J. Mater. Environ. Sci., 2025, Volume 16, Issue 5, Page 925-936

Journal of Materials and Environmental Science ISSN : 2028-2508 e-ISSN : 2737-890X CODEN : JMESCN Copyright © 2025, University of Mohammed Premier Oujda Morocco

http://www.jmaterenvironsci.com



Assessment and Comparison of Heavy Metals Pollution in Sediments from Effluent Channels and New Gusau Reservoir, In North-western, Nigeria

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Received 05 Mar 2025, **Revised** 03 May 2025, **Accepted** 04 May 2025

Keywords:

- ✓ Effluent;
- ✓ Heavy metals;
- \checkmark Contamination;
- ✓ Pollution;
- ✓ Geoaccumulation index;

Citation: Abubakar S, Sani, Z. R., Muhammed S. S. (2025) Assessment and Comparison of Heavy Metals Pollution in Sediments from Effluent Channels and New Gusau Reservoir, In North-western, Nigeria, J. Mater. Environ. Sci., 16(5), 925-936 particularly in developing countries such as Nigeria. Heavy metals analysis done using acid digestion method and Flame Atomic Absorption Spectrophotometer (AA224FS). The concentration of Lead (Pb), Copper (Cu), Cadmium (Cd), Zinc (Zn) and iron (Fe) in sediments from effluent channels of Yar Dantse (YDS), Yar Dole (YDL) and Yar Rumfa (YRF) and New Gusau Reservoir (NGR) were analyzed from February 2018 to July 2020. The data were analyzed using one way ANOVA, with significance difference set at p<0.05. The results of sediment metals analysis indicated a clear pattern between channels and New Gusau Reservoir. This can be related to the discharging of a channel effluent into the reservoir. The sediment Cu ranges from 0.03-5.66mg/kg, Cd 0.91-2.98mg/kg, Fe 7.09-63.77mg/kg, Pb 1.02-2.45mg/kg and Zn has 1.79-25.67mg/kg. The heavy metals in sediment showed relatively higher values in channels compared to the receiving reservoir. The concentration of heavy metals in sediment did not exceed maximum accepted limit for WHO. The contamination factor (CF) confirmed the sediment was not polluted by heavy metals except Fe with very high degree of contamination in all composite sites. The Pollution load index (PLI) and The Geo-accumulation Index (Igeo) values were lower than one (< 1)in all the composite sites, indicating good sediment quality except inYRF with considerable degree of pollution level. Therefore, the sediments in YDS, NGR and YDL had low PLI and hence not polluted by heavy metal

Abstract: Contamination of sediment by heavy metals is one of the global issues

1. Introduction

In many chemical tests needing a high degree of precision and accuracy, such as in food and drug safety, clinical diagnosis, and environmental monitoring, atomic absorption spectroscopy is a widely used technique (Abdel-Ghani & Elchanghaby, 2007). Atomic absorption spectrometers are extremely useful tools in any laboratory setting since they can be used to measure the concentration of more than 70 different elements in a particular sample solution. But as a result of adsorption, precipitation, diffusion processes, chemical reactions, biological activity, and a combination of those phenomena, sediments are the final destination of trace metals (Ramirez *et al.*, 2005; Belbachir *et al.*, 2013). Sediments can become a source of metals, releasing them into the overlying water column (Jones and Turki, 1997). Contamination of heavy metals in the aquatic environment has attracted global

