Application of Multivariate Statistical Methods for Assessment of Sediment Quality in Selected Locations of the Flood Plain of River Kaduna in Niger State, Nigeria

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ABSTRACT

Multivariate statistical methods were applied to assess the pollution status of sediments in flood plain of River Kaduna in Niger State. Heavy metals (Cu, Cd, Cr, Zn and Pb) were analyzed using ICP/OES. Concentrations of Polychlorinated Biphenyls (PCBs) in sediments were determined by GC – MS while physicochemical parameters were determined by standard methods. Multivariate statistical techniques (Cluster Analysis (CA), Principal Component Analysis (PCA), Discriminant Analysis (DA), and its Canonical plots) were used to assess temporal and spatial variations in the quality of sediment at the sampling locations.

The levels of heavy metals (Cu, Cr, Zn and Pb) in sediment shows slight increase after flooding, but were within the acceptable limits set by WHO, while PCBs and Cd were below detection limits. All the heavy metal pairs show strong positive correlations before and after flooding, while PCA generated only one component which explained 82.05% and 76.04% of the total variance before and after flooding respectively. Two distinct clusters were identified before flooding, (1) Zn – Pb – Cr (2) Cu. and after flooding; (1) Cu – Pb (2) Cr – Zn. DA result suggested that Cr, Zn and Pb were needed to account for most of the expected spatial variations in the sediment before flooding while Cu, Cr and Zn responsible for the expected spatial/temporal variations in the sediment quality after flooding. The multivariate analysis shows slight difference between the two sampling seasons, which reflects the dynamic nature of the sediment parameters of the study area.

(Keywords: sediments, heavy metals, multivariate, Polychlorinated Biphenyls, cluster analysis).

INTRODUCTION

Most pollutants enter the environment as emissions to the atmosphere or discharges to water bodies or dumps on land. The emitted materials eventually return to the earth either as particulate fallout or with rain or snow. They may be absorbed by soil and eventually vegetation or may be washed into waterways and become incorporated into sediments or may be metabolized by plants and animal life in water and thus enter the food chain (Pedro *et al*., 2004).

Sediments have high potential for accumulation of contaminants such as persistent organic and inorganic chemicals which are particularly sensitive to anthropogenic impacts when introduced into aquatic environment. They also play an important role in releasing sorbed contaminants back to the overlying water and to the indigenous biota (Baudo and Muntau 1990). In highly polluted situations, contaminated sediments may be directly toxic to certain organisms (Swartz *et al*. 1985) and/or bioaccumulate to excessive levels within others (Mance *et al*. 1984).

Commonly offending contaminants in this regard are heavy metals and polychlorinated biphenyls (PCBs). The partitioning behavior of these contaminants is such that they tend to accumulate in sediments to levels that are several orders of magnitude higher than in the surrounding water. Moreover, their deposition rates are generally related to their rates of input into the immediate area (Förstner, 1990). The chemical analysis of sediments can, therefore, provide a useful and convenient measure of environmental quality and is frequently

incorporated into pollution monitoring surveys. However, it also plays an important part on impact assessment of environmental quality especially to assess the long term impacts of anthropogenic activity. The presence of these contaminants might be one of obstacles to achieving "good ecological status" for a water body.

Sediment acts as a sink for pollutants. Environmental contaminants such as hydrocarbons, heavy metals and pesticides have been known to have direct toxic effects when released into the aquatic environment (Forstner. *et al.,* 1998; Fleeger, *et al.,* 2003). There is a direct link between surface water and sediment contamination. Adekola and Eleta (2007) have carried out studies on the heavy metal pollution of Asa River in Ilorin, Kwara state of Nigeria. Higher enrichment factors (EFs) were calculated for zinc, chromium, manganese and iron in the sediment samples. High values of EFs indicates anthropogenic source of contamination. Elevated levels of Fe, Ni, and V have been observed in the sediments of Iko River, a gas flaring station in the Niger Delta areas of Nigeria (Benson and Etesin, 2008).

