SORPTION KINETICS OF LEAD AS INFLUENCED BY SOIL AGGREGATES OF TWO BENCHMARK SOILS OF SOUTHEASTERN NIGERIA.

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Abstract

It is important to examine mechanisms of lead (Pb) sorption on soils to understand their bioavailability. The ability of soil aggregates of two tropical soils to adsorb Pb was evaluated. The objectives of this study were to determine the extent to which Pb sorption on soil aggregate-sizes depends on contact time and initial metal concentration, and investigate the nature of reactions between the metal and soil aggregates surfaces. Lead adsorption was shown to be biphasic; having an initial rapid reaction rate and a subsequent slower rate. Such biphasic kinetics could suggest the presence of two types of sites such as external readily accessible sites and internal, difficult to access sites, with differing reactivities. Equilibrium adsorption occurred faster on 4 mm Ajata-Ibeku soil aggregates (120 min) compared to 180 min equilibrium time obtained by 0.25 mm soil aggregates. On Amakama soil aggregates, 1 and 4 mm aggregates achieved equilibrium adsorption in 180 min, while 0.25 mm aggregates achieved equilibrium adsorption in 210 min. Lead was sorbed more by Ajata-Ibeku soil aggregates and consistently have higher metals' distribution coefficients (K_d) than by Amakama soil aggregates, making the metal less bio-available in Ajata-Ibeku soils. Adsorptive efficiencies of the aggregates for Pb from the kinetics study varied in this order in both soils: 0.25 mm > 1 mm > 4 mm. Pseudo-second-order kinetics model described well the adsorption of Pb (on Ajata-Ibeku soil aggregates), while pseudo-first-order kinetics model fitted well Amakama adsorption data obtained from the experiment. Lead adsorption on Ajata-Ibeku soil aggregates appears to more readily undergo both innersphere and outersphere surface complexations, while the adsorption on Amakama soil aggregates was due to outersphere surface complexation.

Keywords: Tropical soils; Bio-availability, Sorptivity, Peds, heavy metals, Contaminations.

Introduction

Soil contamination by heavy metals is of great concern because of their toxicity to humans (US EPA, 1992). These metals are used in many industrial, urban, and agricultural applications (Kabata-Pendias and Pendias, 1992; Adriano, 2001) and are often found at contaminated sites. One of the most potentially toxic heavy metals is lead (Pb)., which is particularly toxic to higher animals, producing kidney and blood diseases among other health disorders. Therefore, it is important for environmental scientists and engineers to understand the adsorption of Pb on soils to understand their bioavailability. The term 'sorption' has been used to describe the removal of Pb in solution by the soil solid phase (Hooda and Alloway, 1994) including any retention mechanism that controls availability and mobility. When heavy metal ions are placed in aqueous soil system the ions tend to be distributed between the liquid and solid phases. Most of the metal ions become attached to the soil surface while some may penetrate the soil matrix. The metal ions attached to the soil surface is said to be adsorbed on the soil while the ones in the soil matrix is said to be absorbed by the soil. The two processes go together and are referred to as sorption. Since the amount of the metal ions absorbed by the soil is insignificant compared to the amount adsorbed on the soil, when a solution of metal ions is shaken with soil and the amount of metal ions left in the liquid phase is determined, the difference between the initial concentration of the metal ions in the liquid phase and the concentration after equilibration is often considered to be due to adsorption of the metal ions on the soil surface. The adsorption on soil particles would therefore be viewed as nature's process of removing the toxic metals from the environment and getting them sequestered. The consequences of heavy metal addition to a soil with high adsorption capacity will be lower than that with lower adsorption capacity. Understanding the sorption characteristics of Pb on soil aggregate sizes of two benchmark soils of southeastern Nigeria can improve management strategies aimed at reducing the bio-availability of these toxic metal in the study soils.

