



Physico-chemical characterization of a farmland affected by wastewater in relation to heavy metals

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Abstract: This study investigated selected properties of soils affected by wastewater and its relationship with some heavy metals. A free survey technique involving target sampling was used in siting soil profile pits. Soil samples were collected based on horizon differentiation and analyzed using routine and special analytical techniques. Soil data were subjected to correlation analysis using SAS program. Results show that all heavy metals studied had values above critical limits in the polluted soils using known standards and that these biotoxic metals decreased with soil depths. Highly significant ($P=0.01$ and 0.05) relationships were established between investigated heavy metals and some soil properties, especially soil pH and organic matter. Further studies involving more edaphic properties, biotoxic metals and their bioaccessibility in crops growing on wastewater soils will surely enhance knowledge and management of these highly anthropogenically influenced soils of the study site.

Key words: Bioavailability, Characterization, Heavy metals, Soil pollution, Wastewater

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INTRODUCTION

In most developing countries, wastewater is disposed without treatment, while treated water is considered an important element in water resources planning (Tchobanoglous and Burton, 1991). Toxic chemicals and pathogenic microorganisms abound in untreated wastewater and have a potential for deleterious health effect and disease transmission (El-Arby and Elbordiny, 2006). Wastewater percolates the soil system where it enriches toxicity levels of heavy metals and humans become victims of its hazardous effect by consuming crops grown on the contaminated soils.

Soils affected by wastewater treatment plant activity exhibited significant toxicity on *Daphnia magna* and *Selenastrum capricornutum* growing within Youngsan River watershed in Soul, Korea (Ra *et al.*, 2007). Many heavy metals have chronic ill-health effects on man particularly children (Body *et al.*, 1991). Abdel Aziz (1992) reported that mean values of total Fe, Mn, Zn, Cu, Pb and Cd in surface parts of

El-Saff soils polluted by wastewater were 15.56×10^{-6} , 2340×10^{-6} , 399×10^{-6} , 1666.5×10^{-6} , 129×10^{-6} and 1.95×10^{-6} , respectively. Total Fe, Zn, Cu, Co, Ni and Pb concentrations in upper 10 cm layer increased to 9.0, 3.3, 10.6, 9.6, 6.9 and 3.2 times when irrigated with sewage water, in comparison with Nile water (Elgala *et al.*, 2003). Nickel in small amounts is essential for maintaining proper health in animals and humans (Egyptian Environmental Affair Agency, 1996), while in concentrations of 2.03×10^{-6} mg/kg or less it may not have adverse health effects (Soltan *et al.*, 2005). Cobalt is beneficial because it is part of B₁₂ and has also been used in the treatment of anaemia, while exposures to high levels of cobalt affect the respiratory system (Soltan *et al.*, 2005). Aerial cobalt inhaled by a worker had led to asthma and pneumonia (ATSDR, 1992). Boron is both essential micronutrient required for optimum crop performance and a toxicant at elevated concentration, and the range between its adequacy and toxicity is the smallest among micronutrients (Su and Suarez, 2004). Cadmium plays no known nutritional role in human but enters

human diets through plant foods (Benson and Ebong, 2006). Yet, uptake of Cd by plants is very high in soils amended with sewage sludge (Peles *et al.*, 1998; Gallardo-Lara *et al.*, 1999).

Heavy metals accumulate in soils in various forms: water-soluble, exchangeable, carbonate associated, oxide-associated, organic-associated and residual (He *et al.*, 2004) and differ in their mobilities (Moore *et al.*, 1998). These attributes of heavy metals influence soil and other natural resources.

Most urban and peri-urban settlements in Nigeria are characterized by indiscriminate disposal of wastes including wastewater. The disposed wastewater ends in natural valleys and plains in such locations. Yet, vegetable and arable farms are established in such wastewater-polluted soils, since crops grown on them yield well. But little (Benson and Ebong, 2006; Onweremadu *et al.*, 2007) has been conducted on characterization of sewage and its influence on soil properties. The major objective of this study was to characterize soils affected by urban wastewater, especially in terms of selected heavy metals concentrations in such soils.