Contamination of sediments and fauna in aquatic ecosystems of Niger Delta by a wide range of toxic and bio-accumulative metals and hydrocarbons have been reported severally (Ebong, *et al.* (2006); Udosen and Benson (2006); Benson, *et al.* (2007)). In another study on the Lagos Lagoon carried out by Adeboyejo, *et al.* (2011), the levels of OCPs in sediment were investigated from three stations along the Lagoon. The findings showed that the organic chemicals studied tend to bind with river's sediment particles with resultant effects on water pollution.

The study area is a low-lying, severely flood prone area along River Kaduna. During flooding, water bodies are contaminated, as thus sediment which severely affects the life of the aquatic animals due to inflow of nutrients, toxic substances, organic foods, plankton, sediments etc. Clean drinking water becomes scarce and unhygienic conditions prevailed which may lead to water – borne diseases. Accumulated heavy metals or organic pollutants in sediments could be released back into the water with deleterious effects on human health (Adekola and Eleta, 2007). The aim of this study is to assess sediment quality of the sampling locations during the sampling periods using multivariate statistical analysis.

MATERIALS AND METHODS

Sediment Sampling and Pre-treatment

Composite samples were collected in triplicate at the banks of the selected sample sites thereby bringing a total of 27 samples and were immediately put in sterilized polythene bags. The samples were transported to the laboratory, air – dried, lump samples were gently crushed and sieved to a particle size of 2.00 mm and transferred into amber glass bottles sealed and labeled before storing in a refrigerator (SAEFL, 2003). This is to ensure reproducibility of result and precision.

Sediment Digestion and Heavy metal Analysis

The digestion of sediment sample was done by dissolving 1.00 g of the dried powdered sediment sample in a clean 100 cm^3 beaker followed by the addition of 20 cm^3 concentrated HCl, 5.00 cm^3 concentrated HNO_3 and 2.00 cm^3 concentrated HF. The mixture was then heated to boil for one hour, filtered hot and made up to the mark with distilled water in 100.00 cm^3 volumetric flask and finally the heavy metals were analyzed using ICP/OES (US EPA, 1999).

Sediment Extraction (PCBs)

Sediment sample of 10.00 g was transferred into a 100 cm^3 beaker and 0.02 µg (i.e. 10 uL of 2 mg/L) of each of the mixed internal standards (C13PCB 52 and C13PCB 153) were added, followed by the addition of 30 cm^3 of 50:50 v/v acetone: dichloromethane mixture and ultra-sonicated for about 3 minutes to allow the sediment particles to settle. The supernatant liquid was transferred into a 250 cm^3 round bottom flask and extracted with 30 cm^3 of the acetone: dichloromethane mixture. The solvent extraction and transfer process was repeated twice and all the extracts were combined together in the round-bottom flask.

A blank sample 73 comprising of similar matrix to the sediment under investigation was similarly treated with the same internal standard and procedure above. The extracts (i.e. blank sample and analyte sample) were dried by filtering through a filter containing 10 g of sodium sulphate. The filter paper containing the sodium sulphate was further washed with 30 cm^3 of

solvent mixture and the filtrate collected in the round-bottomed flask. The volume of dried extract was reduced to between 3 and 5 cm^3 using a rotary evaporator; and was further reduced to 1 – 2 cm^3 using a stream of air. Exactly 1 cm^3 of the reduced dried extract was quantitatively transferred to a glass vial after rinsing with 2 cm^3 of hexane while the washing was allowed to stand in the vial. The volume of the vial was finally reduced at ambient temperature to approximately 1.00 cm^3 (SAEFL, 2003 and Roose and Brinkman, 2005).