Materials and Methods

Top soil (0 – 20 cm) of two tropical benchmark soils of southeastern Nigeria, represented by Amakama soil (Fine-loamy, mixed, semi-active, isohyperthermic Rhodic Kandiudult) and Ajata-Ibeku soil (Fine, mixed, semi-active, isohyperthermic Aquic Haplustalf) (Chikezie et al., 2010), were used in this study. Amakama is located on Lat: 5° 26′ 40″ N and Long: 7° 28′ 49″ E, while Ajata-Ibeku is located on Lat: 5° 32′ 51″ N and Long: 7° 33′ 34″ E. Amakama soil is formed on coastal plain sands, while Ajata-Ibeku soil is formed on Bende-Ameke (clay shales) formation. The two soils which will be referred to subsequently, as Amakama and Ajata-Ibeku soils, were chosen for this study because they represent benchmark soils of southeastern Nigeria and have contrasting parent materials and chemical properties. Soils were collected at the 0 – 30 cm layer using soil auger.

Each sample was composited from five samples taken from five locations across the same field. Field-moist soil samples were air-dried and sub-samples of the dry soil screened and fractionated into five different aggregate-size fractions. The remaining soil samples were ground and passed through a 2-mm sieve for background Pb determination. Size fractions were separated by manually moving the sieve in an up and down motion, for about 50 times. This procedure was repeated until all the aggregate-size fractions (4, 2, 1, 0.5, 0.25 mm) have been obtained. All aggregate fractions were weighed and used for studies of Pb sorption. Table 1 presents selected physico-chemical properties of the soils used in this study, which were determined using standard procedures (Appel and Ma, 2002; Appel et al., 2003). The mineralogy of clay particles (< 0.002 mm) as summarized in Table 2 was adapted from the work of Chikezie et al. (2010).

Kinetics of metal sorption
In carrying out the kinetics study, three aggregate sizes (4, 1 and 0.25 mm) were considered at ten contact times (30, 60, 90, 120, 150, 180, 210, 240, 270, and 300 mins). Batch experiments were performed by adding 40 ml of the metal solution (100 mg/L Pb) to 2 g soil aggregate. The soil suspensions for each initial metal solution concentration were shaken on an end-over-end shaker (30 rpm) in a controlled room temperature ($25^oC \pm 2$) for 30, 60, 90, 120, 150, 180, 210, 240, 270, and 300 mins. The suspensions were centrifuged at 6640 X g for 20 mins and the supernatants removed by filtration (Whatman No. 42) before the solutions were analyzed for Pb using UNICAM 919 solar atomic absorption spectrophotometer (AAS). The adsorption amount was calculated as follows:
$$qe = (Ci – Ceq)V/m \ ....(1)$$
Where qe the quantity of the adsorbate (metal) in milligram held by 1 g of the adsorbent (soil), Ci is the initial concentration (mg/l), Ceq is the equilibrium concentration (mg/l), V is the volume of solution (l), and m is the mass of adsorbent (g). Blank solutions were also prepared and analysed.

In order to analyze the rate of adsorption and possible adsorption mechanism of lead onto soil aggregates, the pseudo-first-order and pseudo-second-order models were applied to the kinetics experiments data. The pseudo-first-order model is given as (Ho and Mckay 1998):
$$In (qe – qt) = Inqe – K_1t \ ....(3)$$
Where qt is the amount of lead ions adsorbed at time t (mg/g), qe is the amount of lead ions adsorbed at equilibrium (mg/g), and K_1 is the pseudo-first-order rate constant (min^{-1}) for the first-order adsorption. The pseudo-second-order model can be expressed as (Ho and Mckay 2000):
$$t/qt = 1/K_2qe^2 + t/qe \ ....(4)$$
Where K_2 is the equilibrium rate constant of pseudo-second-order adsorption (g/ mg/min). Values of K_2 and qe were calculated from the slopes and intercepts of the linear plot of t/qt against t. The rate constants K_1, K_2, and qe and the correlation coefficient R^2 of Pb(II) were calculated from the relevant plots. If the r^2 value is nearer to 1, it indicates that the respective equation better fits the experimental data, and the calculated q_e should also approximate the experimental $q_{e.}$.

