



Kinetics and isotherm modeling of Pb(II) and Cd(II) sequestration from polluted water onto tropical ultisol obtained from Enugu Nigeria

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Abstract

Deterioration in soil–water environment severely contributed by heavy metal bioavailability and mobility on soil surface and sub-surface due to irrational increase in wastewater discharge and agrochemical activities. Therefore, the feasibility of adsorption characteristics of the soil is paramount in curbing the problem of micropollutant contamination in the farming vicinity. Soil from a farming site in a populated area in Enugu, Nigeria was collected and tested to measure the lead and cadmium contents using atomic absorption spectrophotometer (AAS). The adsorption potency of the ultisol soil was estimated for identifiable physicochemical properties by standard technique. The mean activity concentration of Pb²⁺ and Cd²⁺ was 15.68 mg/kg and 3.01 mg/kg. The pH, temperature, metal concentration and contact time adsorptive effect on the Pb²⁺ and Cd²⁺ uptake was evaluated by batch adsorption technique. The Langmuir, Freundlich and Temkin models were fitted into equilibrium adsorption data and the calculated results depict a better and satisfactory correlation for Langmuir with higher linear regression coefficients (Pb²⁺, 0.935 and Cd²⁺, 0.971). On the basis of sorption capacity mechanism of the soil, pseudo-second-order model best described the kinetics of both metal ions retention process. The results of the present study indicated that the soil being a low cost-effective adsorbent can be utilized to minimize the environmental risk impact of these metal ions.

Keywords Adsorption · Heavy metals · Ultisol · Farming site · Isotherm · Kinetics

Introduction

Composite structured soil medium constitute the environmental basis for plants and other biomass survival as well as interaction of minerals and organic matter in ionic formation being instigated by various industrial, agricultural and other anthropogenic activities. In this recent economic face and urbanization, the soil surface is being endangered via assimilation or uptake of some hazardous metallic ions that can affect human health inclusively in the food chain.

Among the metallic toxic inorganic micropollutants with unique properties heavy metals are natural elemental constituents of parent materials in rocks and sediments being needed by the soil in trace amount for optimum performance of plant physiological process (Emenike et al. 2016) whereas the non-essential parts are venomous to plants and humans with increased toxicity in water, soil and air accumulations at very low concentrations tolerance management (Ezeudo 2014). They form precipitates or complex ions with other compounds in mobile phase of the soil diffusing into the food chain infusing carcinogenic threat through translocation to plants. The extensive utilization of agrochemical, wastewater irrigation and other anthropogenic sources through agricultural activities have protrude to an unending release of some toxic heavy metals (Sharma et al. 2006) into the environment which is huge global concern in relation to ecology and human health menace (Wuana and Okieimen 2011; Dube et al. 2011; Hegazi 2013; Tang et al. 2009; Raymond et al. 2014). Increasing mobility rate of these metallic ions in the soil can lead to polluted growing crops and plants. Hence, the solubility of heavy metals in deeply

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farming soil solutions is of increasing apprehension to the populace as a result of physiological negative influence on the plants as regards to food safety issues, potential health risks such as kidney damage, neurological disorder, cancer, fragile bone and other devastating effects on soil ecosystem (Musa et al. 2017). The capacity of the soil to control excessive uptake of the toxic metals to the root and shoot of the plants readily depends on the type, morphology and porosity of the soil. The productivity of heavy metal polluted agricultural soil can be severely affected by inhibiting the rate of photosynthesis in plants yielding poor crop output and also deteriorating the quality of air and water environments (Umeh et al. 2019, Raymond et al. 2014). The studied town is widely known for farming activities especially some places at Nsukka inducing poorly treated wastewater and other soil-enriching practices that keep metallic ions in soluble and mobile states. Very high concentration of lead and cadmium has been reported so far in tropical Ultisol soils in Enugu tropical and subtropical soils (Oluwasola et al. 2019). Several proposed applied treatment techniques to reduce and control the mobility of these heavy metals in the soil solution have been compared and adsorption process have tendency to abstract and retain toxic metallic ions from aqueous media owing to its economic feasibility, accessibility and efficient purification (Ramachandran and Souza 2013).

