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RESEARCH ARTICLE

Heavy metals accumulation and ecological risk assessment in asa river sediments, Ilorin-Nigeria, dredged sediment toxicity potential

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Abstract

The reuse of dredged river sediments for agricultural purposes offers a sustainable solution despite environmental risks. This study investigated selected heavy metals content in Asa River sediments and assessed potential environmental risks. Samples were collected from four locations along the Asa River and analyzed using total metal, pore water, and acid leachate procedures. Data obtained were subjected to statistical processing using descriptive and inferential methods. The total metal concentrations were evaluated against established threshold limits for each element. A two-way ANOVA was performed to determine significant variations in elemental concentrations across different sampling locations and extraction methods. Using atomic absorption spectrophotometry, metal concentrations were found to be below permissible levels according to Sediment Quality Guideline (ISQG) and National Oceanic and Atmospheric Administration (NOAA) sediment quality guidelines: Cd (0.080-0.102), Ni (0.049-0.499), Cr (0.113-0.183), Pb (0.581-1.982), Cu (9.02-9.64), Co (0.113-0.183), Zn (0.250-0.680), Mn (0.90-1.42), and Fe (65.4-94.1) mg/kg. Significant positive correlations were found among metals, indicating complicated identical behavior during transport. Pore water analysis indicated potential bioavailability and toxicity concerns for Pb, Cd, and Ni. These findings highlight the need for careful assessment and management to minimize environmental risks associated with sediment reuse.

Keywords**:** acid leachate, bioavailability, heavy metals, pore leachate, readily toxicity

1. *Introduction*

Swift industrialisation and economic growth have led to a steady influx of heavy metals into soils and sediments. Various pathways contribute to this contamination, including the use of fertilizers and irrigation systems (Solihu and Bilewu, 2022), as well as discharge of industrial wastewater, sewage, and atmospheric pollutants (Adekola and Eletta, 2007; Ibrahim *et al*., 2013). The presence of polluted sediments poses a significant environmental threat to both human health and ecosystems, particularly in urban areas of developing countries (Ale *et al*., 2024). This is often a consequence of inadequate planning and unchecked development, leading to irresponsible practices that harm the environment (Shetaia *et al*., 2023). Asa River serves as a primary source of water for Ilorin cities and its surrounding areas, with the water undergoing treatment at Asa Dam treatment plant (Solihu and Bilewu, 2022). This water body flows through a south-north direction and divides the city of Ilorin into almost two equal parts (Ibrahim *et al*., 2013), consequently, heavy metals and organic pollutants enter into it from both terrigenous – weathering of rocks resulting in geochemical recycling of heavy metals and anthropogenic sources (Adekola and Eletta, 2007; Ibrahim *et al*., 2013). Thus, the sediments of Asa River are contaminated with heavy metals and organic substances, including hydrocarbons, polyaromatic hydrocarbons, and polychlorinated biphenyls (Shetaia *et al*., 2023). This level of contamination is consistent with observations from urban areas worldwide, where industrial and anthropogenic activities often lead to environmental pollution (Miranda *et al*., 2021). Consequently, the quest for this study aimed to assess the status of sediments along the Asa River in Ilorin, Nigeria, to determine their suitability for

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sustainable agricultural reuse. This would proffer answers to the following questions: What is the level of heavy metal contamination in the sediments of the Asa River, and what are the implications for their use in sustainable agriculture?

2. Materials and Methods

2.1 Study Area

This research focused on the analysis of dredged sediment samples from Asa River in Ilorin, Kwara state. Sediments were collected from four locations: Coca cola (N080 28’ 26.7”, E004033’40.6”), Unity (28’ 28’50.3”, E004033’38.1”), Post office (N080 28’29’ 16.6”, E004033’39.6”) and Amilegbe (N08029’ 42.33”, E004033’53.9”). A scaled geographical map of the course of Asa River showing the study area and sampling points is shown in Figure 1. The climate of Ilorin is characterized by both wet and dry seasons which begins towards the end of April and last till October and begins in November and ends in April, respectively (Ajadi *et al*., 2016). Temperature ranges between 33°C to 35°C from November to January and 34°C to 37°C from February to April. The cumulative annual rainfall ranges from 990.3mm to 1318mm while the relative humidity varies from 75% to 88% between May to October. In the dry season, however, it ranges from 35% to 80% (Ajadi *et al*., 2016).