PCBs Analysis

Aliquot samples of 10 g were spiked with labeled PCBs as internal and recovery standards, extracted with 30 cm3 mixture of 50:50 v/v acetone: dichloromethane in a microwave. The extracts were then dried by filtering through a filter containing 10g of anhydrous sodium sulphate. The dried extract was cleaned up with florisil and analyzed by GC-MS in the ion recording mode, performed at a mass resolution of 5000. The GC-MS was operated to monitor a minimum of two mass fragmentation ions per level of chlorination based on US EPA method (1999).

Statistical Treatment of Data

High variability associated with environmental data makes Multivariate statistics the choice of competence in this study to evaluate the large number of data in order to decipher patterns within the dataset that otherwise was not observed. The multivariate techniques used include correlation analysis, hierarchical Cluster Analysis (CA), Principal Component Analysis (PCA), and Discriminant Analysis (DA) and were performed using the Statistical Package for the Social Sciences (SPSS) Version 18.0 Software. Basic statistics (Descriptive) were carried out in order to give initial information for all the parameters under investigation.

RESULTS AND DISCUSSON

Sediment Quality Parameters

Tables 1 and 2 show the descriptive statistics of the sediment quality parameters before and after flood. However, the PCBs, suite of ''ICES 7' congeners in the sediment samples were not detected (BDL) throughout the period of investigation.

Copper (Cu) was found to be in the ranges from 4.00-14.00 (10.70±2.45), and 7.00-16.00 (11.56±2.71) mg/kg for the months of March and September, respectively. The levels of Cu in the samples increased after flooding. The result of the study revealed that the levels were relatively low, compared to the standard (18.7 mg/kg). The values are in agreement with those reported at the confluence of river Niger and Benue by Isuwa, (2016).

Cd in the sediment samples was not detected (BDL) throughout the sampling periods.

The mean levels of Cr (mg/kg) in the sediment samples fall within the ranges of 11.00 mg/kg - 29.00 mg/kg (19.81±5.86) mg/kg, and 12.00-39.00 mg/kg (24±7.81) mg/kg, for the months of March and September respectively. These values indicate a slight increase from March to September. These value were all below the recommended limit of 37.3 mg/kg and the values were in agreement with those reported by Isuwa, (2016), and exceeds those reported by Adekola and Eleta (2007) in their works on the sediment from Asa river in Ilorin, Nigeria where Cr levels was found to range 3.0-11.3 mg/kg.

The levels of Zn in the sediment as shown in Table 1 and 2 had mean values of 26.26±9.60 mg/kg and 33.48±10.16 mg/kg before and after flooding, respectively. However, the values were below TEL of 124 mg/kg as recommended by ISQG.

From the results, the sediments from the river recorded Pb in the ranges of 2.00-18.00 mg/kg with the mean of 10.41±5.06 mg/kg before flooding and 5.00-19.00 mg/kg with the mean of 12.85±4.46 mg/kg after flooding. The mean values of Pb increased from March to September. However, the sediment samples were not polluted as the values obtained were far below the critical limit of 30.2 mg/kg.

The PCBs levels in the sediment samples were not detected (BDL) with respect to sampling periods.

Table 1: Descriptive Statistics of Heavy Metals in Sediment (before flood).

Parameter	WHO	Minimum	Maximum	Range	Mean	SD	CV%
Cu(mg/kg)	18.7	4	14	10	10.7	2.45	22.89
Cd(mg/kg)	0.676	BDL	BDL	BDL	BDL	BDL	0
Cr(mg/kg)	37.3	11	29	18	19.81	5.86	29.58
Zn(mg/kg)	124	10	40	30	26.26	9.6	36.56
Pb(mg/kg)	30.2	2	18	16	10.41	5.06	48.61
PCBs (ug/kg)	0.005	BDL	BDL	BDL	BDL	BDL	0

BDL = Below detection limit; Cd > 1 mg/kg, PCBs > 0.05 µg/kg

Table 2: Descriptive Statistics of Heavy Metals in Sediment (after flood).