MATERIALS AND METHODS

Study area

The soil samples were collected from the farmlands of Imo State University, Owerri, Nigeria before the rains in 2006. Owerri is the capital city of Imo State in the southeast of Nigeria and lies between latitudes 5°15' N and 5°50' N, and longitudes 6°30' E and 7°15' E. Soils of the area are formed from Coastal Plain Sands (Benin Formation) of the Oligocene-Miocene geological era. It has a lowland geomorphology, and is of humid tropical climate, with an average annual rainfall of about 2500 mm and mean annual temperature ranging from 26 to 29 °C. It is situated within the highly depleted rainforest vegetation, characterized by a variety of vegetal forms although dominated by trees and shrubs. Owerri is an urban area with farming being practiced on any available land space. In the study site, wastewater arising from residential homes, students hostels, cottage industries, commercial eating houses and automobile service stations flow towards a lowland portion of the university farm. Residents cultivate on less

waterlogged soils of the site for vegetable and arable crop production.

Field studies

Field sampling was conducted using a free survey technique involving target sampling of soils from two sites, namely untreated wastewater-affected soils and soils from control pits, that is, soils unaffected by untreated wastewater. On each soil category, five profile pits were sunk and soil samples were collected at fixed depths, namely surface layer (0~10 cm), subsurface layer 1 (10~30 cm), subsurface layer 2 (30~60 cm), subsurface layer 3 (60~100 cm), subsurface layer 4 (100~150 cm) and subsurface layer 5 (150~200 cm). These soil samples were air-dried and sieved using 2 mm sieve in readiness for various laboratory analyses.

Laboratory studies

Particle size distribution was determined by hydrometer method according to the procedure of Gee and Or (2002). Bulk density was measured by core method (Grossman and Reinsch, 2002). Electrical conductivity (EC) was determined from the filtrate obtained from the suspension for the pH analysis with a conductivity meter. Total carbon in both soil and water was estimated by combustion on a Leco model 21-273 combustion (Leco Corporation, Svenka AB Uplands Vasby, Sweden). The pH was determined using 1:2.5 soil-liquid ratio (Thomas, 1996). Exchangeable basic cations were estimated by inductively coupled plasma atomic emission spectrometer (ICP-AES) (Integra XMO, GBC, Arlington Heights, IL, USA). Total nitrogen was determined by Kjeldahl digestion with a Keltec Auto 1030 system (Tecator, Hoganas, Sweden).

Cation exchange capacity (CEC) was measured by repeated saturation using IM NH₄OAC followed by washing, distilling and titrating (Soil Survey Staff, 1996). Available phosphorus was determined by Olsen method (Emteryd, 1989). Sodium adsorption ratio (SAR) was computed as

$$SAR = C_{Na^+} / \sqrt{(C_{Ca^{2+}} + C_{Mg^{2+}})^{1/2} / 2},$$

where *SAR* is sodium adsorption ratio, C_{Na^+} , $C_{Ca^{2+}}$, $C_{Mg^{2+}}$ are cationic concentrations of sodium, calcium,

and magnesium, respectively.

Nitrates and phosphates were determined by phenoldisulphonic acid and ascorbic acid molybdenum blue methods, respectively, using UV/Visible spectrophotometer.

Total Fe, Mn, Zn, Cu, Co, Ni, Cd and Pb were measured by SP 1900 Pye Unicam Recording Flame Atomic Absorption spectrophotometer at their respective wavelengths after wet digestion with a mixture of HCl and HNO₃. Total dissolved solids (TDS) and chemical oxygen demand (COD) were estimated according to the procedure of Vogel (1978).

Statistics

Linear regression analysis was performed on soil data and heavy metals concentrations using SAS computer software (SAS Institute, 2001).

Results and discussion

Characterizations of the untreated wastewater are shown in Tables 1 and 2. Micronutrients and heavy metals concentrations (mg/L) were 1.7 (B), 5.4 (Fe), 0.7 (Mn), 0.8 (Cu), 3.6 (Zn), 0.4 (Ni), 0.2 (Cd) and 15.0 (Pb), exceeding FAO (1976) and WHO (2003) maximum permissible limits of less than 1.0, 5.0, 0.2, 0.2, 2.0, 0.2, 0.01 and 5.0 mg/L for B, Fe, Mn, Cu, Zn, Ni, Cd, and Pb, respectively. These results suggest a high possibility of heavy metal contamination in the soils tested. It implies that repeated passage of this untreated wastewater may increase heavy metal load of affected soils, which is agreed with the findings of Li *et al.* (1997). However, soil and heavy metal properties as well as environmental factors may affect level of contamination (He *et al.*, 2004) and soil retentivity.