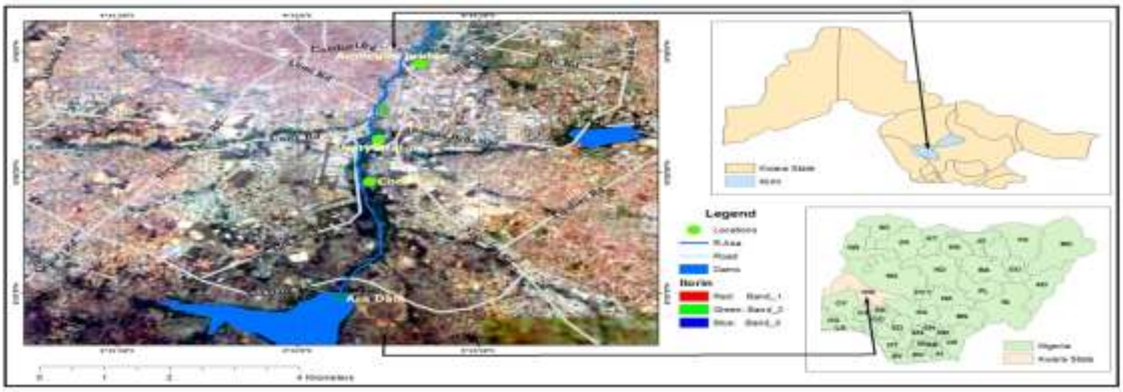

Figure 2.1: *Scale map of study area showing the four locations.*

Ilorin is mainly drained by Asa River which flows in a south-north direction with a dendritic drainage system (Ajadi *et al*., 2016). This water body divides Ilorin into two parts: eastern and western parts. It consists of Precambrian basement complex rock whose soils are mostly loamy with medium to low fertility status (Ajadi *et al*., 2016). The soil texture coupled with the high rainfall and temperature results in leaching loss of minerals nutrients of the soil, thus, the inclination of the typical lateritic soils. Population pressure, an important element of change in land-use has resulted in dwelling increases which apart from the containment of industrial effluents from several manufacturing plants within the estate, also makes the river serves as a recipient of domestic (sewage) and agricultural (run offs along the bank of the river) wastes. Along the area of the river segment are shopping complexes, a hospital, banks, a car park and a mini market for the sale of fresh vegetables and fish (Ajadi *et al*., 2016).

2.2 Leach tests

Two single-stage leach tests with water and dilute sulphuric acid were designed to help assess the likely hydrogeochemical consequences of flushing Asa River sediments with rain or river water. 20 grams portions of soil samples from each collection location were weighed using electronic weighing balance in the Soil and Plant Laboratory of the University of Ilorin in duplicates for water and acid extraction. A set was mixed with 40ml distilled water and the second set mixed with 40ml

0.01 M sulphuric acid (pH ≈ 2.0) in a solid: fluid weight ratio of 1:2. The mixture was then transferred into an airtight container of about 100ml and agitated continuously for 48 hours using mechanical shaker [ASTM, (4)]. The pore water and acid leachate were then removed from contact with the sediment by decanting and then filtered through a 0.45 µm filter and analysed for Lead, Cadmium, Iron, Chromium, Copper, Manganese, Nickel, Zinc and Cobalt using the Atomic Absorption Spectrophotometer (BUCK Scientific ACCUSYS 211) (United States Environmental Protection Agency, 2013).

2.3 Total metal test

Total metal concentration was determined in samples following ternary acid (HNO_3, HF, HCl) digestion. The samples were then analysed for Lead, Cadmium, Iron, Chromium, Copper, Manganese, Nickel, Zinc and Mercury [United States Environmental Protection Agency, (15)].

2.4 Statistical analysis

Statistical analysis of the data including analysis of variance (ANOVA), correlation and regression calculations, were carried out by using SAS 17.0 version. Relationship between pore water and acid leachate metal concentration with total metal was analysed using correlation analysis. The slope coefficient, R^2 were used in determining which of the two - pore water and acid leachate metal concentration could be used in predicting the level of the respective metal toxicity. Total metal concentration was subjected to two-way ANOVA to assess significant differences between the mean element levels in different location and extraction technique. A P-value of less than or equal to 5% indicates that significant relationship exists between the corresponding variables. Spearman correlations between element concentrations were computed. Principal Component Analysis (PCA) was used to categorize and reduce the variables to identify such associations without sacrificing a lot of information. Based on the relative proportions of the variance explained, the number of components to be kept is determined. VARIMAX rotation with Kaiser normalization was employed to make the results easier to understand. It was therefore feasible to determine the most important variables within the component group and the extent of their interrelationship based on the rotated component matrix (Mohammed *et al*., 2014).

3. Results and discussion

3.1 Total sediment concentrations

The concentration of heavy metals (Cd, Cr, Mn, Fe, Ni, Pb, Zn, Co, and Cu) in the samples that were gathered is measured in Test 2.1 and compared to limit values. Table 1 presents the findings. Additionally, differences were seen between sites for all elements except Co and Cd, indicating some degree of human influence (1). Compared to freshwater sediments, the concentration of Co in this investigation was lower (20 mg kg^{-1}) Nagpal (2004). For magnesium and manganese, toxicological data were not provided. Comparisons were made with literature data for the elements lacking toxicological guidelines. Calcium, magnesium, manganese, iron, copper, cobalt, chromium and zinc are nutrient and are likely to present ecological problems in the area where they were elevated (Bhuyan *et al*., 2023).

3.2 Pore water composition and Sediment leaching tests

Tests 2 and 3 investigated the level of heavy metal concentrations in pore waters which gave a measure of their bioavailability and potential toxicity and a leaching test that measured metals that are easily desorbed from sediments upon a change in pH (acidification), respectively. Pore water and leaching results are presented in Table 2 for samples from the four locations.