Parameter	WHO	Minimum	Maximum	Range	Mean	SD	CV %
Cu (mg/kg)	18.7	7	16	9	11.56	2.71	25.44
Cd (mg/kg)	0.676	BDL	BDL	BDL	BDL	BDL	0
Cr (mg/kg)	37.3	12	39	27	24	7.81	32.54
Zn (mg/kg)	124	20	58	38	33.48	10.16	30.35
Pb (mg/kg)	30.2	5	19	14	12.85	4.64	36.11
PCBs (ug/kg)	0.005	BDL	BDL	BDL	BDL	BDL	0

BDL = Below detection limit; Cd > 1 mg/kg, PCBs > 0.05 µg/kg

Correlation Analysis

Pearson correlation coefficient (r) was used to show relationship between the heavy metals. The results presented in Table 3 shows strong positive correlations between all the heavy metal pairs in March (before flooding): Cr and Cu (r = 0.627), Zn and Cu (r = 0.806), Zn and Cr (r = 0.817), Pb and Cu (r = 0.663), Pb and Cr (r = 0.817) and Pb and Zn (r = 0.829).

The same scenario was repeated after flooding as all the heavy metals were strongly correlated positively (Table 4). Significant correlations between metals suggested high possibilities that they may come from the same sources. These sources might be from pollution imposed by human activities (anthropogenic source), or the nature (geochemical source), or both (Yisa, *et al*., 2011).

Anthropogenic source increases heavy metal load beyond permissible limit which has adverse effect on fish and other aquatic organisms, and eventually man through the food chain. The negative effect of heavy metals on aquatic organisms has been previously reported (Canli and Atli, 2003; Nussey, *et al*., 2000; Widianarko, *et al*. 2000).

Table 3: Correlation Coefficients between Heavy Metals in Sediments before Flooding.

	Cu	Cr	Zn	Pb
Cu	1			
Cr	0.627*	1		
Zn	0.806*	0.813*	1	
Pb	0.663*	0.817*	0.829*	1

*Correlation is significant at the 0.01 level (2-tailed)

Table 4: Correlation Coefficients between Heavy Metals in Sediments after Flooding.

	Cu	Cr	Zn	Pb
Cu	1			
Cr	0.535*	1		
Zn	0.539*	0.721*	1	
Pb	0.941*	0.675*	0.655*	1

*Correlation is significant at the 0.01 level (2-tailed)

Principal Component Analysis (PCA)

PCA generated only one component for all the sediment samples throughout the period of investigation with communalities indicating that variances of all the parameters were well described (either positive or negative) as indicated by the only component (Table 5).

Component loading of the PC before and after flood explained 82.05% and 76.04% of the total variance. However, it can be seen that the only component (PC1) in each case give information about the variation in Cu, Cr, Zn, and Pb which indicates that it stem from the same source through natural weathering erosion of the bed parent rock and granular minerals at the bottom and bank of the river (water sediment solution).

The decrease in the % variance after flooding could be as a result of leaching effect on the sediment properties. The Eigen variance before flooding was larger than variance extracted after flooding. These suggest that after flooding there was a decrease in correlation structure among the parameters which may be linked to some environmental effects. The highest communality explained in each case corresponds to the most strongly correlated parameter, and could be a strong sediment quality indicator in the study area.

Table 5: Communality and Cumulative Variance of PCA for Heavy Metals In Sediment.

