# Chemical Speciation of Selected Heavy Metals In Roadside Soils At Intersections And Terminuses In Harare, Zimbabwe

Thulani Ndhlovu<sup>1</sup>, Cleopus Machingauta<sup>2</sup>

<sup>1</sup>(Department of Chemistry, Bindura University of Science Education, Private Bag 1020 Bindura, Zimbabwe) <sup>2</sup>(Department of Chemistry, Bindura University of Science Education, Private Bag 1020 Bindura, Zimbabwe) Corresponding Author: Thulani Ndhlovu

**Abstract:** Sequential extraction technique was applied for the speciation and evaluation of the bioavailability of trace heavy metals in the roadside soil of major highways and terminuses in Harare, Zimbabwe. Heavy metals studied are Cd, Pb, Zn, Cu, Cr, Fe, Ni and Mn. Analysis of these metals was done using the AAS. Results obtained from the sequential extraction technique were compared with the results obtained from aqua regia digestion and Canadian soil quality guidelines. Recoveries were found to be relatively good except for lead which was on the lower side. Speciation species were grouped into non-resistant and resistant fractions. Cd, Pb, Zn, Cu and Mn were found mostly in the non-resistant fraction that is potentially bioavailable to human beings. Fe, Cr and Ni on the other hand were mostly abundant in the more bound crystalline lattice. Results of the analysis indicate that Cd, Pb and Zn did not exceed the maximum permissible limits at all the sites while Cr exceeded the maximum limit at all sites. Ni and Cu exceeded the maximum permissible limits at some of the sites. Speciation of Cd, Cu and Ni was in the order residue > organic > carbonates > exchangeable > oxides of Fe-Mn.

Keywords: chemical speciation, heavy metals, exchangeable fraction, ambient atmosphere, resistant fraction

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#### I. Introduction

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The high population of vehicles on Harare roads has the potential to pollute the city and its surrounding environment during peak hours. A large volume of slow moving traffic on narrow roads spent increased time on the roads with engines running thereby polluting the environment.From the time a motor vehicle engine is engaged, the largest source of catalytic converter particulates are being deposited onto the road surface, resulting in several heavy metals being released especially platinum, palladium and rhodium [1], [2], [3]. The main reason therefore why heavy metal levels within the city are high is the stop-start of vehicles observed. Wear and tear of vehicle tyres and brake linings, congestion in areas like round about, traffic lights and junctions all contribute a lot to therelease of heavy metals to the roadside [4].

In this research, levels of heavy metals on roadside dust were used as an indication of the environmental pollution [5]. Heavy metals pollution occurs when the quantities of these elements in soils are higher than the maximum permissible concentrations, and this is potentially harmful to biological life at such locations. Their presence in the ambient air is of great concern since it poses a serious threat to human health. It was noted that finer particulate matter has the most serious threat to health effects[6], [7]. Vehicle derived particulates were studied using magnetic properties of leaf dust and found out that they are very harmful to human health[8].

The method intended for use in this project is speciation. Speciation is the identification and quantification of all the forms and species in a sample [9]–[13]. Only a few attempts have been made to evaluate the speciation of particulate metals. Previous studies have assumed that all forms of the species are equally toxic to humans. However, in this research paper, the residual specie was assumed not to contribute to the bioavailabity. The heavy metals chosen for this study were chromium, lead, copper, nickel, cadmium, zinc, manganese and iron. The reason behind their choice is that these heavy metals are extremely toxic to humans if available at high concentrations [14]. The aim of the present work was to study the speciation of selected heavy metals of top soils in the city of Harare roads and terminuses with the view of understanding the levels of potentially harmful species.

## **II. Experiment**

#### a. Materials

Sodium acetate, hydroxylamine hydrochloride, acetic acid, nitric acid, standard potassium chloride, hydrochloric acid used were of analytical grade and was purchased from Associated chemicals enterprises of

South Africa. The 1000 mg/L standards of chromium, lead, copper, nickel, cadmium, zinc, manganese and iron were purchased from Anacon cc (SA).

#### b. Equipment

The following equipment was used during the experiments; pH meter from Adwa AD1020 (Romania), Analytical balance from Adam Equipment, Hot plate from Lasany,AA spectrometerAA240 Varian Australia, Water bath Haake D1 Fisons, Germany.

#### c. Study area

The samples were collected in Harare the capital city of Zimbabwe, located in the Highveld plateau about 1500 m above the sea level. Harare is a sub Saharan city situated in Southern Africa with a latitude of 17° 49, 75' N and Longitude 31° 3.24' W.