Sorption Isotherms as a Function of Initial Lead Concentration
Five soil aggregate-sizes were agitated in ten metal concentrations (50, 100, 150, 200, 250, 300, 350, 400, 450, 500 mg/l Pb) - ranges which cut across concentrations regarded as 'normal' and toxic in real agricultural soils (Kabata-Pendias and Pendias, 1992) of the metal. 2 g soil aggregate was weighed into 100 ml soft bottle. 40 ml of Pb solution was added to each sample and agitated on an end-over-end mechanical shaker for 210 mins, filtered through a Whatman No 42 paper, stored and analyzed on the AAS. The adsorption amount was calculated according to equation (1).

Results and Discussion
Characterization of adsorbents
Selected physico-chemical properties of the study soils are presented in Table 1. All the soils were moderately acidic in the surface horizons with differences between Amakama pH and Ajata-Ibeku pH being less than 0.5. In Amakama and Ajata-Ibeku soils, exchangeable acidity accounted for 86% and 72% of the ECEC, respectively, while Ca was the dominant cation in the two soils accounting for the 10 and 21% of the ECEC,

Table 1. Physico-chemical properties of the study soils at 0 – 30 cm depth

Soil property	Amakama soil	Ajata-Ibeku soil
Sand (%)	74.1	23.3
Silt (%)	3.3	20.5
Clay (%)	22.6	56.3
Texture*	SCL	C
pH (H_2O)	4.6	5.0
TN (%)	0.087	0.269
P (mg/kg)	45.9	5.6

OM (%)	0.86	2.14
Ca^{2+} (Cmol/kg)	1.15	5.35
Mg^{2+} (Cmol/kg)	0.41	1.25
K^+ (Cmol/kg)	0.06	0.35
Na^+ (Cmol/kg)	0.08	0.20
TEB (Cmol/kg)	1.70	7.15
EA (Cmol/kg)	10.05	18.0
ECEC (Cmol/kg)	11.75	25.15
BS (%)	14.5	28.4
Background Pb (mg/kg)	17.55	26.30

*Texture: SCL = Sandy clay loam; C = Clay

respectively, of the two soils studied, Amakama soils had lower ECEC, pH, clay and organic atter contents. The initial Pb in the two soils studied were 17.55 and 26.30 mg/kg in Amakama and Ajata-Ibeku soils, respectively. Total background Pb content of the Alfisol (Ajata-Ibeku soil) was greater than that of Ultisol (Amakama soil). It was expected that the amounts of Pb in Ajata-Ibeku soils would be proportionally higher in Ajata-Ibeku soils due to their higher CEC value (Table 1). The fact that greater CEC in Ajata-Ibeku soil results in greater background Pb suggests that exchangeable sites and not high affinity sites contributed to high metal levels in the soils. The mineralogy of clay particles (< 0.002 mm)

as summarized in Table 2 showed that Amakama soil has mixed mineralogy dominated by kaolinite. Small quantities of goethite, gibbsite and haematite are also present. In the Ajata-Ibeku soil, the mineralogy is kaolinite in the upper 100 cm of the profile and mixed clay mineralogy at the lower depths. The mineralogy of Ajata-Ibeku soil is also dominated by kaolinite but has detectable quantities of montmorillonite, and small quantities of goethite and gibbsite. The dominance of kaolinite in these soils agrees with previous studies by Jungerius and Levelt (1964) and Igwe *et al.* (1999) on the soils of eastern Nigeria. The presence of

Table 2. Mineralogy of clay particles (< 0.002 mm) of the study soils (Chikezie *et al.* 2010)

