



## Chemical Speciation of some Heavy Metals in Sand Filter Media [SFM] Waste Dumpsite in Yenagoa, Nigeria

<sup>1</sup>\*IKOKO, IL; <sup>2</sup>OSU, CI; <sup>2</sup>HORSFALL, M

<sup>\*1</sup> Bayelsa State Ministry of Water Resources, Isaac Boro Expressway, Okaka, Yenagoa, Bayelsa State, Nigeria

<sup>2</sup>Department of Pure & Industrial Chemistry, University of Port Harcourt, Choba, Port Harcourt, Nigeria

\*Corresponding Author Email: [ifemiikoko@gmail.com](mailto:ifemiikoko@gmail.com); Tel: +234 803 671 3260, +234 705 862 8765

**ABSTRACT:** The chemical speciation of five heavy metals [HMs] in spent sand-filter-media [SFM] waste dumpsite was investigated with a view to ascertain their distribution and chemical availability of the heavy metals in the dumpsite. Samples were collected at the surface, 100cm depth of the dumpsite and 100m away from the dumpsite and analysed using FAAS for total concentration of Cr, Fe, Mn, Ni and Pb. Speciation analysis was carried out using modified Tessier's sequential extraction method. The percentage concentrations showed that Pb occurred more at the exchangeable fraction with 90.6% at the top, 91.34% at 100cm depth. This signifies that Pb is biochemically available for in-take by living organisms. Fe had the highest concentration by mass 12mg/g but its occurrence of between 22-28% is in the residual/inert fraction. Ni had 40% in the exchangeable and carbonate fractions at the surface and at 100cm depth. Cr had highest exchangeable fraction of 46.29% and 45.59% at the surface and 100cm depth of the SFM. Mn had 37.30% and 42.26% at the surface and at 100cm depth of the SFM. Although Fe had highest concentration by mass, it is least biochemically available occurring more at the residual/inert fraction while the other four metals [Pb, Ni, Cr, Mn] are all biochemically available for intake by organisms and may enhance biochemical accumulation overtime and finally become harmful to living organisms around that dumpsite.

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Among the underground water contaminants, heavy metals are known to be of most concern to environmentalists because their compounds [ligands] are indeed still in reaction and are capable of many more reactions while the central metals themselves are relatively inert and can accumulate from small quantities to quite larger quantities (Dube *et al.* 2001). Further environmental concern of heavy metals reported by Wu *et al.*, (2011), are due to their prolonged persistent stay in the environment unchanged, their ability to be recycled biochemically in the same form that they were, their ability to accumulate and increase their concentration biologically, their biological toxicity to flora and fauna, their capability to undergo reduction & oxidation reactions with or without availability of oxygen, their ability to undergo precipitation, solubilisation, to aggregate or flocculate in solutions, the ability of their ligands to undergo chelation and finally their ability to put all the above together and be toxic in nature (Ademoroti, 1996). Some are carcinogenous, damage digestive systems (Fawell *et al.*, 2003), cause hallucination to even forgetfulness (Du *et al.*, 2011), lung cancer or nasal tumours and even affect DNA (Fawell *et al.*, 2007). Pb affects haemoglobin in blood and can even transfer into foetus through umbilical cord and causes adverse mental situations in small amounts (Cotruvo *et al.*, 2011). Water treatment chemicals are also a huge source and contributors to reaction by-products that lead to health

risk. Depending on pH and temperature, sedimentation/flocculation processes produces  $Al^{3+}$ ,  $Fe^{2+}$ ,  $Fe^{3+}$  by-products of organic and inorganic anions are formed (Sielechi *et al.*, 2010). Disinfection process using chlorine or Sodium hypochlorite [HTH] powder effectively produces hypochlorous acid that enables the disinfection but with other significant organic by-products like trihalomethanes [THM], haloacetic acids, haloacetonitriles, chloral hydrate, etc, while ozone disinfectant produces by-products like bromoform, monobromoac, etc are amongst the contaminants effectively in the spent SFM dumpsites. The surface mobility, biochemical redox reactions and underground bioaccumulation of heavy metal contaminants in spent or used sand filter media [SFM] and their related impact in the ecosystem is of considerable concern to chemists and other environmentalists around the world (Leizou *et al.*, 2015). The phenomenon of sorption and migration are the basic principles of the study of physical speciation or distribution in the sand filter media dumpsites (Templeton *et al.*, 2000). The study of speciation is therefore very important in the field of chemical, biological, environmental and geochemical researches. Specifically, speciation is necessary in the determination of the concentration of an element in different spots in a sampled area and gives a defined conclusion about the mobility and bioavailability of species (Leizou *et al.*, 2015). It helps to determine toxicity by measuring the concentration of some each