#### d. Sample analysis

## i. Sample pre-treatment

The samples were dried in the oven at 105 °C. The drying process was stopped when the mass of soil samples was constant. The samples were then ground using a pestle and mortar before passing through a 2 mm sieve.

#### ii. Physicochemical analysis of the soil sample

The soil pH in water was analysed using a Jenway 3510 pH meter. A Jenway conductivity meter was used to analyse the conductivity of the soil. Distilled water was added to the homogenised sieved sample in a beaker in a 1:5 soil water suspension[5].

#### iii. Sequential heavy metal extraction

The procedure of Kabala and Singh, (2001) which is a modified version of Tessier et al. (1979) was further modified and used for this study. The five fractions considered for fractionation are tabulated below:

#### Table 2.1: Five fractions considered for fractionation

Fraction	Description
F1	Exchangeable form
F2	Specifically sorbed and carbonate bound
F3	Fe-Mn oxide bound form
F4	Organic matter bound form
F5	Residual form (resistant fraction)

A mass of  $2.000 \pm 0.005$  g of homogenised ground soil dust was weighed and placed in a 250 ml Erlenmeyer flask. This was followed by sequentially extractions [15]. The F1 sample was extracted with 20 ml of 1 M NaOAc at pH 7 with stirring for a period of 1 hour at room temperature before filtration under gravity using Whatman filter paper (Cat No 1541 125). This was followed by the washing of the solid with 10 ml of distilled water before the next extraction step. The washes were collected and analysed together with the filtrate from the extraction. Sample F2 was prepared by extraction from the residue using 20 ml of 1 M NaOAc at pH 5 and room temperature for 5 hours with stirring. From the residue remaining after extracting F2, 20 ml of 0.04 M hydroxylamine hydrochloride (NH<sub>2</sub>OH.HCl) in 25 % acetic acid (HOAc) (1:1) was used for the extracting F3 was treated with 15 ml of 30 % H<sub>2</sub>O<sub>2</sub> at pH 2 before the mixture was placed in a water bath at 80 °C for 5.5 hours with constant stirring. The mixture was then cooled and 5 ml of (3.2 M NaOAc + 20 % HNO<sub>3</sub>) was added and the mixture was swirled for 30 minutes followed by dilution to 20 ml with distilled water, lastly the mixture was filtered to obtain F4. The residue from F4 was dried before it was digested in a conical flask with 20 ml of aqua regia on a hot plate. The conical flask was removed from a hot plate when about 1 ml of extractant was left before diluting with distilled water to the 20 ml mark and filtering to obtain F5.

#### e. Assessment of metal contamination

#### i. Contamination factor

Assessment of the extent of contamination of the roadside dust was done using the contamination factor principle.

Contamination factor CF = heavy metal sample concentration (Cx) divided by background heavy metal concentration (Cb),

where contamination factor CF < 1 refers to low concentration;  $1 \le CF < 3$  means moderate contamination;  $3 \le CF \le 6$  indicates considerable contamination and CF > 6 means very high contamination. [5], [16], [17].

#### ii. Estimation of bioavailability of heavy metals

The results were interpreted based on their bioavailability. To explain this, species obtained from the concentrations of fraction F1, F2, F3 and F4altogether make up the non-resistant fraction while the F5 fraction make up the residual fraction. The non-resistant fraction consists mostly of the labile and easily bioavailable heavy metal species whereas the residual fraction consists of the least bioavailable heavy metal species[18].

## iii. Statistical analysis

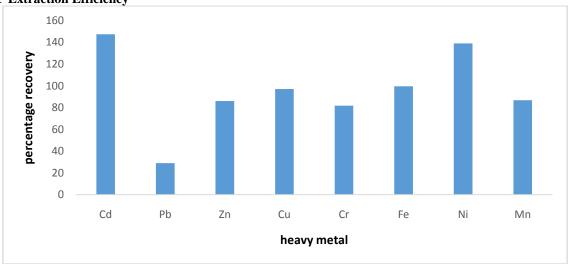
Statistical analysis of the results was done using GenStat Release 12.1. One way ANOVA was used to determine the means, standard error and list of significant differences (LSD) at 95 % confidence level. A comparison of values of the same roadside dust quality parameter was done using the LSD of means [19].