Horizon	Depth(cm)	X-ray Peak size*				TGA %			Interpretation**
				Amakama soil					
Ap1	0 – 13	KK 4	GI 1	HE1	GE 1	KK69	GI 2	GE18	CMIX
Bo1	27 – 63	KK 4	HE1	GE1	GI 1	KK60	GE15	GI2	CMIX
Bo2	63 – 99	KK4	GE1	GI1	HE1	KK62	GE13	GI2	CMIX
Bo4	127 – 159	KK4	HE1	GE1	GI1	KK62	GI2	GI2	CMIX
Bo6	210 - 240	KK4	HE1	GI1	GE1	KK56	GE9	GI2	CMIX
				Ajata-Ibeku soil					
Ap1	0 – 9	KK5	MT2	QZ1	GE1	KK44	GE22	GI2	KAOL
Bt	28 – 60	KK5	MT2	GE1		KK55			KAOL
2Btg	60 – 80	KK5	MT2	GE1		KK55	GE50	GI1	KAOL
2Bssg2	101 – 128	KK5	MT3	GE1		KK51			CMIX
2Bgss	164 - 190	KK5	MT4	GE1	QZ1	KK39	GI1		CMIX

GI = Gibbsite, HE = Hematite, KK = Kaolinite, VR = Vermiculite, GE = Goethite. *peak size – 5 = very large, 4 = large, 3 = medium, 2 = small, 1 = very small, 6 = no peak. **interpretation – CMIX = clay mixture, KAOL = kaolinite.

montmorillonite and plagiociase feldspar in Ibeku soil showed that the soil contains some weatherable minerals (Chikezie et al. 2010).

Sorption Kinetics
Figure 1 represents the variation in lead (II) ions adsorption with contact time. From the figure, it can be determined that the adsorption of the metal was rapid initially; however the adsorption rate becomes slower with passage of time up to 210 min. Adsorption efficiency of Pb was less on large soil

aggregates than on small soil aggregates. This was attributed to the limited surface area/sites available for adsorption in large aggregates and the ease with which those adsorption sites were occupied by Pb. Small particles with large surface-area-to-mass ratios allow more adsorption than an equivalent mass of large particles with small surface-area-to-mass ratios. Rate of Pb adsorption on 1 and 4 mm soil aggregates was faster (180 mins) than the rate of Pb adsorption on 0.25 mm (210 min) Amakama soils aggregates. However, in
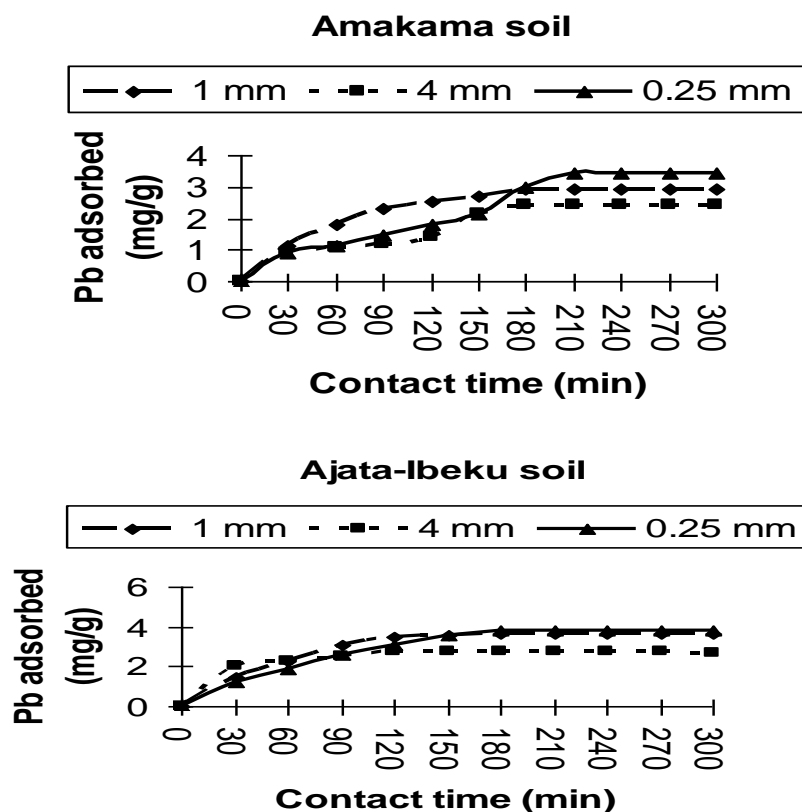
Amakama soil

Ajata-Ibeku soil

Fig. 1. The effect of soil aggregate-sizes on the equilibrium time for Pb(II) and Cd(II) removal from single element solution at pH 5.0, initial metal conc. 100 mg/l Pb and 4.5 mg/l Cd, soil aggregate dosage 2g/40 ml and temperature 27°C.