The ionic retention at the boundary crust between the adsorbate specie and soil adsorbent is due to some forces of attraction and chemical bondings between the two phases (Siti et al. 2013) and is being influenced by some soil properties such as texture, density, pH, organic matter, CEC, type, clay minerals present in the soil. It occurs as a result of existing unsteady residual forces at the external surface of the liquid or solid that attracts and uphold the molecular species which comes in contact with the adsorbing surface liberating energy to the surroundings (Babel and Kurniawan 2003). The deleterious effect and fate of heavy metals to soils in temperate regions (Al-Hamdan and Reddy 2006) have been severally researched on while significant outcome of data analysis on these toxic micropollutants in African tropical soils are minimal.

In this part of African country, massive population growth and development in industries have forcefully contributed to continual use of agricultural lands with high level of health risk heavy metals harbor. The food security issues emanating from heavy metal polluted soil ecosystem are unbearable which require urgent monopoly.

Presently, the studied area entails largely agricultural practices that can build-up unhealthy heavy metals leading to food chain degradation. Therefore, the aim of this research is to determine the mean concentration of lead and cadmium and physicochemical characteristics of tropical ultisol from an intensely agricultural area as to estimate its adsorption

potential for these contaminants using sorption models and kinetics.

Materials and methods

Sampling analysis and physicochemical evaluation

The top soil samples were obtained randomly from varying spots during rainy season at Opi in Nsukka town of Enugu State, Nigeria to a depth of 30 cm from the farming location and carefully mixed together in a homogenized setting to achieve a composite sample. Soil sample was pulverized gently with a pestle and mortar after drying the for some hours (72 h) to remove moisture and thereafter, sieved through a 2-mm mesh size sieve before being stored in a closed cylindrical glass sterile container.

The quantitative spectroscopic analysis was adopted for the tropical ultisol characterization. The heavy metal content in the soil sample was evaluated using the atomic absorption spectrophotometer (AAS) after soil digestion using concentrated nitric acid and hydrochloric acid (1:3) (Ure et al. 2003). The method of Bouyoucos hydrometer was used to ascertain the particle size distribution (Bouyoucos 1964). The potentiometric method being stated in literature (Mclean 1982) was applied to determine soil pH in water and KCl. Adopted literature procedures were utilized to discern the Cation exchange capacity (Chapman, 1965), Organic carbon (OC) and organic matter (Nelson and Sommers 1996) in the agricultural tropical soil sample. The colorimetric estimation of K^+ and Na^+ was performed using Flame photometer. Exchangeable acidity i.e. Al^{3+} , H^+ (Mclean 1982) and bases, i.e., Ca^{2+} , Mg^{2+} (Thomas 1982) were evaluated by titrimetric analysis. The soil textural codification was estimated by calculation (Soil survey, 2006).

Adsorption procedure

The studied heavy metals adsorptions by Enugu soil were determined using an equilibrium batch technique. An exact concentration of lead and cadmium was prepared by dissolution of 1.598 g of $Pb(NO_3)_2$ and 2.744 g of $Cd(NO_3)_2$ in 1L of distilled water. Some factors affecting the uptake of the adsorbate species by an adsorbent were varied. The effect of initial adsorbate concentration (15, 30, 50, 100, 150 mg/L), pH (2.0, 4.0, 6.0, 7.0, 9.0), temperature (303, 308, 318, 323, 333 K) and contact time (5, 15, 30, 50, 60 min) were verified. The sorption studies were carried out by adding 0.1 g of soil sample into 20 mL of each metal ion solution and agitated on a circular mechanical magnetic shaker at 120 rpm room temperature for an hour.

The equilibrated soil–liquid contents were centrifuged using a centrifuge (US M-512) at 3000 rpm and filtered at the end of the given contact time. At the variation of each equilibrium adsorption parameter, other sorption factors are kept constant at optimal condition. The removal efficiency of the soil adsorbent for Pb^{2+} and Cd^{2+} metal ions was determined gravimetrically using Shimadzu atomic absorption spectrophotometer (model AA-7000) at a wavelength of 283.3 nm and 228.8 nm respectively. Each experimental stage was done in triplicate and the mean calculated results were computed. The quantity of lead and cadmium ions removed at equilibrium (mg/g) per unit mass by the tropical soils and the percentage uptake was determined and calculated by the described equation (Shaban et al. 2018):

$$Q_e = \frac{C_0 - C_e}{M} V \quad (1)$$

$$A_e (\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

where C_0 and C_e are the initial concentrations of metal ions solutions (mg/L) and equilibrium concentrations (mg/L). V is the volume of metal ion—soil solution, while M is the mass of soil adsorbent (g). A_e and Q_e are the adsorption efficiency and quantity adsorbed.

