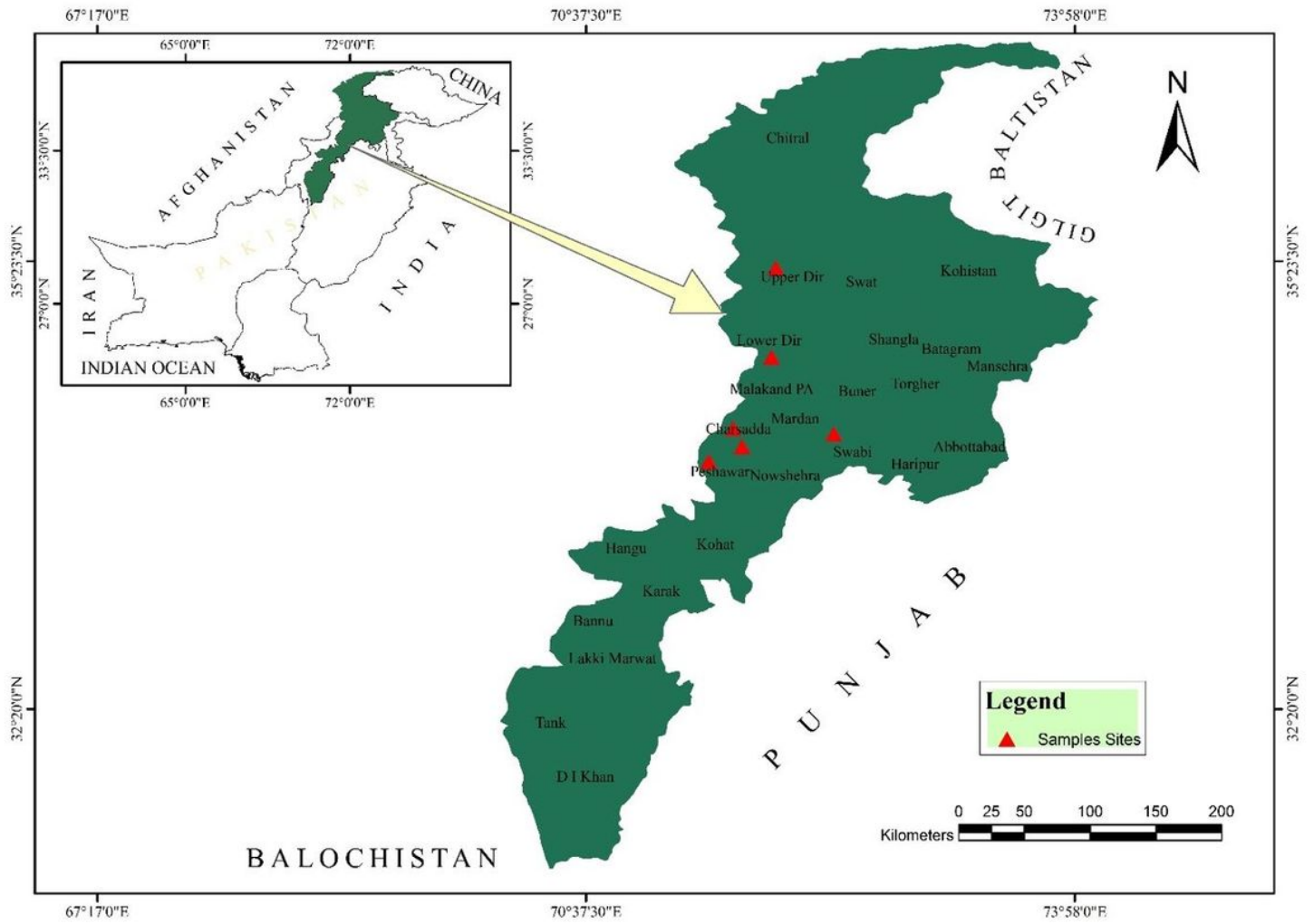


Figure 1

Map of the study area for soil sampling locations

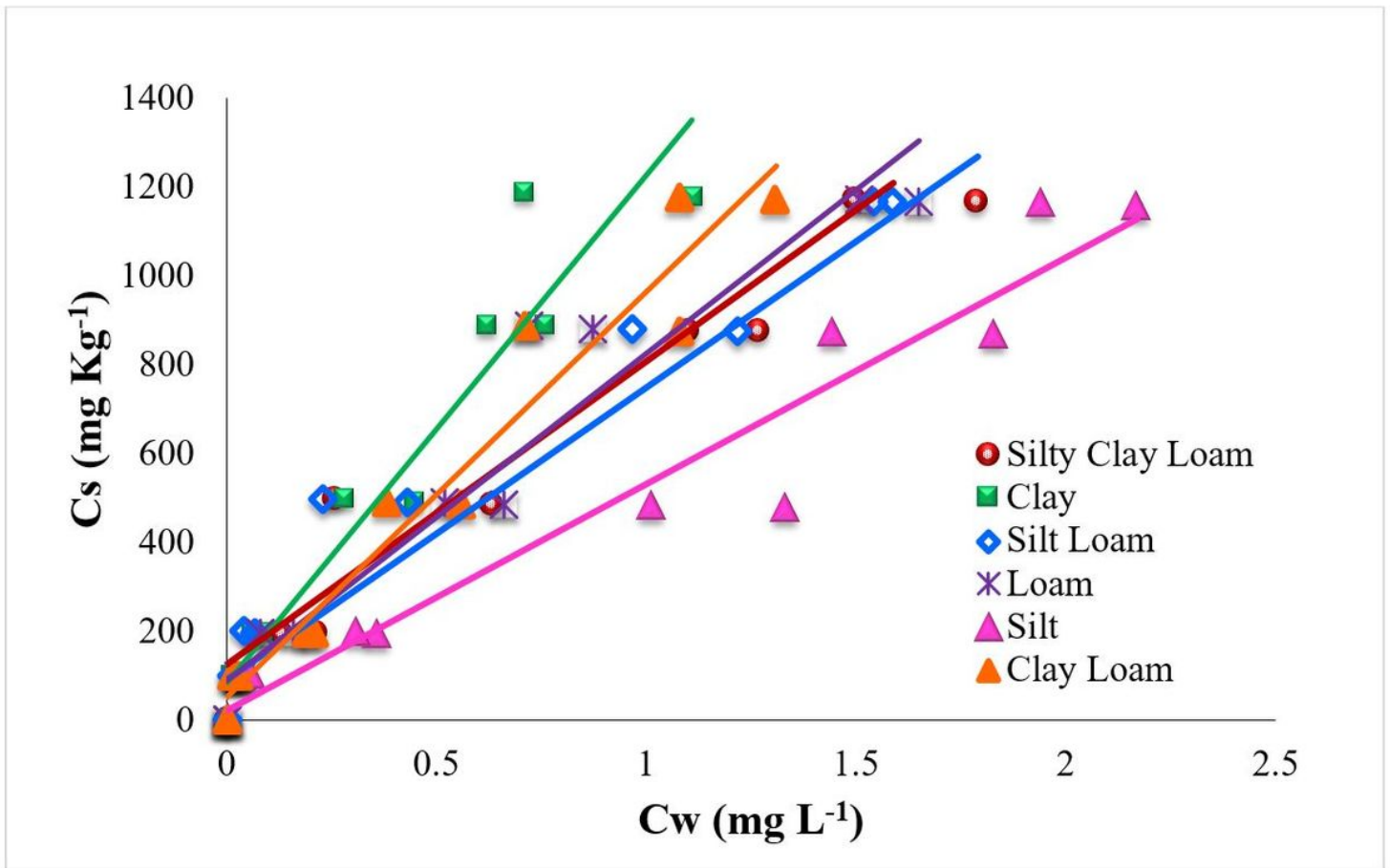


