Chemometric Analysis of Physico-Chemical Parameters and Heavy Metals Accumulation in Bosso Dam, Minna

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Abstract
Water quality is important the protection of humans and marine ecosystems. Over the past two decades, considerable attention has been given to heavy metals in both terrestrial and aquatic ecosystems because they can be accumulated by biota and at high concentrations are potentially toxic. Traditional biological indices or direct measurements of water quality, which are based on in situ data collection are often spatially or temporally limited. On the other hand, the complexity of information requires new analysis techniques that allow for identification of the components and possible causes of spatial and temporal variability. Bosso dam is a major source of water for both domestic and agricultural purposes in Minna metropolis. In this study, the concentration of toxic heavy metals such as Pb, Ni, Mn, Zn, Cd, Cu and Cr were determined in water from Bosso dam during wet season. One hundred water samples were collected and homogenized into ten composite samples. Water and sediment samples were digested according to the method described by Türkmen and Ciminli (2007). Techniques of Chemometrics such as Principal Component Analysis (PCA), Hierarchical Component Analysis (HCA) and Correlation Analysis (CA) were used to analyze pollution data sets from heavy metal determination and assessment of the physicochemical parameters such as pH, temperature, turbidity, Si, conductivity, NO_3^- –N, NH_4^+–N, NO^{-2} –N of water. Pb was below detection limits while Cr was detected at a value of 0.02mg/l and 0.01mg/l for Cd, Zinc, Cu, Ni, Mn in the water samples. Application of PCA and CA helped to identify the underlying pollution sources and signature at the monitoring site.

Keywords: Water quality, Heavy metals, Chemometrics, Pollution, Bosso dam

1.0 Introduction

Heavy metals refer to the, dense and metallic elements that occur in trace levels, but are very toxic and tend to accumulate, hence are commonly referred to as trace metals (Adelekan *et al.,* 2016). They are metallic elements with high atomic weight and density greater than that of water. The major anthropogenic sources of heavy metals in the environment are industrial wastes from mining sites, electroplating manufacturing and finishing plants, domestic waste-water and run off from roads. Many of these trace metals are highly toxic to humans, such as Pb, Cd, and Cr. Exposure to Pb can cause kidney and brain damage. Cd has been reported to cause damage to the adrenals and homopoietic system. Cr has also been reported to cause lung cancer (USDFFS 2018). Their presence in surface and underground water at above background concentrations is undesirable (Adelekan *et al.,* 2016).

Bosso dam is situated within Minna metropolis and is a major source of water for domestic, agricultural and industrial uses in Minna metropolis. Residents along the bank of the river often cultivate crops such as sweet potatoes, maize, tomatoes and vegetables, and use water from the river to irrigate the crops. Water from this dam is also being used for domestic and recreational (swimming) purposes and fishing. These usage expose the downstream users to some health hazards, thus the need to determine the level of heavy metals and mineral elements for the safety of the users.

Chemometric statistical analysis provides useful tools for the study of spatial uncertainty and hazard assessment. In recent times, many researchers used sample site data to model the spatial distribution of heavy metals in sediment. Also, methods of multivariate data analysis are powerful tools for the evaluation and interpretation of river pollution data. Much information is lost using only univariate graphical or statistical methods for data evaluation and interpretation. In the last decade, methods such as principal component analysis (PCA) have become accepted in the identification of temporal and spatial variation and sources of pollution in river water (Biancolillo *et al*., 2018). However, applications of different chemometrics methods to the analysis of water bodies are not common in Nigeria. Thus, the monitoring of heavy metal levels in water bodies using chemometric techniques is of great importance for protection and ensuring future sustainability of aquatic environments. However, few environmental studies on spatial distribution, identification of pollution sources, including risk assessment of trace metals in Bosso have been conducted in Nigeria (Igiri, *et al*., 2018).
Deming *et al.,* (2019) determined the spatial distribution, potential risks and sources of seven heavy metals in Yangtze River Estuary. Analyses of 55 sediment samples revealed that the distributions of metals within the river were determined by the combined effects of their sources, hydrodynamic conditions, pH and Eh. According to the geoaccumulation index (Igeo) and sediment quality guidelines, Pb, Cd and Cr were present at low levels of pollution, with Cd posing the largest ecological risk. Positive Factor Matrix (PMF) results indicated that Hg, Zn, As, Pb and Cr mainly originated from natural geological background sources, while Cu originated from anthropogenic activities and atmospheric deposition was the source of Cd. These three sources contributed to 53.0%, 32.8% and 14.2%, respectively of total heavy metal concentrations.

