Chromium (III) in Relation to Soil Properties and Bioaccessibility in Soils Polluted by Animal Wastes

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Abstract: Research on the concentration of chromium (111) (Cr 111)) was conducted in soils polluted by livestock wastes before the onset of the rainy seasons in 2006. Soil samples were randomly collected from surface soil horizons of 5 locations near university livestock farm. Soil samples were air-dried, crushed and sieved before laboratory analysis for some soil properties and chromium (Cr) analysis. Results showed that bioaccessibility of Cr (111) was constantly higher for I day aging than in 100 days aging. The amount of Cr (111) oxidized to Cr (V1) was less than 10 gm kg $^{-1}$ in the studied soils. Soil properties influenced both Cr (111) sorption and bioaccessibility, with clay, total organic carbon (TOC), total inorganic carbon (TIC), double deionized pH (pH DD1), and cation exchange pH capacity (CEC) giving a high r^2 value of 0.8. In all the models, variance inflation factor (VIF) was less than 4.0, suggesting insignificant collinearity and high reliability. The model can therefore be used as a good decision support tool in remediation of soils in the area. [Nature and Science. 2007;5(2):30-36] (ISSN: 1545-0740).
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Introduction

Heavy metals, such as chromium, lead, calcium and mercury, have not been shown to be essential for life. (Isirimah et al., 2003) but they are continuously introduced into the environment by anthropogenic activities. The principal sources of heavy metals pollution in the environment are domestic sewage sludge, industrial processing of ores, waste dump s, animal and human excrete, and these heavy metals are released in assorted and unjustifiable amounts (Nweke and Ekpete, 2003). Chromium (Cr) is one of such biotoxic heavy metals whose presence in the environment is widespread due to usage in many agro- industrial processes.

Chromium is thermodynamically stable in two oxidative states, namely chromium (111), which is cationic, and chromium (IV), which is anionic

It has been reported that cationic chromium is more stable and less problematic while the reverse is the case for the latter (Patterson et al., 1997). Isirimah et al., (2003) observed that Cr (VI) moves in well-aerated soils of moderate to high pH. These two major forms of Cr pose health risks when ingested (Skoworonski et al., 2001) but Cr (V1) is considered most harmful being carcinogenic and mutagenic at low concentration (Levis and Bianchi, 1992). However, the presence of Cr (111) in an environment is not healthful due to its potential to oxidize to hexavalent forming (Fendorf et al., 1992) thus become more bioaccessible (Rodriguez et al., 1998; Skoworonski et al., 2001). Decreased mobility of Cr (111) form in soils has been attributed to strong adsorption to the charged soil surface, formation of iron and aluminum oxides and hydroxides as well as formation of complex molecules with organic compounds in the soil. These organo metallic derivatives are often volatile and may concentrate in fatty tissue and cause chromosomal damage (Nweke and Ekpete, 2003).The main aim of this study was to assess the distribution of chromium Cr (111), relating it to some properties of polluted soil proximal to a livestock farm in Owerri municipality southeastern Nigeria.Specific objective was to relate the findings in the main objective to bioaccessibility.

Materials and Methods

Study area: The study was conducted before the onset of dry seasons in 2006 at Owerri southeastern Nigeria, lying on latitude 5^0 43'14", 623 and longitude 7^0 39' 34".490 (Handheld Global Positioning System) Receiver (Garmin LTD, Kansas, USA). Coastal plains sands (Benin Formation) are the

predominating geological material in the area (Orajaka, 1975). The area is dominated by lowland geomorphology (Ofomata, 1975) and the site is specifically 55 meters above sea level (Readings of Handled Global Positioning System) Receiver (Garmin, LTD, Kansas, USA). Owerri has mean annual rainfall ranging from 2250 to 2500 mm and mean annual temperature range of 27-28^{0}C (Federal Department of Agricultural Land Resources, 1985). Three months of dryness and bimodal rainfall peak characterize the study area.

