HEAVY METAL SPECIATION IN ROADSIDE SURFACE SOILS AND VEGETATION AS A MEASURE OF ENVIRONMENTAL POLLUTION IN ILE-IFE, OSUN STATE.

BY

OGUNWALE Taiwo Olusegun

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CERTIFICATION

We certify that this research work was carried out by OGUNWALE Taiwo Olusegun with registration number (SCP10/11/R/0088) under our supervision, and approved in accordance with the partial fulfillment of the requirements for the award of Master of Science (M.Sc.) degree in Environmental Control and Management of the Obafemi Awolowo University, Ile - Ife, Osun State, Nigeria.

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_____ _____

Prof. A. O. Ogunfowokan
Supervisor

Dr. J. A. O. Oyekunle
Co-Supervisor

_____

Prof. O. O. Awotoye
Chief Examiner

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DEDICATION

This work is dedicated to the Almighty God for His love, faithfulness, kindness, provision and protection uncommonly experienced in my life especially throughout the period of this study.

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LIST OF ABBREVIATIONS, ACRONYMS AND SYMBOLS

%R	Percentage Recovery
µg/l	microgram per litre
µg/m^3	mcrogram per cubic meter
µm	micrometer
µS/m	Micro Siemens per meter
AAS	Atomic Absorption Spectrometer
AASHTO	American Association of State Highway and Transportation Official
ATSDR	Agency for Toxic Substances and Disease Registry (USA)
BCF	bioconcentration factor
BDH	British Drug House
CF	Contamination Factor
CV	Coefficient of variation
DETR	Department of Environment, Trade and the Region
Directive EC	Directive European Communities
DTPA	diethylenetriaminepentaacetic acid

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EDTA	ethylenediaminetetraacetic acid
Eh	Redox Potential
GPS	Geographic Positioning System
H_3PO_4	Orthophosphoric Acid
HNO_3	Trioxonitrate(v) Acid
HOAC	Acetic Solution
HSDB	Hazardous Substances Data Bank
IARC	International Agency for Research on Cancer
I-geo	Geoaccumulation Index
ILO	International Labour Organisation
IPCS	International Programme on Chemical Safety
Maneb	Manganese ethylene-bis-dithiocarbamate
MF	Mobility Factor
$MgCl_2$	Mangnesium (II) Chloride
MMT	methylcyclopentadienyl Manganese Tricarbonyl
Mn_3O_4	Manganese Tetraoxide
$MnCO_3$	Manganese (II)trioxocarbonate(IV)

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$MnSO_4$	Manganese (II) tetraoxosulphate(VI)
MSWI	Municipal Solid Waste Incinerator
NaOAC	Sodium Acetate Solution
NAS	National Academy of Science
$NH_2OH.HCl$	Hydroxylamine Hydrochloric Acid
NHOAC	Ammonia Acetate
NICNAS	National Industrial Chemical Notification and Assessment Scheme
NOEC	No Observed Effect Concentration
NRC	National Research Council
OECD	Organisation for Economic Co-operation and Development
$Pb(C_2H_5)_4$	Tetraethyllead(iv)
$PbBr_2$	Lead (II)bromide
$PbBrCl_2$	Lead (II) bromochloride
$PbCl_2$	Lead (II) chloride
$PbCO_3$	Lead (II) trioxocarbonate(IV)
PbO	Lead (II) oxide
PbS	Leadsulfide

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$PbSO_4$	Lead (II) tetraoxosulphate(VI)
PEC	Predicted Environmental Concentration
pg/g	picogram (10^{-12}) per gram
pH	Potential of hydrogen (Hydrogen ion index)
PLI	Pollution Load Index
PM 2.5	particulate matter with an aerodynamic diameter less than or equal to 2.5µm
PNEC	Predicted No Effect Concentration
ROS	Reactive Oxygen Species
s.d	standard deviation
SE	Water soluble plus exchangeable
TEL	Tetraethyllead(IV)
TF	Transfer Factor
TRI	Toxics Release Inventory (USA)
UKEPA	United Kingdom Environmental Protection Agency
UNEP	United Nations Environment Programme
USCS	Unified Soil Classification System
USDA	United State Department of Agriculture

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USEPA	United States Environmental Protection Agency
WHO	World Health Organisation
WSA	Weakly Specifically Adsorb

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ABSTRACT

This study investigated the speciation, mobility and pollution indices of heavy metals in the roadside surface soils of Ile-Ife and the translocation factor in some of the associated plants. This was with a view to evaluating the heavy metal pollution status of the roadside surface soils of the study area.