attention owing to its abundance, persistence and environmental toxicity (Islam et al., 2015a; Ahmed et al., 2015; Bazzi et al., 2020; Errich et al., 2023). Both natural and anthropogenic activities are responsible for the abundance of heavy metals in the environment (Wilson and Pyatt, 2007). However, anthropogenic activities can generate heavy metals in sediment and water that pollute the aquatic environment (Ahmed et al., 2015c). The increasing pollution by heavy metals has a significant adverse health effect for invertebrates, fish and humans (Yi et al., 2011). The metal pollution of aquatic ecosystems is increasing due to the effects from urbanization and industrialization (Mir et al., 2016). Metal distributions in River sediments are mainly influenced by industrial wastes. The assessment of heavy metal pollution in estuarine sediments is a complex process in which physical, chemical and biological data are required. Since in many areas the necessary biological data are not available, in environmental geochemistry, the assessment of contamination status is normally based on Sediment Quality Guidelines (SQGs) or quantitative indexes such as Geo-accumulation Index (Igeo) and contamination Factor (CF), etc with the respect to reference values of regional/local background values or average concentrations of shale, continental crust or upper continental crust (Aksu et al., 1997; Rubio et al., 2000; Woitke et al., 2003; Samir et al., 2006; Chen et al., 2007). Various studies have proposed methods for removal of heavy metals to avoid contamination environment and health (Razzouki et al., 2015; Errich et al., 2021; El Hammari et al., 2022; Laoye et al., 2025;) The objectives of this study is to compare the distribution of sediment heavy metals (i.e. Cu, Cd, Fe, Pb and Zn) in effluent channels of Yar Dantse, Yar Dole, Yar Rumfa and receiving new Gusau reservoir, and to evaluate the geochemical factors that control the distribution pattern of the studied heavy metals and assessment of contamination status of the sediments.

2. Materials And Methods

2.1. Study Area

The new Gusau reservoir is located in Gusau Local Government Area of Zamfara State, Nigeria. Between latitude 12°17'02.40" N and longitude 6°39'50.83" 6°66'41.20" E, and occupies an area of 3,364km (1,298.8sqmi). Gusau Local Government had a population of 383,162 people (NPC, 2006). The new Gusau reservoir is gated in the channel of Sokoto River; it is made up of a mass concrete weir surmounted by five steel gates which is operated by electric motors and bar-link chains with provision for emergency manual operation. Concrete walls are also provided at each end of the reservoir, to protect the ends of the reservoir from erosion (Jabbi *et al.*, 2018).

2.2. Experimental Design

The experimental based studies are quantitative in nature. Sampling was based on purposive sampling method, for easy accessibility and their suitability for future studies. The study involved sampling of effluents from nine (9) sampling points of 500m distance YDS, YDL and YRF. Five (5) selected sampling points of 500m distance between them in the receiving reservoir (NGR) (Fig. 1) and (Table 1) A total 756 samples were collected and the sampling was done on monthly basis in triplicate.

2.3. Determination of heavy metals in sediments

The method described by APHA (2005) and Udo*et al.* (2009) was used in determination of selected heavy metals (viz; cadmium, copper, iron, lead and zinc). This involved the use of Ground sediment sample of 1g mass placed in a 250 ml conical flask, 10 ml of concentrated nitric acid was added and mixture digested on a hot plate to near dryness. The residue was further digested with a mixture of

concentrated acids containing HNO₃ (5 ml), and HCl (5 ml) for 10 minutes at room temperature, for breaking down organic matter and minerals and further digested on a hot plate in a fume hood to a volume of 5 ml and filtered using What man no1 filter paper, and made to 100 ml in a volumetric flask using distilled water. The filtrate was used for the determination of heavy metals, using AA240 FS Atomic Absorption Spectrophotometer (AAS). The degree of contamination from heavy metals could be evaluated by determining the contamination factor (CF), pollution load index (PLI), and geo accumulation index (Igeo).