\*Corresponding Author Email: [ifemiikoko@gmail.com](mailto:ifemiikoko@gmail.com); Tel: +234 803 671 3260, +234 705 862 8765

species of that element as the total concentration of the heavy metal alone does not give the complete idea of the potential risk of that element in the area studied (Cui and Du, 2011, Ayadin and Kucuksezgin 2012, Beltran, et al. 2010). The Chemical Sequential Extraction CSE procedure, was first developed by Tessier *et al.*, 1979 and modified in other form by several researchers (Oake *et al.*, 1984, Campbell *et al.*, 1995). The 5-stage sequential extraction procedure established by Tessier and modified by Carliel showed extraction of stages 1 - 5; Exchangeable metal fraction, Carbonate metal fraction, Fe/Mn oxide fractions. Organic matter/Sulphide fraction and the Residual fraction. This paper focuses on the investigation of the distribution and speciation of Cr, Fe, Mn, Ni and Pb in sand-filter-media [SFM] waste dumpsites in Yenagoa, Nigeria and the biochemical availability of the metals for intake by living organisms.

## MATERIALS AND METHODS

The study city, Yenagoa is the capital city of Bayelsa State, Nigeria and lies between latitude 4° 50' to 5° 00' North and longitude 6° 11' to 6° 25' East (Amos-Tantua *et al.*, 2001, Agbalagba *et al.*, 2014). The water facility and SFM dumpsite is located at Geographic Positioning System [GPS] 4.934245, 6.266297 interpreted to read 4°56'03.3"N 6°15'58.7"E.

**Sampling:** Three distinct soil samples were obtained. Sample A<sub>TM</sub> was 2kg deposit of Sand-Filter-Media [SFM] collected on the surface of the dumpsite between 0-10cm in five (5) different points in the sample location using a soil auger. Half of these were put separately in a black plastic bag, labelled sample A<sub>TM</sub> 1, 2-5, stored in a cool place for digestion and analysis of total metal of Pb, Fe, Ni, Cr and Mn. The other half of the five portions were mixed together to get a representative sample of the entire top surface of the SFM dumpsite and put into a plastic bag, labelled as Sample A<sub>TM</sub> and stored in a cool place. Sample B was collected at a depth of 100cm in a 100cm x 100cm x 200cm vertical hole dug with a shovel down the centre of SFM dumpsite. Four separate samples were collected after the 100cm marked depth at the four corners of the 100cm x 100cm x 200cm hand-dug hole. The collected four samples from the four-corners were mixed together to get a representative sample at 100cm depth and 1kg of the representatively mixed sample was put into a plastic bag and stored in a cool place and labelled Sample B. Sample C was collected at the surface of the top soil of an unoccupied land space at a distance of about 100m away from the SFM dumpsite, as a control sample. About excess of 1kg of each sample was collected, stored in a cool place

**Sequential Extraction:** Chemical speciation using 5-stage extraction procedures and the extraction reagents were prepared using standard procedure and analytical grade reagents as specified in Tessier *et al.*, (1980) and modified by Oake *et al.*, (1984) and summarised by