Yunquian *et al.,* (2019) analyzed surface sediment samples collected from 18 sites in Dongping Lake for selected heavy metals including As, Cd, Cr, Cu, Hg, Pb, and Zn to determine their spatial distribution, source, and potential ecological risks. The enrichment degree of the studied metals decreased in the order of Cd N Hg N As N Pb N Cu N Cr N Zn, and the average concentrations of Cd, Hg and As were 3.70, 3.69 and 3.37 times their background values.

With the exception of Cd, the concentrations of heavy metals decreased progressively from the southeast to the north and west within the lake. Based on the enrichment factor (EF) and the potential ecological risk index (PERI), As, Cd and Hg were the heavy metal contaminants of most concern in surface sediments. Moreover, referencing to the results of multivariate statistical analyses, it was deduced that anthropogenic As and Hg were mainly from industrial and mining sources within the Dawen River watershed, whereas, Cd originated from agricultural sources.

Although heavy metal pollution in Bosso dam has been investigated, assessing the contamination and ecological risks of heavy metals in the water using multiple approaches that are based on multivariate analytical tools is meaningful. The assessment would help characterize the contamination sources in river and surface sediments and provide a tool for effectively protecting the river environment. Thus, this study focused on the analysis of the physico-chemical quality and accumulation of selected heavy metals in water from Bosso dam, and application of chemometric techniques to the pollution data. Methods of multivariate data analysis were applied for the evaluation and interpretation of data. This analysis could be helpful for Nigerian Government to optimize the marine water monitoring plan and enhance their pollution control actions

1.1 Study Location
Bosso Dam is located at latitude 9˚ 39N and longitude 6˚ 33E in Minna, Niger state. It is a small water body with a mean depth of 6.1M (20.2FT). The dam is shaded by shrubs, trees and bushes especially in the rainy season. The main use of the dam is for water supply (portable water) for domestic and irrigation purpose. The resources in the dam are conserved and protected from poachers by making it a restricted area. Most changes in the dam were brought about by flooding

of terrestrial ecosystem with organic and inorganic materials, defecation of animals and siltation. The dominant fish species in the dam is Tilapia zilli while crocodiles also inhabit the dam.

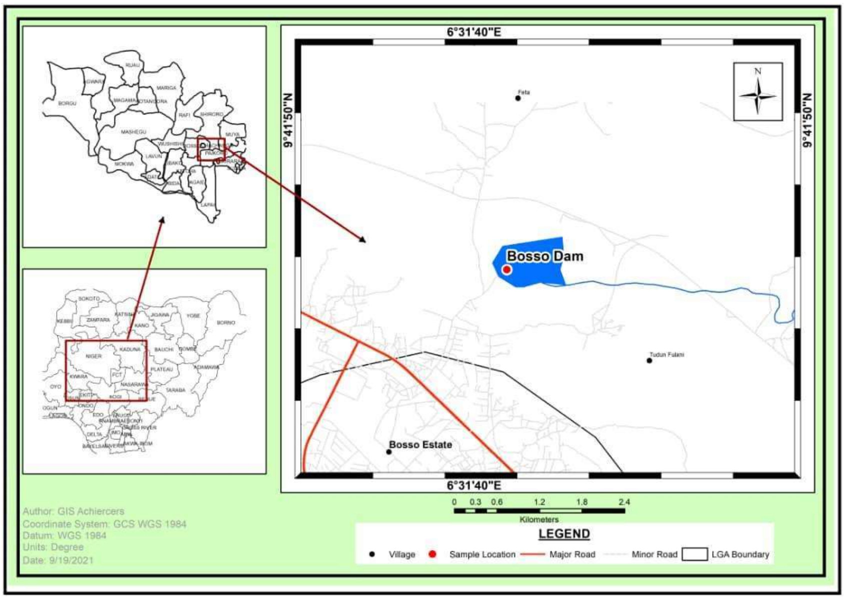

Figure 1: Map of Minna Metropolis showing location of Bosso dam in Minna

2.0 Methodology

One hundred surface water samples were collected at different points and homogenized into ten composite samples coded W1 to W10. Water samples were gently collected in clean plastic containers and were subjected to preliminarily acidification. This was done by adding 5cm^3 of nitric acid to each water sample. On-site analyses of pH, conductivity, and turbidity were carried out at the site of sample collection following the standard protocols and methods of American Public Health Organization (APHA) (APHA, 2005) and American Society for Testing and Materials (ASTM) using different calibrated standard instruments (J. DeZuane, 1997). The pH of the water samples was measured by using a pH meter. The pH meter was calibrated, with three standard solutions (pH 4.0, 7.0, and 10.0), before taking the measurements. The value of each sample was taken after submerging the pH probe in the water sample and held for a couple of minutes to achieve a stabilized reading. After the measurement of each sample, the probe will be rinsed with deionized water to avoid cross contamination among different samples.