Figure 1: Map of the Study Area showing Channels and new Gusau reservoir sampling points

2.4. Pollution load index (PLI) and contamination factor (CF)

To evaluate the sediment quality, combined approaches of pollution load index of the four metals were calculated according to (Islam *et al.* 2015) (CF). PLI = (Cf1×Cf2×Cf3.....Cfn) 1/n

Where CF is the contamination factor and n is the number of parameters .Where CF metals is the ratio between the content of each metal in the sample to the background values (background value from the average shale value) in sediment. (Ana and Zoran 2010): Cf = Cm sample/ Cm background. However, PLI value of zero indicates excellence, a value of one (1) indicates baseline level of pollutants and values above one indicate progressive deterioration of the site and estuarine quality (Tomilinson *et al.*, 1980). The PLI gave inside of the overall toxicity status of the sample and also it is a consequence of the contribution of the studied five metals. The ratio of the measured concentration to natural abundance of a given metal had been proposed as the contamination factor (CF) being classified into four grades for monitoring the pollution of one single metal over a period of time (Islam *et al.*, 2015c, and Mir *et al.*, 2016): low degree (CF < 1), moderate degree ($1 \le CF < 3$), considerable degree ($3 \le CF < 6$), and very high degree (CF ≥ 6). Thus, the CF values can be used to monitor the enrichment of a given metal in sediments for a given period of time.

2.5 Geo-accumulation index (Igeo)

The degree of sediment contamination from the heavy metals could be assessed by measuring the Geoaccumulation index (Igeo). This index has been widely used for the assessment of sediment contamination (Saleem *et al.* 2015; Ghrefat and Yusuf, 2006).

Sampling site	Coordinates: Latitude/ Altitude	Brief description			
NGR/ SP1	6.66833 12.151	It's located within the reservoir at point it received discharge from Yar Dantse effluent channel.			
NGR/SP2	6.66398 12.1522	Characterized by few floating planktons and macrophytes. Irrigation and fishing are carried out in this catchment area within the reservoir.			
NGR/SP3	6.6918 12.1476	Irrigation, farming and livestock watering are carried out here.			
NGR/SP4	6.69578 12.1494	This study area is characterized by few floating planktons. Fishing is the only human activity that is carried out in this point			
NGR/SP5	6.69205 12.1438	SP 5 is located south west from SP 4 of the reservoir, which is characterized by Irrigation and washing of clothes are the human activities carried out in this area.			
YDS/SP6	6.70017 12.1398	Sampling point 6 is located behind Abu Magaji bridge, Yar Dantse effluent channel; it received effluents from various houses with a lot of garbage and grasses around the area.			
YDS/SP7	6.66471 12.1435	This sampling point is located near water board 500m away from sampling point number 6, water treatment plans and painting of tricycle, vehicle and daily vehicle maintenances take place around the area, in Yar Dantse.			
YDS/SP8	6.66255 12.1381	This station is characterized by Big garbage dumping site, with effluent discharge from bakery house, restaurant and brick industries along Yar Dantse effluent channel.			
YDL/SP9	6.66477 12.1331	This point is per from any significant source in Yar Dole effluent channel			
YDL/SP10	6.6706 12.1458	This point is characterized by oily discharge from the garage and small agricultural activity 500 meter away from point above in Yar Dole.			
YDL/SP11	6.6712 12.1408	Its characterize by illegal refuse dumping with lots of human faeces and lots of grasses along Yar Dole channel			
YRF/SP12	6.66005 12.1547	Local Irrigation farming is the only activity carried out here along Yar Rumfa			
YRF/SP13	6.70083 12.1500	Leakages from the dumping waste site of chicken feather and digestive organs. Toxic water from photo laboratory, in addition to market refuse dumping site 500 meter away above point			
YRF/SP14	6.70017 12.1398	Characterized by few floating planktons, Human activities such as washing and Blocks production and local quarry are located in this area, along Yar Rumfa effluent channel			

Table 1. Composite Sites Sampling Point's Description and GPS coordinates

To characterize the level of pollution in the sediment, geo-accumulation index (Igeo) values were calculated using the equation, (Islam *et al.*, 2015a and Ahmad *et al.*, 2015) Igeo = Log_2 (Cn/1.5Bn), where Cn is the measured concentration of metal in the sediment and Bn is the geochemical

background value of element n in the background sample (Santos *et al.*, 2003; Yu *et al.*, 2011 and Islam *et al.*, 2015a). The factor 1.5 was introduced to minimize the possible variations in the background values which maybe qualified to lithogenic effect

2.6 Data Analysis

IBM SPSS Statistics software version 23.0 was used for the analysis of data generated from the studies. One-way Analysis of Variance (ANOVA) was used to determine the significant difference among the concentrations of the heavy metal's sediments (p<0.05). Microsoft office excel, 2012 was used for the construction of graphs/charts.

