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Heavy Metal Speciation Study of Water and Bottom Sediments from River Wujam in Chip District of Pankshin Local Government Area, Plateau State, Nigeria

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ABSTRACT

Heavy metal species of water and bottom sediment samples from River Wujam in Chip district of Pankshin L.G.A. were investigated in the dry and wet seasons. The analysis was done using standard procedures. The results for the total metal concentration of the water samples ranges, thus, 0.02 -0.03 mg/L for copper, 0.01 – 0.11 mg/L for lead, 0.02 - 0.07 mg/L for manganese, 0.01 – 0.06 mg/L for cadmium. But nickel and zinc were not detected within the limits used. The total metal concentration of metals analysed (Cu, Pb, Mn, Cd, Zn, and Ni) in the sediments revealed a generally higher concentration in the dry season except for cadmium which revealed a higher concentration in the wet season. Speciation of the metals was also carried out on the sediments using Tessier method to determine their distribution in the sediment of the river. The percentage ranges of the fractions for the dry season are, 12.24 – 46.09%, 15.80 – 24.01%, 11.47 – 21.91%, 10.83 – 22.80%, and 7.61 – 75.00% for exchangeable, carbonate, Fe-Mn oxide, organic, and residual fractions respectively. Ni was not detected within the limits used in the carbonate, Fe-Mn oxide and organic fractions. The range of fractions for the wet season are, 4.25 – 33.77%, 10.82 – 24.21%, 15.91-38.35%, 10.65 – 51.06%, and 19.01 – 26.77% for exchangeable, carbonate, Fe-Mn oxide, organic, and residual fractions respectively. Ni was not detected within the limits used in the carbonate and organic fractions. With change in the environmental conditions Cadmium and lead bound to such labile phases in both the dry and wet season, can easily remobilize in the water system. A moderate positive correlation existed in the total metal concentration of water samples between dry and wet seasons. A strong positive correlation existed in the total metal concentration in the sediments samples between dry and wet seasons. T-test results indicated that there is no significant variation in the concentrations between the seasons in both cases.

1. Introduction

Heavy metals among other indicators of water and sediment pollution has remained a major source of concern to researchers [1-5]. This is because they could cause a negative effect to human health and the aquatic system. Heavy metals are persistent in nature, toxic, and have a very high tendency to accumulate in living organisms and get adsorbed on sediment particles [6, 7]. A good number of researchers [2,4, 8-10] have reported that sediments/soils are not only basic components of our environment as they provide nutrients for living organisms, but also serve as an important sinks for pollutants that are deleterious to health, particularly heavy metals. They also play a significant role in re-mobilization of contaminants in aquatic systems when certain conditions prevail eg., pH, redox potential, desorption or (bio) degradation of the sorptive substances [11]. Therefore it is very appropriate to study it, to know its level of contamination with metals and to determine its interaction with the water body. The determination of only total element concentration in sediments does not give an accurate estimate of the likely environmental impacts regarding their bioavailability, mobility, toxicity and reactivity [1,11,9]. Hence, speciation analysis (sequential extraction), the partitioning of metal contaminants between specific forms, to investigate their geochemical composition and distribution in the sediments is used [12-14] whereby, components loosely held in the soil are extracted first, followed by those that are more tightly bonded, the sequential solubilisation of the various substrates that make up the sediments [11,15]. It has the following advantages over the determination of total metal concentration as it tells the source of metal, weather natural (lithogenic) or anthropogenic [10], its toxicity to living organism (biota) and a higher knowledge of metal-sediment interaction [16]. The sequential extraction was based on the five-stage sequential extraction procedure [17] which has been used to divide heavy metals into different binding forms: exchangeable, carbonate-bound, Fe-Mn Oxide-bound, Organic matter/Sulfide bound and Residual [1]. The aim of the present research is to study the status of heavy metal speciation of river Wujam. The results obtained from this study will provide information on the level of concentration of heavy metals in water and sediment, thereby contributing to the effective monitoring of environmental quality and health of the ecosystem.

2. Experimental Methods

2.1 Study Area

Mhiship community or Chip community is comprised of people whose tribe is called Mhiship or Chip. It is located around the hills in southern part of Pankshin local government area of Plateau state. It is located between kilometers 50 and 58 along Panyam – Shendam road. They are surrounded by hills and mountains in the north, which has the local government council secretariat/headquarters and plain land that leads to Shendam and Quan-Pan local government areas in the south [11]. The main occupation of the inhabitants of the area is farming and tapping of palm wine. The major sources of water in Chip are the rivers with a very few boreholes. The source of water identified for this project work is River Wujam. The source of water is under consistent attack by human and animal activities such as washing, bathing, excreting, urinating, agricultural activities and cattle rearing.

