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BATCH STUDIES FOR THE INVESTIGATION OF THE ADSORPTION OF LEAD ONTO BENTONITE AND SOIL OF OWERRI, NIGERIA

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ABSTRACT: *Variability in lead adsorption by soil and bentonite was analyzed and results indicated that adsorption of lead increased as volume of* Pb^{2+} *solution increased. At 25ml adsorption capacity of bentonite and soil were 2494.19mg/kg and 2372.89mg/kg respectively. Maximum adsorption was observed at 100ml of* Pb^{2+} *in both soil and bentonite which were 9031.75mg/kg and 9935.75mg/kg respectively. At all volumes of* Pb^{2+} *solution, a significant positive correlation at (*$p \leq 0.05$ *and* $p \leq 0.01$*) probability levels were observed between clay, pH, CEC and organic matter content and adsorption capacity in soil and bentonite. While sand and silt content correlated negatively. Clay, sand, pH, CEC and organic matter content, were indicated to be the vital properties controlling adsorption in soil and bentonite. Bentonite and soil adsorption data were analyzed with both freudlich and langmiur adsorption isotherm, freudlich isotherm showed a better fitting of the adsorption data in both soil and bentonite with correlation factor (*$R^2 > 0.9$*).*

KEYWORDS: Batch Studies, Adsorption of Lead, Bentonite, Sand, Silt Content, Nigeria**.**

INTRODUCTION

Heavy metals are natural components of the earth's crust which cannot be degraded or destroyed. Heavy metals are one of a number of elements that exhibit metallic chemical properties, which has a relatively high density and is toxic or poisonous at low concentration. The term heavy metals chiefly arose with discussion of pollutants discharged to the environment in the form of air, water or soil contaminants. While many heavy metals have considerable toxicity, others are considered not to have significant toxic properties and in fact, several of these elements including zinc, iron, copper, chromium and cobalt are necessary in metabolic function for a large class of organisms. Although some heavy metals are essential micronutrients for animal, plant any microorganism depending on the route and dose, all heavy metals demonstrate toxic effects on living organism via metabolic interference and mutagenesis (Hogan, 2010).

Lead can be introduced into the soil environment through both pedogenic (orgeogenic) and anthropogenic process (U.S Department of Health and Human Services, 2004; Schulin et al., 2007).When lead is deposited in soils from anthropogenic sources, it does not biodegrade or decay and is not rapidly absorbed by plants, so it remains in the soil at elevated levels. Lead is estimated to have a half-time of residence in soil of 1,000 years (Benninger et al., 1975).

Clays are hydrous aluminum phyllosilicates broadly defined as those minerals that make up the colloid fraction (<2 µ) of soils.Clays play an important role in the environment by acting as a natural scavenger of pollutants by taking up cations and anions either through ion exchange or adsorption or both. Thus, clays invariably contain exchangeable cations and anions held to the surface (Van Olphen, 1977).Bentonite is a montmorillonite type clay formed by alteration or weathering of volcanic ash. It varies in composition and is usually highly colloidal and plastic (Richard. 2006). Large specific surface area, chemical and mechanical stability, layered structure, high cation exchange capacity (CEC), etc., have made the clays excellent adsorbent materials (Van Olphen. 1977).

Metal ions are retained in soil mainly by sorption, precipitation and complexation reactions (Bolan et al., 1999; Adriano et al., 2004). While metal ions are released from the soil sediment's surface mainly by desorption.Adsorption is a sorption mechanism, responsible for accumulation of heavy metals.Although metal ions interact with soil minerals and organic matter, the fate of metals in the soil environment is dependent on factors such as pH, cation exchange capacity, insoluble organic matter and clay content, competition from other ions.

The objectives of the study were to determine the adsorption of Pb^{2+} onto soil and bentonite and the effect of solution volume on adsorption.

MATERIALS AND METHODS

Bentonite

Bentonite was collected from oxbow concepts and resources LTD, Portharcourt Rivers State. The bentonite sample collected was crushed using pistol and mortar, air dried and sieved using 2mm mesh sieve. The bentonite sample was stored in a labelled polythene bag in preparation for laboratory analysis.

Soil

Bulk soil sample was collected via surface sampling at a depth of 0-15cm from Southeastern Nigeria; Nekede, Owerri-west L.G.A in Imo state (parent material- coastal plain sand), which lies between $5^0$26 and $7^0$01E'. soil sample was air dried and sieved using 2mm mesh sieve. The soil was stored in a labeled polythene bag in preparation for laboratory analysis.