Carliel and Wheatley (1997) based on the five pillars of extraction being the exchangeable metals, adsorbed metals, organic-bound metals, metal carbonates and Sulphides. The fractions were obtained using 1g of each portion of samples A, B, and C. 1g of the samples were sieved, weighed and put into 100ml sampling bottles. To each sample bottle, 16ml of MgCl<sub>2</sub> at pH 7 was added and allowed for 1 hour at room temperature. The mixture was shaken carefully throughout the period and the extract carefully decanted into 15ml analytical vessel and labelled A1, B1 and C1 representing the fraction of Exchangeable Metals. The extractant removed the metals bound by exchange sites in the SFM such that when the sample is saturated with Mg<sup>2+</sup>, exchangeable metals would be displaced from the exchangeable sites. To the residue after decanting, 16ml of 1M Sodium acetate was added and then acetic acid buffer was added to bring the pH to 5. This mixture was continuously shaken at room temperature for a period of 5 hours and then extracted to remove the Metal Carbonate fraction. The extract was decanted into 15ml sample bottles and labelled A2, B2 and C2. This fraction represents mainly the microbial uptake of metals as it evolves due to microbiological activities in the sediments/sludge due to available CO<sub>2</sub>. For the Fe/Mn oxide fraction, 20ml of 0.4M NH<sub>2</sub>OH.HCl hydroxylamine Hydrochloride in 20% v/v acetic acid HOAc was added to the residues of A, B and C after decanting fraction 2. The mixture was heated to about 96°C in a water bath and kept steady for 6 hours. The extract was decanted into 15ml sample bottles, labelled A3, B3 and C3. To the residue from fraction 3, 3ml of 0.02M HNO<sub>3</sub> was added to 5ml of 30% v/v H<sub>2</sub>O<sub>2</sub> and adjusted to pH 2. The mixture was added to the residue from fraction 3 and heated in a water bath to 85°C for a period of 2 hours with occasional shaking and then allowed to cool. Thereafter, another 3ml of 30% H<sub>2</sub>O<sub>2</sub> which was adjusted to pH 2 with HNO<sub>3</sub> was added to the mixture. This was then heated again to 85°C for another 3 hours with occasional shaking and allowed to cool. Lastly, 5ml of a mixture of 3.2M NaOAc mixed with 20% HNO<sub>3</sub> which was diluted to 20ml volume with de-ionised water, was added. The mixture was decanted and the extract was put into 15ml sample bottle, labelled as A4, B4, and C4 representing the Organic/Sulphide fraction. Lastly, the residual or inert residual from fraction 4 was transferred to a crucible and dried in an oven at 105°C. Thereafter, a mixture of 5ml conc. HNO<sub>3</sub> 70% w/w with 10ml HF w/w 40% with 10ml of HClO<sub>4</sub> 60% w/w, was added in a fume chamber. The mixture was allowed to cool and 10 ml of distilled water was added. The mixture was filtered into 100ml volumetric flask and de-ionised water was added to make it to 100ml mark. A portion of 15ml was again transferred to a 15ml sample bottle and labelled A5, B5 and C5 representing the residual metal fractions. Analysis of the heavy metals concentration for lead (Pb), Iron (Fe), Nickel (Ni), Chromium (Cr) and Manganese (Mn) was done using a Buck Scientific

210 VGP Flame Atomic Absorption Spectrophotometer FAAS following prescribed procedures.

*Statistical Evaluations:* Evaluation of relationship of results using calculations of the Pearson's Correlation Coefficient is given as thus;

$$\text{Correlation Coefficient, } r = \frac{\sum d_A d_B}{\sqrt{\sum d_A^2 \sum d_B^2}}$$

Where,  $d_A$  and  $d_B$  are concentration results at the sample points A and B. Calculated Correlation Coefficient,  $r$  is 0.9959611. Correlation Coefficient,  $r$ , was read in the standard table with  $n = 3$  [being 2 less

than the 5 heavy metals as variables considered] are 0.991 at probability of 0.001, 0.959 at probability of 0.01, and 0.878 at probability of 0.05. For correlation to be significant, the calculated  $r$  value should exceed the tabulated value  $r$  value. The calculated Correlation Coefficient,  $r$ , is higher than the tabulated values at all probabilities. This implies that the correlation in the results at the top and 100cm depth below the SFM waste dumpsites show significant correlation (Horsfall *et al.*, 1994; Horsfall 2014).

## RESULTS AND DISCUSSION

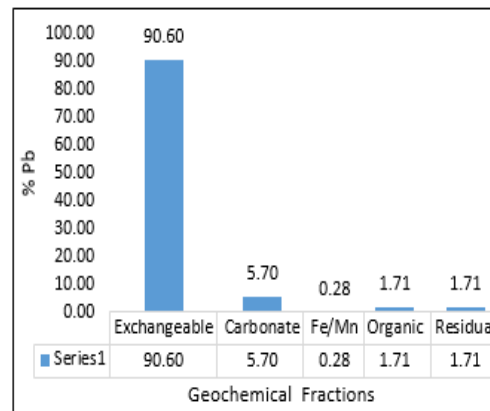
The cumulative speciation results obtained from all the analysis done with 210 VGP FAAS for the five selected heavy metals is showed in table 1.