Table 1 Elemental content of studied wastewater (mg/L)

Parameter	Value
Ca	82.7
Mg	49.8
K	23.2
Na	428.6
B	1.7
Fe	5.4
Mn	0.7
Cu	0.8
Zn	3.6
Ni	0.4
Cd	0.2
Pb	15.0

The concentrations of wastewater parameters are as follows: TDS (150.0 mg/L), COD (165.0 mg/L) and BOD (385 mg/L). Values of COD and BOD contrasted widely with results (COD 700~1368 mg/L and BOD 434~721 mg/L) obtained by Banu *et al.* (2007) from domestic wastewater collected from Nessapakam Sewage Treatment Plant, Chennai in India. Total dissolved solids (TDS) exceeded FAO (1976) permissible limits but were lower than 2000 mg/L for land application in Nigeria (FEPA, 1992). Also, values of PO₄³⁻ and Cl⁻ exceeded FAO (1976) critical limits of 8.6 mg/L and <140 mg/L, respectively, while NO₃⁻ (6.9 mg/L) status was lower than FAO (1976) limits of 10 mg/L.

SOIL PROPERTIES

There were variations in the properties of soils affected by wastewater when compared with unaffected (control) soils (Table 3). There were slight changes in textural characteristics except in silt and clay contents, possibly resulting from fineness of the later particle size fractions, and this encouraged their transportability by wastewater. The characteristic sandiness of soil could be attributed to the nature of parent material (Coastal Plain Sands) from which soils are formed, coupled with aggressive tropical climate and land use history of the study site. Sandiness of these soils suggests easy leaching of heavy metals and nutrients through the pedosphere to groundwater. Increased biotoxicity occurs in soils with less clay content (Babel and Opiso, 2007) as clay

Table 2 General properties of studied wastewater

Parameter	Value
EC (dS/m)	4.6
TDS (mg/L)	150.0
COD (mg/L)	165.0
BOD (mg/L)	385.0
pH	5.9
TN (mg/L)	53.2
NO ₃ ⁻ (mg/L)	6.9
PO ₄ ³⁻ (mg/L)	18.4
Cl ⁻ (mg/L)	922.0
SAR	8.8

EC: electrical conductivity; TDS: total dissolved solids; COD: chemical oxygen demand; BOD: biologic oxygen demand; TN: total nitrogen; SAR: sodium absorption ratio

surfaces are negatively charged thereby attracting cationic heavy metals. Lower bulk density value was recorded from wastewater-influenced soils, and this could be attributed to higher organic matter content when compared with unaffected soils. Soil organic matter reduces availability of these heavy metals by chelation (Ekundayo and Fagbami, 1996). This could be a reason that vermicompost (Carrasqueros Durán *et al.*, 2006) and hulls of palm tree (Gueu *et al.*, 2006) were suggested for bioremediation, as photocatalytic process is expensive (Desrosiers *et al.*, 2006). Soils were strongly acidic, similar to the findings of Aroh (2003) that soil pH ranges from 3.8 to 4.1 in 1 mol/L KCl, implying high potentials for bioavailability of these heavy metals. Soil pH value was slightly higher in the soils affected by wastewater and this possibly caused the increased value of cation exchange capacity (13.6 cmol/kg) in the soil when compared with 9.2 cmol/kg value of the same parameter in the unaffected soils.

Table 3 Some properties of studied soils(surface soils)

Parameter	Soils affected by wastewater	Control
EC (dS/m)	3.2	3.6
pH (water)	5.0	4.8
CEC (cmol/kg)	13.6	9.2
OM (g/kg)	35.0	23.7
Sand (g/kg)	848.8	850
Silt (g/kg)	31.2	40
Clay (g/kg)	120.0	110
Textural class	SL	SL
BD (mg/m ³)	1.40	1.44
Av. P (mg/kg)	44.2	12.8
TN (g/kg)	16.3	2.8

EC: electrical conductivity; CEC: cation exchange capacity; OM: organic matter; BD: bulk density; Av. P: available phosphorus; TN: total nitrogen

Heavy metals concentrations in soils

Data on total content of heavy metals are given in Table 4, indicating greater concentrations in soil profiles affected by wastewater. Generally, concentrations of these heavy metals were greater in surface horizons than those in the subsurface counterparts. Increasing total amounts of these metals in upper horizons is attributable to repeated supply of untreated wastewater. However, in the unaffected soils, greater concentrations of heavy metals were noticed,

suggesting possibility of fallen forms of these metals on the soils unaffected by wastewater. This could be due to proximity of sources of the wastewater to the study site. Similar findings were made by Soltan *et al.*(2005) in soils proximal to a ferrosilicon production factory at Edfu, Aswan in Egypt.