Ajata-Ibeku soil aggregates, rate of adsorption was faster on 4 mm aggregates (120 min), but uniform (180 min) on 1 and 0.25 mm aggregates(Fig. 1). However, the quantity of Pb (3.40 mg/g) adsorbed by Ajata-Ibeku soil aggregates was higher than value

obtained (2.93 mg/g) on Amakama soil aggregates (Table 3). The higher adsorption capacity obtained for Ajata-Ibeku soil relative to Amakama soil may be as a result of the clay

Table 3 Pseudo-first-order and pseudo-second-order adsorption rate constants and calculated $q_{e\ calculated}$ values of Pb(II) ions onto three soil aggregates (0.25, 1 and 4 mm) of two benchmark soils of southeastern Nigeria at 27° C.

Soil	Aggregate-size	q_e experimental (mg g^{-1})	Pseudo-first-order			Pseudo-second-order		
			K_1 (min^{-1})	q_e calculated (mg g^{-1})	R^2	K_2 (g mg^{-1} min^{-1})	$q_{e\ calculated}$ (mg g^{-1})	R^2
Amakama	0.25 mm	3.43	0.006	3.184	0.9772	0.00046	7.246	0.7426
Amakama	1 mm	2.93	0.018	3.128	0.9985	0.00531	3.556	0.9902
Amakama	4 mm	2.44	0.012	2.811	0.7100	0.00200	3.693	0.8599
Ajata-Ibeku	0.25 mm	3.83	0.019	5.740	0.9318	0.00219	5.200	0.9717
Ajata-Ibeku	1 mm	3.62	0.032	7.130	0.9620	0.00613	4.216	0.9865
Ajata-Ibeku	4 mm	2.74	0.020	1.441	0.9487	0.03370	2.844	0.9982

composition of the two soils. Chikezie *et al.* (2010) reported that the clay fraction of Ajata-Ibeku top and sub-soils were dominated by kaolinite and to a lesser

extent by montmorilonite and very small goethite (Table 2). They also reported that Amakama top and sub-soils contained clay mixture with less kaolinite

and very small gibbsite, hematite and goethite. Sorption reactions are often more rapid on clay minerals such as kaolinite with mixtures of expandable clay minerals like montmorillonite. This is in large part due to the availability of sites for sorption. Kaolinite has readily available planar external sites and sorption on the planar sites is almost complete in minutes. The presence of montmorilonite, a 2:1 expanding clay mineral provides Ajata-Ibeku soil with greater cation exchange capacity than Amakama soil. The presence of montmorilonite in the clay mineralogy of Ajata-Ibeku soil ensures high metal sorption capacity (Veeresh *et al.*, 2003) as it provides the soil with multiple sites for retention of metal and high cation exchange capacity, an established factor regulating the sorption of heavy metals by soils (Kuo and Baker, 1980; Hooda and Alloway, 1998; Gomes *et al.*, 2001). The initial faster rate of removal of the metal ion may be due to the availability of the uncovered surface area of the adsorbents, since adsorption kinetics depends on the surface area of the adsorbent (Veeresh *et al.*, 2003). The variations in percentage removal of Pb by the three aggregate-sizes (4, 1 and 0.25 mm) of both soils show slight increase as the aggregate-sizes decrease. The sequence of increase in removal of metal by the aggregates is: 4 mm < 1 mm < 0.25 mm in both soils. The trend obtained is in the order of the surface area per unit mass of the soil which has direct relationship with the number of adsorption sites on the soils.