Figure 2

Sorption isotherms for Pb retention by the agricultural soil samples

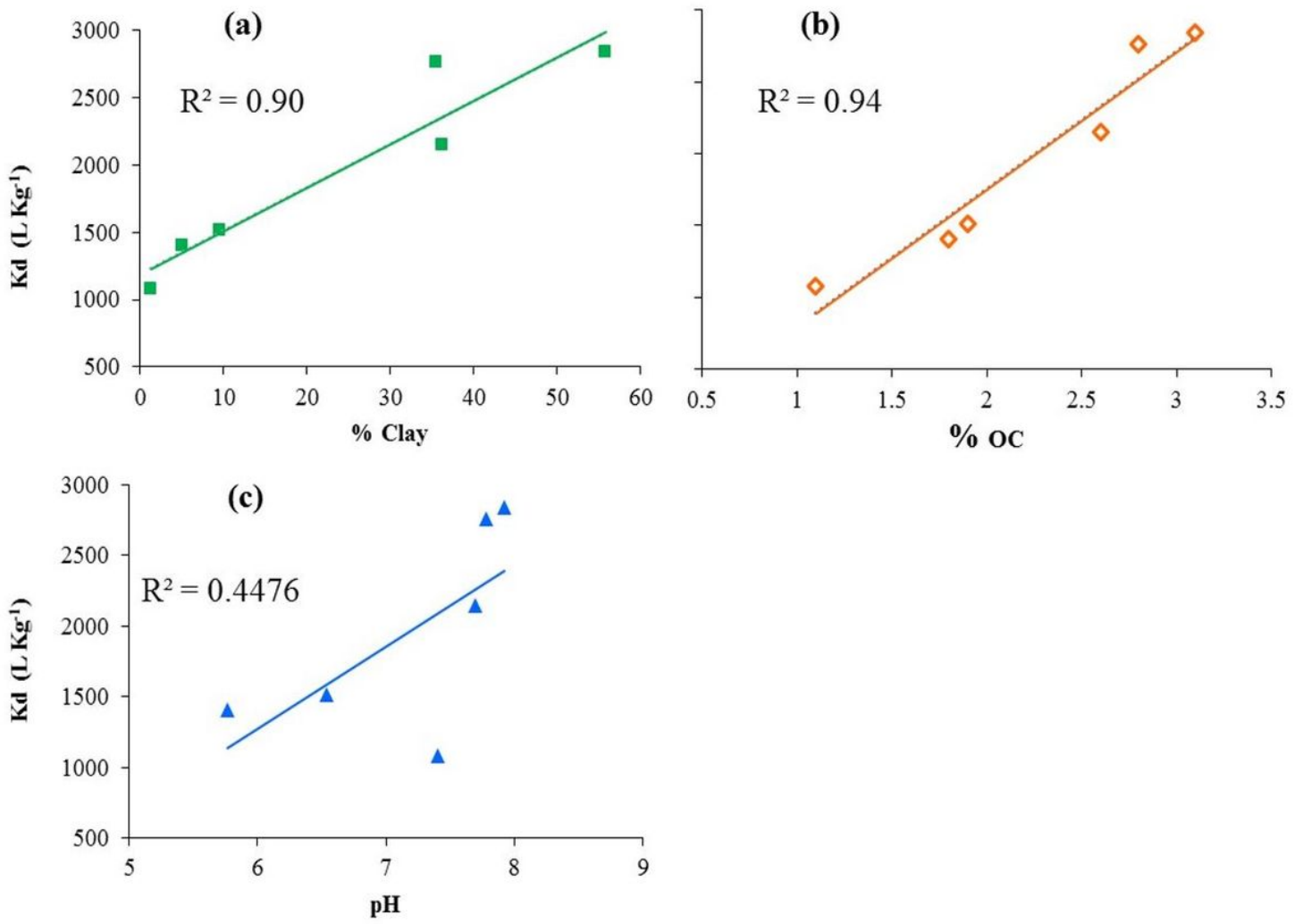


Figure 3

Linear correlation among the partition coefficients (K_d) of various soils with soil properties (a) clay, (b) OC and (c) pH