**Table 1.** Heavy Metals Levels in Sand Filter Media [SFM] Waste Dumpsite in Yenagoa, Nigeria

Sample ID	Lead, Pb [mg/kg]	Iron, Fe [mg/kg]	Nickel, Ni [mg/kg]	Chromium, Cr [mg/kg]	Manganese, Mn [mg/kg]
Sample A <sub>TM</sub>	0	12.5	0.2	1.91	1.25
Sample B <sub>TM</sub>	0	12.3	0.29	2.14	0.28
Sample C <sub>TM</sub>	0	12.1	0.28	2.29	0.85
Sample A1	3.18	11.8	2.59	7.67	1.6
Sample B1	2.53	3.2	2.65	7.92	1.42
Sample C1	3.08	1.6	2.71	8.01	1.55
Sample A2	0.2	10	2.36	3.56	0.63
Sample B2	0.12	5.9	2.4	3.71	0.86
Sample C2	0.06	1.3	2.44	3.9	0.89
Sample A3	0.01	10.9	0.65	2.61	1.28
Sample B3	0.02	8.8	0.22	2.35	0.43
Sample C3	0.06	5.9	0.28	2.22	1.06
Sample A4	0.06	10.7	0.17	2.55	0.42
sample B4	0.03	9.8	0.21	2.64	0.32
sample C4	0.11	5.9	0.11	2.85	0.41
Sample A5	0.06	10.9	0.16	0.18	0.36
Sample B5	0.07	10.9	0.26	0.38	0.33
Sample C5	0.8	10.6	0.06	0.59	0.61

*Speciation of Lead:* The speciation pattern for Pb at the top of SFM dumpsite as shown in figure 1 revealed that Pb occurred most in the exchangeable fraction with 90.06%, followed by Carbonate fraction with 5.70%, the Organic and Residual fractions has 1.71% respectively while the lowest was Fe/Mn fraction with 0.28%. This result indicates that Pb which accumulates most in the exchangeable and carbonate fractions was readily biochemically available for intake or exchange on the surface of the SFM dumpsite and so it is a potential risk to biota around the vicinity of the dumpsite (Leizou *et al.*, 2015). The speciation pattern of Pb at 100cm depth below the surface of the dumpsite is presented in figure 2. The results showed that Pb in the exchangeable fraction increased to 91.34% from 90%. This higher value indicates that there is slight migration of some heavy metals down the dumpsite due to leaching. The high percentage of Pb 100cm below the surface of the dump also means that Pb is available for biological uptake at this depth. Thus plant roots and soil animals are likely to accumulate Pb in their system more than those on the surface (Cui and Du, 2011). However, the carbonate fraction of Pb decreased from 5.2% on the top layer (figure 1) to 4.33% at 100cm in Fig 3. This difference in concentration could be attributed to lower pH values

at the 100cm depth of the SFM dumpsite and the carbonates or metals co-precipitated with carbonates go into solution as the carbonate equilibrium is altered due to the pH drop (Namiesnik and Rabajczyk, 2010). The Fe/Mn fraction increased from 0.28% to 0.72%. The Residual inactive fraction also increased from 1.71% to 2.53%. It can be deduced that some of the Carbonates and Organic fractions reduced down the formation to cause an increase in the Residual fraction.



**Fig 1.** Percent Distribution of Pb at the surface layer of Sand Filter Media [SFM] Waste Dumpsite

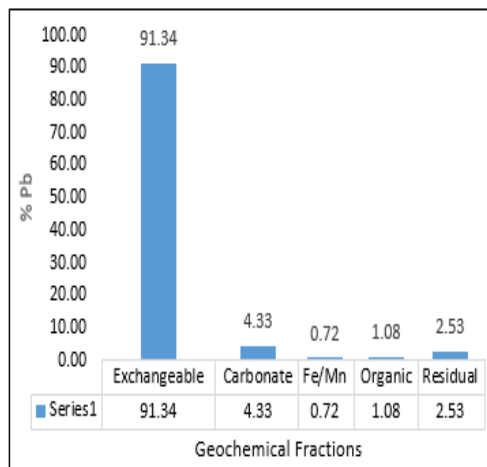


Fig 2. Percent Distribution of Pb at 100 cm depth of the Sand Filter Media [SFM] Waste Dumpsite

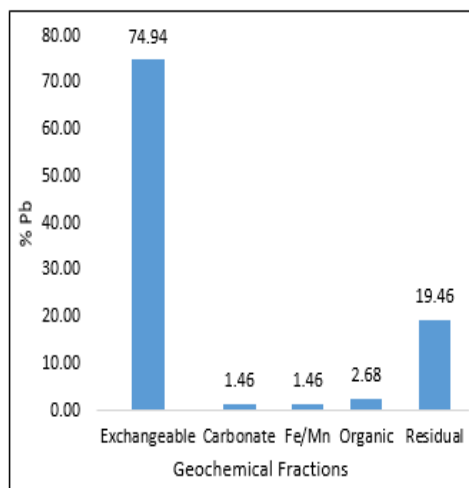


Fig 3. Percent Distribution of Pb at 100meters away from the Sand Filter Media [SFM] Waste Dumpsite