The conductivity of the samples was measured using a conductivity meter. The probe was calibrated using a standard solution with a known conductivity. The probe was submerged in the water sample and the reading was recorded after the disappearance of stability indicator. The turbidity of the water samples was measured using a turbidity meter. Each sample was poured in the sample holder and kept inside for a few minutes before the result was recorded. Elemental analysis of water samples for Cr, Cu, Zn, Cd, Mn and Pb was carried out with Atomic Absorption Spectrometer (acetylene air), according to the ASTM standard method (ASTM, 2012) approved by APHA (2005) while the physicochemical characteristics of the water samples were carried out According to the standard methods of APHA (APHA, 2005) and Sawyer *et al.* (2005) by the filtration process. Silica was determined using the Standard Test Method for Silica in Water as described by ASTM D859-16. A test of the molybdenum-blue method was used for silica measurement.

For nitrite-nitrogen determination, the Nitrate/Nitrite-N in Water and Biosolids by Manual Colorimetry method as described by U.S. Environmental Protection Agency (2001) was used. 2cm^3 of color reagent (prepared by adding 100 cm^3 85% phosphoric acid (H_3PO_4) and 10 g sulfanilamide was added to about 800 mL reagent water and mixed to completely dissolve the sulfanilamide. 1 g

N-(1-naphthyl)-ethylenediamine dihydrochloride was added and mixed to dissolve. It was diluted to 1 L with reagent water and added to 50.0 mL of sample. It was allowed ten minutes for the color to develop, the absorbance of the sample was measure at 540 nm. Techniques of Chemometrics such as Principal Component Analysis (PCA), Hierarchical Component Analysis (HCA) and Correlation Analysis (CA) were used to analyze pollution data sets from heavy metal determination and assessment of the physicochemical parameters such as pH, temperature, turbidity, Si, conductivity, $NO_3^{-,}$ NO_2–N, NH_3, NO_2^- –N of water.

3.0 Results and Discussions

3.1 Physiocochemical parameters and heavy metals in water samples from Bosso dam

The statistical result with respect to mean and standard error of mean values for Bosso dam water quality parameters and heavy metal levels are summerized in Table 1. All water samples contained Ammonia at values significantly ($P \leq 0.05$). This could be due to the increased agricultural activities in the study location during rainy season. All the nitrate levels noticed in all samples were below the World Health Organisation (WHO 2018) recommended maximum safe level (10mg/l) for human consumption. Elevated levels of nitrate and nitrite in surface waters usually result from human activities such as overuse of chemical fertilizers and improper disposal of human and animal nitrogen-containing wastes, which are converted to nitrates in the soil. Nitrogen enters waterways either from the breakdown of dead organic matter or via atmospheric nitrogen gas fixation by specially adapted plants. Excess nitrogen in rivers enhances nutrient enrichment leading to algal blooms, fish kills and weed infestation (Yahaya *et al*. 2019). High levels of dissolved forms of nitrogen (nitrate, nitrite and ammonia) can also be toxic to many aquatic organisms and can prevent the water from being used as potable supply.

Heavy metals such as Pb and Cr were below detection limit in all the water samples. However, Cd was found in water samples W1 above WHO's permissible limit with 0.008mg/l. This could be due to the direct release of effluent from Minna Water Board treatment plant located next to Bosso dam. This corroborated the suggestion by Cotman *et al.* (2019) that surface waters are used for disposal of treated effluents from wastewater treatment plants. These effluents usually contain only small amounts of various contaminants that accumulate over time in the river, especially in sediments (Cotman *et al.,* 2019). Muhammad *et al*. (2018) reported similar result and attributed such occurrence to the fact that the discharge of heavy metals into rivers by domestic and industrial activities causes their rapid association and dissociation with particulates and incorporation into bottom sediment.