Laboratory Analysis

The particle size distribution of samples were determined by using the hydrometer method. (Gee and Or, 2002).Organic carbon was determined using walkey and Black wet digestion method (Nelson and sommers, 1982). The cation exchange capacity was determined by the method of Rhoades and Polemio (1977). Soil pH and bentonite pH was determined both in water and in potassium chloride (KCl) using the pH meter at a soil/water ratio of 1:2.5 (Hendershot et al, 1993)). Organic carbon was determined by wet digestion method (Walkey and Black, 1962) as modified by (Nelson and sommers, 1982). .Organic matter was determined by multiplying the value of organic carbon by 1.724 (Van Bermellen factor.

Adsorption Study

Weight of 1g of soil and bentonite sample were placed in 100ml polyethylene bottles. Different volume (25, 50, 75 and 100ml) of 0.01M KCl solution containing 100mg/L Pb^{2+} was added. The solutions were adjusted to pH 5.5±0.1 with either 0.1M NaOH or HCl. These were agitated at230rpm for 2h at 25^{o}c, then equilibrated for 22hrs. the suspension were centrifuged at 500rpm for 10min and filtered. The supernatant solution taken for Pb^{2+} ion analysis using AAS (Atomic Adsorption Spectrometer), and the amount adsorbed by the soils (adsorption capacity) was calculated from the difference between the total amount added and the amount remained in the equilibrium solution, i.e. $\frac{(Ci-Ce)v}{w}$

Determination of lead adsorption was calculated from the equations;

- Adsorption capacity = $\frac{(Ci-Ce)v}{w}$
- % adsorption = $\frac{Ci-Ce}{Ci} \times 100$
- Distribution coefficient = $\frac{Ci-Ce}{Ce}\frac{V}{W}$

C_i = initial conc. of lead in soil/bentonite solution (mg/l)

C_e = equilibrium conc. Of lead in soil/bentonite solution (mg/l)

V = volume of solution (ml)

W = weight of soil/bentonite (g)

Two commonly used mathematical expression for describing the adsorption equilibrium, namely langmuie and freudlich isotherm models were tested with experimental data (Table 6)

Statistical analysis

Data collected was subjected to statistical method of analysis. Simple linear correlation analysis was used to determine the relationship between soil properties and lead adsorption capacity, bentonite properties and lead adsorption capacity. Data was subjected to analysis of variance (ANOVA) and significant mean was separated using least significant difference (LSD) at 0.05 probability level.all experiment was replaced three times and only mean values were used.

RESULTS AND DISCUSSION

Soil physico-chemical properties

From table 1, sand particles were 858.00g/kg and 513.20g/kg for soil and bentonite respectively. The higher sand particle in the soil could be attributed to the nature of parent material which is coastal plain sand. Difference in the sand particle of soil and bentonite were significant (57.34) at ($p \leq 0.05$).

Silt particle were 44.80g/kg and 64.20g/kg in soil and bentonite respectively. Silt content in soil and bentonite showed a significant difference (12.66) at ($p \leq 0.05$).

Clay particle content were 97.20g/kg and 420g/kg in soil and bentonite respectively. Higher clay particle in bentonite could be attributed to bentonite being a montmorillonite type clay. Clay particle in soil and bentonite showed a significant difference (67.07) at ($p \leq 0.05$).From the particle size distribution, the texture of the soil and bentonite were determine from a textural triangle. The texture of the soil was sandy while bentonite was sandy clay loam.

pH in water were 5.66 and 6.04 in soil and bentonite repectively, while pH in KCl were 4.99 and 5.53 in soil and bentonite respectively. Both pH in H_2O and KCl showed significant difference (0.37) (0.49) at ($p \leq 0.05$) respectively.

Organic matter was 20.87g/kg and 14.06g/kg in soil and bentonite respectively. Higher organic matter in soil could be attributed to the accumulation and decomposition of plant and animal residue. Both soil and bentonite showed significant difference (6.06) in organic matter at ($p \leq 0.05$).