BEFORE FLOOD			AFTER FLOOD		
Parameters	**PC1**	**Communality**	**Parameters**	**PC1**	**Communality**
Cu (mg/kg)	0.850	0.723	Cu (mg/kg)	0.874	0.763
Cd (mg/kg)	-	-	Cd (mg/kg)	-	-
Cr (mg/kg)	0.901	0.812	Cr (mg/kg)	0.834	0.696
Zn (mg/kg)	0.953	0.909	Zn (mg/kg)	0.829	0.687
Pb (mg/kg)	0.916	0.839	Pb (mg/kg)	0.945	0.893
PCBs (µg/kg)	-	-	PCBs (µg/kg)	-	-
PC1= 3.28; 82.05%			PC1= 3.04; 76.004%		

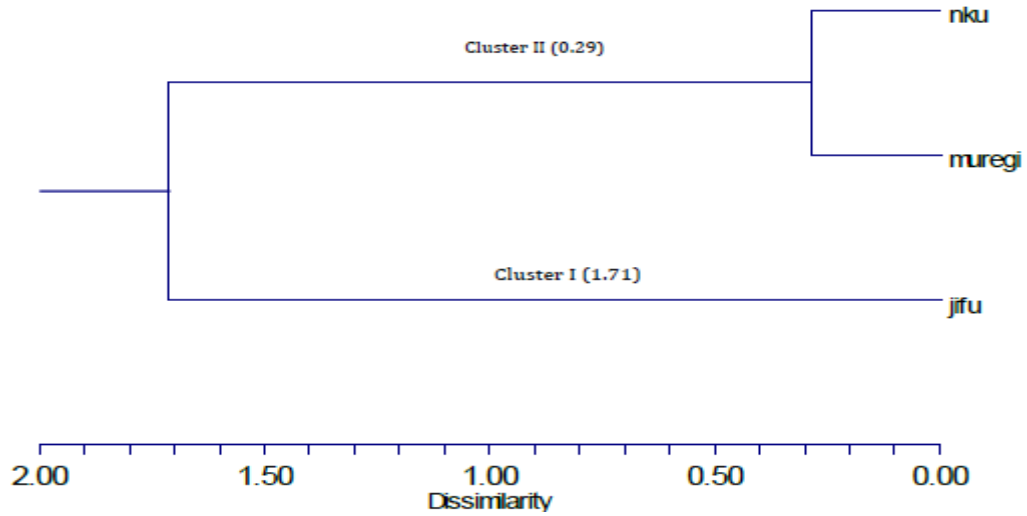

Figure 1: Dendrogram of Cluster Analysis of Sampling Locations.

Cluster Analysis (CA)

The dendrogram resulting from hierarchical agglomerative CA for the sediment sampling period (Figure 1) for clustering of cases (locations) displayed the presence of two major clusters; Cluster I includes Muregi and Nku at a distance of about 0.42; cluster II includes Jifu as the only singleton within the cluster and was linked to cluster I at about 1.69 presenting moderately large similarity distance with the other group.

In order to classify the heavy metal variables, cluster analysis was employed. Cluster analysis is an efficient tool for metal source identifications as it assigns a set of observations into subsets (called cluster) so that observations in the same cluster are similar in some sense (Yisa *et al*., 2011; Han *et al*., 2008) .

Figure 2 and 3 show the dendrogram of the hierarchical cluster analysis of metal contents using Average Linkage (Between Group) before flooding (March) and after flooding (September), respectively. Two distinct clusters were identified before flooding, namely: (1) Zn – Pb – Cr (2) Cu. Although all the metal pairs showed significant positive correlations, cluster analysis indicates that cluster 1 with rescaled distance of less than 5 are more related than cluster 2 which has only Cu. This result is similar to that obtained by Yisa *et al*, (2011).

