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Determination of some Heavy Metals Speciation Pattern in Typha domingensis invaded Soil in Bauchi, Nigeria

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ABSTRACT

The speciation of some heavy metals on Typha domingensis invaded soil along Gombe road, Bauchi were evaluated. Soil samples from the Typha domengensis invaded soil and the control site were collected and analysed for exchangeable bound metals, carbonate bound metals, manganese bound metals, iron-manganese bound metals, organic/sulphide bound metals and residual bound metals. The results on the Typha domingensis invaded soil were found to be exchangeable bound metals; Fe 1.47 ± 0.21 mg/dm3, Zn 1.45 ± 0.02 mg/dm3 and Pb 0.16 ± 0.04 mg/dm3. Carbonate bound metals Fe 26.10 ± 1.01 mg/dm3, Zn 2.66 ± 0.17 mg/dm3 and Pb 0.89 ± 0.03 mg/dm3 and Manganese bound metals Fe 14.50 ± 0.45 mg/dm3, Zn 4.03 ± 0.78 mg/dm3 and Pb 1.22 ± 0.06 mg/dm3. Iron-manganese bound metals Fe 120.40 ± 19.15 mg/dm3, Zn 6.79 ± 1.12 mg/dm3 and Pb 2.16 ± 0.05 mg/dm3. Organic/sulphide bound metals Fe 5.90 ± 0.50 mg/dm3, Zn 4.14 ± 0.68 mg/dm3 and Pb 3.58 ± 0.07 mg/dm3. Residual bound metals Fe 13.10 ± 0.55 mg/dm3, Zn 6.12 ± 0.17 mg/dm3 and Pb 4.48 ± 0.09 mg/dm3.The results of the control sample (without Typha domingensis) shows that the exchangeable bound metals Fe 9.40 ± 1.89 mg/dm3, Zn 1.71 ± 0.45 mg/dm3 and Pb 0.28 ± 0.05 mg/dm3, Carbonate bound metals Fe 0.70 ± 0.12 mg/dm3, Zn 2.20 ± 0.71 mg/dm3 and Pb 0.46 ± 0.12 mg/dm3, Manganese bound metals Fe 2.50 ± 0.21 mg/dm3, Zn 1.90 ± 0.09 mg/dm3 and Pb 1.68 ± 0.04 mg/dm3, Iron-manganese bound metals Fe 221.30 ± 21.12 mg/dm3, Zn 7.46 ± 0.48 mg/dm3 and Pb 2.75 ± 0.06 mg/dm3, Organic/sulphide Fe 4.20 ± 0.62 mg/dm3, Zn 3.70 ± 0.80 mg/dm3 and Pb 3.93 ± 0.04 mg/dm3 and Residual bound metals Fe 30.10 ± 5.80 mg/dm3, Zn 7.38 ± 0.61 mg/dm3 and Pb 0.53 ± 0.03 mg/dm3. Statistical analysis using pooled standard deviation methods of computing t indicates significant difference in all the fractions at (p=0.05). This research work shows that the plant (Typha domingensis) have negative impacts on the soil samples.

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Introduction

Soil is a heterogeneous mixture of organic and inorganic substances. The binding mechanisms for metals vary with the composition of the soil. The ecological effects of heavy metals in soil are closely related to the distribution of species in the solid and liquid phases of the soil. The presence of heavy metals in soil is of great ecological significance owing to their toxicity at certain concentrations through food chains and non-biodegradability which is responsible for their accumulation in the biosphere. Heavy metals like iron, tin, copper, manganese and vanadium occur naturally in the soil and could serve as plant nutrients depending on their concentrations (Opaluwa 2012). The levels of heavy metals in the soil have been seriously increased during the last decades due to human activities. Metals like mercury, lead, cadmium, silver, chromium and many others that are indirectly distributed as a result of human activities could be very toxic even at low concentrations. Soil management is the basis of sustainable agricultural production. In the tropical soils, most soil nutrients required by plants especially nitrogen, potassium, phosphorous, calcium and magnesium are often found deficient at the root zones (Kolo *et al.*, 2009).