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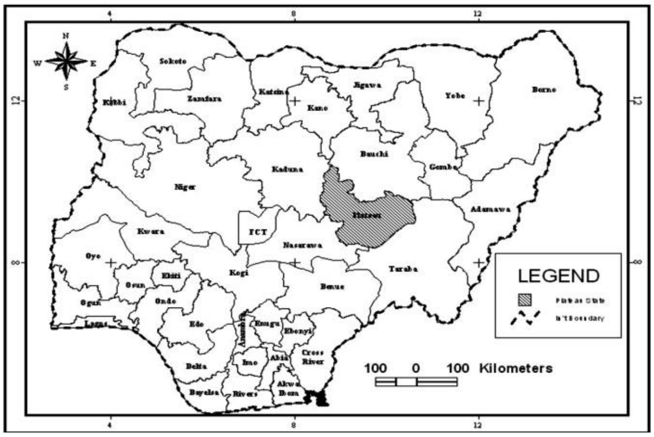

Fig. 1 Map of Nigeria showing Plateau State

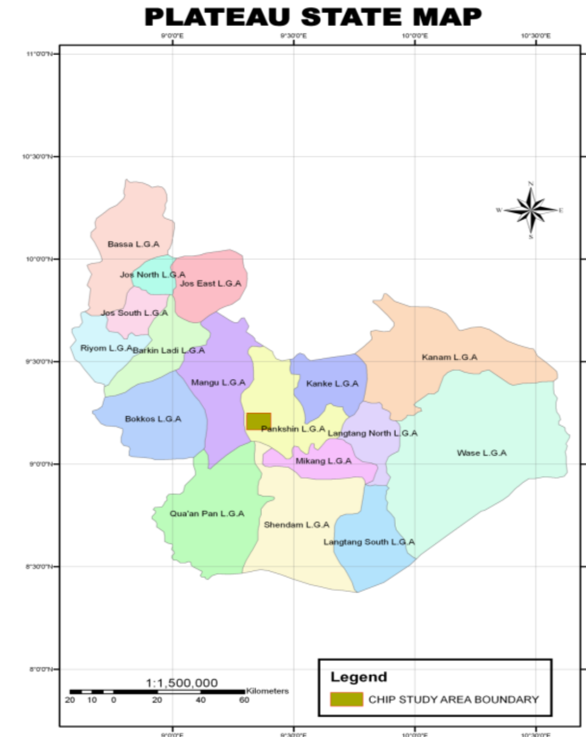

Fig. 2 Map of Plateau State showing Pankshin L.G.A.

2.2 Sample Collection

2.2.1 Water Sample Collection

The sample containers (2 L plastic containers with a screw cap) were washed with detergent, leached with concentrated HNO_3, rinsed with distilled water until acid free and finally with the water sources. And were labelled appropriately. Grap samples of water were collected from four different locations singly (at about 500 meters interval) along the rivers into the prepared containers manually. This was done in January, Febuary and March for the dry season and June, July and August for the wet season of the year. The samples were collected, a few drops of Hydrogen Trioxonitrate (v) acid was added at the point of collection and then preserved at 4 °C prior further analysis [5, 18].

2.2.2 Sediment Sample Collection and Treatment

Sediments samples were collected alongside the water samples using a stainless steel scoop into a polyethylene bags previously soaked with dilute nitric acid for 24 hrs, rinsed with distilled water and dried. The grab samples of surface sediments were taken at a depth of 0-5 cm for the various months for the dry and wet seasons [19]. These samples were then transported to Postgraduate Chemistry Laboratory University of Jos, Nigeria, and air dried for about two weeks at room temperature and grinded with mortar and pestle. The ground soil samples were sieved using 0.2mm sieve size and kept in a poly ethylene bottle for further analysis.

