

**HEAVY METALS MOBILIZATION AND BIOAVAILABILITY
IN A TROPICAL ESTUARINE LAGOON IN SOUTH-WEST
NIGERIA**

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CERTIFICATION

**This is to certify that the Thesis
“HEAVY METALS MOBILIZATION AND BIOAVAILABILITY IN A
TROPICAL ESTUARINE LAGOON IN SOUTHWEST NIGERIA”**

Submitted to the
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**For the award of the degree of
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is a record of original research carried out
By
USESE Amii Isaac
in the Department of Marine Sciences**

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CERTIFICATION

We certify that the work embodied in this thesis for the award of the degree of Doctor of Philosophy (Marine Biology) is a record of original research carried out by USESE Amii Isaac under our supervision.

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DEDICATION

This work is dedicated to the El-Shadai God, the Incomparable Father and the Memory of my Beloved mother, Late Mrs Roseline Isaac Usese (Nee Anike) whose desire was to see her girls excel academically and be exceedingly great in life!

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LIST OF ABBREVIATIONS

APHA	American Public Health Association
AWWA	American Water Works Association
As	Arsenic
Cd	Cadmium
Cr	Chromium
Cu	Copper
Co	Cobalt
Fe	Iron
Mn	Manganese
Ni	Nickel
Pb	Lead
SPSS	Statistical Package for Social Sciences
Zn	Zinc
XRD	X-ray Diffractometry
SEM	Scanning Electron Microscopy
DORM-3	Dog Fish Muscle
CRM	Certified Reference Materials
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
HPLC	High Performance Liquid Spectrometry
SQG	Sediment Quality Guidelines
ASV	Average Shale Value
WHO	World Health Organization
USEPA	United State Environmental Protection Agency
FMENV	Federal Ministry of Environment
BSAF	Bio sediment Accumulation Factor
BWAF	Bio water Accumulation Factor

ABSTRACT

Environmental issues related to heavy metals and their biological effects continuously heightens investigation into the risk associated with their presence and accumulation in aquatic ecosystems globally. This study investigated the mobilization, partitioning, bioavailability and risk associated with the heavy metal levels in the Lagos Lagoon between September 2013 and February 2015. It entailed the division of the lagoon into five zones and sampling carried out on a seasonal basis. The physicochemical and geochemical characteristics of bottom water and sediment were determined using standard methods and procedures. X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) was used to evaluate factors responsible for metals immobilization from sediment. Heavy metal (Chromium (Cr), Manganese (Mn), Iron (Fe), Cobalt (Co), Nickel (Ni), Copper (Cu), Zinc (Zn), Arsenic (As), Cadmium (Cd) and Lead (Pb)) levels in bottom water, pore-water, sediment, demersal fish (*Liza falcipinnis*, *Bathygobius soporator*, *Mugil cephalus*, *Chrysiichthys nigrodigitatus*) and macrobenthic invertebrate (*Tympanotonus fuscatus*) were analysed by An Agilent 7500c Inductively Coupled Plasma-Mass Spectrometry (ICP-MS, Agilent Technologies, Tokyo, Japan). The quality of procedure was checked using Certified Reference Materials (DORM-3 and NIST 27111). Potential mobility and bioavailability of heavy metals was determined after the fractionation of heavy metals into operationally defined phases using a five step sequential extraction procedure while Arsenic speciation in *C. nigrodigitatus* was achieved by 1:1 extraction with Methanol: Water. Risk indices and empirical Sediment Quality Guidelines (SQGs) were used to evaluate ecological and public health risks. The results from physicochemical analysis showed significant variations ($P < 0.05$) for a number of parameters and most parameters fluctuated with seasonal precipitation. Temperature although higher in the dry season did not significantly ($P > 0.05$) change as compared to salinity which had a seasonal bias. X-ray diffractometry (XRD) and Scanning electron microscopy implicated pH, salinity, redox potential (Eh), FeS, alumino-silicate and silty-clay minerals in enhancing metals immobilization and bioavailability. There were no regular or consistent seasonal variations in the levels of heavy metals in all zones sampled but heavy metals with relatively higher concentrations were also found to be the most abundant in all investigated environmental and biological media. Significantly lower concentrations of heavy metals which varied from -0.0001 mg/L for Cadmium (Cd) and 2.5550 mg/L for Iron (Fe) were recorded in all zones for bottom water irrespective of season. There was also significant variation ($P < 0.05$) in the levels of heavy metals in pore-water with relatively higher concentrations (0.005 mg/L for Cd and 72.08 mg/L for Fe) as compared to bottom water. The concentrations of observed heavy metals in sediments which rarely exceeded threshold element levels for analysed metals was in the decreasing order; $Fe > Mn > Zn > Cu > Cr > Pb > Co > Ni > As > Cd$. Generally, with only few exceptions, concentrations of heavy metals in zones 2 and 3 and some stations in zones 1 and 4 (adjoined by Iddo – Oworonshoki- Ikorodu) which are closest to the diverse pollution sources in the Lagos Lagoon was significantly higher than zones 4 and 5 (adjoined by Ikoyi – Ajah- Ibese). There were significant variations ($p < 0.05$) in metals concentration in tissues of biota except for Mn and Zn. The levels of metal bioaccumulated in biota were in the order: $T. fuscatus > L. falcipinnis > B. soporator > M. cephalus > C. nigrodigitatus$. Bio-sediment Accumulation Factor (BSAFs) showed that *T. fuscatus* accumulated higher levels than demersal fish species. Although, metals accumulation in fish did not exceed the international permissible standards suggesting that these species are suitable for human consumption, arsenic speciation in *C. nigrodigitatus* revealed bioaccumulation of toxic inorganic arsenic species (As III) in varying concentrations and calls for further investigation. From geochemical fractionation of metals in sediments, observed order of potential mobility and bioavailability of metals was; $Cd > Zn > Mn > Co > Ni > As > Cu > Pb > Fe > Cr$. Metals like Cr, Ni, Fe and Pb showed the strongest association with the residual fractions (60%–92%, 53%–67%, 24%–85% and 35%–67%) indicating significant geogenic source while Cd and Zn were found to be predominantly associated with the mobile phase (75%–99%) from anthropogenic inputs. Thus, Cd and Zn represent metal of greatest concern due to the high mobility which by implication, may enter the food chain and pose ecological risk or adverse effects to ecological receptors and humans. The evaluated pollution indices only implicated Cd and Zn around the western axis of the lagoon with very significant enrichment (Zn-19.95-32.30; Cd-14.23-134.82), high degree of contamination (C_d) for Cd (16.88-21.56) and high risk from the Risk Assessment Code (RAC) (Zn- 82.53% and Cd, 89.47%). However, based on applied sediment quality guidelines (SQG), pollution load index (PLI) and geochemical accumulation ($I_{geo} < 0$) and low bioavailability for almost all metals, the Lagos Lagoon can be said to be relatively unpolluted. This study therefore, establishes the significant role of anthropogenic sources of metals, geochemical fractionation, and primary crystal structure of sediments in controlling the release, distribution and bioavailability of metals in the Lagos Lagoon ecosystem.

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