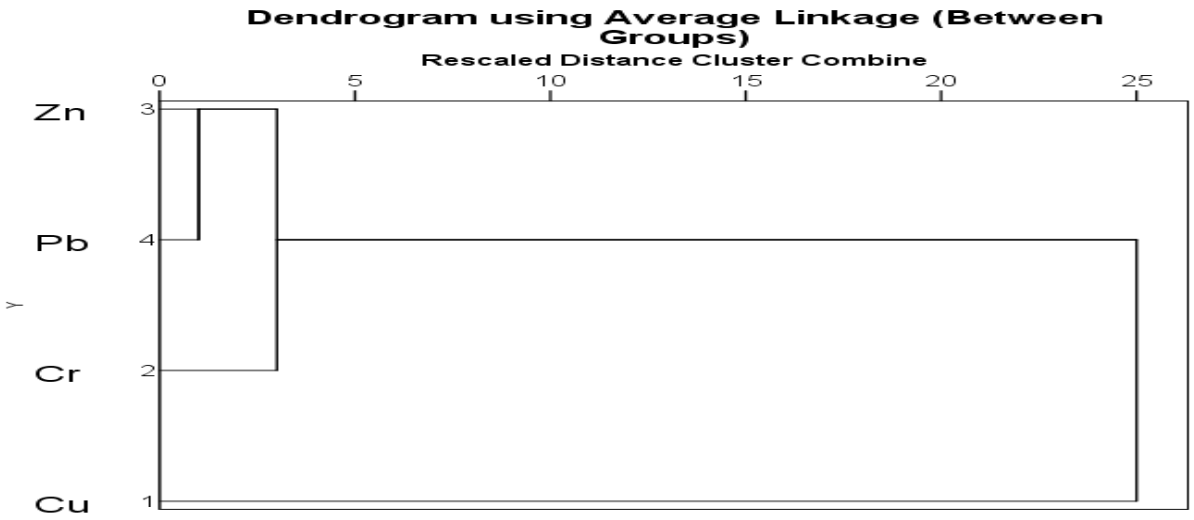

Figure 2: Dendrogram of the Hierarchical Cluster Analysis of Metal Contents (before flooding) using Average Linkage (between group).

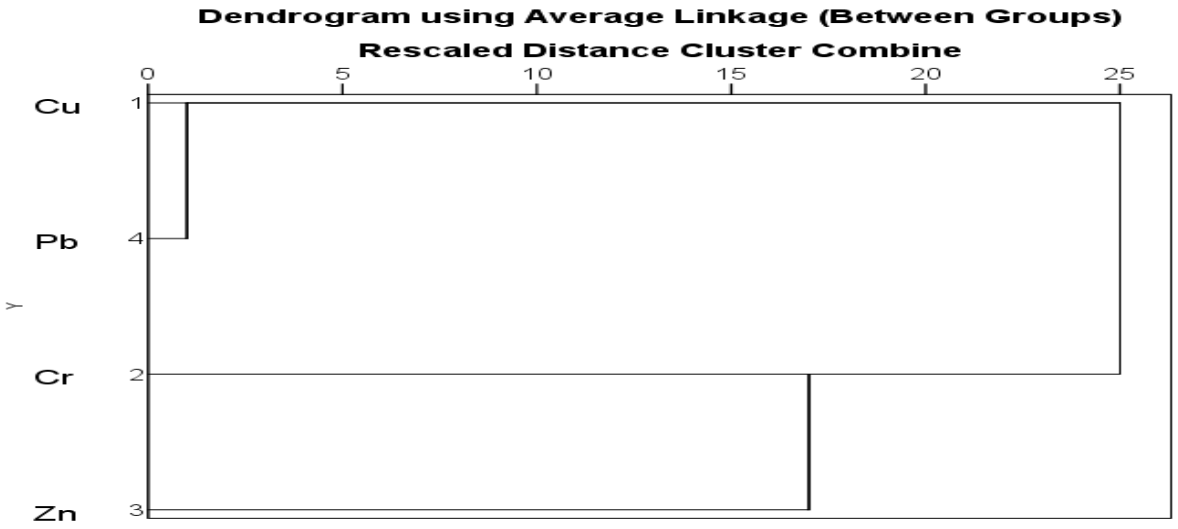

Figure 3: Dendrogram of the Hierarchical Cluster Analysis of Metal Contents (after flooding) using Average Linkage (between group).

