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A Study of Heavy Metals Pollution in the Coastal Marine Sediment of Ondo State, Nigeria

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Authors' contributions

This work was carried out in collaboration among all authors. Author APO designed the study and carried out the sampling. Authors APO and OLT performed the statistical analysis, wrote the protocol, managed the literature searches and wrote the first draft of the manuscript. Author AIF managed the analyses of the study. All authors read and approved the final manuscript.

Article Information

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Original Research Article

ABSTRACT

The present study was carried out to assess the pollution status of heavy metals in the marine sediment in Ondo coastal area. A total of 36 sediment samples were collected from the three locations (Awoye, Abereke and Ayetoro) once in a month in 2015 covering both dry and wet season. The sediment samples were subjected to digestion and Atomic Absorption Spectrophotometer (AAS) was used to measure the concentrations of As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, V and Zn. The measured concentrations data were used for evaluating the contamination level and accumulation status of heavy metals in sediment by employing several pollution indices: contamination factor (CF), degree of contamination (*Cdeg*), pollution load index (PLI), enrichment factor (EF) and index of geoaccumulation (*Igeo*). The results showed that Fe had the highest average concentration values of 248.00*§* 20.00 and 324.33*§* 5.80 mg/kg at Awoye sampling site for wet and dry season, respectively while the highest mean concentration of Mn were 271.77*±*9.50 and 295*±*10.06 mg/kg at Abereke and Ayetoro, respectively. The contamination factor (CF) values for As, Cd and Mn were 1

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< CF *<* 3, indicating a moderate contamination. The results of *Cdeg* were ranged from 8.6 to 12.5 for both dry and wet season except in Ayetoro site that had 7.8 in the wet season. The PLI values were 0.47, 0.50 and 0.54 (*<*1) for Abereke, Awoye and Ayetoro sites, respectively, denoting unpolluted conditions. The EF values for Pb and Ni obtained from all the sites ranged from 20.8 to 38.9, showing their significantly enriched in sediment. The *Igeo* values of As, Cr, Fe, V, Zn and Cu were less than one (*Igeo <*1) for the three sampling locations in both seasons, implying that the sediments in the study area are practically uncontaminated by these metals. Pb had the *Igeo* values of 2.80 and 2.85 during dry and wet season at Awoye sampling site while Ni had 2.57 at Abereke only in the dry season. The heavy metals in sediment might be derived from the upstream rivers from the top soil, mechanically weathered rock materials and anthropogenic activities.

Keywords: Sediment; heavy metals; pollution; indices.

1. INTRODUCTION

Sediment is a mixture of weathered materials and mineral species from the parent rocks. It contains organic debris that are transported by detrital process from the upstream and deposited at the river bed [1]. Sediment, because of their variable physical and chemical properties acts as repository for heavy metals in the aquatic environment [2]. Naturally, se-diment pollution due to heavy metals are caused by geologic weathering of the bedrock and direct atmospheric deposition. The interactions between water and crustal materials with which water is in contact is a major process by which heavy metals content in the sediment can be increased [3]. The anthropogenic activities such as sea transportation, energy generation and utilization, dredging, fishing, oil exploration, farming, infrastructural oil exploration, farming, infrastructural development and mining are crucial forsocial-economic reasons [4], however, they are probable sources of sediment pollution when their wastes are discharged into the river body by urban surface water runoff [5]. The increased concentration and accumulation of heavy metals alter sediment quality as well as the food stuff available for the aquatic organism, leading to loss of aquatic biodiversity [6].

In recent years, heavy metals pollution of the aquatic environment has become a worldwide problem due to their persistence in nature and their capacity to accumulate in living organism [7]. Ahmadipour et al. [8] reported that heavy metals have negative health implications due to their toxicity potential and tendency to accumulate in environmental media such as water, soil, sediment and other biological media. Several heavy metals persist in the sediment with varied concentrations most especially in fine grain [9]. The increased concentration of the heavy metals in the sediment has adverse health implication due to possibility of entering the food chain through aquatic organism uptake and direct consumption of aquatic organism by man [10]. Polluted sediment can act as a metal pool. thereby release metals to the overlying water column via natural or anthropogenic processes, causing potential adverse health effects to the ecosystem and degrade the sediment quality [11]. Heavy metals are not destructible and nonbiodegradable, thus they may exist in an environmental medium for a long period of time [12]. Moreover, heavy metals in the sediment can undergo sorption and complexion, ion exchange, dissolution and precipitation reactions which influence their behavior and bioavailability during transportation [13].