Table 1: Physiocochemical parameters and heavy metals in water samples from Bosso dam

Parameters	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	WHO
pH	6.91 ± 0.01^{b}	6.91 ± 0.01^{a}	6.92 ± 0.01^{a}	6.92 ± 0.01^{a}	6.86 ± 0.07^{a}	6.75 ± 0.01^{a}	6.72 ± 0.01^{a}	6.75 ± 0.01^{a}	6.75 ± 0.01^{a}	6.85 ± 0.01^{a}	6.50-8.50
Temperature	27.70 ± 0.06^{a}	27.70 ± 0.06^{a}	27.70 ± 0.06^{ab}	27.70 ± 0.06^{a}	27.70 ± 0.06^{b}	27.40 ± 0.10^{a}	27.40 ± 0.06^{a}	27.40 ± 0.06^{a}	27.40 ± 0.06^{a}	27.60 ± 0.06^{a}	12– 25
Silica	1.90 ± 0.06^{a}	6.60 ± 0.06^{c}	1.70 ± 0.06^{b}	1.70 ± 0.06^{a}	9.40 ± 0.06^{c}	8.60 ± 0.06^{b}	4.80 ± 0.06^{b}	4.70 ± 0.06^{a}	9.60 ± 0.06^{c}	9.10 ± 0.06^{c}	10.00
Turbidity	18.00 ± 0.06^{a}	18.00 ± 0.06^{a}	14.00 ± 0.06^{a}	14.00 ± 0.06^{a}	12.00 ± 0.06^{a}	21.00 ± 0.06^{a}	19.00 ± 0.06^{a}	34.00 ± 0.06^{a}	30.00 ± 0.06^{a}	61.00 ± 0.06^{a}	5.00
Conductivity	74.00 ± 0.06^{a}	71.00 ± 0.06^{a}	71.00 ± 0.06^{a}	71.00 ± 0.06^{a}	76.00 ± 0.06^{a}	80.00 ± 0.06^{a}	33.00 ± 0.06^{a}	91.00 ± 0.06^{b}	76.00 ± 0.06^{a}	95.00 ± 0.06^{a}	1000
Nitrite	0.02 ± 0.00^{b}	0.02 ± 0.00^{c}	0.02 ± 0.00^{a}	0.02 ± 0.00^{a}	0.01 ± 0.00^{a}	0.01 ± 0.01^{a}	0.01 ± 0.00^{a}	0.02 ± 0.00^{a}	0.01 ± 0.00^{a}	0.02 ± 0.00^{a}	1.00
Nitrite as N_2	0.02 ± 0.00^{b}	0.01 ± 0.00^{c}	0.01 ± 0.00^{a}	0.01 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.01 ± 0.00^{a}	0.00 ± 0.00^{a}	0.01 ± 0.00^{a}	0.04
Ammonia	8.30 ± 0.06^{c}	6.60 ± 0.06^{c}	6.30 ± 0.06^{c}	6.30 ± 0.06^{c}	4.40 ± 0.06^{b}	6.70 ± 0.06^{c}	6.20 ± 0.06^{b}	5.90 ± 0.06^{b}	5.600 ± 0.06^{c}	5.90 ± 0.06^{c}	0.40
Copper	0.00 ± 0.00^{b}	0.00 ± 0.00^{b}	0.01 ± 0.00^{c}	0.01 ± 0.00^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.01 ± 0.00^{c}	0.00 ± 0.00^{ab}	0.01
Nitrate	0.70 ± 0.06^{a}	0.44 ± 0.01^{b}	0.38 ± 0.01^{a}	0.38 ± 0.01^{a}	0.500 ± 0.06^{a}	0.60 ± 0.06^{a}	0.13 ± 0.01^{a}	0.11 ± 0.01^{a}	1.60 ± 0.06^{a}	1.44 ± 0.01^{a}	10.00
Zinc	0.00 ± 0.00^{a}	0.01 ± 0.00^{b}	0.01 ± 0.00^{a}	0.01 ± 0.00^{a}	0.01 ± 0.00^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.01 ± 0.00^{c}	0.00 ± 0.00^{b}	0.01 ± 0.00^{c}	0.01
Manganese	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.01 ± 0.00^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{b}	0.10
Cadmium	0.01 ± 0.00^{c}	0.01 ± 0.00^{b}	0.00 ± 0.00^{b}	0.00 ± 0.00^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00
Lead	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.01
Chromium	0.02 ± 0.01^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{aa}	0.05

Values are reported as mean ± standard error of means. Values along the rows with the different alphabetic superscripts are significantly different at $p\leq0.05$ while values with the same alphabet are not significant at the same confidence level.