Table 1: Selected physico-chemical properties of soil and bentonite

Material	sand g/kg	silt g/kg	Clay g/kg	TCL	pH (H2O)	pH (KCl)	OC g/kg	OM g/kg	TEA Cmol/kg	TEB Cmol/kg	CEC Cmol/kg
Soil	880.00	60.00	60.00	S	5.98	5.12	13.10	22.82	1.20	2.39	3.59
Soil	820.00	40.00	140.00	S	5.02	4.66	10.80	18.81	0.80	3.20	4.00
Soil	864.00	40.00	96.00	S	5.74	5.01	9.70	16.90	1.00	2.90	3.90
Soil	860.00	40.00	100.00	S	5.63	5.00	12.20	21.25	1.20	2.92	4.12
Soil	866.00	44.00.	90.00	S	5.92	5.21	14.10	24.56	1.1	2.84	3.94
Mean	**858.00**	**44.80**	**97.20**	**S**	**5.66**	**4.99**	**11.98**	**20.87**	**1.06**	**2.85**	**3.91**
Bentonite	480.00	64.00	456.00	CL	6.21	5.50	5.70	9.93	1.20	3.28	4.48
Bentonite	544.00	60.00	390.00	SCL	5.88	5.88	10.2	17.77	1.30	6.92	8.22
Bentonite	520.00	72.00	408.00	SCL	6.04	5.42	8.00	13.94	0.90	5.55	6.45
Bentonite	490.00	60.00	450.00	SCL	6.11	5.40	6.44	11.22	1.20	6.80	8.00
Bentonite	532.00	68.00	400.00	SCL	6.00	5.45	10.00	17.42	1.10	5.8	6.90
Mean	**513.20**	**64.20**	**420.80**	**SCL**	**6.048**	**5.53**	**8.07**	**14.06**	**1.14**	**5.67**	**6.81**
LSD (0.05)	**57.34***	**12.66***	**67.07***		**0.37***	**0.49***	**3.48***	**6.06***	**0.30**	**1.48***	**1.62***

*Significant at 0.05 probability level

OC=ORGANIC CARBON, OM=ORGANIC MATTER, TEA=TOTAL EXCHANGEABLE ACID, TEB=TOTAL EXCHANGRABLE BASE, CEC=CATION EXCHANGE CAPACITY, TCL= Textural class, S= Sand, CL= Clay loam, SCL= sandy clay loam.

Total exchangeable acidity was 1.06cmol/kg and 1.14cmol/kg soil and bentonite. Total exchangeable acidity in soil and bentonite were not significant (0.30) at ($p \leq 0.05$).

Total exchangeable base were 2.85cmol/kg and 5.67cmol/kg in soil and bentonite respectively. Both soil and bentonite showed significant difference with TEB (1.48) at ($p \leq 0.05$).

CEC were 3.91cmol/kg and 6.81cmol/kg in soil and bentonite. Low CEC in soil could be attributed to leaching while higher CEC in bentonite could be attributed to presence of montmorillonite. CEC in soil and bentonite were significant (1.62) at ($p \leq 0.05$).

Effect of sorptive volume on lead adsorption

In table 2; adsorption capacity of lead by soil was 2372.89mg/kg, 4721.75mg/kg, 6845.06mg/kg and 9031.75mg/kg at 25ml, 50ml,75ml and 100ml solution respectively, bentonite were 2494.19mg/kg, 4984.14mg/kg, 7462.88mg/kg and 9935.75mg/kg. Adsorption capacity of Pb^{2+} by soil and bentonite showed increase with increase in volume of solution. Bentonite had higher adsorption capacity for Pb^{2+} than soil, this could be attributed to bentonite having a finer texture than soil which was coarse, this is in line with Jeanne & Roy (1991) who concluded that fine textured soil contain higher amount of Pb (3889mg/kg) and coarse textured soil contains (530mg/kg).

TABLE 2: Lead adsorption capacity in soil and bentonite(mg/kg)

	25 (ml)	**50(ml)**	**75(ml)**	**100(ml)**
Soil	2372.89	4721.75	6845.06	9031.75
Bentonite	2494.19	4984.14	7462.88	9935.75
LSD (O.05)	**11.53***	**75.36***	**124.92***	**117.91***

* significant at 0.05 probability level.

Table 3: Lead percentage adsorption in soil and bentonite(%)

	25 (ml)	**50(ml)**	**75(ml)**	**100(ml)**
Soil	94.71	94.44	91.26	90.32
Bentonite	99.77	99.68	99.51	99.36
LSD (O.05)	**0.89***	**1.51***	**1.66***	**1.18***

* significant at 0.05 probability level.