The capacity of the sediment to accumulate heavy metals make them relevant indicators for monitoring purposes as well as the evaluation of their pollution levels and patterns of the aquatic system [14]. Based on these, several studies had been conducted on degradation of sediment quality due heavy metals using several pollution indices [15,16,17,18] while few studies were based on heavy metals speciation and potential bioavailability in the sediment. In the study area, quite a few studies had been undertaken on the distribution of heavy metals in the sediment [19]. In this study, the concentrations of heavy metals in the sediment samples were determined using Atomic Absorption Spectrophotometer (AAS) technique. This was with a view to assessing the pollution status due to heavy metals in the sediment. The main objective of this work was to assess the level of heavy metal pollution in the sediment samples by employing the combination of pollution indices: contamination factor (CF), degree of contamination (D*deg*), pollution load index (PLI), index of geo-accumulation (I*geo*) and enrichment factor (EF) analysis.

2. MATERIALS AND METHODS

2.1 Study Area

Ondo State coast is strategically located along the gulf of guinea as the Transgressive Mud Beach (TMB) lying in the east of the West African lagoon system and on the west of the Benin Flank of the Niger Delta Basin. Most of the creeks and rivers (Omila and Edo) in the inland area are drained into the Atlantic Ocean through Awoye and Abereke estuaries. Also, the sandy beach materials of the Nigeria coastal plain are replaced by mud and lacustrine deposits in Ondo state coast. Other unique features of this coast are overlying mud of about 60 m and massive incursion of the sea into the inland swamps. It is one of the longest coastlines in Nigeria (more than 10 % of the country coastline) which favors fishing activities in the riverine areas. The major anthropogenic activities around the study area are transportation services, seismic investigations, crude oil exploration, commercial and agricultural activities. The study area lies within the tropical rain forest zone and bounded by the latitude 5.56 - 6.30[°]N and longitude 4.40 -5.43[°]E with 21.38 m above the sea level. The study area is characterized with two distinct seasons namely: dry and wet season. The dry season spans through the month of November to March while wet season occurs between April to

October with the annual rainfall of about 3000 mm [20].

2.2 Sample Collection and Preparation

Sediment samples were collected at approximately 2.0 nautical mile from the Ondo state coastline at 10 m depth using a VanVeen grab sampler. A representative samples were taken with plastic spatula from the middle of the grab bulk sediment. Samples were collected once in a month from January to December, 2015. Three sampling locations were chosen based on their proximity to various anthropogenic activities such as oil exploration, ship breaking yard, market activities and fishing and the areas of incursion of the estuaries into the Atlantic Ocean. Fig. 1 shows the map of the Ondo section of the southwest coast of Nigeria with the study locations (Abereke, Ayetoro and Awoye) and other settlements along the coast. After sampling, each sample was transported to the laboratory and air dried at room temperature for two weeks, then grounded in a pre-washed agate mortar and pestle. In order to remove coarse debris, the samples were screened through a 100 µm stainless steel sieve. The samples of the fine-grained sediment were retained for chemical analysis. All the sampling materials were washed with water and clean with acetone after each sampling to avoid possible contamination.

Fig. 1. Map of the study area showing the sampling sites

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2.3 Elemental Analysis

The samples were subjected to wet digestion in Foss Tecator digestion vessel. One (1) gram of sediment sample was added in a 15 mL mixture of nitric (HNO3) and perchloric acid (HClO4) in ratio 1:3. The whole content was placed on a heating electric plate at about 334 - 350 $\mathrm{^0C}$ for 2 hours. The mixed solution was boiled until the evaporation of the acid solution and a clear solution was obtained. Upon cooling, the solution was filtered to a 100 mL volumetric flask and then diluted to 50 mL with distilled de-ionized water and kept in the sample bottle at 4^0C before analysis. The concentrations of As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, V and Zn were determined using Atomic Absorption Spectrophotometer (Buck Scientific VGP 210 Model) at the Central Science Laboratory (CSL), Obafemi Awolowo University, Ile-Ife, Nigeria. All reagent were of analytical grade. The operating conditions consisting of maximum wavelength *λmax*, energy, current and slit width of the ASS instrumental analytical set up are presented in table 1. The detection limits of As, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Ni were 0.05, 0.001, 0.005, 0.001, 0.005, 0.001, 0.003, 0.003 and 0.002 mg/kg, respectively. The heavy metal concentrations data were subjected to descriptive statistic using the statistical package for social science software (SPSS 16.0).