Notes: W1 to W10 represents water samples collected from ten different points

3.1.1 Chemometric Analysis

Chemometrics analysis was performed on the pollution data generated and the PCA results obtained for the elements are shown in figures 2 to 7. Two principal components having eigenvalues greater than 1 were considered. According to Liu et al. (2018), strong and moderate factor loadings range from >0.75, and <0.5 to 0.3, respectively. The first principal component (PC1) in the datasets explains 30.03% of total variance and is moderately positively loaded with Nitrate, Zn, turbidity, Silica, Conductivity, Cr, Mn and Nitrite, indicating both natural and anthropogenic sources. The dominant factor loading of Zn in the first PC1 strongly suggests that the origin of Zn could be associated to the leaching fertilizers into underground water as reported by Mmolawa *et al.* 2017). The most important anthropogenic sources zinc in soil come from discharges of smelter slags and wastes, mine tailings, coal and bottom fly ash, and the use of commercial products such as fertilizers and wood preservatives that contain zinc (Mmolawa *et al.* 2017).

The PC2 in the datasets shows 29.72 % of variance and positively loaded with Cu, Cd, Ammonia, Temperature and pH, indicating anthropogenic sources. The long-established agricultural practices near the study site can be regarded as the sources of Ammonia and Cu, while the waste water effluent can be regarded as the major source of Cd at the study site. Fertilizers can also be regarded as anthropogenic sources of ammonia and Cu.

Figure 3 shows the Hotelling $T^{^2}$ plot which allows us to identify outlying samples. From the result obtained, Samples W8 and W10 were observed as the exceptional samples with unusual pollution status. The PCA scores as seen on figure 4 is about sample grouping. The plot differentiated the samples into three groups. Figure 6 represents the Biplot shows samples and variables simultaneously. The result obtained showed the different groups and their variables. HCA performed on the data reveals three major clusters (Fig. 7). Cluster 1 comprises 1, 2 and 3. The interrelated association among these samples shows similar positive loadings in PC1. Cluster 2 includes 4, 5, 6, 7, and 9. The interrelated association shows similar positive loadings in PC1. Cluster 3 contains 8 and 10, and its positive loadings are similar to PC2. HCA showed Pair 8 and 10 share similar pollution status. Their similarity is seen in their Ammonia contamination level which is the same (5.90mg/l) as well as nitrite and nitrite as nitrogen levels which are 0.02mg/l and 0.01 respectively.