Since the toxicity of heavy metals is increasingly attracting more attentions. Soil organic matter and Fe – Mn oxide have been found to be the most important soil properties and components influencing the biological optics of heavy metals. Depending on their origin, trace elements exist in different mineral forms and chemical compounds in different combinations with mineral and organic components of soil and sediments which may vary according to various conditions (Calmano *et al.*, 2001) *Typha domingensis* which is the species that is widespread in Bauchi is considered to be among the first wetland plants to colonize areas of newly exposed wet mud, with its abundant wind dispersed seeds. It survives in the soil for a long period with buried seeds (Albertoni *et al.*, 2005). It also germinates best in sunlight and fluctuating temperature which is typical of many wetlands that regenerates on mud lands. It spreads by rhizomes, forming large interconnected stands.

Sanders (1999) used the ion exchange equilibrium technique to determine complexes form of Co, Mn and Zn in aqueous extracts of five soils. (Olumu *et al.*, 1973) found that practically all the soluble Fe in some blooded soils was complexes with organic matter, whereas Mn was either not complexes or weakly complexes. Cottenie *et al.*, 1982, use the combination of cation and anion exchange resins to determine the species of selected metals in soluble fractions of a sandy soil. Copper and ion were largely present as stable complexes, manganese was largely presenting the free ionic form and zinc was evenly distributed between these two forms. In other word, a dilating resin in the calcium form, and containing a sufficient amount of the trace element to maintain a constant activity in solution, was used to determine free and complex form of Zn, Ca and Cd sludge-amended soil.

Due to human and industrial activities, the presence and concentration of heavy metals tend to increase in areas where they were not present some years ago. Today, gross symptoms of some heavy metal poisoning are seldom met with except in those exposed to extreme occupational hazards. This has given reason to seek out ways to remedy the presence of these metals in the soil. This research work is aimed at assessing some heavy metals redistribution among the exchangeable fractions caused by changes in the soil properties associated with *Typha domingensis* invaded soil along Gombe road, Bauchi. The objectives are to determine Iron, Zinc and Lead in the following fractions; Exchangeable bound metals (F1), Carbonate bound metals (F2), Manganese oxide bound metals (F3), Iron-manganese oxides bound metals (F4), Organic/sulphide (F5) and Residual bound metals (F6) respectively.

MATERIALS AND METHODS

Samples Collection

Soil samples were collected in polythene bags using auger from *Typha domingensis* invaded soil along Gombe road, Bauchi. Samples were taken randomly round the invaded soil from depths of 0-30cm were taken at each sampling point. And the control soil sample (without *Typha domingensis*) was also randomly taken few kilometres away from the *Typha domingensis* invaded soil. The samples both from the soil and control were brought to the laboratory.

Metal Analysis

The concentration of Iron, Zinc and Lead were determined from the soil extracts using a BUCK SCIENTIFIC Atomic Absorption Spectrophotometer Model 210 VGP. The spectrophotometer was calibrated by aspirating standard salt solutions of Iron, Zinc and Lead separately at their various wavelengths and Acetylene-air mixtures in the ratio of 6:8. Measurements were made after calibration, instrument settings optimized and blue flame obtained. Concentrations of analytes were obtained automatically from the read out on extrapolation of the absorbance of the analytes on the calibration curve by the machine, while maintaining the same condition for both soil samples and the control samples

Sample Pre-treatment

Soil samples(both soil and control samples) were air-dried in the laboratory before being crashed in a ceramic pestle and mortar then sieved in a 2mm screen plastic sieve. The soil samples were stored in plastic bottles and labelled appropriately. The metals were fractionated using the method of Tessier *et al.,* 1999 and Elsokkary *et al.,* 1980. The metal species were classified into six fractions (both soil and control) as indicated in the table below:

Fraction (Soil and Control)	**Symbols**
Exchangeable metals	F1
Carbonate bound metals	F2
Manganese oxide bound metals	F3
Iron-Manganese oxides bound metals	F4
Organic/sulphide bound metals	F5
Residual bound metals	F6

Determination of Exchangeable metals

A 1 g of the Soil sample weighed into a 250cm^3 conical flask and 10cm^3 of 1M Sodium acetate adjusted to pH 8.7 with acetic acid was added. The mixture was shaken for 2 hours using Edmund Bahler Swip Mechanical shaker before being filtered into a 100cm^3 volumetric flasks using a Whattman Filter No. 1. The filtrate was made up to mark with water from where the metals were determined using AAS. The residue was reserved for further fractionation.

Determination of Carbonate bound metals

The residue from the exchangeable metals was leached for 3 hours with each of the sodium acetate adjusted to pH 5.0 with acetic acid. The leachate was transferred to a 100cm^3 volumetric flask and made up to mark with water. The leachate was then analysed for metals using AAS and the residue reserved for further analysis.

Determination of Manganese oxide bound metals

The residue from carbonate bound soil was leached with $10cm^3$ 0f 0.10M hydroxylamine hydrochloride and 0.01M nitric acid (adjusted to pH 2.0 with acetic acid) after shaking for 3 hours using an Edmund Balder Swip mechanical shaker. The leachate was transferred quantitatively into a $100cm^3$ volumetric flask and made up to the mark with water. The leachate was then analysed for metals using AAS and the residue reserved for further analysis.

Determination of Iron-Manganese oxide bound metals

The residue from the manganese oxide bound soil was extracted with $10cm^3$ oxalate buffer of pH 3.0 after shaking for 12 hours at 90℃ in a water bath. The extract was filtered into a $100cm^3$ volumetric flask and water added to mark. The extract was then analysed for metals using AAS and the residue reserved for further analysis.

Determination of organic matter-sulphide bound metals

The residue from the iron-manganese oxide bound soil was extracted by shaking with $100cm^3$of 30% hydrogen peroxide that has been adjusted to pH 2.0 with drops of nitric acid for 6 hours at 90℃ in a water bath. It was then re-extracted at room temperature with $10cm^3$of 1M ammonium acetate that has been adjusted to pH of 2.0. After shaking for 3 hours, into the first extract in a $100cm^3$ volumetric flask and made to mark with water. The extract was then analysed for metals using AAS and the residue reserved for further analysis.

Determination of Residual metals

The residue from the organic and sulphide bound soil was digested with $10cm^3$ aqua regia by heating in a digestion tube at a digester temperature of 250℃. The clear digest was removed and allowed to cool before transferring quantitatively into a $100cm^3$ volumetric flask. It was then made up to the mark with water and the solution analysed for the metals of interest.

RESULTS AND DISCUSSIONS

Results

The results from the sequential extraction carried out on the *Typha domengensis* invaded soil were obtained into the following fractions (exchangeable, carbonate bound, manganese oxide bound, iron-manganese bound, organic matter sulphide, residual fractions) and presented in Table 1 while the result of the control soil are equally presented in Table 2.

Table 1: Soil Samples

Fractions	Metal Concentrations		
	Fe	Zn	Pb
Exchangeable bound metals	1.47 ± 0.21	1.45 ± 0.02	0.16 ± 0.04
Carbonate bound metals	26.10 ± 1.01	2.66 ± 0.17	0.89 ± 0.03

Manganese bound metals	14.50 ± 0.45	4.03 ± 0.78	1.22 ± 0.06
Iron-manganese bound metals	120.40 ± 19.15	6.79 ± 1.12	2.16 ± 0.05
Organic/sulphide	5.90 ± 0.50	4.14 ± 0.68	3.58 ± 0.07
Residual	13.10 ± 0.55	6.12 ± 0.17	4.48 ± 0.09

Values are mean ± standard deviation (n=4)

Table 2: Speciation pattern of the levels of some heavy metals (mg/dm^3) in non *Typha domingensis* invaded soil.