Ten roadside surface soil samples were collected for each of the wet and dry seasons. The soil samples were dried to constant weight and powdered using agate mortar and pestle. Sequential extraction procedure was used to fractionate seven heavy metals (Al, Cd, Cu, Mn, Ni, Pb and Zn) in the soil samples into five operationally defined geochemical fractions (exchangeable, bound to carbonates, bound to iron and manganese oxides, bound to organic matter and residual).The soil physico-chemical parameters such as pH, electrical conductivity, organic matter content and particle size distribution were also determined for each season. The concentrations of the metals studied were quantitatively determined using Atomic Absorption Spectrophotometre (AAS). Quality control measures included blank determination, recovery analysis and calibration of standards. Descriptive and inferential statistical methods were adopted for data analyses.

Among the five chemical fractions, organic matter fraction had the highest pool for the seven metals studied for both seasons. Order of decreasing bioavailability of the metals followed the trend: Organic matter > residual >exchangeable> carbonates bound >bound to iron and manganese oxides for wet season while for the dry season, the trend was: Organic matter > residual > bound to iron and manganese oxides > carbonates bound > exchangeable fractions respectively. The mobility and bioavailability of Al, Cd, Cu, Mn, Ni, Pb and Zn in the soil

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samples for wet season followed the order: Al > Cu > Mn > Cd > Ni > Pb > Zn while for dry season, the order was Pb > Ni > Al > Mn > Cu > Cd > Zn. The total metal concentrations of roadside surface soils for wet season ranged from 1.85 μgg^{-1} Al to 5331.56 μgg^{-1}Mn while for dry season, heavy metal concentrations ranged from 1.05 μgg^{-1} Al to 4945.55 μgg^{-1} Mn. Values of enrichment factor for the metals showed that the roadside soils of Ile-Ife was highly enriched with Cd, Mn, Zn and Cu. The values of translocation factor of metals in the plant samples ranged from 0.002 μgg^{-1} for the wet season to 0.713 μgg^{-1} for the dry season. The pH of the soil samples collected from roadside ranged from 2.90 at Toll Gate to 5.40 at Olonade site for wet season and dry season values ranged from 3.10 at OAU Sports Complex to 5.10 at Our Lady Junction site. The electrical conductivity (EC) for wet season of roadside surface soils ranged from 52.60 μSm^{-1} at Our Lady Junction to 65.20 μSm^{-1} at Toll Gate site, while the EC values for dry season ranged from 31.90 μSm^{-1} at OAU Sports Complex to 58.80 μSm^{-1} at Oduduwa College road, of Ile-Ife were less alkaline. The loamy sand and sandy loam textural classes predominated in the soil samples for both seasons and the percentage organic matter of the roadside surface soils of Ile-Ife for both seasons were low ranging from 0.06% to 0.13%.

The study concluded that soils of Ile-Ife were polluted with respect to Cd, Mn, Zn and Cu and unpolluted with regard to Al, Ni and Pb in all the studied metals.

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CHAPTER ONE

1.0 INTRODUCTION

1.1. Background to the Study

Globally, roads have been identified as a source of social and economic development (Bai *et al*., 2008). According to Adefolalu (1980) and Mabogunje (1980) in developing countries like Nigeria, improved road accessibility creates variety of socio-economic activities which range from mobile shops, cafés, vehicle repairs, vulcanizers and dealers in other facilitators of motor transportation. These activities send metals into the air in particulate form and the metals subsequently are deposited into nearby soils (Okunola *et al.,* 2008). Among the sources of metals in road environment, traffic emissions from vehicles, lorries, motorcycles have been identified to introduce a number of toxic metals into the environment, which are later deposited on roadsides (Ogunfowokan *et al.,* 2004, 2009; Okunola *et al*., 2011).

Highway run-off contains numerous potential environmental pollutants that can adversely affect fauna and flora adjacent to roads, the effects of which have been shown to extend a greater distance from increased daily vehicle use (Trombular and Frissel, 2001; Pliejal *et al*.,2004). The dispersal of vehicle emissions and particulates from wear and tear also varies from place to place being influenced by factors such as wind direction, local climate, topography and seasonal salt applications in severe winter weather (Marsaleki *et al.,* 1999). Potential traffic-derived pollutants include poly aromatic hydrocarbons, volatile organic compounds, heavy metals and particulates such as rubber from tyres, nitrogen oxides, ammonia and nitrous acid (Bignal *et al*., 2007).

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The term heavy metal refers to any metallic chemical element that has a relatively high density and is toxic or poisonous at low concentrations (Skoog*et al.,* 1991). Examples of such heavy metal include Hg, Cd, Ar, Cr, Cu, Pb, Zn, Mn, Fe, etc that may exist as natural component of the earth's crust. They cannot be biodegraded or destroyed and to some extent, may enter our bodies through food, drinking water and air. As trace elements, some heavy metals such as Cu, Fe, Se and Zn are essential to maintain the metabolism of the human body. However, at higher concentration, they lead to poisoning. Heavy metal poisoning could results for instance, from drinking water contaminated (eg lead pipes), high ambient air concentration near the heavy metal emission sources or through intake via the food chain (Alloway, 1990).