In table 3; percentage of adsorbed Pb^{2+} were 94.71%, 94.44%, 91.26% and 90.32% onto soil and 99.77%, 99.68%, 99.51% and 99.36% onto bentonite at 25ml, 50ml, 75ml, 100ml of solution. Percentage Pb^{2+} adsorbed by soil and bentonite decreased with increase in volume of $Pb(NO_3)_2$ solution. This could be attributed to increasing concentration of Pb(ii) in equilibrium solution with increase in the volume of Pb^{2+} solution.

Relationship between adsorption capacity and selected soil and bentonite properties

Table 4 and 5 shows the relationship between adsorption capacity and selected soil and bentonite properties. Soil and bentonite properties were correlated with adsorption capacity at 25ml, 50ml, 75ml and 100ml of Pb^{2+}solution.

In both soil and bentonite, sand particle showed a negative relationship with adsorption capacity, as sand particle increase adsorption capacity of lead by soil and bentonite decreases. Onto soil, at 25ml and 100ml of Pb^{2+} solution significant negative correlation was observed between lead adsorption capacity and sand (r= -0.63748) ($p \leq 0.05$) and (r= -0.94841) ($p \leq 0.01$) respectively. Onto bentonite a significant negative relationship was observed between adsorption capacity and sand particle (r=-0.7882) ($p \leq 0.01$) at 75ml of Pb^{2+} solution.

Clay particle, pH, organic matter, CEC correlated positively with adsorption capacity, indicating that lead adsorption increased with increase in these properties in both soil and bentonite. Onto soil, a significant positive relationship was observed between clay and adsorption capacity (r= 0.679013) ($p \leq 0.05$) and (r= 0.938516) ($p \leq 0.01$). Onto bentonite, relationship between clay and adsorption capacity was significant at 75ml (r=0.796997)

($p \leq 0.01$). Lead adsorption increases with clay content this is in line with Soldatini*et al.* (1976) who concluded that soil organic matter and clay content were two major factors which influence adsorption.

Table 4: Relationship between soil properties and adsorption capacity

SOIL PROPERTY	**ADSORPTION CAPACITY (mg/kg)**			
	25 (ml)	**50 (ml)**	**75 (ml)**	**100 (ml)**
Sand	-0.63748*	-0.33801	-0.48399	-0.94841**
Silt	0.183499	0.278904	-0.98318**	-0.6326*
Clay	0.440875	0.178261	0.679013*	0.938516**
pH	0.67343*	0.34333	0.36605	0.80554**
Organic matter	0.265212	0.685347*	0.57166*	0.4644
Cation exchange capacity	0.85523**	0.028603	0.869956**	0.48941

*and** = significant at 0.05 and 0.01 probability levels respectively

Table 5: Relationship between bentonite properties and adsorption capacity

BENTONITE PROPERTY	**ADSORPTION CAPACITY (mg/g)**			
	25 (ml)	**50 (ml)**	**75 (ml)**	**100 (ml)**
Sand	-0.2583	-0.3045	-0.7882**	-0.29677
Silt	0.562122	-0.89623	-0.0339	-0.88982
Clay	0.201419	0.411787	0.796997**	0.4004
pH	0.82877**	0.240184	0.86397**	0.303432
Organic matter	0.31496	0.17619	0.79785**	0.19937
Cation exchange capacity	0.010474	0.222997	0.79232**	0.153426

*and** = significant at 0.05 and 0.01 probability levels respectively

Onto soil, relationship between pH and adsorption capacity was significant (r= 0.67343) ($p \leq 0.05$) at 25ml and (r= 0.80554) ($p \leq 0.01$) at 100ml of $Pb(NO_3)_2$ solution. Onto bentonite, pH and adsorption capacity showed a significant relationship (r= 0.82877) ($p \leq 0.01$) at 25ml and (r= 0.86397) ($p \leq 0.01$) at 75ml of $Pb(NO_3)_2$ solution. A number of adsorption studies indicate that within the pH range (4 to 11), lead adsorption increases with increasing pH (Bittel and Miller, 1974; Braids *et al.*, 1972; Griffin and Shimp, 1976; Haji-Djafari*et al.*, 1981; Hildebrand and Blum, 1974; Overstreet and Krishnamurthy, 1950; Scrudato and Estes, 1975; Zimdahl and Hassett, 1977).