Table 1. Comparison between the averages detected level of elements in mg kg^{-1} dw with NOAA and ISQGs Marine sediment quality guideline values

Elements	Average results	ERL	ERM	ISQG/TEL	PEL
Cd	0.07	1.2	9.6	0.7	4.2
Ni	0.30	21	52	Non polluted <20 Moderatelypolluted 20-50 Heavily polluted>50	-
Cr	0.15				
Pb	1.30	47	220	*30.2 Nonpolluted <40 Moderatelypolluted 40-60 Heavily polluted>60	112
Cu	9.31	34	270	*18.7 Nonpolluted <25 Moderatelypolluted 25-50 Heavily polluted>50	108
Co	0.15				
Zn	0.49	-		*124 Non polluted <90 Moderatelypolluted 90-200 Heavilypolluted>200	
Mn	1.13	460	1100		0.78
Fe	79.41			LEL:20 SEL:40	

ERL =effects range low

ERM =effects range median

ISQG/TEL =Interim marine sediment quality guidelines/threshold effect level

PEL =Probable effect levels.

Table 2. Descriptive statistics of concentration of metals in the Asa River sediment as obtained by Pore-water and Acid leachate analysis

Element	*Mean*	*Std. Dev.*	*Variance*	*Minimum*	*Maximum*	*Median*
Mn	26.52	35.43	1255.31	0.10	84.20	0.68
Fe	183.35	240.27	57728.57	0.38	615.50	26.17
Cu	9.37	0.77	0.59	8.63	11.04	9.06
Zn	12.29	22.77	518.48	0.04	70.95	0.26
Co	0.22	0.20	0.04	0.004	0.55	0.11
Pb	1.08	0.88	0.78	0.23	2.34	0.66
Cd	0.06	0.02	0.00046	0.02	0.09	0.06
Ni	0.37	0.17	0.03	0.11	0.67	0.34
Cr	0.07	0.06	0.0035	0.01	0.21	0.07

The pore water procedures used on the sediment investigation in this study resulted in Cadmium (Cd) metal concentration values ranging from 0.02 to 0.082 mg kg^{-1} while, acid leachate procedures recorded Cd metal values ranging from 0.04 to 0.09 mg kg^{-1} (Table 2). These values are below the Interim Sediment Quality Guidelines (ISQG) maximum permissible level of 0.60 mg kg^{-1}. Pore water Nickel (Ni) metal concentration ranged from 0.113 to 0.400 mg kg^{-1} while the acid leachate samples recorded Ni metal values ranging from 0.28 to 0.67 mg kg^{-1} (Table 2). The pore water procedures used on the sediment investigation in this study resulted in Chromium (Cr) metal concentration values ranging from 0.02 to 0.07. Also, the acid leachate procedures recorded Cr metal values ranging from 0.01 to 0.21 (Table 2). The pore water procedures used on the sediment investigation in this study resulted in Lead (Pb) metal concentration values ranging from 0.23 to 0.92. Also, the acid leachate procedures on the sediment investigation in this study resulted in Copper (Cu) metal concentration values ranging from 8.63 to 9.24. Also, the acid leachate procedures recorded Cu metal values ranging from 8.96 to 11.04 (Table 2). The pore water procedures used on the sediment investigation in this study resulted in Cobalt (Co) metal concentration values ranging from 0.004 to 0.120. Also, the acid leachate procedures recorded Cu metal values ranging from 0.10 to 0.55 (Table 2). The pore water procedures used on the sediment investigation in this study resulted in Zinc (Zn) metal concentration values ranging from 0.04 to 0.31. Also, the acid leachate procedures recorded Zn metal values ranging from 0.02 to 70.95 (Table 2). The pore water procedures used on the sediment investigation in this study resulted in Manganese (Mn) metal concentration values ranging from 0.10 to 1.10. Also, the acid leachate procedures recorded Mn metal values ranging from 0.30 to 84.20 (Table 2). The relationship between pore water and acid leachate and total metal concentration for Cd is presented in Figure 1. A fair linear relationship was observed between the variables for pore water based on the slope (1.0488). While a very weak and negative relationship was observed for acid leachate and total Cd metal concentration. This implies that Cd metal toxicity could be predicted using pore water concentration. This is because the greatest percentage of the total metal concentration is in the exchangeable form (Maurya and Kumari, 2021; Zou *et al*., 2021). Also, the positive slope displayed for pore water metal concentration implied a tendency for Cd ions to increase proportionately with an increase in total metal concentration. Thus, Cd metal is readily available to plants (Sojka and Jaskula, 2022); and increases with total metal concentration whose higher portion is in the exchangeable form. The relationship between pore water and acid leachate and total metal concentration for Ni is presented in Figure 2, a weak linear relationship was observed between the variables for pore water based on the slope (-0.9473) and a strong relationship was obtained between the variables for acid leachate as shown by the slope (0.5731). Also, the R^2 values from the graph show that Ni toxicity is better predicted using the acid leachate analysis as against the pore water analysis. Nickel will adsorb to clays, iron and manganese oxides, and organic matter and is thus removed from the soil solution. The formation of complexes of Ni with both inorganic and organic ligands will increase Ni mobility in soils.