The predominant vegetation is evergreen rainforest whose density has been reduced by farming and deforestation. Small/ holder livestock production is common with over 80% of rural families keeping West Africa Dwarf ruminants and mixed breeds of local and exotic chicken (Ejiogu, 1990). But, urbanization has led to the establishment of commercial farms, especially poultry and piggery while facilities of Agriculture in five tertiary institutions located in the town resulted in the mounting of livestock demonstration farms, including the Teaching and Research Farm of the Federal University of Technology Owerri, Nigeria. Consequent upon, dump sites are found near livestock farms. The livestock farm influences about. 10,000m^2 of arable farmland: Random surface soil sampling field studies was done at the dumpsite Six sampling sites were identified during a reconnaissance visit to the area, and 5 soil samples were collected from each sampling site. Soil samples were triplicates giving a total of 90 soil samples for the study. Soil samples were in air-dried, crushed and sieved using 2-mm sieve. Sieved soils were further disaggregated by gently grinding them in a mortar using a pestle, and this was re-sieved to provide a particle size fraction less than 250 mm (Rodriguez et al, 1999).

Laboratory analysis: Silt and clay fractions of soil samples were determined by hydrometer method (Gee and Or, 2002). Soil pH was estimated using double deionized (DDI) water in soil/solution ratio of 1:2. The pH of the clear supernatural was measured with a microprocessor ionalyzer/901 9orion Research, Beverly, MA) using a combination of glass and calomel electrode (Beckman Fullerton, CA). Total organic carbon (TOC) and total inorganic carbon (TIC) were determined by combustion method using Perkin-Elmer 2400 Series 11 CHNS/O analyzer. Before the determination of TOC, soil samples were pre-treated to remove TIC, which involved near boiling, 3 M HCL extraction method on agitated samples. Soil TIC was calculated as a deference between total soil carbon, which involved no pretreatment, and TOC. Extractable iron oxide (FeO) and Manganese oxide (MnO) were estimated using dithionite – citrate-bicarbonate (DCB) methods (Mehra and Jackson, 1960). Total chromium (Cr) in soils was estimated using a modified EPA method 3052. In this method, the soil was digested in a CEM microwave; model MDS-8ID, with hydro fluoric and nitric acid. Earlier, boric acid was added before sample analysis to facilitate the removal fluoric from solution through the formation of fluoroboric acid.

These soil samples were stored and analyzed for total Cr_T using inductively coupled plasma (ICP).

Bioaccessibility: Ten grams of sieved soil was weighed and placed in 200-ml glass centrifuge vessel along with 100 ml of 500 mg kg $^{-1}$ Cr (111) as Cr CL at pH 4.The slurry was agitated on a reciprocal shaker for 48 hours, centrifuged and the supernatant decanted for analysis. This was done 4 times and after the fourth addition of Cr, soils were washed 3 times with DDI water and air-dried. After air-drying, soils were crushed, homogenized and wetted with DDI water to achieve a 25% moisture content. Thereafter, soils were kept in a container out of direct light and 25% moisture content was maintained. This was followed by the inclusion of soils for 100 days (duration of study).In vitro bioaccessibility was attempted in which a physiologically based extraction test (PBET) was adopted from Ruby et al. (1999). Triplicate 0.4 g moist samples were placed in 50 ml polyethylene tubes to which 30ml 0.4 m glycerine at pH 1.5 and temperature of 37^0 agitated at 30+_ 2 revolutions per minute (rpm) for 60 minutes. The supernatant was separated from the solid through centrifugation. The pH of the supernatant was measured to ensure that the pH was within +_0.5 units of the initial pH. Then bioaccesibility was computed as follows:

% Bioaccessibility = $\dfrac{\text{(Cr in PBET supernatant (Ug mL}^{-1}) \times 30 \text{ mL/0.3g dry soil)}}{\text{(Cr in soil surface (mg kg} -1)}$

After this, Cr analysis was performed. The PBET supernatant, soil spiking solution and equilibrium solution were measured for Cr and equilibrium solution were measured for Cr (VI) and Cr_T. The value of Cr(VI) was estimated using a modified s-diphenyl- carbohydrazide colourimetric method (Bartlett and James, 1979) using a UV-VIS Spectrophotometer at a wavelength of 540 Nm (HP model

8453(VI)immediately to prevent reduction of Cr (VI) to Cr(III), Palo Alto, CA). Rapidly cooled PBET solutions were used to perform analysis of Cr (III) by glycine decoding to Jardine *et al., (*1999). Total chromium was estimated using Perkin Elmer Analyst 800 atomic absorption spectrophotometer (Wesley P.A.). Value of Cr(III) was computed as a difference between Cr_T and Cr (Vi).

