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Effect of different pH levels on adsorption and desorption of Pb in contrasting parent materials of south eastern agro climatic zone of Nigeria.

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ABSTRACT

Lead adsorption and desorption at different pH levels in acid soils of diverse parent materials were evaluated. The soil samples were collected from soils underlain by olivine basalt (Ikom), coastal plain sands (Ihiagwa) and false bedded sandstone (Ishiagu). The collected samples were air -dried, crushed, sieved with a 2 mm sieve and analyzed in the laboratory. The adsorption of lead (Pb) increased with increasing solution pH. At pH 3, 4 and 6, the adsorption of Pb was higher in false bedded sandstone than the other soils. At pH 5, adsorption of Pb was higher in olivine basalt than the other soils. Except at pH 3, desorption of Pb was higher in coastal plain sands than the other soils. At the same time it was lower in false bedded sand stone than the other soils at all pH. At pH 5, adsorption of Pb had a significant positive correlation with organic matter ($r = 0.774481$, $p \leq 0.05$), while at pH 6, it had a significant positive correlation with soil pH ($r = 0.738401$, $p \leq 0.05$). Organic matter and soil pH are the most critical soil properties affecting adsorption-desorption of Pb on these soils.

1.0. Introduction

Heavy metal pollution is a fast- growing problem for the environment in areas with intensive industries (Afridi, 2010; Udoh and Chukwu, 2017). Heavy metals exist naturally in soil systems and their concentrations are variable depending upon parent materials. Most occur at low concentrations and are incorporated into soil minerals by isomorphous substitution in the crystal lattice (Alloway, 1998).

Lead is a naturally occurring metal found in the environment (Brady and Weil, 2002). Lead is a wide spread contaminant that represents a significant challenge to the environment. In the environment, it arises from both natural and anthropogenic sources and it is significantly harmful to humans and living things (Ojebah and Uwague, 2018; Onwudike *et al*., 2017). Lead is considered as the most critical environmental pollutant in agricultural soils, because of the potentially harmful effects they may have on food quality and health of soil (Onweremadu and Duruigbo, 2007).

Sorption is regarded as one of the most important chemical processes that affect heavy metal availability (Antoniadis *et al*., 2007; Azeez *et al.,* 2018). Sorption studies involving either batch or column techniques are usually employed to evaluate the extent of metal's retention by the soil (Abdu and Mohammed, 2016). Adsorption is defined as the accumulation of ions/materials at the inter-phase between a solid phase and an aqueous phase (Alloway, 1990). Metal availability in the soil may vary considerably depending on the nature of the adsorption-desorption process in the soil. Adsorption-desorption can be affected by many factors such as cation exchange capacity (CEC), organic matter, clay content, competition from other metal ions. However, it is greatly affected by pH Stevenson

(1991) defined pH as the negative logarithm of the hydrogen ion concentration. This refers strictly to proper solution in which the ions are entirely dissociated. The primary value of a pH measurement is not that it shows a soil to be acidic or alkaline, but it also gives information about nutrient mobility, availability etc. The pH of the soil is an essential parameter which directly influences the adsorption process. In general, maximum retention of cationic metals occurs at pH< 7 and maximum anionic retentions occur at pH> 7. However, because of the complex nature of the soil systems such a generalization may not hold. In a condition where oxidation reactions are involved, the solubility of heavy metals increases with decreasing pH. Soil pH is crucial for lead adsorption, since lead availability is relatively low when pH is about 6.5 -7. At higher pH, ions hydrolysis (MOH) is favoured, and the energy barrier that must be overcome when these ions approach the surface of the soil particles is decreased (Yu *et al*., 1997). As pH increases, the adsorbed lead becomes much less exchangeable. An increase in pH makes the surface electrostatically more suitable for adsorption (Mckenzie, 1980).

Soils of the humid tropics and indeed southeastern Nigeria are acidic with pH value between 4-8, and contain appreciable mineral oxides (Hakim, 2002; Opara-Nadi, 1988).These soils contain a higher proportion of positively charged elements such as Cu^{2+}, Fe^{2+}, Pb^{2+} and other minor elements. Thus, at very low pH values, the soil may contain the toxic quantity and it is considered that this induced toxicity is responsible for poor growth rather than acidity per se. Understanding the mechanisms of metal adsorption -desorption in soils is essential as these reactions control the strength of metal-soil surface interactions. Although several authors have reported in the influence of pH on metal adsorption (Silber *et al*., 2012; Najafi and Jalali 2016).However data on influence of pH levels on adsorption and desorption of metal from these tropical soils are scarce. Hence this study to investigate the adsorption and desorption of lead at different pH levels .Most studies have focused on the adsorption of heavy metals in soils, desorption data are also needed to address contaminant transport model assumptions of reversibility and to determine the feasibility of using chemical enhancement techniques for in-situ removal of metals at hazardous waste sites.