3. Results and Discussion

The results of four (4) different sampling sites from sediment heavy metals were presented in Table 1 so as to establish their concentrations. Five heavy metals viz; Copper, Cadmium, Iron, Lead, and Zinc were monitored. The mean annual and seasonal variations for the period of study of heavy metal are presented.

Parameters/	Yar Dantse	Yar Dole	Yar Rumfa	NGR	P-Value	WHO
site						
Cu (mg/kg)	$2.32^{b} \pm .127$	$3.55^{a} \pm .168$	$2.30^{b} \pm .132$	$2.31^{b} \pm .053$.000	25
Range	1.22-5.39	1.79-5.66	1.11-5.61	0.03-5.0		
Cd (mg/Kg)	$0.42^{b} \pm .053$	$0.36^{b} \pm .050$	$0.33^{b} \pm .042$	0.13 ^a ±.022	.000	0.05
Range	ND-0.97	ND-0.99	ND-0.91	ND-2.98		
Fe (mg/Kg)	$9.94^{d} \pm .278$	30.26 ^b ±1.742	21.65 ^c ±2.079	34.92 ^a ±.640	.000	50
Range	10.23-52.31	6.30-14.55	7.09-53.77	16.30-63.77		
Pb (mg/Kg)	$0.38^{a} \pm .085$	$0.24^{b} \pm .041$	0.39 ^a ±.074	$0.18^{b} \pm .019$.000	35
Range	ND-1.02	ND-1.99	ND-2.45	ND-1.54		
Zn (mg/Kg)	5.59 ^c ±.201	6.67 ^c ±.292	17.28 ^a ±1.086	12.71 ^b ±.342	.000	123
Range	2.69-9.67	3.99-11.77	3.10-27.22	1.79-25.67		

Table 2: Mean ± Standard Error and Range Values of Composite Sites Heavy Metal C	concentrations
in Sediments Recorded from February 2019–July 2020	

Means in same row with different superscripts are significantly different at p < 0.05 levels. ND – not detected, NGR –new Gusau reservoir

Copper can get into aquatic ecosystems from diverse sources such as from Cu compounds used in fungicides, algicides, insecticides, wood preservatives, electroplating and azo dye manufactures (Akan *et al.*, 2010). Also, from Cu compounds added in fertilizers and animal feeds as a nutrient to support plant and animal growth. Copper (Cu) concentration measure during dry season at Yar Dantse was $4.10\pm.237$ mg/kg, Yar Dole $2.20\pm.255$ mg/kg, Yar Rumfa $2.58\pm.220$ mg/kg and in new