Data analysis: Statistical analyses were conducted in SAS version 8.0 (SAS Institute, 2001). Logarithmic functions were used to minimize soil variables, which typically spanned several orders of magnitude. Pearson correlation analysis with uncorrected probabilities was used to examine relationships between bioaccessibility of Cr (III) and selected soil properties, Soil parameters with P- values < 0.10 were considered in a stepwise multiple linear regression analysis to develop models predicting bioaccessibility of Cr (III) from soil properties

RESULTS

Soil properties: Results of selected soil properties in the studied soils are shown in Table 1. Soils were generally acidic and of low cation exchange capacity. Soil pH values were slightly higher than those reported by Mbagwu *et al.* (2001) in the same region. Silt and clay contents were low and these results are consistent with the findings of Igwe et al. (1995). In all soil locations, total organic carbon (TOC) dominated total inorganic carbon (TIC). The same trend was exhibited by extractable iron oxide (FeO) over extractable manganese oxide (MnO). Earlier studies in the region (Igwe and Stalr, 2004) reported least values of MnO when compared with oxides of iron, aluminum and potassium.

Soil properties and chromium sorption

Chromium sorption (Cr III) in soils are related according to the data on Table 2. Soil pH influenced Cr adsorption and desorption, and consequently surface precipitation. The Cr (III) was increasingly precipitated as pH rose above 5.0 while it was complexed at lower pH value. Similarly, TIC increased Cr(III) sorption but there was no correlation between soil pH and TIC, suggesting a local effect of soil pH in Cr(III) sorption. The model also suggested that cation exchange capacity (CEC) promoted Cr (III) sorption, that is the higher the (CEC), the greater the Cr (III) sorption. Among the particle sizes determined, clay content had a positive correlation with Cr (III) adsorption and this could be attributed to the predominantly negatively charged sites of clay minerals. This condition is created by isomorphic substitution taking place at the charged sites. (Klein and Hurlbut, 1993). Negatively charged sites attract Cr^{3+} leading to the formation of an electrostatic bond. Relating this to CEC, it implies that greater availability of negatively charged sites increases CEC and as a greater propensity for Cr^{3+} to sorb. Again, clay sized particles have largest specific surface suggesting greater ability to attract and accommodate Cr^{3+}. In a similar study, Kaiser *et al.* (1996) observed increased sorption of dissolved organic carbon with increased surface area. By these results, the independent variables of soil pH, Clay content, CEC and soil carbon component can be used to predict Cr (III) sorption as dependent variable. Value of variance Inflation factor (VIF) was slightly larger than no multicollinearity among tested variables. The relationship describing Cr (III) sorption was:

Cr (III) (g kg $^{-1}$ on soil) = -186.3 + (82.9 x clay) +113.3x CEC) +(180 X TIC) +(184.4 pHDDI)…..(1)

The Cr (III) sorption was strongly correlated with soil properties (r^2 =0.6).

Aging, soil properties and chromium bioaccessibility: Aging influenced the amount of Cr (III) that is soluble and available in soils. Bioaccessibility values were consistently higher for 1 day aging compared with 100 days (Table 3), with values ranging from 12.2 to 21.4 g kg^{-1} and 5.1 to 8.6 g kg $^{-1}$ for 1day and 100 days, respectively. As obtained from the PBET extractment, a value of Cr (VI) following the oxidation of Cr (III) was less than 10 g kg^{-1} (that is less than 1%).Bioaccessibility of Cr (III) was influenced by clay content, TIC, TOC, pH, pH_{DDI} (DDI) and CEC (Table 4). The model attributes show that Cr (III) bioaccessibility decreases as TIC increases and clay content decreases. In other words, as the clay content increased, the quantity that is bioaccessible also increased. Step-wise multiple regression analysis also suggested that Cr (III bioaccessibility was significantly correlated with, clay (P<_0.001) and TOC (P<_ 0.001) of soils (Table 4). The relationships explaining Cr (III) bioaccessibility is given as follows:

Cr (III bioaccessible =17.6 +(0.5 x clay
-         (4.1 x TOC)……………...(2)
Cr (III) bioaccessible = 17.6 +(6.3 X pH) + (7.1 X CEC
               -   (8.2 x TIC) …………………(3)

With an r^2 value of 0.8, the P value was less than 0.01, implying that parameter attributes were statistically rigorous at 99% confidence, with VIF values approximately 1.000(Table 4)

Table 1. Selected properties of soils studied (mean Values)