The concentrations of Fe, Mn, Zn, Cu, Ni, Pb, Cd and B in surface horizon of wastewater soils were 1.53, 2.35, 2.22, 4.60, 2.02, 2.45, 8.00 and 2.33 times more than the concentrations of the same heavy metals in the unaffected soils. Generally, these metal concentrations declined with depth. The abundances of Fe, Zn and Cu in wastewater soils may be good for crops so long as their concentrations do not exceed tolerance limits. Similarly, Dumontet *et al.*(1990) and El-Gendi *et al.*(1997) reported the use of treated drainage water for enhancing these micronutrients in soils. But, higher concentrations in the plough layer suggest that most arable plants may get these micronutrients by interception and mass flow while tree crops may suffer deficiencies due to inadequacy in the deeper horizons.

Table 4 shows toxicities of micronutrients and heavy metals. They exceeded FAO (1976) permissible limits of 5.0 (Fe), 0.2 (Mn), 2.0 (Zn), 0.2 (Cu), 0.2 (Ni), 5.0 (Pb), 0.01 (Cd) and 1.0 (B) mg/kg. However, critical maximum levels of these heavy metals and uptake by crops may depend on metal form and soil type (He *et al.*, 2004), soil management (Chen and Lee, 1999) and crop type (Kabata-Pendias and Pendias, 1992; Wang and Liao, 1999).

Relationships of heavy metals and selected edaphic properties between studies

Relationships between micronutrients, heavy metals and some soil properties are shown in Table 5. Soil chemical parameters had very significant ($P=0.05$) relationship with studied heavy metals. Soil pH had the highest influence on these heavy metals in the pedosphere, indicating highly significant ($P=0.01$) relationship with Fe ($r^2=0.68$), Zn ($r^2=0.59$), Cu ($r^2=0.77$) and B ($r^2=0.61$). The coefficient of determination obtained when pH was correlated with Mn in this study ($r^2=0.38$, $P=0.05$) contrasted with the findings ($r^2=0.48$) of Negra *et al.*(2005) in the northwest of Vermont. Lead showed the highest significance ($r=-0.96$, $r^2=0.92$, $P=0.01$) relationships with soil pH, implying that as pH decreases, Pb concentration in

Table 4 Distribution of heavy metals (mg/kg) in studied soils (mean values) (N=60)

	Depth (cm)	Total Fe	Total Mn	Total Zn	Total Cu	Total Ni	Total Pb	Total Cd	Total B	
Wastewater	Surface layer	0~10	48.6+5.11	27.18+3.23	11.28+1.71	7.00+1.02	1.90+0.08	6.92+1.03	0.80+0.058	1.4+0.05
	Subsurface layer 1	10~30	46.3+5.06	22.28+1.79	11.16+1.01	6.44+0.91	1.62+0.08	6.82+0.82	0.40+0.06	1.2+0.03
	Subsurface layer 2	30~60	46.1+5.08	21.56+1.61	10.96+0.96	5.72+0.56	1.34+0.06	6.42+0.88	0.10+0.04	0.8+0.03
	Subsurface layer 3	60~100	43.8+4.96	19.18+1.16	9.38+0.92	4.08+0.38	1.02+0.07	4.22+0.68	0.08+0.04	0.3+0.02
	Subsurface layer 4	100~150	39.6+4.86	17.08+0.96	7.24+0.36	3.96+0.28	0.92+0.07	3.20+0.36	0.03+0.03	0.6+0.02
	Subsurface layer 5	150~200	35.8+4.37	12.92+0.97	4.56+0.24	1.90+0.24	0.62+0.06	2.46+0.29	0.03+0.02	0.2+0.01
Control	Surface layer	0~10	31.6+1.19	11.56+1.18	5.06+1.12	1.52+0.11	0.94+0.02	2.82+0.08	0.01+0.01	0.6+0.02
	Subsurface layer 1	10~30	31.4+1.16	11.40+1.16	4.32+1.08	1.50+0.08	0.90+0.02	2.80+0.06	0.01+0.01	0.5+0.02
	Subsurface layer 2	30~60	30.8+1.15	10.36+1.15	4.16+0.92	1.46+0.06	0.88+0.02	2.56+0.06	—	0.3+0.02
	Subsurface layer 3	60~100	30.7+1.09	10.28+1.09	4.12+0.82	1.44+0.04	0.86+0.01	2.20+0.06	—	0.1+0.01
	Subsurface layer 4	100~150	30.2+0.98	10.22+0.98	4.11+0.36	1.43+0.02	0.40+0.01	1.96+0.03	—	0.1+0.01