2.3.1 Contamination factor and degree of contamination

The contamination factor (CF) is a single pollution index which indicates the contamination level of a particular toxic substance or elements in a given environmental medium [21]. In this study, the sediment contamination due to As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, V and Zn were assessed using the contamination factor (CF). The CF of *i th* heavy metal was calculated as:

$$
C_{\ j}^i = \frac{C_{\ s}^i}{C_{\ m}^i} \tag{1}
$$

where C_s^i is the mean concentration of the element i^{th} . $C \frac{i}{m}$ is the background concentration which is the maximum level of that metal in a given, beyond which the medium is said to be contaminated [22]. In this study, Taylor and McLennan [23] continental crustal average data was used as the background concentration. The CF is classified into four main groups: CF *<*1 means low contamination; 1*≤* CF *<* 3 means moderate contamination; 3 *≤* CF *≤* 6 implies considerate contamination and CF *>* 6 indicates very high contamination [10]. The degree of contamination (C_{deg}) is an indicator that shows the extent to which the environmental media is polluted. The C_{deg} is estimated as the sum of the contamination factors $(C_{deg} = CF1 + CF2 + CF3$ + *CFn*) of all the heavy metals in the sediment for a site. Four major category of the degree of contamination have been identified: *<* 8, 8-16, 16-32 and *>* 32 indicate low, moderate, considerable and very high degree of contamination, respectively [22].

2.3.2 Pollution load index

The pollution load index (PLI), as proposed by Tomlinson et al. [24], is a simple mathematical model use to determine the pollution status of a particular site taking into consideration the metal concentrations of that site as a single value. In this study, the PLI was evaluated as:

$$
PLI = \sqrt[n]{CF_1 * CF_2 * CF_3 *CF_n}
$$
 (2)

CF is the contamination factor of each of the heavy metals obtained from equation 1 while n is the number of heavy metals under consideration. The PLI values could be < 0 , > 0 and 1 implying unpolluted condition, progressive degradation in the sediment quality and the occurrence of baseline pollution level, respectively [24].

Elements	λ _{max} (nm)	Current	Energy	Slith width
As	193.00	10	75	0.7
Cd	228.80		69	2.7/0.80
Cr	357.87	25	97	2.7/0.80
Cu	324.75	15	80	2.7/0.80
Fe	248.33	30	66	1.8/1.35
Mn	279.48	20	62	1.8/1.30
Ni	232.00	25	67	1.8/1.35
Pb	283.31	10	75	2.7/1.05

Table 1. The operating conditions of the AAS instrumental set up

2.3.3 Enrichment factor (EF) analysis

The Enrichment Factor (EF) is used to evaluate the magnitude of the elements in air, water, soil and sediment samples. It provides information on the relative abundance of species in a given medium to the background values. It is also employed to assess the degree of pollution and to differentiate the elements of anthropogenic and natural sources [25]. Its approach is based on the standardization of the measured element against a reference element. The *EF_x* was estimated using the equation:

$$
EF_x = \frac{\left[X \frac{1}{2} E_{s (ref)}\right]}{\left[X \frac{1}{2} E_{s (ref)}\right]}
$$
(3)

Where EF_x is the enrichment values for the element x. *Xs* and *Xc* are concentrations of the element of interest in the sample and in the crust while $E_{s(ref)}$ and $E_{c(ref)}$ are the concentrations of the reference element used for normalization in the sample and in the crust. A reference element is often characterized by low occurrence variability and stable chemical properties. The common reference elements for EF analysis are Sc, Mn, Ti, Al, Si and Fe. In this study, Fe was selected as the reference element due to its lithogenic origin and abundance. Taylor and McLennan [23] continental crustal average data was adopted as the background concentration. The EF values were categorized as *<* 2, 2-5, 5- 20, 20-40 and ≥40 and considered as deficiency to minimal, moderate, significant, very high and extremely high enrichment, respectively [26].

2.3.4 Index of geoaccumulation

The index of geoaccumulation (*Igeo*) was originally proposed by Muller [27]. It is a quantitative index which indicates the metal contamination in soil, water and sediment [1]. The (I*geo*) compares the measured concentration *cm* of the elements in a given sample with the geochemical background concentrations B*^m* for the element *m* in the sample [27]. The *Igeo* was calculated using the logrithimic function:

$$
I_{geo} = \log \left[\frac{C_m}{1.5_{Bm}} \right]
$$
 (4)

Taylor and McLennan [23] continental crustal average data was used as the background elemental concentrations. The factor 1.5 was introduced to minimize the effect of possible variations in the background values, B_m , which may be attributed to the lithogenic variations in the sediment [1,22,27]. In this study, *Igeo* was estimated using the seasonal average values of the measured elemental concentrations. The categories of *Igeo* values are *<*0, 0-1, 1-2, 2-3, 2- 4, 4-5 and *>*5 and their respective interpretations are practically unpolluted, unpolluted to
moderately polluted, moderately polluted, moderately polluted, moderately polluted, moderately to strongly polluted, strongly polluted, strongly to extremely polluted and extremely polluted [28].