Gusau reservoir 2.54±.054mg/kg. While during rainy season the result are 3.12±.208mg/kg, 2.42±.106mg/kg, 2.08±.152mg/kg and 2.13±.082mg/kg for Yar Dantse, Yar Dole, Yar Rumfa and Gusau reservoir respectively. The values are below WHO (2008) permissible limit of 25mg/kg for survival of aquatic organisms USEPA (16 mg/kg), Cu mean value did not exceed the limit. In comparison with previous studies, Cu concentration is greater Atonements shoreline (0.133 - 0.623)mg/kg) but lesser than Calabar River (21.13 mg/kg), Euphrate River (18.91 mg/kg) and World average (122.9 mg/kg). And were found to have significant difference among sampling sites, season and year at p<0.05 .it was found to be lesser than the values. In comparison with WHO (25 mg/kg), Copper compounds are also used in food additives and copper salts in water supply systems to control biological growths in the reservoirs and distribution pipes (Eaton, 2005; WHO, 2004). Copper is an essential substance to human life, however, in high concentrations, it can cause anaemia, liver and kidney damage, stomach and intestinal irritation (Turnlund, 1998). The high Cu levels in Yar Dantse site could be attributed to agricultural activities in the catchment especially the use fertilizers, fungicides and insecticides. During the rainy season Cu compounds added in fertilizers and animal feeds get into the channels and the receiving reservoir via surface runoff. The mean levels of Cu in the study area were below the WHO standard values of 25 mg/kg for the survival of aquatic organisms (WHO, 2004). In Lake Kanyaboli, mean Cu concentration levels ranging from 1.80 -30.27 mg/kg have been observed (Ochieng et al., 2008). Heavy metal contamination studies done in Lake Victoria, found higher mean levels of Cu in sediments (3.90 – 150.2 mg kg-1) than obtained in Masinga reservoir (Ochieng et al., 2008) In this study Cadmium(Cd) concentration measured during dry season was found to be 0.44±.081mg/kg, for Yar Dantse, Yar Dole 0.53±.085mg/kg, 0.44±.077mg/kg Yar Rumfa and new Gusau reservoir was 1.28±.092mg/kg, the values decreases during the rainy season due to dilution factor where we have 0.31±.061mg/kg Yar Dantse, 0.32±.063mg/kg Yar Dole, 0.25±.040mg/kg Yar Rumfa and 0.19±.084mg/kg for YDS, YDL, YRF and NGR respectively Table 1. The differences in Cd concentration may be due to difference in sampling season. Both season results are higher than WHO 2006/NESREA 2007 standard of 0.05mg/l. Analysis of variance showed significant variation existed between seasons, sites and year (p<0.05)This high Cd. concentration above standard may be as result of oil discharge from Allah gatankowa mechanic village in the area, fertilizer from point and non-point source and metal base pesticide. The value is higher than previous study by Lekwot et al., (2012) where it was found that cadmium was very low with upstream value 0.002mg/l, downstream 0.0004mg/l and at discharge point 0.024mg/l. Iron (Fe) concentration measure during dry season at Yar Dantse was 28.49±2.314mg/kg, Yar Dole 8.84±.291mg/kg, Yar Rumfa 14.67±2.116mg/kg and at new Gusau reservoir 28.84±.779mg/kg Table 4.9. While during rainy season the result are 34.08±2.336mg/kg, 10.82±.375mg/kg, 27.23±2.995mg/kg and 39.78±.766mg/kg Table 4 for YDS, YDL, YDF and NGR respectively. The result shows it is lower than NESREA 2007 and WHO 2006 standard (50mg/kg). When compared with the world surface rock average (35900 mg/kg) and mean shale concentration (46700), it was found to be lesser than the values. In comparison with USEPA (30 mg/kg), Fe mean value did not exceed the limit. In comparison with previous studies, Fe concentration is greater Asejire Lake (2.392 mg/kg) and Calabar River 6.00 mg/kg) but lesser than Ase River (2.392 mg/kg), Ibeno coastal (22.18 mg/kg), Euphrate River (2249.47 mg/kg) and World average (57405.9 mg/kg). The CF values of Fe are > 6 in all sampling sites, this suggest that these sites are highly contaminated. The Igeo values of Fe ranged from 1.64 to 2.73 which is greater than 0. This implies that sediment quality of this study sites is polluted in all sampling sites. Analysis of variance showed significant variation existed between seasons, sites and year (p<0.05). From previous study by Abui