Kinetic Modeling
Kinetic equations have been developed to explain the transport of metals onto various adsorbents. The pseudo-first-order rate equation and pseudo-second-order rate equation were applied to study the adsorption kinetics and possible adsorption mechanism of Pb onto 4, 1 and 0.25 mm soil aggregates. Figure 2 presents the pseudo-first-order reaction kinetics and pseudo-second-order reaction kinetics for Pb (II) ions on Amakama and Ajata-Ibeku soils aggregates. Linear regression (r^2) is frequently used to determine the best fitting kinetic model for metal sorption. In this study, a comparison of the kinetics of Pb (II) sorption on the individual soil aggregates was evaluated the differences in the mineralogical using the coefficient of determination (r^2) as the fitting parameter. Table 3 presents the values of the parameters calculated from the linearised forms of the pseudo-first-order and pseudo-second-order kinetics model equations. Application of the pseudo-first-order model showed that the calculated values of the amount of Pb adsorbed at equilibrium agreed with the values obtained experimentally in Amakama soils but did not agree in Ajata-Ibeku soils, despite the high values of r^2 obtained on Ajata-Ibeku aggregates. Thus, suggesting that adsorption process of Pb followed the pseudo-first-order model in Amakama soil aggregates (Table 3). In comparison to first-order kinetic model, pseudo-second-order model described well the adsorption kinetics of Pb on Ajata-Ibeku soil aggregates. These suggest that Pb adsorption data are well represented by pseudo-second-order kinetics in Ajata-Ibeku soil aggregates, and this supports the assumption that the rate-limiting step of Pb adsorption onto soil aggregate sizes of both soils may be due to outer sphere and inner sphere complex sites. The pseudo-second-order rate constant (K_2) increases with decrease in surface area in Ajata-Ibeku soil aggregates. The reason for this behaviour can be attributed to the high competition for the limited surface sites of large soil aggregates. Aggregates that are small in size will provide high active sites for adsorption, and the competition for the active sites will   be
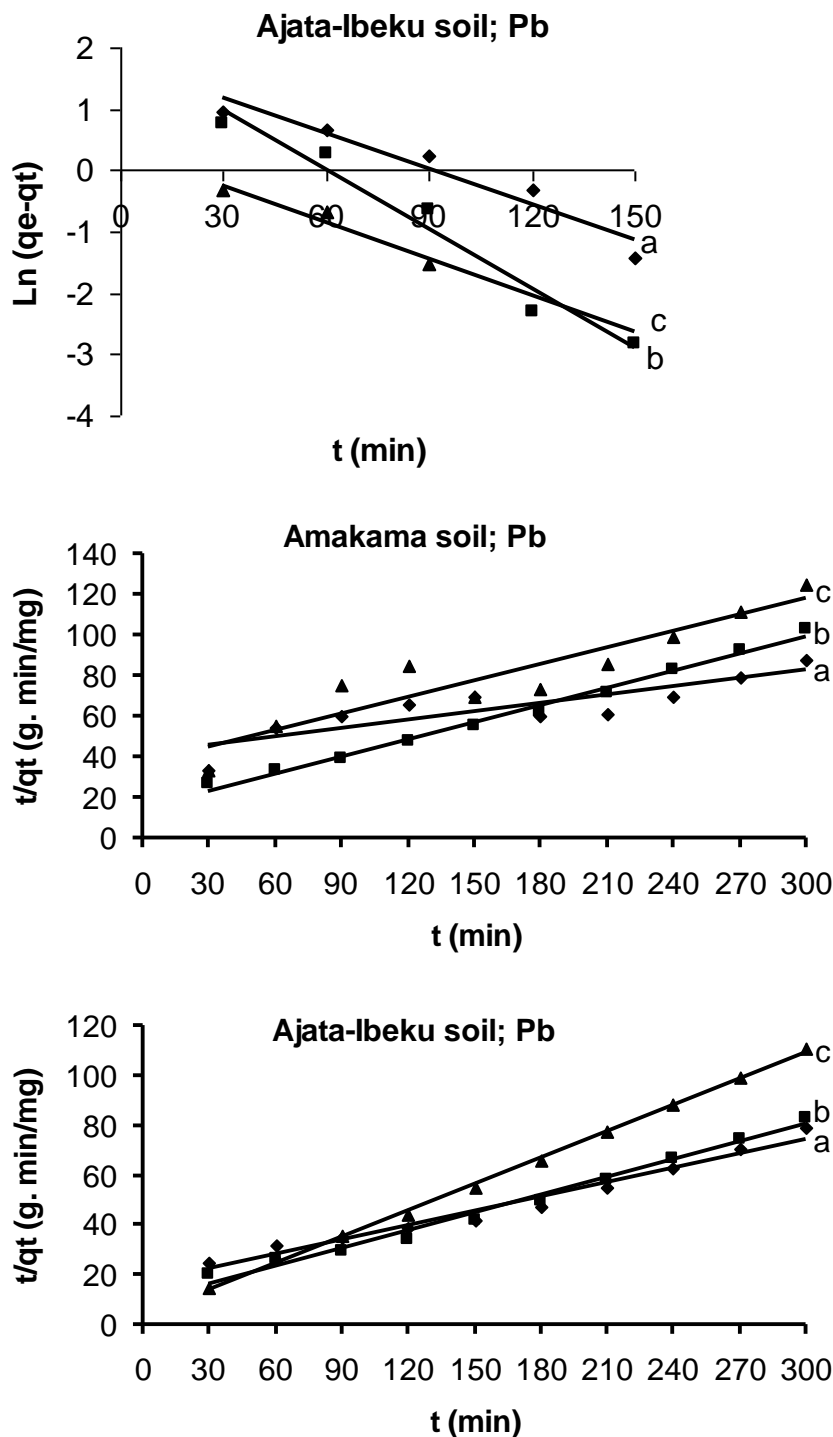