Two distinct clusters were also identified after flooding; (1) Cu – Pb (2) Cr – Zn. It shows that the flooding has changed the distribution pattern of the metals making Pb to be more related to Cu than its previous cluster before flooding.

Discriminant Analysis (DA)

DA obtained from the standard, forward and backward selection modes of MD_5 are shown in Table 6. The standard, forward and backward modes were constructed using four parameters (Cu, Cr, Zn and Pb). The distribution showed that with the exception of Cu, all others were the discriminating parameters in the spatial/temporal variations.

Table 6: Discriminant Analysis (DA) of Heavy Metals in Sediment in March (before flooding).

	Standard	Forward	Backward
Constant	-78.30		
Cu	2.02		
Cr	2.99	46.75	37.23
Zn	0.69	25.89	25.89
Pb	3.95	89.50	59.08

Wilks' lambda and Chi-Square test of DA Sediment				
Modes	**Fun.(s)**	**R**	**Wilks' lambda**	***P* level**
Standard	1	0.92	0.02	0.001
Forward	1	0.95	0.0001	0.001
Backward	1	0.47	0.06	0.001

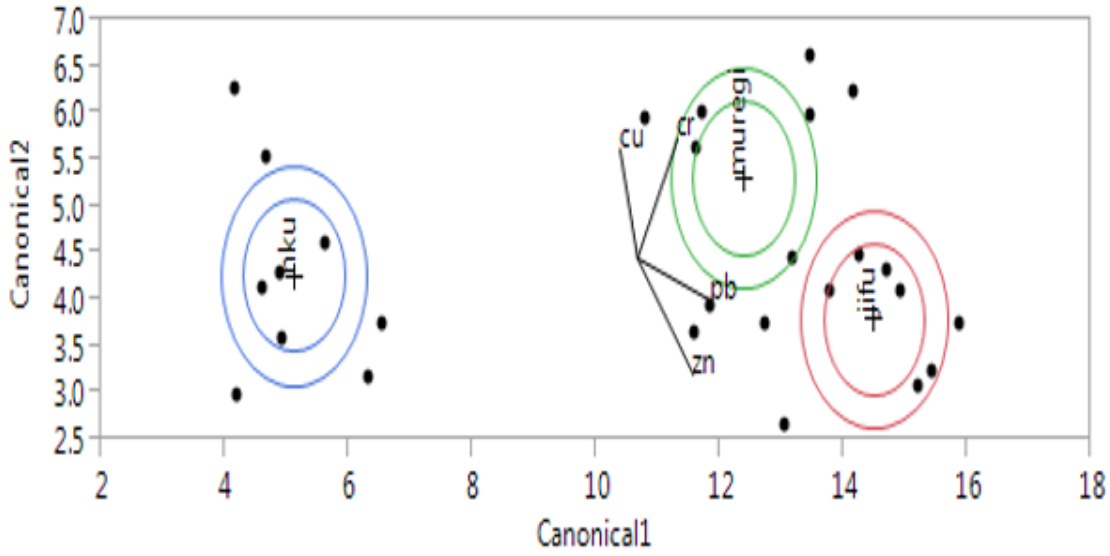

Figure 4: Canonical Plot of Sediment Sample in March.

Thus, the spatial DA result suggested that Cr, Zn, and Pb were needed to account for most of the expected spatial variations in the sediment with higher accuracy for classification using the standard (R=92%, lambda=0.02 and P= 0.001), forward (R= 95%, lambda=0.0001 and P=0.001) while backward selection mode had moderate accuracy for classification (R=47%; lambda=0.06 and P=0.001). The value observed in this study was within the range of 0.00-0.401 as reported by Ladipo *et al.,* (2012).

Figure 4 shows the canonical plot of sediment sample in March. It can be noted that Muregi had Cr and Pb as the major and significant biomarker for sediment quality while Jifu and Nku had a null effect for the major discriminators

Table 7 showed the DA of sediment sample in September and revealed that among the four parameters, Cu, Cr and Zn had the highest prediction accuracy for discriminating efficiently and are therefore, responsible for the expected spatial/temporal variations in the sediment quality of these locations.