Location	TOC G kg^{-1}	TIC G kg^{-1}	FeO G kg^{-1}	MnO g kg^{-1}	Silt G kg^{-1}	Clay G kg^{-1}	CEC cmol kg^{-1}	pH DD1
A1	1.6	0.6	5.1	1.0	20	50	6.1	5.6
A2	0.9	0.2	4.8	0.4	18	80	7.7	5.8
A3	1.7	0.5	2.8	0.3	19	87	78	5.6
A4	2.0	0.4	5.2	1.0	21	92	9.2	5.7
A5	1.1	0.6	2.2	O.2	20	82	6.2	4.9
B1	1.2	0.4	4.6	0.9	22	79	6.3	4.8
B2	0.9	0.4	4.0	0.7	18	68	5.8	4.9
B3	2.7	0.5	3.6	0.6	19	70	8.8	5.3
B4	2.1	0.3	1.9	0.2	16	68	8.2	5.6
B5	0.8	0.5	2.4	0.3	14	81	4.8	5.8
C1	2.2	0.3	1.9	0.2	18	70	8.3	5.8
C2	1.6	0.5	1.6	0.1	25	74	6.0	4.8
C3	1.1	0.5	1.9	0.3	2.0	68	5.8	4.9
C4	1.6	0.4	4.8	0.8	21	82	5.9	4.9
C5	1.9	0.5	5.2	1.0	18	79	6.0	5.0
D1	1.2	0.3	2.3	0.3	30	90	10.6	5.4
D2	0.8	0.4	2.6	0.5	22	6.8	4.8	4.8
D3	0.9	0.4	2.1	0.2	28	72	4.8	5.0
D4	2.6	0.5	2.6	0.4	24	68	8.8	5.8
D5	1.8	0.5	4.9	0.8	21	71	6.0	4.8
E1	0.9	0.6	5.1	0.9	20	72	4.6	4.7
E2	1.2	0.4	4.2	0.6	21	73	4.8	4.7
E3	1.5	0.3	6.1	1.1	18	80	8.6	5.8
E4	2.8	0.5	2.9	0.4	22	69	2.8	5.3
E5	2.1	0.4	2.2	0.3	26	72	7.6	5.7
F1	1.6	0.5	3.1	0.6	20	83	8.9	5.8
F2	1.9	O.4	2.2	0.3	18	82	9.6	5.6
F3	2.1	0.5	1.8	0.2	19	78	8.6	5.5
F4	2.0		2.0	0.3	22	82	10.0	5.3
F5	1.8		2.6	0.3	18	81	9.6	5.2

TOC = total organic carbon, TIC =total inorganic carbon, CEC=cation exchange capacity, DDl = double deionized, FeO = extractable Fe oxide, MnO = extractable Mn oxide.

Table 2．Parameter estimates, standard errors and other statistics got from a multiple regression analysis relating Cr(III) sorption to some soil properties

Parameter	Value	SE	P	V/F
Intercept	-186.4	64.1	<0.001	-
Clay (g kg^{-1})	82.9	17.8	<0.001	1.016
CEC (cmol kg^{-1})	113.3	23.2	0.015	1.080
TIC (g kg^{-1})	180.2	56.6	0.042	1.524
pH (DDl)	184.4	32.4	<0.001	1.801
r^2	0.6		<0.001	

Table 3. Solid phase concentrations of Cr (III) in soils with their corresponding bioaccessibility after 1 and 100 days incubation (mean –values)

Location	Cr- in soil (g kg^{-1})	Cr (III) bioaccessibile 1 day. (g kg^{-1})	Cr(III) 100 days (g kg^{-1})	Boiaccessible Value of Oxidized Cr (VI) (g kg^{-1})
A	38.3	16.4	7.6	4.2
B	42.1	19.6	8.2	4.5
C	53.6	21.4	8.6	4.7
D	36.8	15.3	6.6	3.8
E	33.2	12.2	5.1	2.9
F	44.6	20.8	8.3	4.1

Table 4. Parameter estimates, standard errors and other statistics obtained from a multiple linear regression analysis that related some soil properties to Cr (III) bioaccessibility.