Amakama soil; Pb
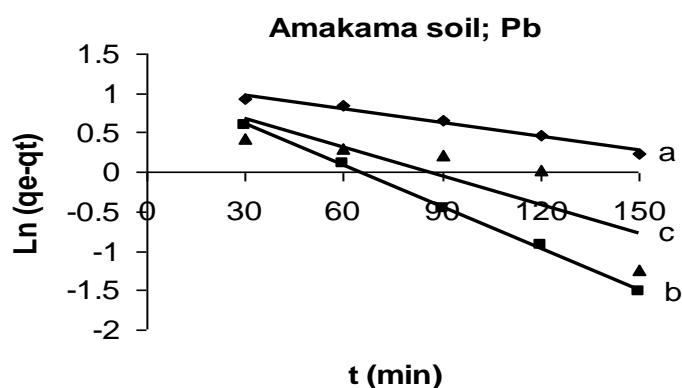
Fig. 2.   Pseudo-first-order reaction kinetics and pseudo-second-order reaction kinetics for Pb(II)) ions on individual soil aggregates (a = 0.25 mm, b = 1 mm, and c = 4 mm) at pH 5.0, soil aggregate dosage 2g/40 ml, initial metal conc. 100 mg/l Pb, temperature 27°C.

low and consequently sorption rate will be low in soils with large soil aggregates. No particular trend was not followed in Amakama soil aggregates. Applicability of pseudo- first-order kinetics model in Amakama soil aggregates suggests that adsorption may have occurred on planar sites only, while the applicability of pseudo-second-order kinetics models

to Ajata-Ibeku soil aggregates suggests that both planar and interlayer sites may be operational.