Fig. 3 River Wujam in the dry season

Fig. 4 River Wujam in the wet season

2.3 Methodology

Water samples were analysed for metallic elements using atomic absorption spectrophotometric method after digestion [5]. The digestion of the sediment sample for the determination of total metal was done using aqua regia. The sequential method was used to determine the speciation of the metals studied in the sediments [17]. These five sequential procedure was used [20] which were,

1. A 1.00 g triplicate dried sieved soil sample from each sampling site was shaken with 20 cm^3 of 1 M $MgCl_2$ at pH 7 for 1 hour, filtered and analysed for exchangeable fraction.
2. The residue of the exchangeable fraction was shaken with 20 cm^3 CH_3COONa at room temperature for 5 h at pH 5, then filtered and analysed for the carbonate fraction.
3. The residue of the carbonate fraction was digested with a mixture of 10 cm^3 0.04 M $NH_2OH.HCl$ and 10 cm^3 of 25% CH_3COOH at 96 °C for 6 h, then filtered and analysed for the Fe-Mn oxide/reducible fraction.
4. The residue of the Fe-Mn oxide/reducible fraction was transferred into 250 cm^3 beaker and 9 cm^3 of 0.02 M HNO_3 and 15 cm^3 of 30% H_2O_2 were added, the mixture was heated for 5 h at 85 °C on a water bath. After 2 h of heating, another 15 cm^3 of 30% H_2O_2 was added. This was then filtered and analysed for the organic fraction.
5. The residue of the organic fraction was digested with aqua regia (7.5 cm^3 of 37% HCl and 2.5 cm^3 of HNO_3) at 85 °C for 1 h and then filtered and analysed for the residual fraction.

2.4 Statistical Analysis

The results obtained for heavy metals in the water and sediments in Tables 1 and 2 were subjected to statistical analysis such as correlation coefficient and t-test. The Spearman's rank correlation coefficient was employed to determine the correlation.

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3. Results and Discussion

Table 1 shows the results for the heavy metal analysis. The mean concentration reveals a lower values for copper and manganese in the wet and dry season based on the WHO standard. Lead was 0.01 mg/L in the dry and 0.11 mg/L in the wet seasons which is slightly higher than the WHO standard. Cadmium also shows a slightly higher values of 0.01 mg/L and 0.06 mg/L in the dry and wet seasons respectively. However the concentrations of zinc and nickel were below the detection limits of the instruments used. The correlation of 0.4286 indicates that there exist a moderate positive correlation in metal concentration of water samples between dry and wet seasons in River Wujam. The critical value of t at 5% significant level with 10 degrees of freedom is ±2.228. Hence, comparing the calculated value (-0.7086) and the critical value, the null hypothesis cannot be rejected. Thus, it can be concluded that there exist no significant variation in the total metal concentration of water samples between dry and wet seasons of River Wujam.

Table 1 The total metal concentration (mg/L) of the water samples of River Wujam

Parameters(Mg/L)	Dry season	Wet season	WHO	SON
Copper	0.02 ± 0.01	0.03 ± 0.01	1.3	1.3
Lead	0.01 ± 0.002	0.11 ± 0.01	0.01	0.01
Manganese	0.07 ± 0.03	0.02 ± 0.001	0.1	0.05
Zinc	BDL	BDL	0.1	5.0
Cadmium	0.01 ± 0.01	0.06 ± 0.03	0.01	0.003
Nickel	BDL	BDL	0.07	0.02

BDL = Below detection limits

Table 2 Total metal concentration (mg/Kg) of the sediments of River Wujam

Sesson	Cu	Pb	Mn	Zn	Cd	Ni
Dry	0.11± 0.02	0.23 ± 0.02	1.56 ± 0.21	3.36 ± 0.31	0.07 ± 0.01	0.12 ± 0.02
Wet	0.10± 0.01	0.19 ± 0.01	0.35 ± 0.01	3.09 ± 0.32	0.09 ± 0.02	0.06 ± 0.02

Table 3 Concentration (mg/Kg)of sequential fraction of metals in sediments of River Wujam at the Dry season (RW_D)

Fraction	Cu	Pb	Mn	Zn	Cd	Ni
Exchangeable	0.03 ± 0.01	0.21 ± 0.01	0.35 ± 0.02	4.06 ± 0.12	0.06 ± 0.02	0.02 ± 0.01
Carbonate	0.05 ± 0.02	0.21 ± 0.01	0.18 ± 0.04	3.93 ± 0.10	0.04 ± 0.01	BDL
Fe – Mn Oxide	0.04 ± 0.01	0.19 ± 0.02	0.09 ± 0.03	4.49 ± 0.49	0.05 ± 0.01	BDL
Organic fraction	0.05 ± 0.01	0.18 ± 0.01	0.08 ± 0.02	4.56 ± 0.13	0.05 ± 0.02	BDL
Residual	0.05 ± 0.02	0.23± 0.01	0.06 ± 0.01	4.77 ± 0.10	0.03 ± 0.01	0.06 ± 0.02