3. RESULTS AND DISCUSSION

3.1 Average Elemental Concentrations of Heavy Metal Results

Table 2 shows the results of the seasonal average concentrations of As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, V and Zn. Fe had the highest average concentration values of 248.00±20.00 and 324.33±5.80 mg/kg at Awoye sampling site for wet and dry season, respectively. The highest mean concentration of Mn is 271.77*±*9.50 and 295*±*10.06 mg/kg at Abereke and Ayetoro, respectively. The high concentrations of Fe and Mn are most likely related to the local mineralogy
and lithogenic origin, rather than the and lithogenic origin, rather than the anthropogenic activities. Fe had been reported as being an elements whose origin is fundamentally natural and one of the most common elements in the earth's crust [29]. The mean concentrations of Fe and Mn were similar to high values of 463.0 and 321.4 mg/kg for both wet and dry season reported by Iqbal and Shah [30] in the sediment samples obtained from Khanpur Lake, Pakistan. A few difference in seasonal concentrations of Cd, Cr, Ni, V and Zn could be related to their regional deposition and the heavy metal accumulation rate in the sediment. The erosion activities, runoff by the action of water and land based sources might introduced heavy metal into the water body. Atmospheric deposition of metals from anthropogenic sources has been reported to be the probable sources for the variation in metal concentrations in the sediment in study area [19]. Most industries such as oil exploration which are located around the sites discharged industrial waste directly into the water without treatment. This might contain heavy metals such Pb, Ni, V, Cr and Cd. The commercial ship and industrial flying boat for the transportation of the workers during oil exploration and seismic investigation could also release significant amount of Pb, Cd, V and Ni-containing contaminants into the sediment. In each site, the average values of Cd, Cu and Cd were lower than the World Health Organization [31] sediment quality guidelines values of 6, 25 and 123 mg/kg, respectively. These show the unpolluted conditions of the sediment by Cu, Cr and Cd. The Ni and Cr mean concentration values in each site exceeded the [31] values of 20 and 25 mg/kg, suggesting that a minute pollution due to Ni and Cr.

3.2 Contamination Factor, Degree of Contamination and Pollution load Index Results

Table 3 shows the results of contamination factor, degree of contamination and pollution load index for the wet and dry season of the three studied areas. In the dry season, Cr, Cu, Fe, Ni, Pb, V and Zn had a CF values to be *<*1, signifying the low contamination status of the sediment. The CF values for As Cd and Mn were 1 *<* CF *<* 3, indicating a moderate contamination. The results of the CF showed similar trend in the three sites both seasons indicating that CF values were less influenced by the seasons. The results of C_{deg} ranged from 8.6 to 12.5 for both dry and wet season except in Ayetoro site that had 7.8. This classified the sediment from the three sites as a moderate degree of contamination. The PLI values were 0.47, 0.50 and 0.54 (*<*1) for Abereke, Awoye and Ayetoro sites, respectively, denoting unpolluted (perfection) conditions [24]. The little seasonal variations in PLI values might be associated with upstream discharged into the water and the alteration in various phases of elements in solution. In the wet season, a similar pattern of CF, C_{deg} and PLI values were observed in the three sites. This shows that the pollution of sediment is most likely less affected by the season. Although, there might be progressive accumulation of the heavy metals in the sediment which depends on the increased manmade activities such as oil exploration and waste discharge into the river from the upstream location. The wet season in 2015 was a period of heavy rainfall, leading to high fluvial inputs. The

transport of the industrial waste alongside with the soil materials by the erosion activities into the ocean should increase the pollution of the sediment by the heavy metals concentration in the sediment during wet season. However, in this study, the results of CF, C_{dea} and PLI showed that the sediment quality was less affected by wet season.

3.3 Enrichment Factor and Index of Geoaccumulation Results

The results of the enrichment factor (EF) analysis for the three locations are presented in the Table 4. The EF values of As and Cr were *<*2 for the three sites, signifying deficiency to minimal enrichment. Zn and V had EF between 2 *<* EF *<* 5 and 5 *<* EF *<* 10 for dry and wet season, respectively, indicating moderate and significant enrichment while Cd and Mn had the EF values of 5 *<* EF *<* 20 in the three sites, classifying them as significant enrichment in both seasons. The moderate and significant enrichment of Zn and V could also be related to the anthropogenic sources such as boat exhaust systems, antifouling paints and fossil fuel from mechanized boat used in fishing and transportation which were common in the study area.