## **III. Results And Discussion**

From the different sites under study, the most important difference was in the content of clay. Though most of the soil dust samples collected was silt loam to sandy, it was however noticed that samples from charge office terminus was clay loam. The clay loom soil at charge office with the lowest pH and highest conductivity compared to the other sites under study suggest that it is acidic and has the highest content of metal ions. The physicochemical properties of the roadside dust samples collected are recorded in the Table3.1. From the pH measurements done, it could be seen that for all the twelve samples the pH did not differ much. The highest recorded was 8.04 at fourth street terminus and the lowest was 6.61 at charge office terminus. The conductivity of the same soil samples was also measured and recorded in the same Table 3.1. The highest conductivity recorded was  $3920 \ \mu \text{Scm}^{-1}$  at the charge office terminus with the lowest recorded at the intersection of second street and Herbert Chitepo Street being  $332.0 \ \mu \text{Scm}^{-1}$ .

Sample number	Sample description	рН	Conductivity (µScm <sup>-1</sup> )
1	Fourth street terminus	8.04	2010.0
2	Market square terminus	7.04	2010.0
3	Charge office terminus	6.61	3920.0
4	Copa cabana terminus	7.57	2480.0
5	Speke zebra crossing	7.39	1790.0
6	Kaguvi street	7.85	1564.0
7	Samora Machel and Chinhoyi streets intersection	7.50	402.0
8	Samora Machel and Leopold Takawira streets intersection	7.77	737.0
9	Julius Nyerere and Kwame Krumah streets intersection	7.68	762.0
10	Second and Herbet Chitepo streets intersection	7.45	332.0
11	Julius Nyerere and Kenneth Kaunda street intersection	7.94	484.0
12	Background correction sample	7.48	128.3

 Table 3.1: pH and conductivity measurements of the samples



# 3.1 Extraction Efficiency

Figure 3.1:Sequential extraction recovery efficiency

The sequential extraction percentage recoveries are represented in the bar graphs of Fig 3.1 above. The trace element fractionation efficiency was determined by calculating the sum of trace elements for each fraction using equation 1 below and compare that to the total concentration of elements determined by aqua regia digestion method. The poor lead recovery can be attributed to filtration loss during sequential extraction. The very high recoveries of cadmium and nickel can be attributed to the efficiency of the method.

: Sum = exchangeable fraction + carbonate fraction + Fe-Mn fraction + organic fraction + residue fraction (1) [20].

## 3.2 Heavy metal speciation concentration in roadsides soils

Heavy metal speciation is discussed with reference to Figs 1 up to 8 for all the elements shown below. Cadmium in road side dust in Harare is available mainly in the organic fraction at Copa Cabana with a high of 2.973 mg/kg. The Fe-Mn oxide bound form of cadmium has the lowest concentration levels at all the sites. Lead concentration at all the sites showed an increasing pattern from fractions F1 to F3 before dropping at F4 to below the concentration level of F1 before again peaking to the highest level at F5. Zinc concentration at all the sites recorded the highest concentrations for fractions F4 and F5. Chromium concentration level at sites 1 to 4 had the highest concentration levels for fractions F3 and F5. Sites 7 to 12 recorded the highest levels for fractions F2 and F5. Nickel concentration at all the sites for fractions F1 to F4 averaged 4 mg/kg. Fraction F5 had the highest concentration at all the sites. Manganese concentration at all the sites had noticeable picks of concentration levels for fractions F2, F3 and F5. Since there are no industries in the studied area, we can attribute the source of the heavy metals to be from the wear and tear, attrition of motor vehicle tyres and oil leaks. Iron recorded extremely high concentration values at all the sites under study. The residual concentration of iron represented by fraction F5 was the highest of all the fraction concentrations ranging from 41 322.3  $\pm$  517.4 to 124 947.5  $\pm$  989.8 mg/kg.

Site	Cd	Pb	Zn	Mn	Cu	Fe	Ni	Cr
1	1.04	3.89	0.62	0.72	1.59	3.19	0.27	0.95
2	1.09	5.76	0.91	0.36	6.16	1.51	0.38	1.26
3	1.16	4.01	0.97	0.30	3.33	1.24	0.37	1.57
4	2.99	4.22	0.85	0.41	1.48	0.64	0.28	1.39
5	0.96	6.48	0.69	0.29	2.11	1.62	0.40	1.08
6	0.91	12.36	1.05	0.34	7.83	5.45	0.61	1.68
7	0.92	6.37	0.33	0.29	1.57	1.50	0.24	0.97
8	0.92	12.18	0.38	0.29	2.05	1.74	0.31	0.91
9	0.95	7.59	0.28	0.29	3.34	1.67	0.42	0.99
10	1.06	9.54	1.04	0.47	3.61	1.72	0.40	0.87
11	0.96	6.51	1.21	0.32	3.42	2.47	0.34	0.92