Sorption of Pb by soil aggregates as a function of initial metal concentrations
The effect of soil aggregates on the removal of Pb(II) from soil solutions was studied with five aggregate sizes (4, 2, 1, 0.5 and 0.25 mm). The amount of metal

adsorbed (mg/kg) when plotted against the equilibrium concentration (mg/l) produced a non-linear regression model as shown in Table 4. These best-fit regression models provide for drawing tangents at the points where adsorption started to drop, to locate the critical equilibrium concentrations of the metals in solution, at which, presence of metal in solution starts to reduce adsorption. The equilibrium concentration of Pb (x) (mg/L) at the maximum kinetics models described well the adsorption of Pb (on Ajata-Ibeku soil aggregates) on both soils. Pb adsorption was obtained by equating the first derivative of Pb adsorbed (y) (mg/kg) with respect to equilibrium concentration (x) to zero, and the value of x substituted in the relevant equation. The data contained in these models were used to calculate a distribution coefficient or the partitioning between the solution phase and the solid phase, which is a ratio of solid phase concentration to solution phase concentration (Table 4). Examination of the data presented in Table 4 indicates that these coefficients were consistently higher in Ajata-Ibeku soils (ranging from 135.0 – 313.2 l/kg) than in Amakama soils, where K_d values ranged from 23.5 – 57.1 l/kg. Thus, suggesting that Pb(II) was removed from solution by Ajata-Ibeku soils aggregates more than by Amakama soil aggregates. The distribution coefficients of the metals on the aggregates did not follow any particular trend in both soils, however, maximum distribution coefficient of Pb for both soils was observed at the 1 mm soil aggregate.

Table 4.   Effect of soil aggregates on maximum adsorbed Pb(II) ions and their distribution coefficients on the aggregates.

Soil	Soil aggregate	Regression model	R^2	Solution phase conc. (mg/l)	Solid phase conc. (mg/kg)	Distribution coefficients (K_d)
			Pb			
Amakama	4 ,m,m	$Y = -0.0953x^2 + 33.891x + 1266.9$	0.958 **	177.81	4280.02	24.1
Amakama	2 mm	$Y = -0.1043x^2 + 41.009x + 1205$	0.837 **	196.59	5236.01	26.6
Amakama	1 mm	$Y = -0.206x^2 + 69.972x + 3750.2$	0.636 *	169.83	9692.05	57.1
Amakama	0.5 mm	$Y = -0.076x^2 + 35.476x + 1335.3$	0.938 **	233.39	5475.26	23.5
Amakama	0.25 mm	$Y = -0.153x^2 + 63.64x + 603.38$	0.951 **	207.97	7221.11	34.7
Ajata-Ibeku	4 ,m,m	$Y = -3.168x^2 + 310.52x + 2082.6$	0.970 **	49.01	9691.71	197.8
Ajata-Ibeku	2 mm	$Y = -3.2563x^2 + 366.6x + 2331.4$	0.917 **	56.30	12649.52	224.7
Ajata-Ibeku	1 mm	$Y = -7.5823x^2 + 505.48x + 2014.6$	0.955 **	33.33	10439.16	313.2
Ajata-Ibeku	0.5 mm	$Y = -2.8945x^2 + 291.44x + 2610.8$	0.932 **	50.34	9946.89	197.6
Ajata-Ibeku	0.25 mm	$Y = -1.7276x^2 + 219.49x + 1605$	0.924 **	63.52	8576.50	135.0

K_d = concentration in the solid phase / concentration in the solution phase.

Conclusion.
A biphasic kinetics was observed, namely, a rapid reaction rate, followed by a much slower reaction time. The rapid reaction rate occurred at the external planar sites of the kaolinitic clay mineral, while the slower reaction occurred on the inter layer sites with differing reactivities. Adsorptive efficiencies of Pb from the kinetics study were more on soil aggregates with smaller surface area, in both soils. Equilibrium adsorption capacities of Pb by soil aggregates in Ajata-Ibeku soils were consistently higher than the values obtained in Amakama soil aggregates. Similarly, the distribution coefficients (K_d) of Pb on Ajata-Ibeku soil aggregates were higher than those for Amakama soil aggregates. The kinetics data for the adsorption process obeyed pseudo-first-order kinetics model on Amakama soil aggregates, but did not obey the pseudo-first-order kinetics models in Ajata-Ibeku aggregates. Pseudo-second-order kinetics model fitted Pb adsorption data of Ajata-Ibeku soil aggregates kinetics. This difference in kinetics was attributed to the clay mineralogy of the study soils.

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