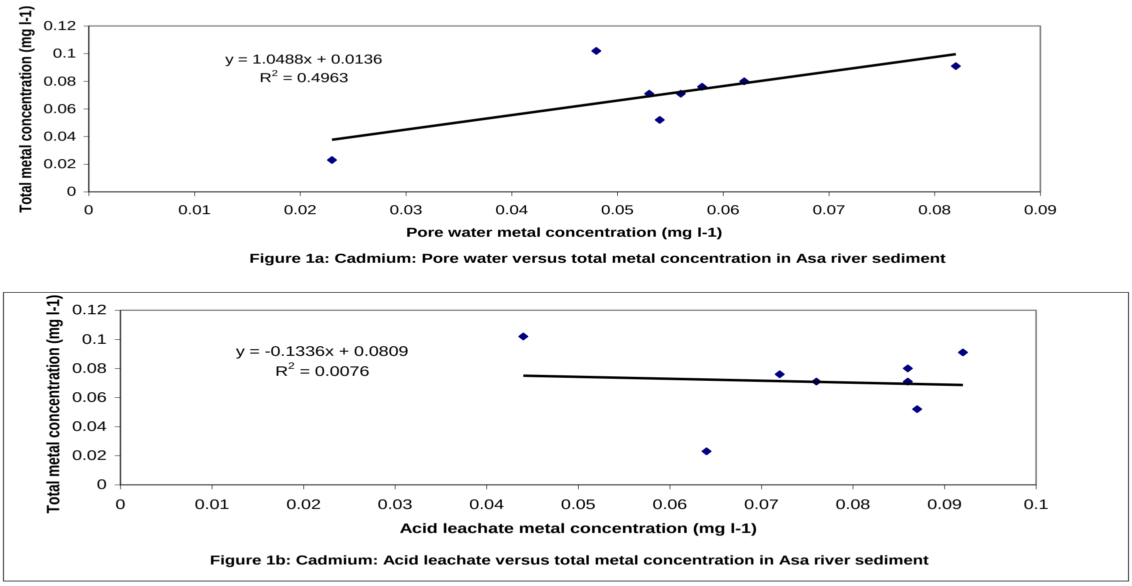

Figure 1a: Cadmium: Pore water versus total metal concentration in Asa river sediment

Figure 1b: Cadmium: Acid leachate versus total metal concentration in Asa river sediment

Figure 1: Relationship between pore water, acid leachate and total metal for Cadmium.

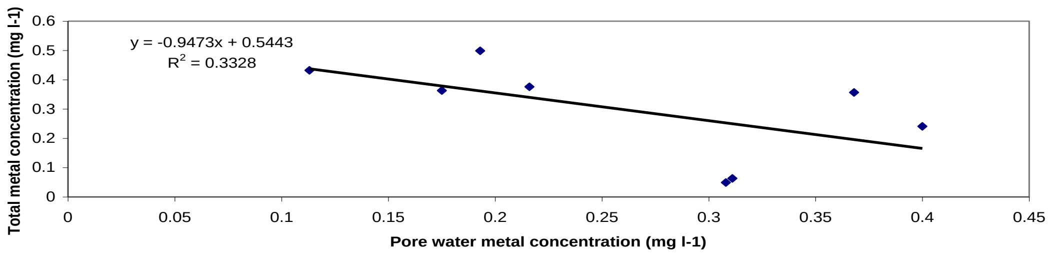

Figure 2a: Nickel: Pore water versus total metal concentration in Asa river sediment

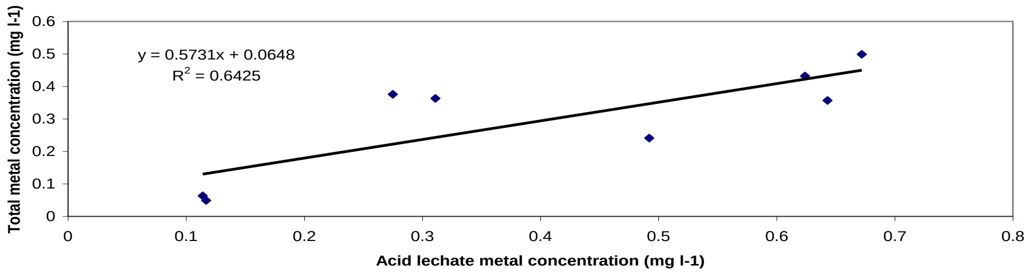

Figure 2b **Nickel: Acid lechate versus total metal concentration in Asa river sediment**

Figure 2: Relationship between pore water, acid leachate and total metal for Nickel.

The relationship between pore water and acid leachate and total metal concentration for Cr is presented in Figure 3, a weak linear relationship is observed between the variables for pore water based on the slope (0.2461). While very weak relationships were observed for acid leachate and total Cr metal concentration. Also, from the R^2 values in these figures, Cr metal toxicity could be predicted using pore water concentration.