At 50ml and 75ml of $Pb(NO_3)_2$ solution, soil organic matter and adsorption capacity showed a significant relationship of (r=0.685347) ($p \leq 0.05$) and (r=0.57166) ($p \leq 0.05$), bentonite organic matter and adsorption capacity showed a significant relationship (r=0.79785) ($p \leq 0.01$) at 75ml of Pb^{2+} solution. Lead adsorption increases with increasing organic matter in soil, Gerritse*et al.* (1982) examined the lead adsorption properties of soils as a function of organic matter content of soils.

In soil CEC showed positive significant relationship with adsorption capacity at 25ml and 75ml (r=0.85523) and (r=0.869956) ($p \leq 0.01$) respectively. In bentonite, CEC showed positive significant relationship with adsorption capacity at 75ml (r=0.79232) ($p \leq 0.01$).

Adsorption Isotherm

Adsorption data were fitted into both the Langmuir and freudlich isotherm equations (1) & (2) respectively.

$$\frac{c_e}{q_e} = \frac{1}{K_L.Q_{MAX}} + \frac{1}{Q_{MAX}}.C_e \qquad (1)$$

$$Inq_e = \frac{1}{n}InC_e + InK_f \qquad (2)$$

C_e (mg/l) was the lead concentration in the solution at equilibrium,q_e (mg/kg) was the amount of lead adsorbed per unit mass of soil and bentonite (absorbent). From the experimental datas the adsorption capacity increased linearly with equilibrium concentration, suggesting that adsorption site on soil and bentonite was sufficient (Taha *et al*., 2010). Q_{MAX} and K_L are Langmuir isotherm constants related to the theoretical maximum adsorption capacity (mg/kg) and the energy of adsorption (l/g) respectively. $K_f$is the freudlich constant representing the adsorption capacity (l/g) and n is the freudlich exponent depiciting the adsorption intensity (dimensionless).

From fig.1 soil and bentonite adsorption data was analyzed and *ce/qe* was plotted against C_e to produce a linearized Langmuir isotherm equation for both soil and bentonite. The equation was analyzed and Langmuir constants were determined for both samples. Fig.1 indicates that both soil and bentonite adsorption data fairly fits into the Langmuir adsorption isotherm with ($0.5>R^2<0.9$), correlation factor for both soil and bentonite were $R^2=0.5385$ and $R^2=0.5671$ respectively.