Adsorption isotherm

Few sorption models were used to evaluate the equilibrium adsorption processes. Langmuir sorption theory described the homogeneity distribution of adsorbed molecules on sorbent active sites (Mbadcam et al. 2011) and is given by the following linear equation (Langmuir 1918):

$$\frac{C_e}{q_e} = \frac{1}{q_L K_L} + \frac{C_e}{q_L} \quad (3)$$

where q_e (mg/g) and C_e (mg/L) are the quantity of solute adsorbed per unit mass of adsorption and equilibrium solute concentration in the bulk solution. K_L (L/g) is constant while q_L (mg/g) is monolayer adsorption efficiency. The dimensionless constant, R_L provide appropriate articulation of Langmuir isotherm (Unuabonah et al. 2007) expressed as:

$$R_L = \frac{1}{1 + K_L C_0} \quad (4)$$

The identification of the stated parameters in the above equation has been earlier described in (Kovo et al. 2015) literature.

Freundlich isotherm interprets the multilayer adsorption on heterogeneous adsorbent active sites and is being expressed by this linear equation (Freundlich 1906):

$$\text{Log } q_e = \text{Log } K_F + \frac{1}{n \text{Log } C_e} \quad (5)$$

where q_e (mg/g) and C_e (mg/L) are the quantity of solute adsorbed per unit mass of adsorption and equilibrium solute concentration in the bulk solution. K_F (mg/g) is the isotherm constant and $1/n$ indicates the intensity of the process of adsorption (Tang et al. 2009).

Temkin isotherm was used to evaluate the level of adsorption temperature and bonding energies of the molecules in layer during sorbate and sorbent interactions and is described by the following equation in its linearized form (Temkin 1940):

$$q_e = B \ln A + B \ln C_e \quad (6)$$

where q_e (mg/g) and C_e (mg/L) are the quantity of solute adsorbed per unit mass of adsorption and equilibrium solute concentration in the bulk solution. The plot of q_e against $\ln C_e$ enables to determine A and B .

Kinetic modeling

The kinetics of adsorption mechanism was analyzed by pseudo-first-order, pseudo-second-order and Weber and Morris Diffusion models.

Pseudo-first-order kinetic entails the comparative to the disparity of the adsorption rate between the capacity of adsorption (q_e) in stability and the capacity at any time (Ibrahim et al. 2006). The linearized form of equation (Wahab and Zaghoul 2007; Jain et al. 2004) is given by:

$$\log (q_e - q_t) = \log q_e - \left(\frac{K_{1t}}{2.303} \right) t \quad (7)$$

where q_e and q_t (mg/g) are the amount of solute adsorbed per unit mass at equilibrium and at time respectively. K_1 (min^{-1}) is the order adsorption constant.

Pseudo-second-order model presumes that the limiting step rate may emanate from adsorbing substance involving valence forces by electrons interchange tween the adsorbent and adsorbate species (Demirbas et al. 2007) and is expressed in linear equation (Debnah and Ghosh 2008) as follows:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \left(\frac{1}{q_e} \right) t \quad (8)$$

K_2 (g/mg min) is the pseudo-second-order adsorption constant at equilibrium and $h = K_2 q_e^2$.

Weber and Morris or intra-particle diffusion model was evaluated to determine the speed of diffusion of molecules and is given by the following equation (Igwe and Abia 2007):

$$q_t = K_{id}t^{1/2} + I \quad (9)$$

where q_t is the amount of metal ions adsorbed (mg/g) at time. K_{id} (mg/g min^{1/2}) is the intra-particle diffusion constant and I is the intercept. The linear plot of q_t versus $t^{1/2}$ shows that during the mechanism of intra-particle diffusion, the exclusive rate-determining step takes place (Guler and Sarioglu 2013).

Results and discussion

Soil sample characterization

The physicochemical properties of a tropical soil from deeply cultivating location are presented in Table 1. The soil sample is classified as sandy loam with slightly acidic pH value in aqueous solution and metals exist as free ionic species or as soluble organometals and are more bioavailable. The organic matter content (OM) and the cation exchange capacity (CEC) of the agricultural soil are high which ensure a dependable heavy metal adsorption capacity. Acidity level of the soil is attributed to the organic content as a result of biological activities and agrochemical processes through metallic complexation (Osakwe and Okolie 2015). There is tendency of the soil sample to provide an active site for cation exchange as a result of its high clay particle content.