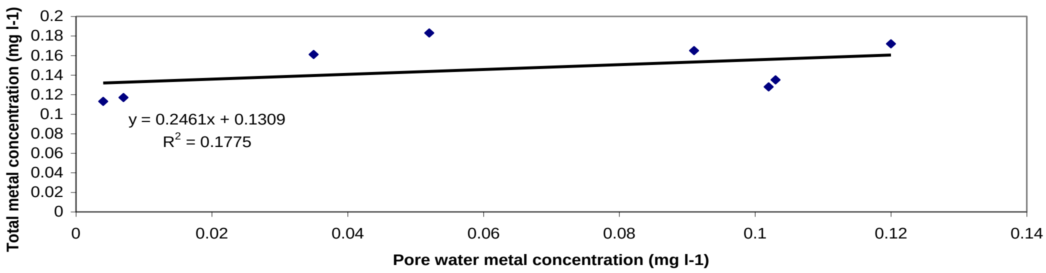

Figure 3a: Chromium: Pore water versus total metal concentration in Asa river sediment

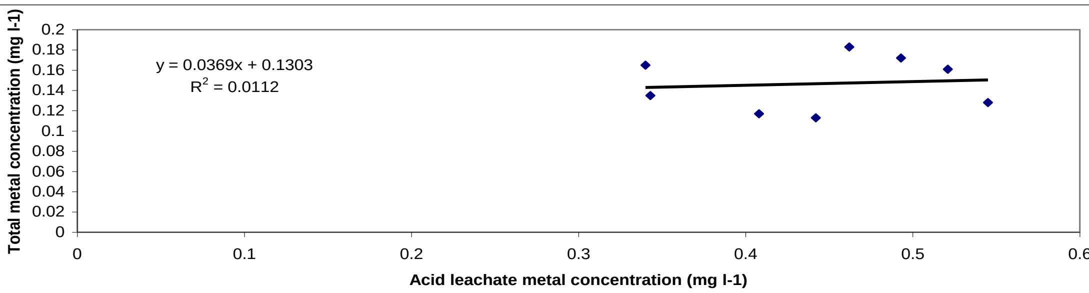

Figure 3b: Chromium: Acid lechate versus total metal concentration in Asa river sediment

Figure 3: Relationship between pore water, acid leachate and total metal for Chromium.

The relationship between pore water and acid leachate and total metal concentration for Pb is presented in Figure 4, a very strong linear relationship is observed between the variables for pore water based on the slope (1.6996). This implies that Pb may be readily available for plants uptake for both the pore water metal concentration analysis and acid leachate metal concentration analysis. Also, a strong relationship was observed for acid leachate and total Pb metal concentration. The R^2 values displayed here shows that Pb metal toxicity could be better predicted using pore water concentration as opposed the acid leachate metal concentration.

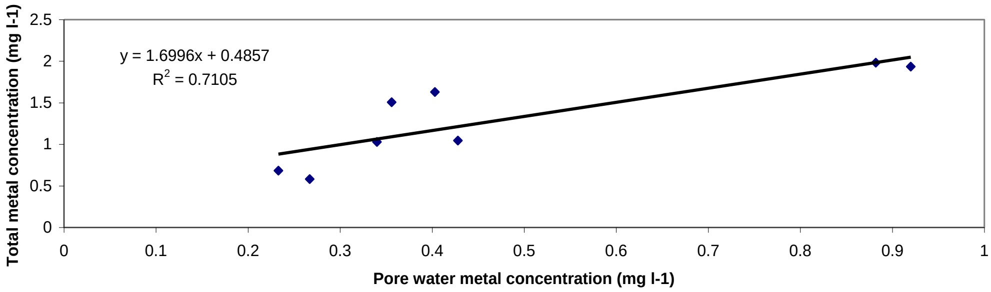

Figure 4a: Lead: Pore water versus total metal concentration in Asa river sediment

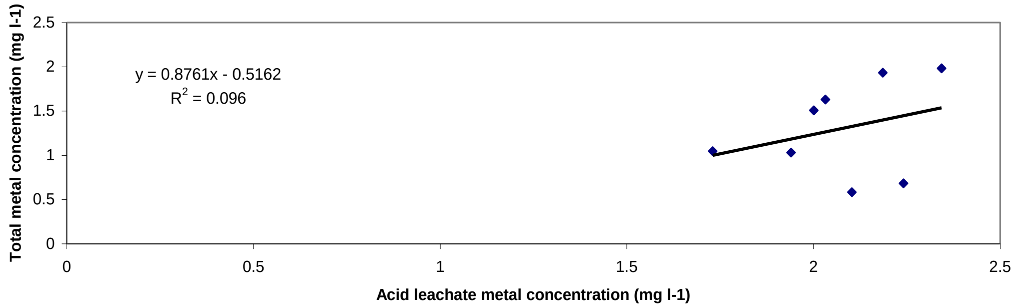

Figure 4b Lead: Acid lechate versus total metal concentration in Asa river sediment

Figure 4: Relationship between pore water, acid leachate and total metal for Lead

The relationship between pore water and acid leachate and total metal concentration for Cu is presented in Figure 5, a very weak linear relationship is observed between the variables for both pore water and acid leachate and total Cu metal concentration based on the slopes shown in Figure 5. This implies that Cu metal may not be readily available for plants uptake. Also R^2 values show that Cu toxicity may not be effectively predicted using both procedures.