Fractions	Metal Concentrations		
	Fe	Zn	Pb
Exchangeable bound metals	9.40 ± 1.89	1.71 ± 0.45	0.28 ± 0.05
Carbonate bound metals	0.70 ± 0.12	2.20 ± 0.71	0.46 ± 0.12
Manganese bound metals	2.50 ± 0.21	1.90 ± 0.09	1.68 ± 0.04
Iron-manganese bound metals	221.30 ± 21.12	7.46 ± 0.48	2.75 ± 0.06
Organic/sulphide	4.20 ± 0.62	3.70 ± 0.80	3.93 ± 0.04
Residual	30.1 ± 5.80	7.38 ± 0.61	0.53 ± 0.03

Values are mean ± standard deviation (n=4).

Discussions

The concentration of metals on exchangeable bond metals fractions of Fe 1.47 ± 0.20, Zn 1.45 ± 0.02 mg/dm^3 and Pb 0.16 ± 0.0.04mg/dm^3 were lower than the concentration of the exchangeable bond metals when compared with the control samples. Though, the values of iron, zinc and lead were relatively higher than the reported literature values of Fe 0.50 ± 0. 12 mg/dm^3 Zn 0.87 ± 0.05 mg/dm^3 and the Pb 0.13 ± 0.004 mg/dm^3. Carbonate bond metals on soil sample with values; Fe 26.10 ± 1.01 mg/dm^3, Zn 2.66 ± 0.17 gm/dm^3 and Pb 0.89 ± 0.03gm/dm^3 was found to be in high concentration compared to the control sample (Fe 0.70 ± 0.12mg/dm^3, Zn 2.20 ± 0.71 gm/dm^3 and Pb 0.46 ± 0.12 gm/dm^3) which shows high effects on the soil when compared with the reported literature values of Fe 19.76 ± 1.04 mg/dm^3 Zn 1.87 ± 0.12 mg/dm^3 and Pb0.89 ± 0.08 mg/dm^3 (Uwumarongie 2008).In Manganese bond metals, the values of Zn and Fe (4.03 ± 0.78 mg/dm^3 and 14.50 ± 0.45 mg/dm^3) were highly distributed in the soil sample with the exception of Pb been in low concentration in the control sample. The highest metal concentration was implicated in the Iron-manganese fraction which is found to be Fe (120.40 ± 19.15 mg/dm^3) in the soil sample and that of the control sample (221.30 ± 21.12 mg/dm^3) which shows higher impacts on the soil when compared with the literature values of Fe 14. 56 ± 2.21 mg/dm^3 (Tessier *et al.,* 1999). Organic/sulphide metals; the values of Fe 5.90 ± 0.50 mg/dm^3, Zn 4.14 ± 0.68 gm/dm^3 and Pb 3.58 ± 0.07 gm/dm^3 from the soil sample were found to be in high concentration than that of the control sample (Fe 4.20 ± 0.62 mg/dm^3, Zn 3.70 ± 0.80 gm/dm^3 and Pb 3.93 ± 0.04 gm/dm^3) but however, found in lower concentration with the reported literature values of (Omuku *et al*., 2009) Fe 3. 02 ± 0.32 gm/dm^3 Zn 1.32 ± 0.12 gm/dm^3 and Pb 2.01 ± 0.03 gm/dm^3.

Conclusion

The investigation carried out along Bauchi-Gombe road shows that the Carbonate bound metals, Manganese bound metals and the Organic/sulphide bound metals values on *Typha domingensis* along Gombe road are relatively high which implies high fertility of the soil while the Exchangeable bound metals and the Residual bound metals appear to be low. When subjected to hypothesis test, the result revealed significant variations amongst the Fractions of soil in the *Typha domengensis* invaded soil and that of the control sample area.

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