(2012) it was observed that iron concentration in both dry and rainy season is very low with 0.9 mg/l and 0.40mg/l upstream, 0.92mg/l for both seasons at discharge point, 2.66mg/l downstream at both seasons because of the difference in sampling period of the study. It is low at upstream and downstream but increases downstream above the permissible limit. The mean variations in lead concentration for all sampling sites are presented in Table 2. The mean values of lead concentrations in dry season were recorded as 0.26±.055 at Yar Dantse, 0.23±.107 Yar Dole, 0.34±.098 Yar Rumfa and $0.16\pm.027$ for Gusau reservoir respectively and analysis of variance showed significant variation existed between seasons, sites and year (p<0.05) Table 2. These values are below (WHO, 2004) the permissible limit of 35mg/kg. Previous study done at river Romi by Lekwot et al. (2012) observed that an average mean value in water samples of lead is 0.11mg/l. Butu (2002), also observed low concentration of lead in Galma dam. High concentration of Pb in Yar Dole effluent channels could be due to some chemicals which contain lead such as petroleum products that have been discharged into the channel from mechanic village and filling stations. The sources of Pb in sediments include industrial wastes and from water pipes (Akan et al., 2010). Other likely sources of Pb are lead acid batteries, solder, alloys, cable sheathing, pigments, rust inhibitors, ammunition, glazes and plastic stabilizers (WHO, 2004). Mean Pb levels in Gusau reservoir (0.18-0.39 mg/kg), were within the range recorded in Rift Valley lakes of 10.92 – 18.98 mg/kg (Ochienget al., 2007). When compared with the world surface rock average (16 mg/kg) and mean shale concentration (20 mg/kg), it was found to be lesser than the values. In comparison with USEPA (40 mg/kg) and CCME (35 mg/kg), Pb mean value did not exceed the limit. In comparison with previous studies, Pb concentration is lower in Abonnema (0.0075- 0.0520), Asejire Lake (0.0740 mg/kg), Ibeno coastal (0.05 mg/kg) and Oil exploration zone (0.045 mg/kg) and lesser than Calabar River (16.58 mg/kg), Euphrate (22.56 mg/kg) and World Average (230.75 mg/kg). Ochieng et al. (2008) found higher mean levels of Pb in sediments of different sampling sites within Lake Kanyaboli (11.42 - 153.90mg/kg) and Winam Gulf (3.09 – 66.05mg/kg). The contamination factor (CF) values of Pb from this study ranged from 0.12 to 0.26. These values for Pb are lesser than 1 in all sampling sites. The mean variations in Zn concentration for all sampling sites are presented in Table 1. The highest and the lowest zinc concentrations in dry season were recorded 5.82±.255 Yar Dantse, 6.34±.282 Yar Dole, 19.61±1.234 Yar Rumfa and 14.38±.404 Gusau reservoir. Analysis of variance showed significant variation existed between seasons, sites and year (p < 0.05). The source of Zn concentrations in sediments to the reservoirs could be from a number of alloys including brass and bronze, batteries, fungicides and pigments (Akan et al., 2010). Zinc is also used in galvanizing steel and iron products hence a possible source from the urban areas within the Yar Dole and Yar Rumfa area and Gusau reservoir catchment area. Another source could be Zn Carbonates used as pesticides (Anglin-Brown et al., 1995) and textile industries' waste waters (Smith, 1988). The elevated Zn values recorded at Yar Rumfa site sediment may be attributed to printing and dyeing processes in Zamfara textile industries, located within the area where zinc is part of the chemical used. The results obtained from the study Zn concentration levels in all the sampling sites did not exceed the WHO recommended limit of 123mg/kg (WHO, 2008). However, sediments have the capacity to accumulate more heavy metals than water, with time and remobilize them back to water and the food chain (WHO, 2008). Compared to other studies, mean Zn levels in Gusau reservoir were lower than 96.2 to 229.6 mg/kg recorded in five Rift Valley lakes, Kenya (Ochieng et al., 2007). But they were within the mean Zn levels recorded in Lake Kanyaboli (65.0 – 146mg/kg) and 23.39 – 350.80 mg/kg at Winam gulf (Ochieng et al., 2008). When compared with the world surface rock average (127mg/kg), mean shale concentration (95 mg/kg), the value did not exceed the limits. In comparison with WHO (123mg/kg),