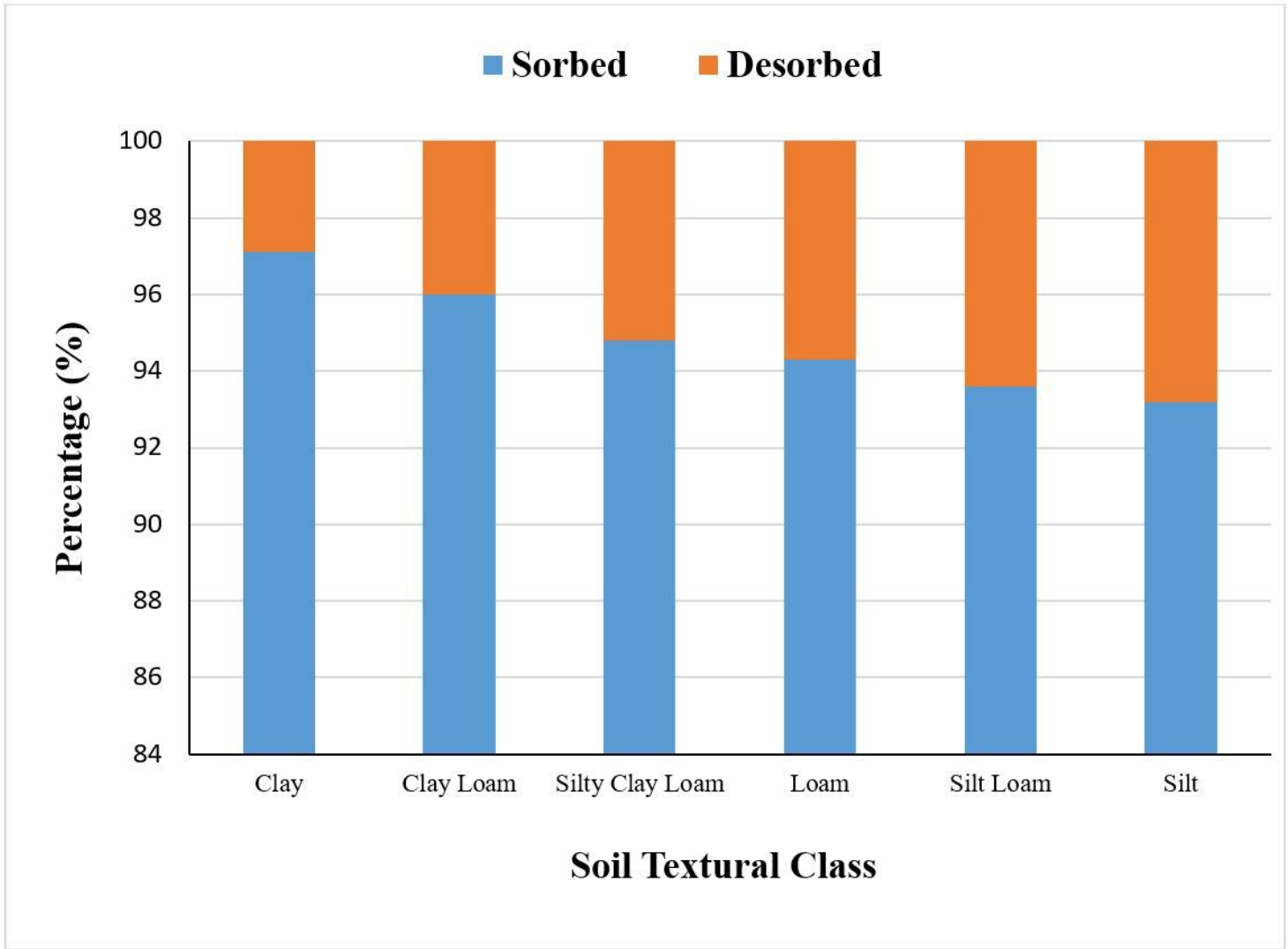


Figure 4

Percentage of Pb sorbed-desorbed by the soil samples