The mean concentration of lead and cadmium in the agricultural tropical soil are 15.68 mg/kg and 3.01 mg/kg

Table 1 Mean physicochemical parameters of studied soil sample

Parameters	Opi
Soil textural class	Sandy loam
Fine sand (%)	45.07 ± 0.02
Silt (%)	11.26 ± 0.01
Clay (%)	15.40 ± 0.02
Coarse sand (%)	42.32 ± 0.02
Soil order	Ultisol
Organic matter (%)	2.30 ± 0.03
pH (H ₂ O)	6.52 ± 0.02
pH (KCl)	4.50 ± 0.01
Cation exchange capacity (CEC) (me/100 g)	12.10 ± 0.01
Organic carbon (%)	1.56 ± 0.04
Exchangeable acidity (me/100 g)	
H ⁺	0.63 ± 0.01
Al ³⁺	ND
Exchangeable bases (me/100 g)	
Na ⁺	0.39 ± 0.01
K ⁺	0.17 ± 0.01
Ca ²⁺	7.40 ± 0.02
Mg ²⁺	2.30 ± 0.02

respectively which are relatively high according to world health organization (WHO 2008) standard as a result of unremitting application of phosphatic fertilizer, untreated wastewater irrigation and other agrochemicals at various stages of crop production.

Effect of pH

Solution pH is among the vital parameters that determine the feasibility of sorption process as it readily influences the degree of ionizable metal ions in their specified chemical state (Sheng et al. 2004). The experimental data on the effect of pH relating to percentage removal of Pb²⁺ and Cd²⁺ from binary solution by Opi soil are shown in Fig. 1. The level of heavy metals uptake increases with increase in pH with maximum adsorption at pH ≥ 6 for Cd²⁺ while there is serial retention of Pb²⁺ onto the adsorbent surface as the pH of the medium increases as a result of sphere coverage complexation and precipitation which enables the adsorptive reactivity of the soil surface (pokrovsky et al. 2012). The deprotonation of the soil coverage site increases at a decreased acidity of soil aqueous solution due to the presence of soluble ligands forming complex compound with the free metals. The removal efficiency of Cd²⁺ was low in an acidic condition as a result of electrostatic repulsion on the bonding location of the adsorbent site. This present study is comparable with some reported literature (Das et al. 2014).

Effect of metal ion concentration

The effect of concentrations on Pb²⁺ and Cd²⁺ adsorption capacity of Opi tropical soil are presented in Fig. 2. Metal ion concentration provides a energy force that overwhelm the extent and effect of transfer of mass resistance of metallic ions between the aqueous and solid phases (Kovo et al. 2015). It was observed from the plot that when the Pb²⁺