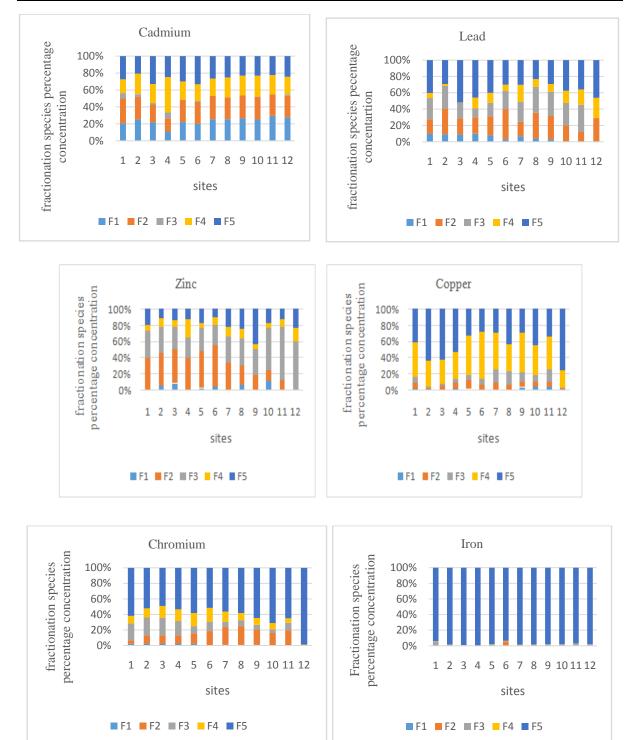
Table 3.2: Contamination factors (CF) of the elements at the eleven sites

# **3.3** Contamination factor

Reference is made to Table 3.2for the contamination factors.For cadmium, sites 5, 6, 7, 8, 9 and 11 were lowly contaminated with the highest CF value of 0.96 for sites 5 and 11. Sites 1, 2, 3, 4 and 10 were moderately contaminated by cadmium with the worst being site 4 with a CF of 2.99. The sites were either considerably contaminated with lead, where CF ranged from 3.89 for site 1 to 5.76 for site 2, or very contaminated at sites 5, 6, 7, 8, 9, 10 and 11.Eight of the sites were lowly contaminated with zinc andthree moderately contaminated. There was only a low level of contamination due to manganese and nickel at all the sites under study. With respect to copper, sites 1, 4, 5, 7 and 8 are moderately contaminated, sites 3, 9, 10 and 11 are considerably contaminated and sites 2 and 6are very highly contaminated with the worst having a CF value of 7.83. For iron, only site 4 is lowly contaminated. Sites 2, 3, 5, 7, 8, 9, 10 and 11 are moderately contaminated and sites 1 and 6 are considerably contaminated. Sites 1, 7, 8, 9, 10, and 11 were lowly contaminated whereas sites 2, 3, 4, 5 and 6 were moderately contaminated by chromium.

# **3.4** Estimation of Bioavailability of heavy metals

Generally, cadmium concentration of all fractions at all the sites averaged 0.7 mg/kg.At all the sites, the non-resistant fraction has the highest concentration of cadmium with a percentage concentration of 73.4 % against 26.6 % for the resistant fraction.Overall, lead is more available in the non-resistant fraction with a mean percentage of 61.6 % compared to 38.4 % for the resistant fraction.At all the sites zinc is more available in the non-resistant form with a percentage mean of 79.9 % compared to 20.1 % for the resistant fraction.Overall, copper is more available in the non-resistant form with a percentage mean of 56.7 % compared to 43.3 % for the resistant form.Chromium is more available in the resistant form with a percentage mean of 61.9 % compared to 38.1 % for non-resistant fraction. However, at site 3, chromium is available mostly in the non-resistant form and can harm people at the charge office terminus (site 3).Overall, nickel is more available in the resistant form with a percentage concentration mean of 54.6 % compared to 45.4 % for the non-resistant fraction. However sites 2 and 9 are mostly polluted with non-resistant nickel fraction which can harm human beings on exposure.At all the sites, manganese is more available in the non-resistant form with a percentage mean of 69.2 % compared with 30.8 % for the resistant fraction.At all the sites, iron is more available in the resistant form with a percentage mean of 97.5 % compared with 2.5 % for the non-resistant fraction and thus making it unavailable to humans.