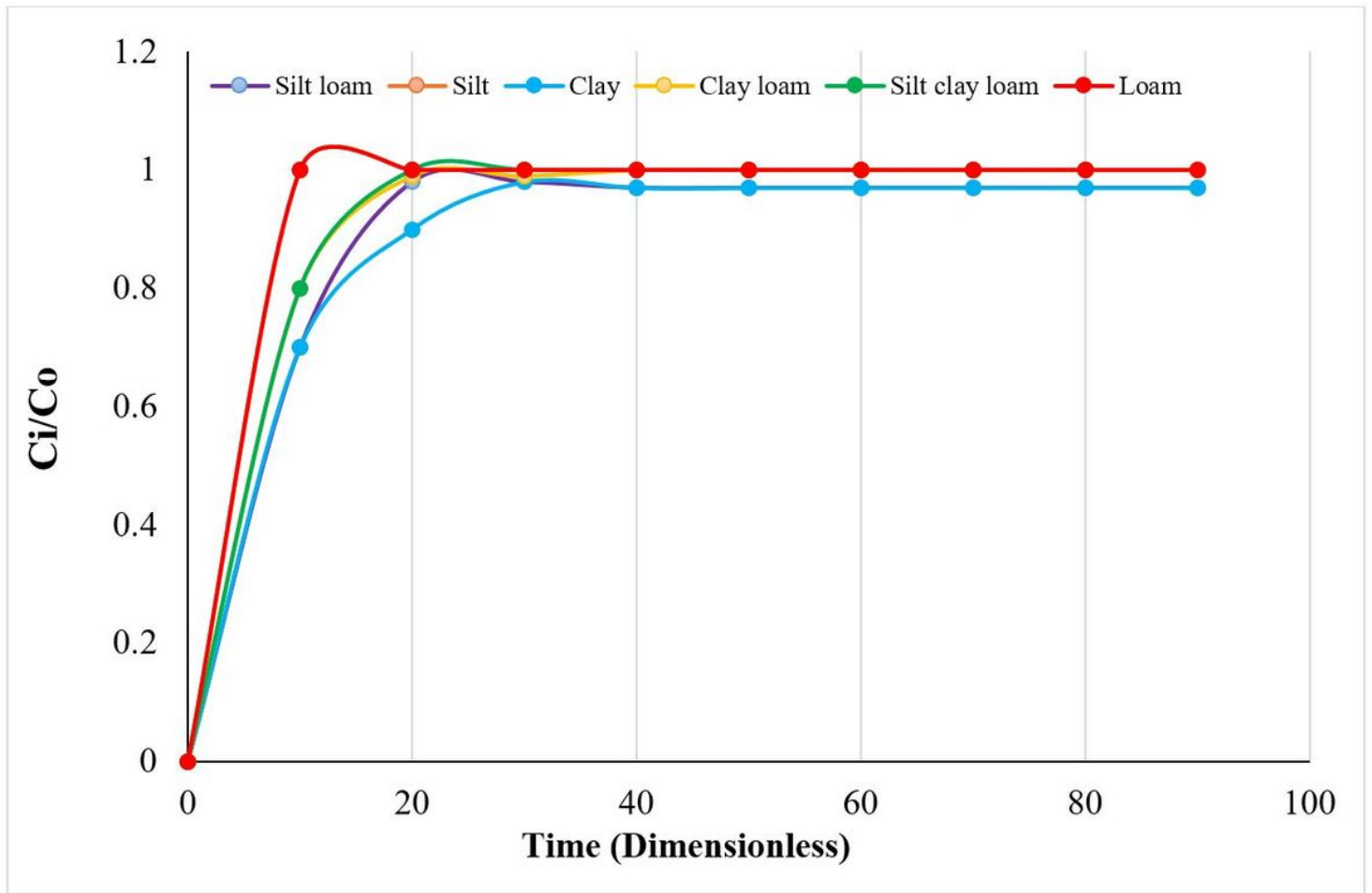


Figure 5

Breakthrough curve plots for soil leaching model of the various soils