USEPA (110 mg/kg) and CCME (123mg/kg), Zn mean value was found to be less than these values. In comparison with previous studies, Zn concentration was greater than Abonnema (1.0535 - 7.0965 mg/kg) and Ibeno (0.05 mg/kg) but lesser than Ase River 12.46(mg/kg), Calabar River (115.58 mg/kg), Euphrate River (48.00 mg/kg) and world average (303 mg/kg). The CF values of Zn ranged from 1.79 - 11.52. These values for Zn were greater than 1 in all sampling sites, this suggests that these sites are range from low degree to very high in contamination.

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Sampling sites/ metal	Cu	Cd	Fe	Pb	Zn	PLI	
YDS	0.26	0.27	6.62	0.25	1.79	0.04	_
YDL	1.54	0.24	20.17	0.16	2.66	0.63	
YRF	2.36	0.22	14.43	0.26	11.52	4.48	
NGR	1.54	0.08	23.28	0.12	8.47	0.58	

 Table 3: Composite Sites Sediments Heavy Metals Contamination Factor (CF) and Pollution Load
 Index (PLI)

The calculated pollution load index (PLI) values of metals in sediments are summarized in Table 3. The PLI values were ranged from 0.04 to 4.48 confirming that the sediment of the studied reservoir was not contaminated (PLI < 1) except the sediment from Yar Rumfa (YRF) which is considerably polluted ($3\leq$ CF<6). The PLI provide pollution status, and information about the quality of the sediment. In addition, it also provide essential information to the decision makers on the pollution status of the study area (Suresh *et al.*, 2012) The contamination Factors of the trace metals in the Gusau reservoir and channels sediments revealed that Cd and Pb have lower contamination factor, while Fe has very high sediments contamination in all the sampling sites High concentration of Fe could be due to some chemicals which contain Fe such as agro chemicals products that have been discharged into the channels and reservoir during rainy and dry season farming (Akan *et al.*, 2010)

Sampling sites/ metal	Cu	Cd	Fe	Pb	Zn	Igeo-total
YDS	-1.17	-1.13	1.64	-1.20	0.50	-1.36
YDL	0.37	-1.23	2.60	-1.59	0.84	0.99
YRF	0.74	-1.31	2.31	-1.17	2.12	2.69
NGR	0.37	-2.19	2.73	-1.84	1.85	0.92

Table 4: Composite Sites Heavy Metals Sediments Geo-accumulation indices (Igeo

The Igeo values of Zn, which ranged from 0.5 to 2.12 and Fe, which ranged from 1.6 to 2.7, are more than 0. According to Table 4, sediment quality effluent channels and new Gusau reservoir sampling locations are comparatively contaminated with iron and zinc.

Conclusion

The mean concentration values of all the heavy metals were below the WHO, world surface rock average, mean shale concentration, Except Cd which is higher than WHO 2006 and NESREA 2007

standard of 0.05 mg/kg. The CF values of Fe and Zn are > 1- 6 in all the sediment sampling sites, ranging from low to a very high degree of contamination. The PLI values were ranged from 0.04 to 4.48 confirming that the sediment of the studied reservoir was not contaminated (PLI < 1) except the sediment from Yar Rumfa (YRF) which is considerably polluted ($3 \le CF \le 6$). The PLI provide information on quality of the sediment. In addition to essential information to the decision makers on the pollution status of the study area.

Acknowledgement: This is to acknowledge the technical support of Mal. Aminu Umar of Soil sciences Laboratory, BUK. Mr. Abdurrahman Babatunde and Loveleen, C.o., of Department of Biological Science laboratory, FUGUS.

Disclosure statement: The authors declared no conflict of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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(2025); http://www.jmaterenvironsci.com