The objective of the study is to investigate the effect of pH levels on the adsorption-desorption of lead from diverse parent materials of acid soils of southeastern Nigeria.

2.0. Materials and methods

2.1. Study area

The study area comprises of three different locations representing three- parent materials in the southeastern agro-climatic zone of Nigeria. The study locations were Ihiagwa (5^0 6' N and 7^0 45' E) with coastal plain sands as the parent material, Ikom (5^0 45' N and 8^0 30' E) with olivine basalt as the parent material and Ishiagu (5^0 57' N and 7^0 34' E) with false bedded sandstone as the parent material. These locations are characterized by rainfall distribution which ranges from 2000 – 2500 mm, temperature ranges from 26 □ - 29□ . The vegetation is the rainforest which is characterized by thick dense forests.

2.2. Sample collection and preparation

Bulk soil samples were collected from the three different locations at 0 – 15 cm depth. The samples were air-dried and sieved using 2 mm mesh sieve. The sieved samples were stored in a labeled polyethylene bag in preparation for laboratory analysis.

2.3. Laboratory analysis

Particle size distribution was determined using the hydrometer method (Gee and Or, 2002). Soil pH in water was analyzed as described by Hendershot *et al*. (1993) at a soil-water ratio of 1:2.5 using glass electrode pH meter. Organic carbon was determined by wet digestion method (Nelson and Sommer, 1982). Organic matter was computed by multiplying the value of organic carbon by a factor of 1.724 (Van Bermmelens factor). Cation exchange capacity was determined by the method described by Rhoades and Polemio (1977).

2.3.1 Determination of adsorption of Pb

A 100 mg/L solution of Pb was prepared from a stock solution of 1000 mg/L using 0.01 mol/L KCl as background electrolyte. Following the procedure of Nkwopara (2012).Metal ion solution of 25 ml was added to 1 g of soil weighed into 50 ml polyethylene bottles. The solutions were adjusted to pH 3 -6 using either 0.1 M NaOH or HNO_3. The suspensions were agitated for 2 hrs. The pH of the solutions was checked and readjusted to the initial pH . The suspensions were equilibrated for 22 hrs. and readjusted to the initial pH after equilibration. The solutions were centrifuged at 5000 rpm for 10 mins. The supernatants obtained after centrifugation were analyzed for Pb using Atomic Absorption Spectrophotometer (AAS) (Model AA 500, PG Instrument, U.K). The amount of Pb adsorbed was calculated by the difference between the total applied Pb^{2+} and the amount of Pb^{2+} remaining in solution.

2.3.2 Determination of desorption of Pb

Following the procedure of Nkwopara (2012). Batch desorption of Pb was accomplished by replacing the supernatant liquid with metal-free KCl solution following adsorption. The operation was conducted at 25□ ± 0.1 by removing the adsorption equilibrium supernatant solution after centrifugation replacing the solution with an equal volume of 0. 01 mol/L KCl background solution of various pH of 3, 4, 5, 6 after adsorption. The suspension was agitated for 2 hrs, equilibrated for 22 hrs and centrifuged at 5000 rpm for 10 mins. The supernatants collected were analyzed for lead using Atomic Absorption Spectrophotometer (AAS) (Model AA 500, PG Instrument, U.K).

The adsorption capacity was calculated using the equation below;

$$\text{Adsorption capacity} = \frac{Ci - Ce}{W} V \quad \text{----------equation (1)}$$

$$\% \text{ Adsorption} = \frac{Ci - Ce}{Ci} \times 100 \quad \text{----------equation (2)}$$

$$\text{Distribution Coefficient} = \frac{Ci - Ce}{Ci} \frac{V}{W} \quad \text{----------equation (3)}$$

Where Ci = initial concentration of lead in solution (mg/L)

Ce = equilibrium concentration of lead in solution (mg/L)

V = volume of lead solution (ml)

W = weight of soil (g)

2.5.1. Statistical analysis

Simple linear correlation analysis was used to determine the relationship between soil properties and lead adsorption. Data collected were subjected to analysis of variance (ANOVA) using Gensat Discovery 3rd edition. Significant mean was separated using the least significant difference LSD at the 0.05 probability level. The experiment was replicated three times and the means are reported.