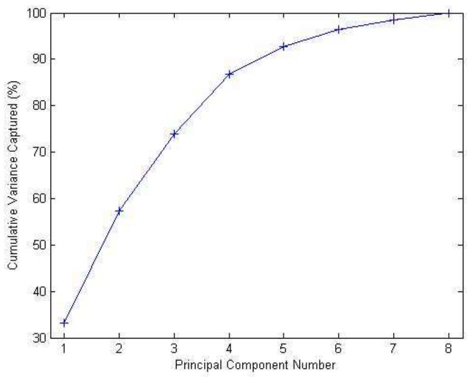

Figure 2: PCA Eigenvalue plot for water samples

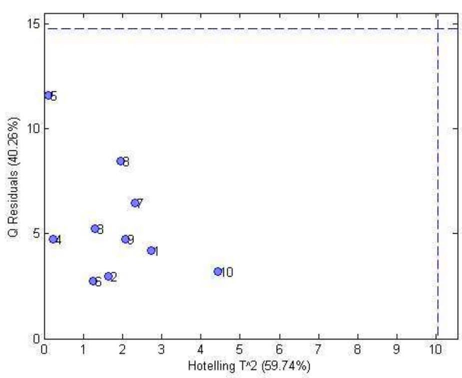

Figure 3: Hotelling $T^{^2}$ plot for water samples from Bosso dam

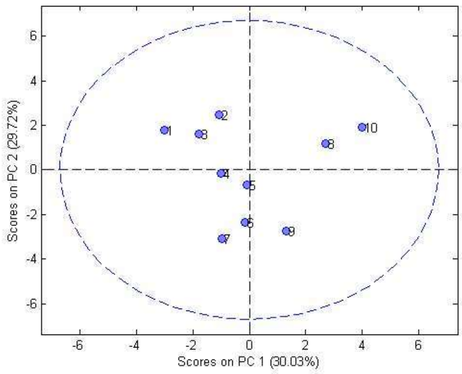

Figure 4: PCA Scores plot for water samples

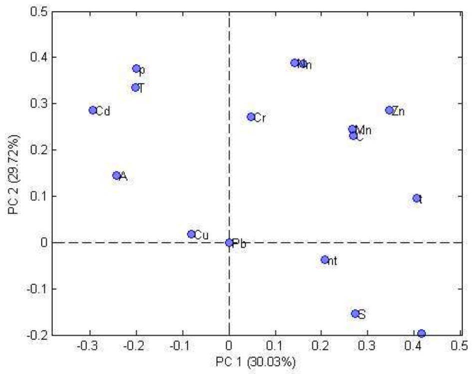

Figure 5: PCA Loadings plot for water sample

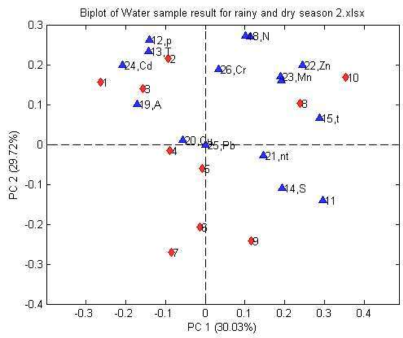

Figure 6: PCA Biplot for water samples and variables

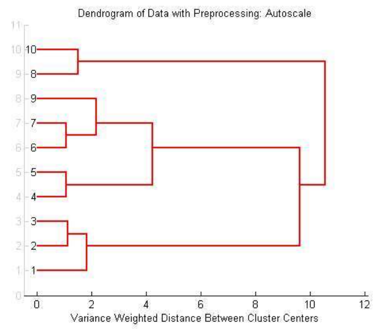

Figure 7: HCA Dendogram for water samples

3.1.2 Correlation Analysis

Correlation analysis of the data was carried out using Microsoft Excel Add-ins. The result shown in Table 2 suggests that most of the variables were negatively correlated. However, strong positive correlation was observed for Cd - NO_2^-, Cd - pH, Cd - Temperature, Pb - Cr, Cd - NH_3, NO_2^- -NH_2^--N, Zn – Mn, Cr – Zn, Cr, Mn and Cr - Pb. Cadmium concentration was strongly correlated with pH, Temperature, Nitrite, Nitrite as Nitrigen and Ammonia at 0.75, 0.66, 0.65 and 0.73 respectively, which may be due to leaching or fertilizer from irrigation water. Silica showed no positive correlation with other variables.

4.0 Conclusion

This work was undertaken to evaluate the pollution status of Bosso dam. From PCA, two principal components suggests possible anthropogenic sources. These components explain 59.79% of the total variance and high positive loading was found in samples W8 and W10. The present investigation clearly indicates that the water from Bosso dam is contaminated with some toxic heavy metals, Ammonia, Nitrite as Nitrogen. Consequently, there is a dire need to reduce/regulate the anthropogenic sources of pollution in the study area.

Table 2: Correlation among different variables

	p	T	S	t	C	n	N	A	Cu	nt	Zn	Mn	Cd	Pb	Cr
p	1														
T	**0.969159**	1													
S	-0.4093	-0.34433	1												
t	-0.62176	-0.7298	0.319241	1											
C	0.185616	0.098174	0.129887	0.334386	1										
n	0.434303	0.297305	-0.30832	0.20901	0.19927	1									
N	0.422745	0.266588	-0.29637	0.265694	0.254967	**0.993111**	1								
A	0.292907	0.142827	-0.51481	-0.01376	-0.05749	0.443681	0.462759	1							
Cu	0.382434	0.298165	-0.00985	-0.29175	0.22225	-0.20192	-0.1478	-0.0873	1						
nt	-0.07575	-0.129	0.441989	0.26257	0.231529	-0.37574	-0.28216	-0.03474	0.435493	1					
Zn	0.173931	0.176391	-0.07952	0.250258	0.557617	0.452345	0.439603	-0.43137	-0.11874	-0.35615	1				
Mn	-0.02896	-0.06036	-0.29473	0.347184	0.463693	0.49332	0.489099	-0.16762	-0.38604	-0.46655	**0.841442**	1			
Cd	**0.751012**	**0.656134**	-0.33289	-0.23578	0.032316	**0.647945**	**0.647392**	**0.732274**	0.015563	0.012771	-0.11733	-0.15297	1		
Pb	0.069012	0.5382	-1.42091	0.21682	0.33986	0.220197	0.10932	-0.83922	0.3274	0.13249	0.44271	-0.31974	0.73921	1	
Cr	0.436376	0.485238	-0.2025	-0.12227	0.471831	0.315439	0.274753	-0.35453	0.028453	-0.43992	**0.865184**	**0.573337**	0.038013	**0.82653**	1

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