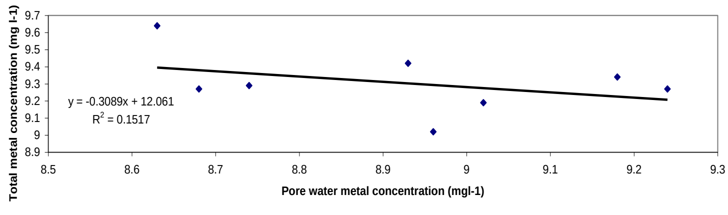

Figure 5a: Copper: Pore water versus total metal concentration in Asa river sediment

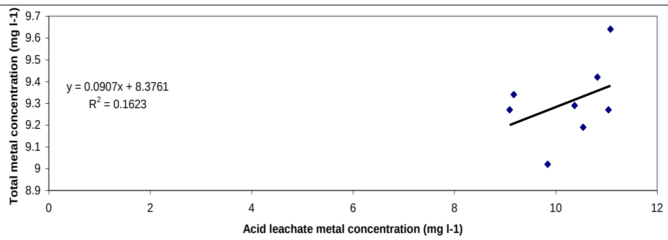

Figure 5b:Copper: Acid leachate versus total metal concentration in Asa river sediment

Figure 5: Relationship between pore water, acid leachate and total metal for Copper

The relationship between pore water and acid leachate and total metal concentration for Co is presented in Figure 6, a weak linear relationship is observed between the variables for both pore water and acid leachate and total Co metal concentration based on the slopes shown in Figure 6 with the values for acid leachate been the weakest. This implies that Co metal may not be readily available for plants uptake. Also, R^2 values shows that Co toxicity will be better predicted using the pore water analysis procedures. However, the predictions by the two extraction techniques would not be accurate based on the relatively low R^2 values recorded.

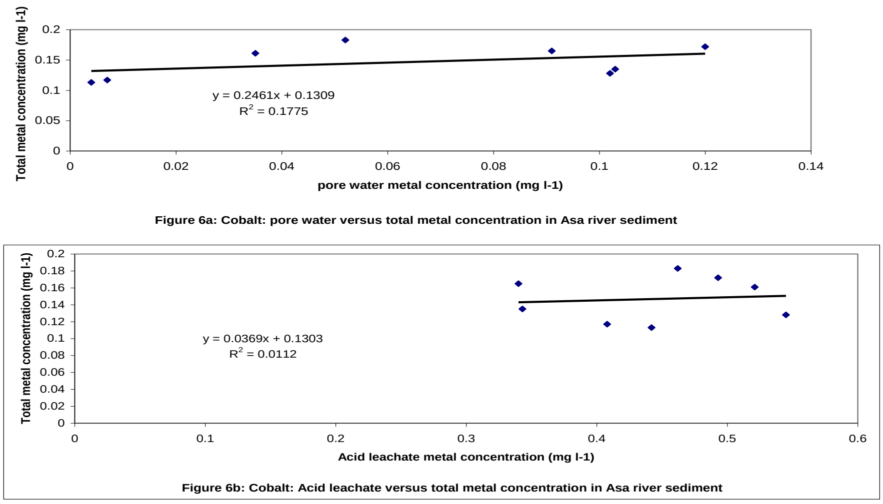

Figure 6: Relationship between pore water, acid leachate and total metal for Cobalt

The relationship between pore water and acid leachate and total metal concentration for Zn is presented in Figure 7, a strong linear relationship is observed between the variables for pore water based on the slope (0.7733) and a very weak relationship is seen for acid leachate as shown by the slope (0.0018). This implies that Zn maybe readily available for plants uptake for the pore water metal concentration analysis. The R^2 values displayed here shows that Zn metal toxicity is better predicted using pore water concentration as opposed the acid leachate metal concentration.