3.0. Results and Discusion

3.1. Physical-chemical properties

The result of physicochemical properties is shown in Table 1. In Ikom, the mean values of sand, silt, clay were 501.88, 225.6 and 272.55 gkg^{-1} respectively. In Ihiagwa , the mean values of sand, silt and clay were 885.2, 42.27 and 72.53 gkg^{-1} respectively. In Ishiagu, the mean values of sand, silt and clay were 741.87, 192.27 and 65.86 gkg^{-1} respectively. There was a significant difference between the sand and clay contents of Ikom and other soils. There was a significant difference between the silt content of all the soils. The textural class of Ikom soils was dominantly sandy clay loam. Ihiagwa soils were dominantly sandy while Ishiagu soils were loamy sand. The variation in texture reflected the difference in parent materials. Ezenwa (1987) and Okoli *et al.* (2016) observed that parent material is a significant factor determining the texture of soils. Soil texture, especially clay content affects the behavior, fertility, water holding capacity as well as the plant root movement (Esu, 1987). The low silt and clay contents were due to high rainfall of the region which favoured high rate of leaching and the influence of the process of translocation in which silt and clay contents were washed down the profile (Onweremadu *et al.,* 2007; Ihem *et al.,* 2017). Ihiagwa soils with high sand (> 76 % or760 gkg^{-1}) will have low moisture retention, high water infiltration and increased mobility (Enwezor, 1999). The mean value of organic carbon for Ikom, Ihiagwa and Ishiagu was 24.3, 17.7, and 19.1 gkg^{-1} respectively. There was a significant difference between Ikom soils and the other soils. The low organic carbon may be attributed to the decomposition rate of organic matter due to agricultural activities (Adjia *et al.,* 2008). It could also be due to varying parent materials especially, coastal plain sands (Ihiagwa soils) having a low inherent organic carbon (Obi et al., 2012). Organic matter reflects the level of fertility in the tropics (Opara-Nadi, 1988). Therefore low organic matter means low fertility. Constant high temperatures are the cause of high biological activity and rapid mineralization of plant debris, preventing an accumulation of organic substances. The mean value of pH for Ikom, Ihiagwa and Ishiagu were 5.35, 5.96 and 5.43 respectively. There was no significant difference between Ikom soils and Ihiagwa soils but there was a significant difference between the two soils and Ishiagu soils.The strong leaching of bases leads to acidic conditions of the soils (Anja, 2005) which resulted in the exchange complex of the soils to be dominated by acidic cations. Similar results have been reported by Eshett et al. (1990), Okoli et al. (2016) and Uzoigwe, (2014). The mean value of CEC for Ikom, Ihiagwa and Ishiagu was 3.89, 2.32, and 2,86 respectively. There was a significant difference in CEC in all soils. The CEC are generally, low due to the predominance of so-called low activity clays such as kaolinite and a lack of 2:1 phyllosilicates (Marques *et al*., 2004).

3.1.1 Adsorption studies

The result of lead adsorption on soils is shown in Fig. 1. Percent adsorption of lead increases as the pH levels increase. Percent adsorption of lead was highest in Ishiagu soils at pH 3, 4 and 6 (74.8, 89.96 and 91.00) respectively.

Table 1: The physicochemical properties of the study locations

Location	Depth (cm)	Sand (g/kg)	Silt (g/kg)	Clay (g/kg)	Textural class	pH	OM (g/kg)	CEC(cmol/kg)
Ikom	0- 20	525.2	225.6	249.2	SCL	5.67	37.8	3.77
	0- 20	445.2	225.6	329.2	SCL	5.15	34.7	3.90
	0-20	535.2	225.6	239.2	SCL	5.24	43.3	4.02
Mean		501.88	225.6	272.53		5.35	41.9	3.89
Ihiagwa	0- 20	885.2	35.6	79.2	S	5.94	30.9	2.25
	0 -20	875.2	45.6	79.2	S	5.95	29.2	2.47
	0 -20	895.2	45.6	59.2	S	6.01	31.9	2.25
Mean		885.2	42.27	72.53		5.96	30.7	2,32
Ishiagu	0 -20	745.2	195.6	59.2	LS	5.45	29.9	2.67
	0 -20	695.2	225.6	79.2	LS	5.48	32.6	3.32
	0-20	785.2	155.6	59.2	LS	5.37	36.4	2.60
Mean		741.87	192.27	65.86		5.43	33.06	2.86
LSD (0.05		52.08	46.89	54.49		0.820	5.443	0.4932

Percent adsorption of lead was highest in Ikom at pH 5. At lower pH (3 and 4), there was significant difference in percent adsorption of lead in all the soils. At highest pH (6), there was a significant difference in percent adsorption of lead between Ishiagu soils and the other soils. Increase in adsorption of