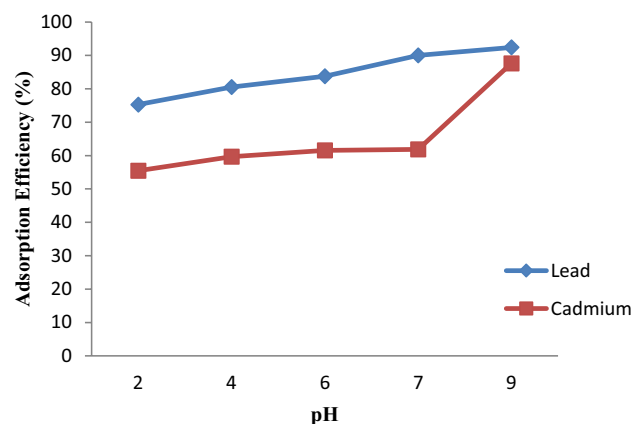


Fig. 1 Effect of pH on Pb²⁺ and Cd²⁺ Adsorption by Opi soil

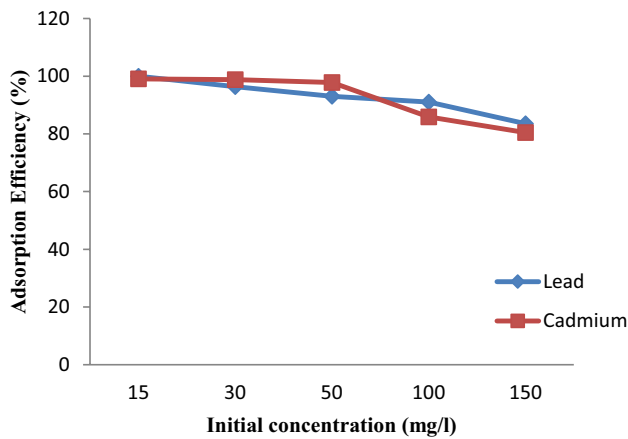


Fig. 2 Effect of concentration on Pb²⁺ and Cd²⁺ Adsorption by Opi soil

and Cd²⁺ concentrations increased from 15 to 150 mg/l, the percentage removal of the studied soil decreased which indicates that the insufficient number of active coverage sites of the adsorbent becomes saturated at higher concentrations. Hence, the increase in heavy metal uptake capacity led to an increase increases in metal ions concentration at equilibrium. The soil affinity of the metallic ions is in the order Pb²⁺ > Cd²⁺ at concentration of 15, 100 and 150 mg/l. Similar trend has been earlier reported on adsorption of Pb²⁺ and Cd²⁺ by lateritic soil (Udoeyo et al. 2010).

Effect of time

The uptake level of Pb²⁺ and Cd²⁺ as a function of contact time are shown in Fig. 3. The experimental plot revealed that the metal ions adsorption rate was rapid at the initial stage and gradually reduces as it approaches equilibrium. Therefore, the sorption potential of the studied soil sample

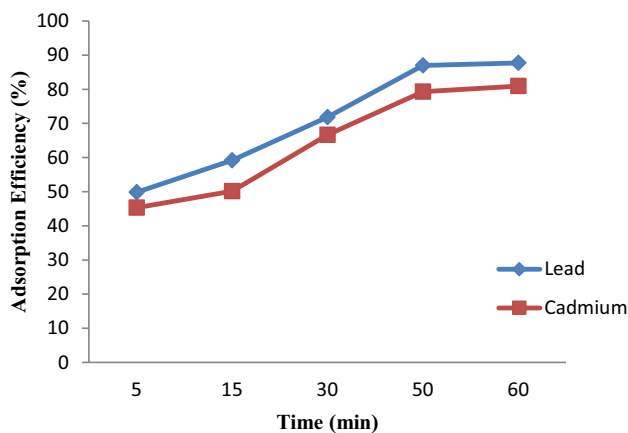


Fig. 3 Effect of Time on Pb²⁺ and Cd²⁺ Adsorption by Opi soil

increases as the time increases. This implies that during the diffusion process of adsorption, the number of pores on the adsorbent active binding centres increases that sustain the adsorbate species from bulk solution to the adsorbent surface minimizing the mobility and availability of lead and cadmium ions in soil aqueous media. Furthermore, the Opi ultisol has been severely undergoing agricultural practices which activated an increased organic matter and cation exchange capacity contents (Table 1) leading to formation of stable coating as the soil ages. The physicochemical constituents of the soil contributed immensely to its metal ion adsorption efficiency by exposing the available binding sites of the adsorbent as time increases. Some related studies (Mbadcam et al. 2011; Umeh et al. 2019; Olaofe et al. 2015) observed similar trend with this present study.

Effect of temperature

The experimental data relating to the varying effect of temperature on adsorption efficiency of Cd²⁺ and Pb²⁺ from solution onto Opi soil are shown in Fig. 4. As observed, an increased temperature from 303 to 333 K gave a notifying increase in metal ions adsorption. It is evident that the kinetic energy of the mobile adsorbate molecules in solution are activated as the temperature rises approaching the pore enlarged adsorbent centre faster to be adsorbed. The removal efficiency of the heavy metal ions is in this array Pb²⁺ > Cd²⁺. Similar study has been documented in literature (Oluwasola et al. 2019; Nappadol and Pongsakom 2014).

Adsorption isotherm analysis

The equilibrium relationship between the concentration of lead and cadmium adsorbates and the soil adsorbent coverage in solution at a given operational units were assessed by