Table 5 Correlation coefficients of soil properties of micronutrients and heavy metals in the study site (N=60)

Property	Correlation coefficients							
	Fe	Mn	Zn	Cu	Ni	Pb	Cd	B
EC	0.57*	0.62*	0.19 ^{NS}	0.23 ^{NS}	0.24 ^{NS}	0.14 ^{NS}	0.25 ^{NS}	0.72*
pH	-0.83**	0.62*	-0.77**	-0.88**	0.92**	-0.96**	-0.89**	-0.78**
OM	-0.73**	-0.76**	-0.86**	-0.91**	-0.91**	-0.88**	-0.82**	0.59**
Clay	-0.79*	-0.81	-0.84*	-0.74*	-0.69*	-0.79*	-0.83*	0.62*
Av. P	-0.88*	-0.68*	-0.89*	-0.66*	-0.72*	-0.72*	-0.92**	-0.75*
BD	0.2 ^{NS}	0.16 ^{NS}	0.14 ^{NS}	0.18 ^{NS}	0.16 ^{NS}	0.19 ^{NS}	0.21 ^{NS}	0.18 ^{NS}

EC: electrical conductivity; OM: organic matter; Av. P: available phosphorus; BD: Bulk density; **: $P=0.01$; *: $P=0.05$; ^{NS}: not significant

soils increases. Similar findings were made by Abdel-Ghani *et al.* (2007). At lower pH values, the hydrogen ions compete with Pb and other heavy metals, which in turn leads to partial releasing of the latter. But Pb may be completely released when soils become extremely acidic. At high pH (alkalinity) as well as on sandy acidic soils, Zn becomes least available, possibly due to leaching (Foth, 1984). Soil organic matter had a negative significant ($P=0.01$) relationship with all the heavy metals except B. Similar results were recorded when clay was correlated with heavy metals (Table 5), showing that an increase in soil organic matter and clay may decrease availability of heavy metals. Organic matter and clay provide negatively

charged surfaces which attract the positively charged heavy metals for adsorption. In a similar study, Clemente *et al.* (1991) reported that organic materials promoted fixation of heavy metals in a mine soil contaminated with mineral sulphides in Aznalcolar, Spain. Available phosphorus had significant ($P=0.05$) negative relationship with all heavy metals, implying unavailability of phosphorus in soils affected by wastewater. Abundance of Fe in these soils increases phosphate sorption (Giesler *et al.*, 2005) and this is significant under acidic soil conditions (Kleinman and Sharpley, 2002). With the exception of Fe, Mn and B, there were non-significant relationships between electrical conductivity and heavy metals. Soils

of the agroecology are sandy (Akamigbo, 2001), which confers macroporosity on soil, thereby reducing electrical conductivity (Mbagwu *et al.*, 1983) and possible interactions between toxic metals and electrical conductivity. Poor relationship was also established between bulk density and heavy metals. One would expect a great influence of bulk density on heavy metals but such may have been masked by other soil properties.

CONCLUSION

The results of heavy metal analysis of soil influenced by wastewater show that values of these toxic metals are higher than limits, suggesting a high degree of bioavailability and biotoxicity. These heavy metals decreased with depth in both affected and control soils. There were strong relationships between these heavy metals and some soil properties, particularly chemical parameters. Effect of these heavy metals on crops growing on wastewater soil needs further studies to determine their tolerance levels as well as hyper accumulating characteristics.

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