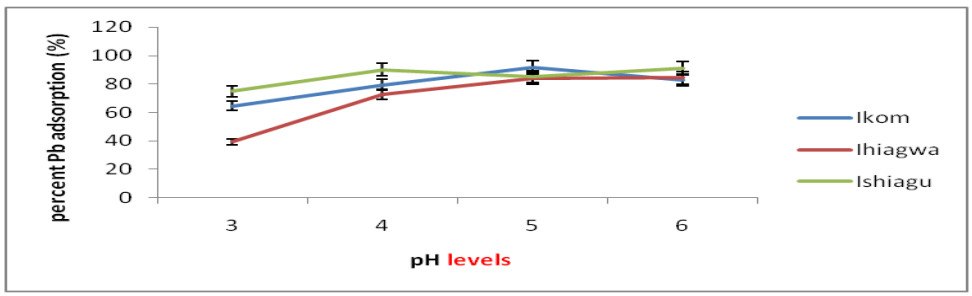


Fig 1: Percent adsorption of lead at different pH levels

lead with increased solution pH can be attributed to several reasons. They include, that soil surface which contains a large number of active sites may become positively charged at low pH, which leads to competition between H^+ and the metal ions for available adsorption sites because of the increased amount of H^+ in solution. As pH increases the competition decreases as these surface active sites become more negatively charged, thereby enhancing the adsorption of the positive charge metal ions through electrostatic force of attraction. Also increasing pH has been shown to decrease the solubility of lead. Increasing pH encourages metal ion precipitation from solution in the form of hydroxides (Uzoigwe, 2014). Adsorption of cationic metals increased with pH and reached 99 % around pH 5.8 (Abdu and Mohammed, 2016).The increase in adsorption of Pb as the pH of solution increased is consistent with previous findings that increased pH generally causes an increase in the metal adsorption (Bradl, 2004; Pagnanelli *et al.,* 2003).The higher Pb adsorption in Ishiagu and Ikom relative to ihiagwa can be attributed to the nature of parent materials. The influence of the clay content on adsorption could be related to the soil parent material and mineralogy (Nkwopara *et al.,* 2016; Nkwopara, 2020). Soils derived from basic-rocks or basalt has high adsorption capacity while soils containing kaolinite have low adsorption capacity (Udo, 1985). The low values observed in Ihiagwa soils could be attributed to the sandy nature of the soil. The higher organic carbon and cation exchange capacity (CEC) in Ikom and Ishiagu implies that the soils had higher potentials for receiving and retaining Pb^{2+} than Ihiagwa soils. These observations agree with reports by Coles and Yong (2002) and Yang *et al.* (2006) that CEC was positively correlated with lead and cadmium retention capacity. In general, the adsorption capacity of these soils (tropical soils) were lower compared to temperate soils and could be as a result of weather conditions of the soils in this region which resulted in high concentration of variou iron and aluminium oxides (Osodeke and Ubah, 2006).

Distribution coefficient (Kd) was higher in Ishiagu soils at pH 3, 4 and 6 (18.72, 22.49, and 22.75 mL/g) respectively (Fig. 2). Distribution coefficient (Kd) was higher in Ikom soils at pH 5 (22.90 mL/g).At lowest pH (3) there was no significant difference in Kd between Ikom soils and Ishiagu soils while at highest pH (6) there was no significant difference in Kd between Ihiagwa soils and Ishiagu soils (Fig.2). The higher distribution coefficient of Ishiagu soils at pH 3, 4 and 6 except pH 5 indicates stronger attraction of Pb^{2+} to these soils at the various pH than the other soils, and it also shows lower susceptibility to leaching losses in Ishiagu soils than the other soils.

3.1.2 Desorption studies

The result of lead desorption from soils is shown in Fig. 3. As expected, desorption percent of lead decreases as pH levels increases. Desorption percent of lead was highest in