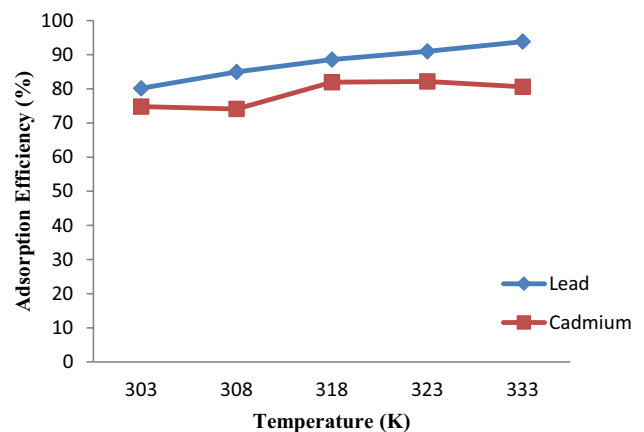
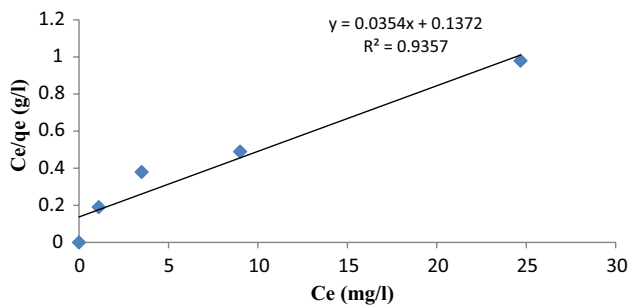
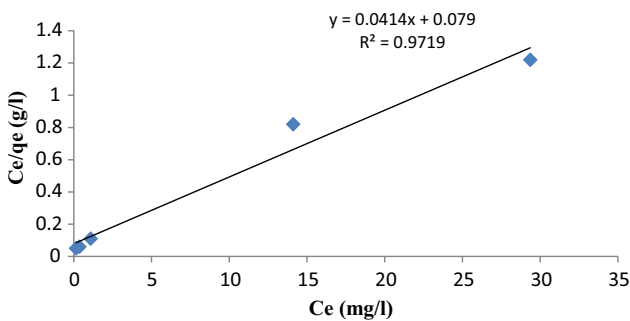


Fig. 4 Effect of Temperature on Pb²⁺ and Cd²⁺ Adsorption by Opi soil

Table 2 Equilibrium isotherm parameters for Pb²⁺ and Cd²⁺ adsorption by studied soil

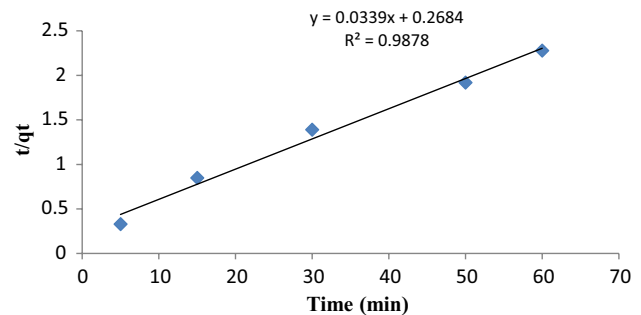
Parameters	Pb ²⁺	Cd ²⁺
<i>Langmuir</i>		
q _L (mg/g)	28.57	24.39
K _L (L/mg)	0.26	0.52
R _L	0.21–0.03	0.12–0.01
R ²	0.935	0.971
<i>Freundlich</i>		
K _F (mg/g)	4.20	7.46
n	1.67	2.82
R ²	0.925	0.950
<i>Temkin</i>		
B (mg/g)	6.57	3.67
A (L/g)	1.68	2.65
R ²	0.933	0.968

**Fig. 5** Langmuir isotherm plot of Pb²⁺ adsorption onto Opi soil**Fig. 6** Langmuir isotherm plot of Cd²⁺ adsorption onto Opi soil

Langmuir, Freundlich, and Temkin isotherms. The studied isotherms provided an insight in optimization of the ultisol adsorbent of its affinity for the metal ions adsorption in addition to the analyzable physicochemical properties of the studied soil. The parameters of the equilibrium isotherms are illustrated in Table 2. A good and satisfactory description for the Pd²⁺ and Cd²⁺ adsorption efficiency of the Opi tropical ultisol was attested by Langmuir isotherm as shown

Table 3 Kinetic model parameters for Pb²⁺ and Cd²⁺ adsorption by studied soil

Parameters	Pb ²⁺	Cd ²⁺
<i>Pseudo-first order</i>		
q _e _{exp} (mg/g)	26.31	24.34
q _e _{cal} (mg/g)	18.58	17.02
K ₁ (min ⁻¹)	0.06	0.05
R ²	0.745	0.877
<i>Pseudo-second order</i>		
q _e _{cal} (mg/g)	30.30	27.78
K ₂ (g/mgmin)	4.06 × 10 ⁻³	3.96 × 10 ⁻³
h (mg/gmin)	3.73	3.06
R ²	0.987	0.984
<i>Intraparticle diffusion</i>		
I (mg/g)	9.70	8.05
K _d (mg/gm ^{1/2})	2.20	2.14
R ²	0.987	0.971