Table 7: Discriminant Analysis (DA) of Heavy Metals in Sediment in March (after flood).

	Standard	Forward	Backward
Constant	-115.81		
Cu	13.83	86.36	80.08
Cr	2.15	66.72	47.71
Zn	1.07	43.03	43.04
Pb	-2.19		

Wilks' lambda and Chi-Square test of DA Sediment				
Modes	**Fun.(s)**	**R**	**Wilks' lambda**	***P* level**
Standard	1	80.08	80.08	80.08
Forward	1	47.71	47.71	47.71
Backward	1	43.04	43.04	43.04

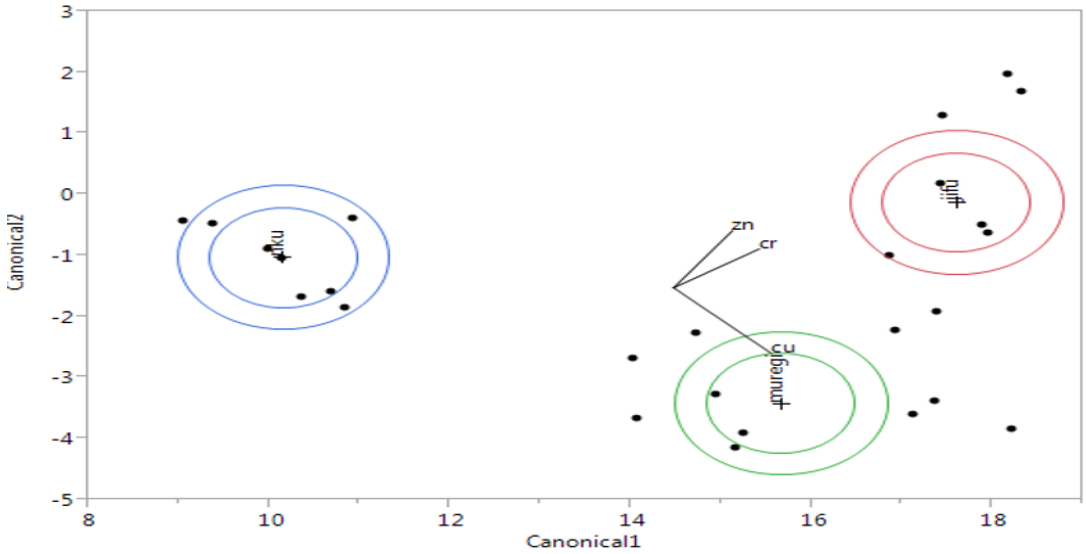

Figure 5: Canonical Plots for Sediment in September.

The correlation coefficients were 0.92, 0.91 and 0.68 in the standard, forward and backward selection modes which indicates reliability in the variable selection. The range of 0.024 – 0.315 in the Wilk's lambda values indicated that the variable selected in the forward and backward selection mode contributed significantly ($p<0.001$) in the discriminant function. This was slightly different from what was observed in MD_5, as Cu now become an important discriminator. It can be noted that Cr and Zn had high repeatability as it form part of the significant discriminators during the period.

Figure 5 shows the canonical plot of sediment sample in September. It can be observed that the sampling stations showed a non-intersecting relationship which implied significant difference among the sampling stations. The plots also indicated Cu, Cr and Zn as the major discriminators, however Cu had a significant effect in Muregi while others had propensity to be a major marker at Jifu.

CONCLUSION

Multivariate statistical methods were effectively used to study the pollution status of the flood plain of River Kaduna. While the PCBs were not detected, the concentration of heavy metals increased after flooding and appears to have a common source. The multivariate analysis shows slight difference between the two sampling seasons, which reflects the dynamic nature of the sediment parameters of the study area.

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