Table 4 Percent (%) of metal sequential fraction of metals in sediment of River Wujam at the dry season (RW_D)

Fraction	Cu	Pb	Mn	Zn	Cd	Ni
Exchangeable	12.24	20.43	46.09	18.63	25.44	25
Carbonate	22.35	20.56	24.01	18.02	15.80	BDL
Fe – Mn Oxide	19.13	18.50	11.47	20.59	21.91	BDL
Organic fraction	21.28	17.79	10.83	20.89	22.80	BDL
Residual	25.00	22.70	7.61	21.86	14.06	75

Table 5 Concentration (mg/Kg)of sequential fraction of metals in sediments of River Wujam at the wet season (RW_W)

Fraction	Cu	Pb	Mn	Zn	Cd	Ni
Exchangeable	0.04 ± 0.02	0.16± 0.08	0.06± 0.02	3.88 ± 0.45	0.10± 0.03	0.02 ± 0.01
Carbonate	0.06 ± 0.03	0.16 ± 0.05	0.28 ± 0.06	4.00 ± 0.89	0.03 ± 0.01	BDL
Fe – Mn Oxide	0.11 ± 0.04	0.17 ± 0.06	0.57 ± 0.05	4.26 ± 0.91	0.05 ± 0.02	0.01 ± 0.001
Organic fraction	0.03 ± 0.01	0.19 ± 0.05	0.29 ± 0.08	3.90 ± 0.84	0.04 ± 0.01	0.04 ± 0.01
Residual	0.06 ± 0.02	0.15 ± 0.01	0.29 ± 0.07	3.96 ± 0.63	0.08 ± 0.03	BDL

Table 6 Percent (%) of metal sequential fraction of metals in sediments of River Wujam at the wet season (RW_W)

Fraction	Cu	Pb	Mn	Zn	Cd	Ni
Exchangeable	13.68	24.66	4.25	19.42	33.77	32.63
Carbonate	20.14	24.21	18.82	20.00	10.82	BDL
Fe – Mn Oxide	36.52	26.91	38.35	21.28	15.91	16.31
Organic fraction	10.65	30.29	19.37	19.50	12.73	51.06
Residual	19.01	24.32	19.21	19.80	26.77	BDL

The results for the total metal concentration of sediments in Table 2 revealed a higher concentration of copper (0.11 mg/kg), lead (0.23 mg/kg), manganese (1.56 mg/kg), zinc (3.36 mg/kg) and nickel (0.12 mg/kg) in the dry season as compared to the wet season. However Cadmium showed a higher concentration in the wet season with 0.09 mg/kg. The results for the metal analysis in the sediments of the river in both seasons were within the guidelines for sediments quality. The correlation coefficient of 0.8143 indicates that there exists a strong positive correlation in total metal concentration of the sediments between dry and wet seasons of River Wujam. The critical value of *t* at 5% significant level with 10 degrees of freedom is 2.228. Hence, comparing the calculated value (0.2632) with the critical value, the null hypothesis cannot be rejected and it can be concluded that there exist no significant variation in the total metal concentration of the water samples between dry and wet seasons of River Wujam. Correlation and t-test analysis of total metal concentration of water and sediment was also carried out. For the dry season, the Correlation value of 0.2143 reveal a very weak positive correlation in total metal concentration between water and sediment. The critical value of *t* at 5% significant level with 10 degrees of freedom is 2.228. Hence, comparing the calculated value (-1.6454) with the critical value, the null hypothesis cannot be rejected and it is concluded that there exist no significant variation in the total metal concentration of water and sediments. For the wet season, the Correlation value of -0.0571 reveal a weak negative correlation in total metal concentration between water and sediment. The critical value of *t* at 5% significant level with 10 degrees of freedom is 2.228. Hence, comparing the calculated value (-1.2303) with the critical value, the null hypothesis cannot be rejected and it is concluded that there exist no significant variation in the water and sediments samples.