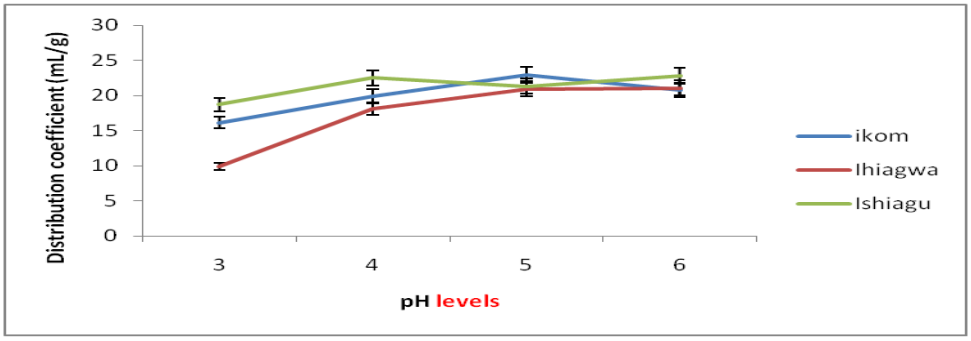


Fig 2: Distribution coefficient of lead at different pH levels

Ikom soils at pH 3 (23.2 %). At pH 4, 5 and 6 it was highest in Ihiagwa soils (3.50, 2.80 and 2.90 %) respectively. At all pH desorption percent was lowest in Ishiagu soils. Generally, desorption percent of lead was small in all the soils at all the studied pH. Desorption percent of lead was highest at lowest pH (pH 3) than the others. The higher desorption observed in Ihiagwa soils could be as the result of the sandy nature of the soils. Sandy soils are highly porous which tend not to retain or hold the heavy metal (lead) tenaciously which may lead to high desorption nature than the other locations. Generally, the desorption percent of Ishiagu is low as a result of high retention of Pb on the soils. Also the sandy soils have a high infiltration rate and large pore space which makes it easier for heavy metal to be flushed out of the soil.

Relationship between adsorption of lead and soil properties at various pH levels

The result of the relationship between soil properties and adsorption of lead at various pH are shown in Table 2, at

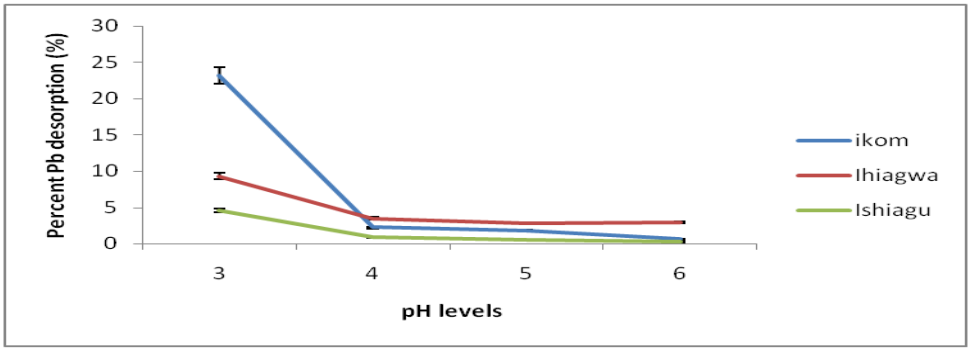


Fig 3: Percent desorption of lead at different pH levels

lower pH, (pH3) all the soil properties except sand content correlated positively with adsorption of lead. However it was only silt content that had a significant positive correlation (r =0.61, p ≤0.01 At higher pH (pH 5 and 6), adsorption of lead had a significant positive correlation with organic carbon and p (r = 0.774 and 0.738, p ≤0.01) respectively. The significant positive relationship between adsorption of Pb and organic matter and soil pH at pH 5 and 6 indicated that organic matter and soil pH are the main properties affecting the adsorption of Pb on all soils.

4.0. Conclusion

Table 2: Correlation between adsorption and selected soil properties at varying pH

Soil property	Different P	Different pH levels		
	pH 3	pH4	pH5	pH6
CEC	0.364^{ns}	0.161^{ns}	0.487^{ns}	-0.295
Clay	0.200^{ns}	-0.025^{ns}	0.582^{ns}	-0.408
OM	0.487^{ns}	0.070^{ns}	0.774**	-0.273
Sand	-0.432^{ns}	-0.148^{ns}	-0.531^{ns}	0.251
Silt	0.613*	0.324^{ns}	0.346^{ns}	0.002
pH	0.536^{ns}	0.541^{ns}	0.121^{ns}	0.738**

*and ** = significant at 0.005 and 0.001 probability levels
CEC=Cation exchange capacity, OM = Organic matter.

From the studies, an increase in solution pH increases the adsorption of Pb and decreases the desorption of Pb on these soils. More Pb was adsorbed on Ishiagu soils and less on Ihiagwa soils .Less Pb was desorbed on Ishiagu soils and more on Ihiagwa soils Organic matter and pH could be attributed to be the properties which affect the adsorption of Pb on these soils. Ishiagu soils had the potential of retaining Pb and releasing less Pb to the environment. Therefore in case of Pb pollution in the area, Ishiagu soils tended being less polluted.

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