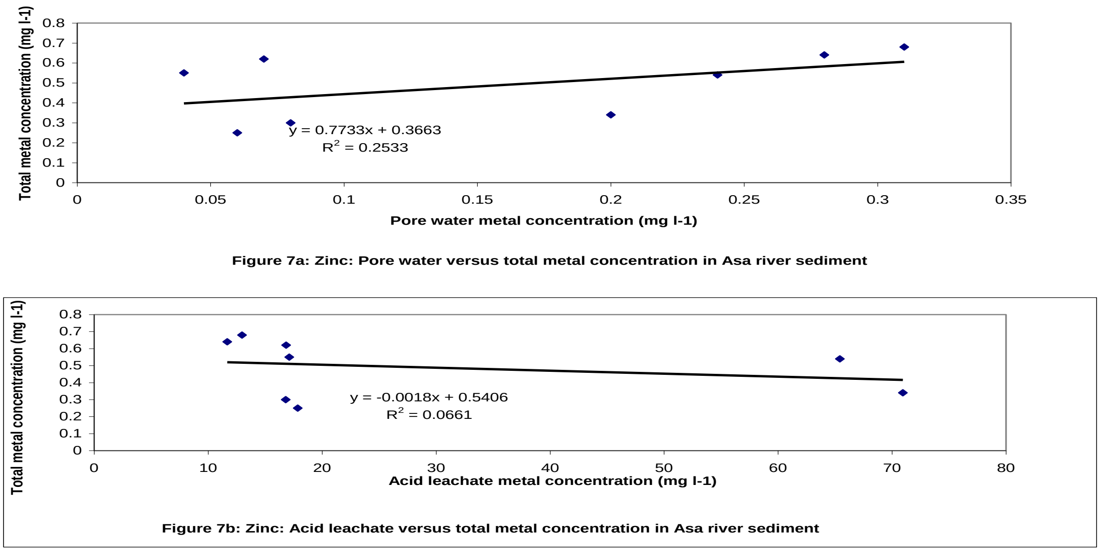

Figure 7: Relationship between pore water, acid leachate and total metal for Zinc

The relationship between pore water and acid leachate and total metal concentration for Mn is presented in Figure 8, a weak linear relationship is observed between the variables for pore water based on the slope (0.1897) and a very weak relationship is seen for acid leachate as shown by the slope (-0.0021). This implies that Mn may not be readily available for plants uptake for the pore water metal concentration analysis. The R^2 values displayed here shows that Mn metal toxicity is better predicted using pore water concentration as opposed the acid leachate metal concentration.

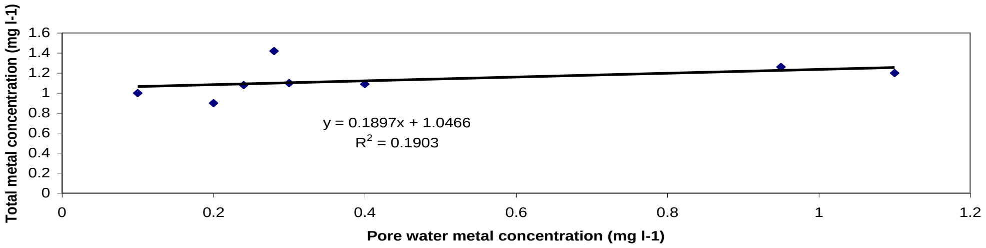

Figure 8a: Manganese: Pore water versus total metal concentration in Asa river sediment

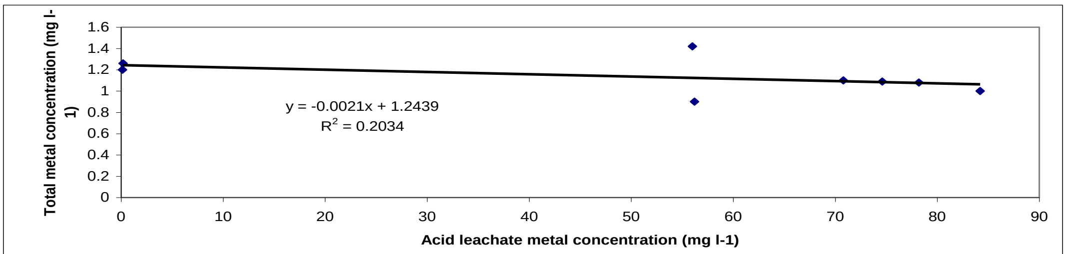

Figure 8b: Manganese: Acid leachate versus total metal concentration in Asa river sediment

Figure 8: Relationship between pore water, acid leachate and total metal for Manganese

3.3. Statistical analysis between levels of metals

3.3.1 Factor analysis of the whole database

Three factors were extracted from the thirteen variables, accounting for about 94.25% of the variance. Factor 1 retains information of about 55.80% of the total variance. Factors 2 and 3 explained 20.24% and 18.21%, respectively of the total variance (Table 3). The factor components (FCs) with eigen values > 1 were retained, since eigen values < 1 indicated the factor could explain less variance than an additional metal concentration.

Table 3: Eigenvalue, proportion and cumulative variance explained by factor analysis using correlation matrix of 13 elements in Asa River sediment

	Eigenvalues	Proportion	Cumulative
Components	←	%	→
1	71708.015	58.82	58.82
2	30378.24	24.92	83.73
3	12819.99	10.52	94.25

FC 1 explained 54.82% which included Ca, Mg, Mn, Fe, Cu, Zn, Co, Pb, Cr and Ni as the major contributing variables (Figure 9). FC 2 explained 24.92% of the total variance which included K and Na while FC 3 explained 10.52% of the total variance included Cd (Figure 9). FC 1 was directly related to 'trace metals', FC 2 to 'monovalent cations' and FC 3 included 'highly toxic' metals.