The EF values for Pb and Ni were *>* 20 in all the sampling sites at dry and wet season, indicating that the sites were highly enriched with Pb and Ni. The high enrichment of Pb and Ni might be linked to the fact that Pb and Ni are mostly anthropogenic elements associated with oil pollution which is prevalent in the study area. The enrichment of Pb might also be attributed stable isotopic nature of Pb in crustal materials and it takes a considerable period of time before an appreciable depletion can occur. The difference in the EF values of heavy metals in the coastal sediment might be related to the difference in the magnitude of input for each metal in the sediment as well as difference in the removal rate of each metal from the sediment [32,33,34]. Largely, the low EF values of As and Cr indicating that they are mostly derived from soil. The high EF values of Zn, V, Pb and Ni might be attributed to contribution from anthropogenic activities such as oil exploration in the study area. Table 4 shows the results index of geoaccumulation (*Igeo*) of As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, V and Zn. The *Igeo* values of As, Cr, Fe, V, Zn and Cu were negative for the three sampling sites in the both seasons. These indicated that the sediment in the study area are practically uncontaminated by As, Cr, Fe, V, Zn and Cu. This is in agreement with the study

of Salah et al. [1] that obtained negative *Igeo* values for most metals in the sediment samples of Euphrates River in Iraq. Also, the negative *Igeo* average concentrations of heavy metals in sediment are lower than the average crustal concentration [1]. The *Igeo* values for Cd, Mn, Ni and V were 0 *< I*geo *<* 1 implying uncontaminated to moderately contaminated [27]. Pb has the *Igeo* values of 2.8 and 2.85 during dry and wet season at Awoye sampling site while Ni has 2.57 at Abereke only in the dry season. The accumulation of heavy metal in sediment might be derived from the discharge of Omila and Edo rivers into the ocean and the mechanically weathered surface materials [35].

Generally, the results of all the pollution indices employed in this study agreed well. However, few differences exist among the pollution indices which could be attributed to their various approach of computation since the same background concentrations data were used. The CF, C_{dea} and PLI results all confirmed the uncontaminated status of most heavy metal in all the sites. The results of EF revealed that sediment were enriched by Pb, Cd and Ni but the *Igeo* values showed that the the sediments were uncontaminated to moderately contaminated with respect to Pb and moderately contaminated with Cd. Also, the EF values of Cd and Mn were 5 *<* EF *<* 20, classifying the sites as significant enriched by Cd and Mn in both seasons. Meanwhile, Cd and Mn had the *Igeo* values in between 0 and 1 (0 *< Igeo <* 1) indicating uncontaminated to moderately contaminated. This implies that the calculations of *Igeo* had more reliability than those of EF. The differences in the results of the *I_{geo}* and EF in this study might be associated with the methods of their associated with the methods of their computations. The nature of the *Igeo* computation, which involved the logarithmic function and matrix correction factor of 1.5 was quite different from enrichment factor which normalized heavy metal concentration as the ratio to another constituents in the sediment. The difference in classification and interpretation of the each class might also contribute to little differences in the results.

4. CONCLUSION

In this study, the bottom sediment samples were obtained in Abereke, Awoye and Ayetoro sites in Ondo state coastal marine area, Southwestern Nigeria in order to study the pollution status of heavy metals contents. The elemental concentration of heavy metals analysis of the sediment samples was performed using Atomic Absorption Spectrophotometer (AAS). The heavy metals concentrations data were processed using statistical analysis and pollution indices: contamination factor (CF), degree of contamination (D_{deg}) , pollution load index (PLI), enrichment factor (EF) and index of geoaccumulation (*Igeo*). Fe had the highest average concentration values of 248.00*±* 20.00 and 324.33*±* 5.80 mg/kg at Awoye sampling site for wet and dry season, respectively. Cr, Cu, Fe, Ni, Pb, V and Zn had a CF value to be *<*1, signifying the low contamination status of the sediment. The PLI values were *<* 1 for Abereke, Awoye and Ayetoro sites, respectively. The EF values of As and Cr were *<* 2 for the three sites. The *Igeo* values of As, Cr, Fe, V, Zn and Cu were negative for the three sampling sites in the both season, implying practically uncontaminated condition. Generally, the results of pollution indices due to heavy metals showed low pollution status. Despite the low pollution level, continuous monitoring of the heavy metals is necessary to provide information on the alteration in the sediment quality due to the possibility of gradual increase in heavy metals loading in the sediment.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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