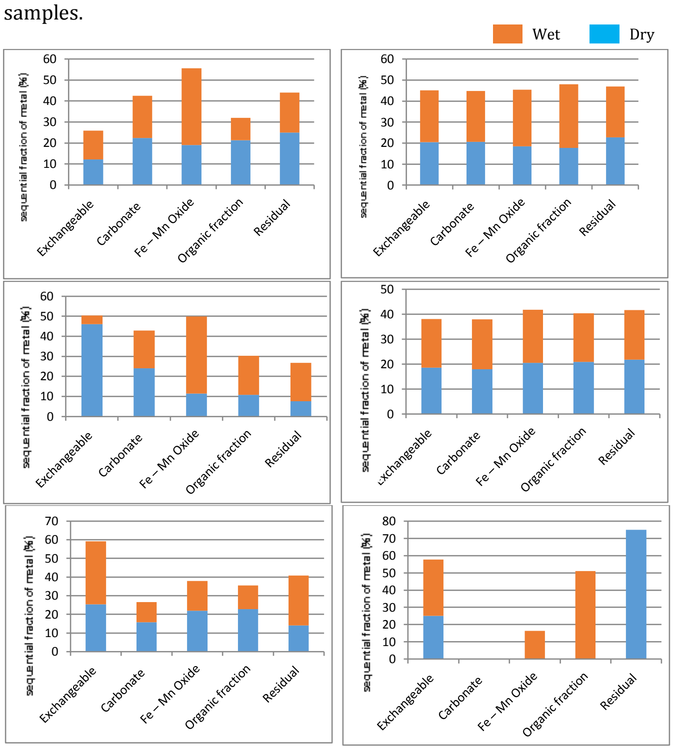

Fig. 5 The speciation pattern of Cu, Pb, Mn, Zn, Cd and Ni respectively in the dry and wet season of the study period presented as percentage fraction of metal species

Tables 3 and 5 show the concentration (mg/Kg) of sequential fraction of metals in the sediments. The results were converted to percent as shown in Tables 4 and 6 and were used to plot graphs for the patterns as shown in Fig. 5. The speciation pattern for copper in the sediments of River Wujam indicates the highest percentage of copper in the Fe-Mn oxide (0.11 ± 0.04 mg/Kg) followed by the residual (0.05 ± 0.02 mg/Kg) in the wet and dry seasons respectively. This indicates that copper is not bioavailable in the river being investigated but certain conditions can remobilize it in the wet season. The speciation pattern for lead in the sediments of River Wujam indicates that lead has the highest percentage in the residual fraction (0.23 ± 0.01 mg/Kg) and organic fraction (0.19 ± 0.05 mg/Kg) in the dry and wet season respectively. Although, the concentrations of the other fractions were considerably high. This indicates that lead may be bio-available to living organism in the study area. The speciation pattern for manganese in the sediments of River Wujam for the dry season indicates an occurrence of the highest concentration of manganese in the exchangeable fraction (0.35 ± 0.02 mg/Kg) and decreasing with the least in the residual fraction (0.06 ± 0.01 mg/Kg). In the wet season the highest percentage of manganese occurred in the Fe-Mn oxide fraction (0.57 ± 0.05 mg/Kg). The speciation pattern for zinc in the sediments of River Wujam indicates that zinc has the highest percentage in the dry and wet season in the residual fraction (4.77 ± 0.10 mg/Kg) and Fe-Mn oxide fraction (4.26 ± 0.91 mg/Kg) respectively. This indicates that zinc will not be released easily into the water layers when environmental conditions changes. This may suggest why zinc was below the detection limits in the water samples analysed from the river in both season. The speciation

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pattern for cadmium in the sediments of the river shows an occurrence of the highest percentage of cadmium in dry and wet seasons in the exchangeable fractions with (0.06 ± 0.02 mg/Kg) and (0.10 ± 0.03 mg/Kg) respectively. This study reveals that the percentage of cadmium in the exchangeable fractions in River Wujam in the dry and wet seasons are labile, highly toxic and the most bioavailable. The speciation pattern of nickel in the sediments of River Wujam reveal that nickel was only detected in the exchangeable (0.02 ± 0.01 mg/Kg) and residual fractions (0.06 ± 0.02 mg/Kg) in the dry season. In the wet season Ni was below the detection limits in the carbonate and residual fractions and the highest percentage of it was in the organic fraction (0.04 ± 0.01 g/Kg). This indicates that Ni is not bioavailable or cannot be mobilized. This may suggest the results obtained for Ni in the water samples from the same river, where it was below the detection limit.

4. Conclusion

There is quite a variation in the heavy metal content of river Wujam in the wet and dry season. The water samples obtained in the wet season have a higher concentration of the metals than the samples obtained in the dry season except manganese that reveals the contrary. Heavy metal content of the sediments samples in the dry season is higher than the sediments samples obtained in the wet season except for cadmium that reveals the contrary. The speciation analysis reveals that Pb, Mn, Cd exist generally more in fractions that can easily be mobilized and are bioavailable in the water system.

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