Figures 1-8: Fractionation species percentage concentration

Table 3.3. Canadian son quanty guidennes, mg/kg				
Element	Maximum concentration accepted			
Cd	22.0			
Cr	1.4			
Cu	91.0			
Pb	260.0			
Ni	50.0			
Zn	360			
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 Table 3.3: Canadian soil quality guidelines, mg/kg

Table 3.3 above indicates some of the maximum concentration levels acceptable according to the Canadian soil quality guidelines for the protection of environmental and human health. Comparing these guidelines with the results obtained from Harare roadside dust gives an indication of how much our soils are contaminated. Roadside dust of Harare has acceptable concentration levels of Cd, Pb and Zn. This means that at all the sites studied the levels of Cd, Pb and Zn were below 22.0, 260.0 and 360 mg/kg respectively. Chromium at all the sites under study exceeded the maximum concentration level of 1.4 mg/kg. This means that the town of Harare is polluted with chromium mainly from vehicular emissions. Sites 2, 3 and 6 have their copper levels above the maximum acceptable limit of 91 mg/kg. The high pollution of soil by copper at these sites is mainly attributable to frictional tear and wear of automobile parts and tyres and breaking [21], [22]. The Ni concentration at sites 6 and 12 exceeded the maximum acceptable limit of 50 mg/kg. Site 6 has 52.6 mg/kg while site 12 has 59.5 mg/kg. These high levels of nickel can be as a result of vehicular emission [13].

## 3.5 Total concentration of trace heavy metals

A comparison of total heavy metal concentrations in the roadside soils of Harare with other cities can be done making use of Table 3.4 below. The lead levels in Harare roadside dust were third highest out of the eight cities recorded with a high of 65 mg/kg. The highest lead content of 120 mg/kg was recorded in Hong Kong and the lowest of 24.0 mg/kg in North Wales. Harare ranked the fourth city out of eight with high cadmium concentration with a high of 1.9 mg/kg and North Wales recorded the highest of 6.8 mg/kg. On Pb and Zn, Harare was less contaminated, with highs of 255.9 mg/kg and 218.7 mg/kg respectively. The highest polluter when Pb and Zn are concerned is North Wales with highs of 1779 mg/kg and 1143 mg/kg respectively.

Table 3.4: Com	parison of heav	v metal levels in i	roadsides of Harare	against other w	orld cities (mg/kg)
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City	Cu	Cd	Pb	Zn
*Harare	65.0	1.9	255.9	218.7
Amman	29.7	0.75	188.8	121.7
Lancaster	19-199	5.2	-	300-530
Hong Kong	120	1.1	991	633
North wales	24.0	6.8	1779.0	1143.0
Auckland	27.0	0.4	1650.0	180.0
London	-	4.2	1354.0	513.0
Birmingham	-	0.70	180.0	205.0

(Jaradat and Momani 1999) \*results from this project

#### **IV.** Conclusion

Cd, Pb, Zn, Cu and Mn were the most prevalent contaminants due to anthropogenic sources since they recorded a higher non-resistant percentage concentration fraction compared to the resistant fraction. Fe, Cr and Ni were most abundant as the resistant fraction available in the more bound crystalline lattice of the residue. Chromium levels at all the sites studied exceeded the maximum while Cd, Pb and Zn fell below the maximum permissible limits according to Canadian soil guidelines of 2007. Only copper levels at sites 2, 3 and 6 exceeded the maximum permissible limit. Ni levels at sites 6 and 12 are the only ones that exceeded the maximum permissible limit. As suggested by theory, sites with low pH and high conductivity values recorded high heavy metal concentrations with site 3 being an example. Harare compares very highly with other major cities when pollution levels are concerned. The chemical speciation of Cd, Cu and Ni at most sites was in the order residue > organic > carbonate > exchangeable > oxides of Fe-Mn. Fe, Mn and Zn were mostly determined in the order Fe-Mn >> residue > carbonate > organic > Fe-Mn > exchangeable. The chemical speciation of Pb and Cr was mostly in the order residue >> carbonate > organic > Fe-Mn > exchangeable. The results therefore suggest that there is a potential human risk from non-resistant fraction through ingestion, dermal adsorption and inhalation of emissions from vehicles.

#### Recommendations

From the research study, it can be recommended that this survey be carried out in many other cities of Zimbabwe. The data from the extended survey will help the regulatory authorities to come up with enough data to develop a national standard. It is this standard and not foreign adopted standards that would be used when regulating. The scope of this research study must be widened to include analysis of human samples from environments that are being covered in this research study. This would help give a co-relationship between the levels of contaminants and the exposed bodies. Guided by the findings of this research study it is recommended that on a predetermined number of times per week, people must use public transportinstead of private cars to reduce the volume of traffic and subsequently the emission which cause pollution.

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