Parameters	Value	SE	P	VIF
Intercept	17.6	2.1	<0.001	-
Clay content (g kg^{-1})	0.5	0.8	<0.001	1.001
TOC (g kg^{-1})	-4.1	0.7	<0.001	1.001
TIC (g kg^{-1})	-8.2	0.9	<0.001	1.002
PH$_{DDI}$	6.3	1.2	<0.001	1.002
CEC (Cmo1kg^{-1})	7.1	0.8	<0.001	1.002
r^2	0.8		<0.001	

SE = Standard error, P = probability, VIF = Variance inflation factor, TOC = Total organic carbon, TIC = Total inorganic carbon.

Discussions

Generally, soils of the study site have high potentials for sorption activities, Presence of organic deposits from livestock pens coupled with presence of FeO, suggest high sorption of Cr (III). Gerke and Hermann (1992) showed that the phosphate sorption in Fe-humic substance mixture was six to seven times larger than amorphous iron oxide, due to the formation of ternary complex between humic substances and phosphate. Similarly, Fe- humic substances create negative charges, which may attract Cr^{3+} in soils. On the other, the more mobile Cr(VI) anions may be attracted to the positively charged ferric substances in the highly oxidized soils of the study area. The presence of MnO in soils although low has great influence on

sorption behaviour of soils of the site. Negra *et al.*(2005) noted that the chemical influence of Mno in the soil system may be much greater than that suggested by its relatively low abundance. Manganese oxides are powerful oxidizers known to oxidize Cr (III) to Cr (VI) (Kozuh *et al.,* 2000; Kim *et al*. 2002). The values of FeO and MnO may have influenced sorption of Cr (III0)although they were not used as independent variables in the study

The determined soil properties had significant effect on Cr (III) sorption (P<_0.05) (Table 2). The values of VIF suggested that collinearity between the independent variables was not significant (Table 2) and it can be inferred from this result that the parameter estimates are reliable. Because, VIF values were less than 4.0, collinearitly does not pose a problem in this model

Bioaccessibility of Cr (III) decreased towards 100 days, suggesting that the aging effect is related to the enhanced stability of Cr (III) on the soil surface with time. Karthein et al. (1991) attributed this sampling with time to structural re-orientation of Cr surface, bonds and slow precipitation reaction . It is also possible that after 100 days, aging effects on bioaccessibility decreases to insignificance. Bioaccessibility of Cr (III) in soils did not show any distinct trend in response to location, and this could be attributed to low intensive field sampling of soils used in the determination.

 Low values of oxidized Cr (III) (that is, Cr (III) to Cr (VI) values, tend to suggest minimal oxidation reactions due to low MnO content of soils. It is also possible that oxidized products of anionic chromium (Cr) were tightly held by the soil exchange complex. As surface soil samples were used in the study, organic matter may have reduced oxidized Cr (Eary and Rai, 1991. Fendorf and Li, 19960).

Model attributes (Table 4) indicate that the parameter had good association; (r^2 – 0.8), hence reliable predictions of Cr (III) sorption in the site. It implies that 80% of the variability in Cr (III) bioaccessibility was explained. Low values of VIF (nearly 1.000) shows that there was no redundant information in soil properties and there was insignificant co- linearity between independent variables. Low standards error (SE) on the estimated values (Table 4) are further indicators of the reliability of parameter estimates.

Based on the results of the step- wise multiple regressions (Table 4), Cr (III) had an inverse relationship with the TIC implying that as TIC, increases bioaccessibility of Cr (III) decreases. Total inorganic carbon promotes the formation of solid phase Cr (III) hydroxides, which are sparingly soluble even undersevere acidic soil milieu. The precipitation of these hydroxides cause them in cover the soil surfaces thereby making Cr (III0) difficulty bioaccessible. Also the relationship between bioaccessibility and TOC and Clay content was smaller to that relating it to TIC that relating increased TOC resulted to decreased bioaccessibility of Cr (III). Organic matter is a major sorbent for heavy metal cations (Sparks, 1995) since it substantially contributes to the generation of negative charges in the soil system. These heavy metal cations when complexed are not easily relased since carboxylic bonds hold them. The implication of these complexation reactions between heavy metal cation and TOC is that groundwater is sparingly contaminated.

Conclusions

Soil properties influenced Cr (III) sorption characteristics in the studied soils. The amount of bioaccessible Cr (III) also depended on the soil properties and decreased with time. Bioaccessiblity of Cr (III) can be predicted with high degree of reliability using clay content, TIC, TOC, pH and CEC, having an r^2 value of 0.8.

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