It has been established in Nigeria that both human being and animals are unnecessarily over-exposed to numerous environmental hazards, often as a result of gross inefficiency and negligence. These poor environmental conditions have resulted in increasingly deteriorating health condition as well as drastic reduction in the developing world (Basta, 2000).

Atmospheric transport and deposition are important processes in the global cycling of heavy metals. The atmospheric flux of heavy metal is a major component in both marine and terrestrial environment. Environmentalpollution deals with the modification of the natural and chemical environment of the earth by human activities and natural activities such as volcanic eruption, bush-fire, decaying process,etc. It has been reported that we may be experiencing a "silent epidemic"of environmental poisoning from the ever increasing amounts of metals waste emitted into the biosphere. The mining, manufacturing and disposal of metals and metal containing materials are inevitable causes of environmental pollution (Chanery *et al.,* 2002).

The trend in trace metal levels have been attributed to automobile emissions which are a major source of heavy metals, as the highest concentrations of lead and zinc were recorded in the

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commercial areas of the city known for the high traffic densities (Kakulu, 2004). Other investigators such as (Tong, 2006) recently confirmed that elevated concentrations of copper in heavily travelled highways were noted suggesting that much of the copper pollutant is probably of automotive origin. The lead concentration which vary with housing age and higher level of the contaminations in the older neighbourhoods were probably due to the accumulation of residues from leaded gasoline and lead-based paint in the past and the use of coal fire or space heating in older houses.

Most of these heavy metals are potentially hazardous. For example, the extensive literature on cadmium toxicity that result to chronic bronchitis, hypertension, renal and cardio vascular diseases has been reviewed (Tong and Lam, 2000; Pagatto *et al.,* 2001). Cadmium, zinc, and nickel originate from oils, pneumatics and old car pieces in general and manganese prevalently from natural sources. Accumulation (and distribution) of anthropogenic heavy metals in soil may depend on wet and dry depositions that convey particles from air to soil. Heavy metals are potentially toxic to human life and the environment.

Several studies have shown that metals such as lead, cadmium, nickel, among others, are responsible for certain diseases that have lethal effects on man and animals (Lawther, 1965; Gidding, 1973; Gustav, 1974). Heavy metals may also impair plant physiology by reducing respiration and growth, interfering with photosynthetic processes and inhibiting fundamental enzymatic reactions if accumulated at high concentrations. The ability of plants to accumulate heavy metals into their organs may hence be used to monitor soil pollution, and in particular the amount of heavy metals available to plants.

In the past, several authors investigated the distribution of heavy metals in roadside soil (Chen *et al.,* 2010; Xia *et al.,* 2011); grass (Caggiano *et al.,* 2001) and leaves (D'souza *et al.,* 2010;

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Huang *et al.,* 2011) emphasizing lead accumulation in soils and vegetation (Elekes *et al.,* 2010; Yangun *et al*., 2004, 2005), near highways (Vandenabeele and Wood, 1972), in small mammals (Nakayama *et al.,* 2011), human (Harmanescu *et al.,* 2011) and invertebrates (Williamson and Evan, 1972). Other authors focused their attention on heavy metals accumulation by higher plants in order to study the urban pollution (Sawidis *et al.,* 2011; Gallagher *et al.,*2008).

Man, animals, vegetation and soil act as "sink" for atmospheric pollutants (Clyde, 1971; Valkovic *et al.,* 1979; Osibanjo and Ajayi, 1980). Biomonitoring studies provide valuable information about the quantity and quality of pollutants in the environment and can be very effective as an early warning system to detect environmental changes (Seaward, 2004). The usefulness of soil and vegetation samples in detecting environmental metals has been reported by many workers. The most economical and reasonable method for monitoring heavy metal levels in the environment is by using vegetation. The high metal accumulative capacity of plants is the reason why plants can be used as biomonitors of heavy metals pollution in soil. Wenzel and Jockwer (1999), Witting (1993) and Markert (1993) worked on the basic criteria for selection of species as a bioindicator. The major criteria are species should be represented in large numbers all over the monitoring area, have a wide geographical range, be possible to differentiate between air-borne and soil-borne heavy metals i.e being able to accumulate heavy metals, be easy to sample and there should be no identification problems.

Determining the chemical form of a metal in vegetation and soil are important to evaluate its mobility and bioavailability. Chemical speciation of heavy metals allows us to determine the availability and mobility of metals in soil and vegetation. It is widely recognized that to assess the environmental impact of soil and vegetation pollution, the determination of metal speciation gives more information about the released heavy metal contaminants and further processes of

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migration and toxicity (Rauret *et al.,* 1988; Usero *et al.,* 1998). Thus, to provide reliable information on the forms of association on heavy metals regarding their availability levels and

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