**Fig. 7** Pseudo-second-order plot of Pb²⁺ adsorption onto Opi soil

in Figs. 5 and 6 followed by Temkin isotherms with high correlation value, R² of 0.935 (Pb²⁺) and 0.971 (Cd²⁺). This implies lead and cadmium monolayer coverage applicability and evenly distribution of bonding energies on the studied soil surface during adsorbent—adsorbate interactions. The higher uptake of Pb²⁺ (28.57) compared to Cd²⁺ (24.39) regarding maximum adsorption capacity is as a result of increasing electronegativity and decreasing ionic radii of lead. Related studies have been documented (Umeh et al. 2019; Fonseca et al. 2009; Oluwasola et al. 2019). Furthermore, the initial lead and cadmium concentrations (15 mg/L to 150 mg/L) showed ranged values of R_L from 0.21–0.03 (Pb²⁺) and 0.12–0.01 (Cd²⁺); thus proposing a favourable adsorption (0 < R_L < 1) for both metal ions affinity onto Opi ultisol soil.

Sorption kinetic analysis

The degree of adsorption rate and possible metal ions sorption mechanism on the adsorbent were evaluated by

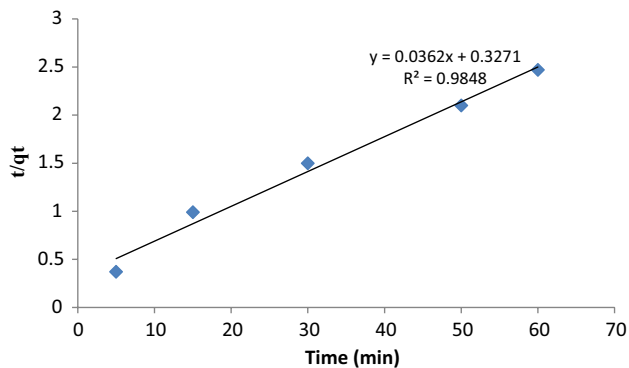


Fig. 8 Pseudo-second-order plot of Cd²⁺ adsorption onto Opi soil

pseudo-first-order, pseudo-second-order and intra-particle diffusion models (Kovo et al. 2015). The relating kinetic parameters of the utilized models are given in Table 3. A better correlation (Fig. 7 and 8) was confirmed by pseudo-second-order theory to the experimental data followed by intra-particle diffusion for Pb²⁺ and Cd²⁺ adsorption on the surface of adsorbent specie being identified with high linear regression (R^2) values. This suggests the existence of certain boundary layer effect on the soil substrate by which the rate-limiting mechanistic step involving chemical adsorption for Pb²⁺ and Cd²⁺ removal is due to the interaction of physicochemical properties between the adsorbent and adsorbate phases. The calculated q_e values were more than the experimental obtained values which additionally specify the acceptable desirable quality of the kinetic theory for adsorption of lead and cadmium onto studied Opi soil.

Conclusion

The adsorption potential of Enugu, Nigerian tropical soil for Pb²⁺ and Cd²⁺ from aqueous media was explored. The efficiency of the adsorption for the selected studied ultisol for the metal ions increases with increasing operational varying parameters. The maximum retention was attained at contact time of 60 mins which was chosen for sorption equilibrium. Amidst the tested isotherms, Langmuir theory gave a satisfactory and better fit to Pb²⁺ and Cd²⁺ experimental plot and data which confirm a homogeneous soil adsorbent surface for the heavy metals uptake. Pseudo-second-order and intra-particle models gave a similar and good conformation for the sorption mechanism of Pb²⁺ because both models emphasized on rate-limiting process for adsorbent–adsorbate physicochemical interactions while the abstraction capacity for Cd²⁺ was best suited to pseudo-second-order kinetic. Hence, the studied Enugu, Nigerian tropical agricultural soil can be useful as a low-cost effective adsorbent to control

Pb²⁺ and Cd²⁺ effects in soil–water ecosystem and as such reducing the environmental human health risk.

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Declarations

Conflict of interest The authors declare no conflict of interest.

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