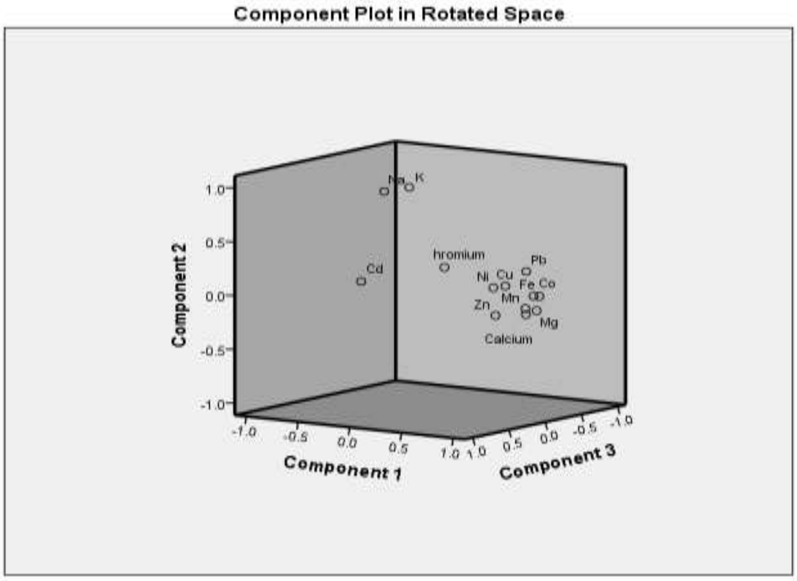

Figure 9. PCA loading plot of selected 13 elements in sediments from Asa River Factor Loading (>0.5) (Varimax normalized)

The 'trace metals' have significant influence on nutrition and physiology of animals and human beings. Highly significant positive ($P < 0.01$) inter-relationships were observed between Mn, Fe, Cu, Zn, Co and Pb, and Ca and Mg (Table 4) which indicate the complicated identical behaviour of these metals during their transport in Asa River that can be influenced by many factors. There were also positive and highly significant ($P < 0.01$) inter-elemental correlations between Na and K since both could exchange for one another in soil-plant reactions. Cd was the "most toxic" metal whose level in the sediments studied increase mainly due to anthropogenic activities [Adekola and Eletta (1)].

Table 4. Spearman correlation matrix between element levels in Asa River sediment at Ilorin, Nigeria

Elements	Ca	Mg	K	Na	Mn	Fe	Cu	Zn	Co	Pb	Cd	Ni	Cr
Ca	1	.788**	-.214	-.341	.666**	.810**	.675**	.643**	.845**	.819**	-.041	.168	.110
Mg		1	-.295	-.416*	.873**	.968**	.772**	.545**	.876**	.725**	-.135	.499*	.096
K			1	.951**	-.274	-.155	.006	-.218	-.097	.166	.285	-.061	.237
Na				1	-.381	-.293	-.087	-.323	-.260	-.018	.438*	-.220	.176
Mn					1	.886**	.554**	.713**	.806**	.686**	-.115	.670**	.379
Fe						1	.701**	.517**	.873**	.788**	-.092	.493*	.107
Cu							1	.550**	.855**	.672**	.043	.316	.247
Zn								1	.780**	.575**	-.105	.461*	.613**
Co									1	.820**	-.129	.491*	.347
Pb										1	-.067	.474*	.382
Cd											1	-.308	.032
Ni												1	.611**
Cr													1

** Significant at <0.01 level of probability
*Significant at <0.05 level of probability

4. Conclusion and recommendation

Result of trace metals such as Cd, Cr, Mn, Ni, Fe, Co, Cr, Cu, Pb and Zn in dredged Asa River sediment shows that it contains heavy metals but only a small fraction of these metals was readily bioavailable. Thus, Asa river sediments contain metals which are contaminants but below the permissible level for them to be toxic. Also, these metals may not be readily available for plant uptake except for Lead and Cadmium with correlation coefficients of 0.71 and 0.50, respectively in relationship between pore water and total metal concentration and Nickel (0.64) in acid leachate regression with total metal concentration in the sediment. Therefore, concerted efforts should be put in place for proper monitoring of Asa River as accumulation of these metals with time could induce toxic effects to both water benthic and human beings. Pore water analysis was a better parameter for measuring the bioavailability and toxicity level of Asa river sediments than the acid leachate analysis as all the metals studied except Ni would be bioavailable with an increase in total concentration while the formation of complexes of Ni with both inorganic and organic ligands will increase its mobility in soils. However, studies on adsorption of metal cations with soil properties such as pH, redox potential, clay, soil organic matter, iron and manganese oxides and calcium carbonates contents are necessary as dredging involves oxidation which has the potential of significantly lowering the pH thereby raise the metal content of associated leachate of Cu, Fe, Mn, Zn, Co and Ni.

Declarations

Authors' Contributions
All authors are contributed in this research. All authors reviewed and approved the final manuscript.

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Institutional Review Board Statement
All Institutional Review Board Statements are confirmed and approved.

Data Availability Statement
Data presented in this study are available on fair request from the respective author.

Ethics Approval and Consent to Participate
Not applicable

Consent for Publication
Not applicable.

Conflicts of Interest
The authors disclosed no conflict of interest.

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