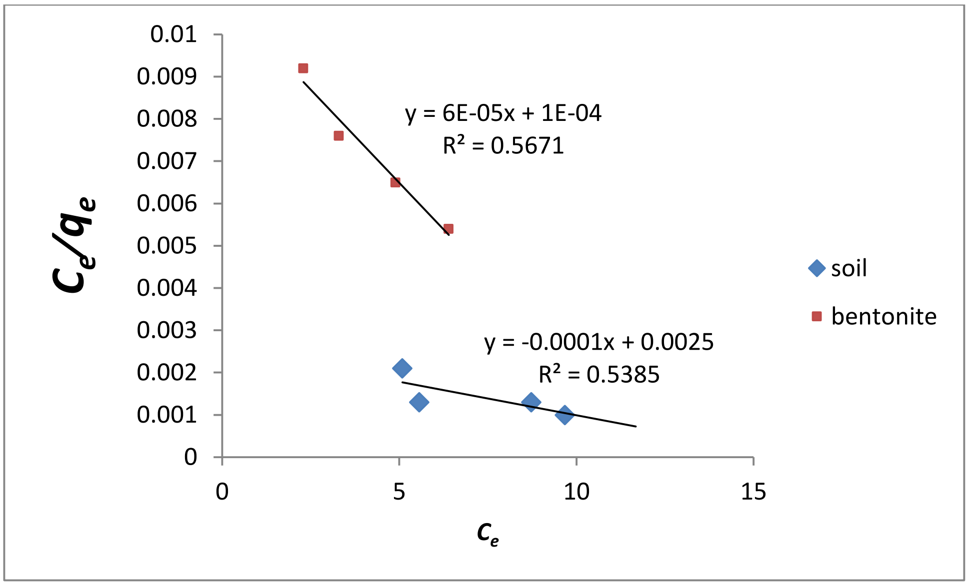

Fig.1 Langmuir Isotherm

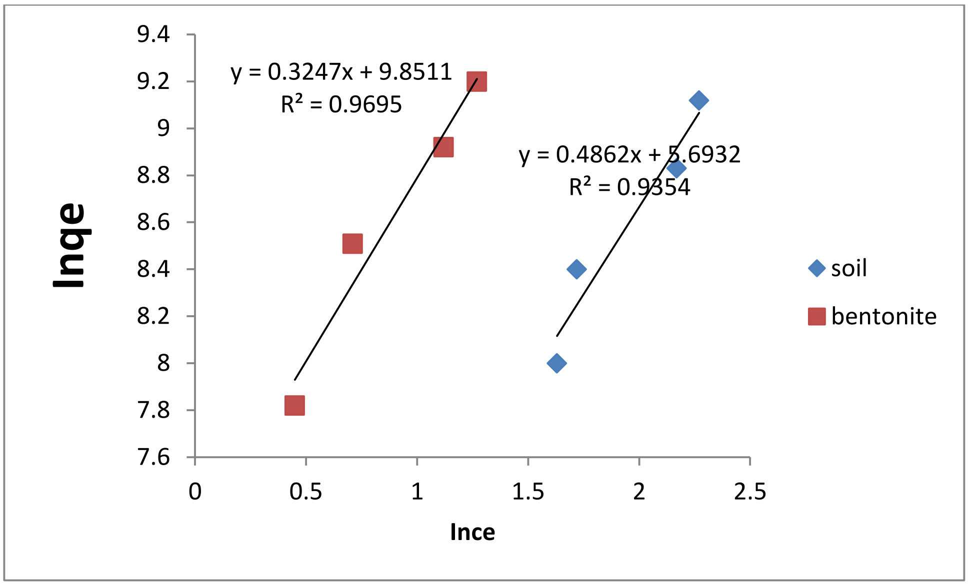

Fig.2 Freudlich Isotherm

From fig.2 soil and bentonite adsorption data was analyzed and *Inq_e* was plotted against *Inc_e* to produce a linearized Freudlich isotherm equation for both soil and bentonite. The equation was analyzed and Freudlich constants were determined for both samples. Fig.2 indicates that both soil and bentonite adsorption data fits perfectly into the freudlich adsorption isotherm with (R^2>0.9), correlation factor for both soil and bentonite were R^2=0.9354 and R^2=0.9695 respectively.

Langmuir and freudlich constants were determined for both soil and bentonite from the linearized equation and were presented in table 7. From the Langmuir constants,bentonite showed a higher maximum adsorption of 16666.67mg/kg than soil which was 10000mg/kg, while the freudlich maximum adsorption for bentonite (18979.20l/mg) which was far greater than that of soil (296.84l/mg), this could be attributed to bentonite having a higher clay content Soldatini*et al.* (1976). The langmiur adsorption isotherm indicated that bentonite (0.6Lmg^{-1}) had a higher energy of adsorption than soil (0.04Lmg^{-1}), freudlich adsorption isotherm indicated that bentonite (3.08) had a higher intensity of adsorption than soil (2.05).

TABLE 6: LANGMUIR AND FREUDLICH ISOTHERM CONSTANTS.

	Langmuir			**Freudlich**		
	K_L (Lmg^{-1})	**Q_{max} (mg/kg)**	**R^2**	**K_f (Lmg^{-1})**	**N**	**R^2**
SOIL	0.04	10000.00	0.5385	296.84	2.05	0.9354
BENTONITE	0.6	16666.67	0.5671	18979.20	3.08	0.9695

CONCLUSION

Soil and bentonite ability to absorb or retain lead was compared at increasing volume of lead nitrate solution. Lead being a bio-toxic heavy metal regarded as an environmental pollutant in agricultural soils was studied because of the potential adverse effects it may pose to soil health and the environment. The selected physical and chemical properties of soil and bentonite were correlated to estimate relationship between lead adsorption and the physic-chemical properties of the soils and bentonite under different volume of lead nitrate solution.

Positive correlation were observed between clay content, pH, organic matter and CEC, indicating that adsorption capacity of lead by soil and bentonite increases with increasing clay content, pH, organic matter and CEC, while sand correlated negatively with adsorption capacity of soil and bentonite. Lower adsorption capacity of lead by soil is attributed to high sand content. It can be concluded that physical and chemical properties was vital in the adsorption of lead by soil and bentonite.

Experimental data showed a better fitting in freudlich isotherm (R^2>0.9) than Langmuir isotherm (R^2<0.9), this indicates that soil and bentonite had multiple adsorption site. Higher lead adsorption capacity, energy of adsorption and intensity of adsorption was observed in bentonite, indicating that bentonite is a better absorbent of lead in the environment than soil.

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