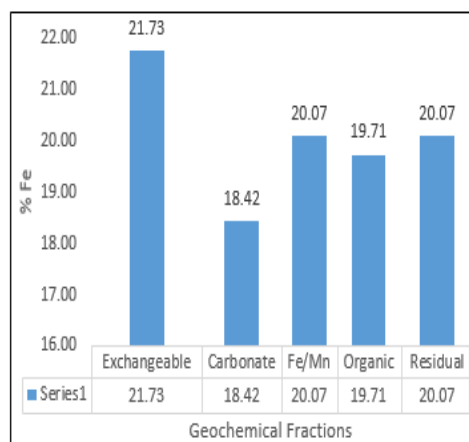


Fig 4. Percent Distribution of Fe at the surface layer of Sand Filter Media [SFM] Waste Dumpsite

The speciation of Pb in the five (5) fractions away from the dumpsite [samples C] is given in figure 3. Exchangeable Pb fraction was 74% which is lower

than those from the dumpsites at the top and below. The Carbonate fraction also decreased to 1.46% thereby adding to the decrease in the bio-available Pb amount in the area. In addition, the Residual amount of Pb increased to 19.46%. This suggests that away from the dumpsite, there was decrease in bio-available Pb caused by the decrease in exchangeable and Carbonate Pb and increase in the residual Pb fraction. Thus, Pb is less available for plant and animal uptake in the area 100m away from the dump site.

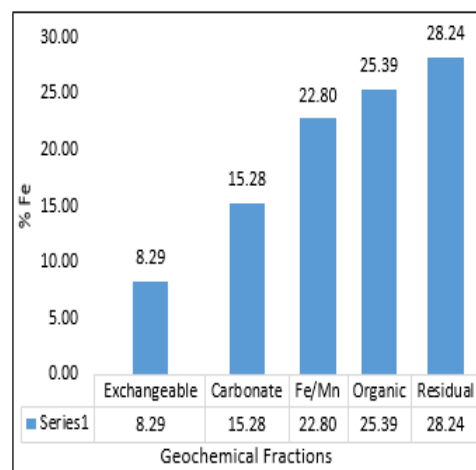


Fig 5. Percent Distribution of Fe at 100 cm depth of the Sand Filter Media [SFM] Waste Dumpsite

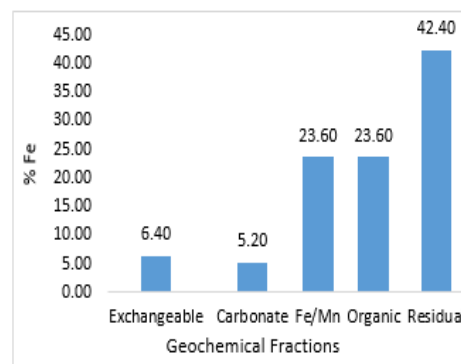


Fig 6. Percent Distribution of Fe at 100 meters away from the Sand Filter Media [SFM] Waste Dumpsite

**Iron Speciation:** The total amount of Fe at the top of the SFM [Sample A] was 10.0mg/kg as against that of Pb which was 0.2mg/kg. The results for Fe fractions at the top of the dumpsite as given in figure 4 was quite close from maximum of 18.42 to minimum of 21.73% unlike Pb. The results showed that the exchangeable fraction of Fe in the sample was 21.73%, the Fe/Mn and Residual fractions was 20.07% each, and the Organic fraction was 19.71% while the carbonate fraction of Fe was 18.42%. The results show a seemingly even distribution when compared with the speciation of Pb with over 90% in the exchangeable fraction. This means that Fe was more concentrated (10mg/kg Fe against 0.2mg/kg Pb), thus it is also

biologically available like Pb. There was increase in other less biologically available fractions, like the Carbonate fraction increased to 18.42 against that of Pb which was 5.70%, Fe/Mn increased to 20.07% against the of Pb which was at 0.28, Organic fraction increased from 1.71% in Pb to 19.71% in Fe while Residual fraction increased from 1.71% in Pb to 20.02% in Fe all from Samples collected at the top of the dumpsite (Kim, et al. 2010). The speciation of Fe at 100cm below the surface of the dumpsite [sample B] gave a decrease in the Fe distribution in the exchangeable Fraction and the Carbonate fraction being 8.29% and 15.28% respectively as against the fractions at the top which were 21.73 and 18.42 respectively. This means that there was decrease in the percentage of Fe that was biologically available at the 100cm depth [figure 5]. This decreased bioavailability was confirmed by the increased percentage of Residual, Organic and Fe/Mn fraction which were 28.24%, 25.39% and 22.80% as against 20.03%, 19, 71% and 20.07% respectively at the top of the dumpsite.

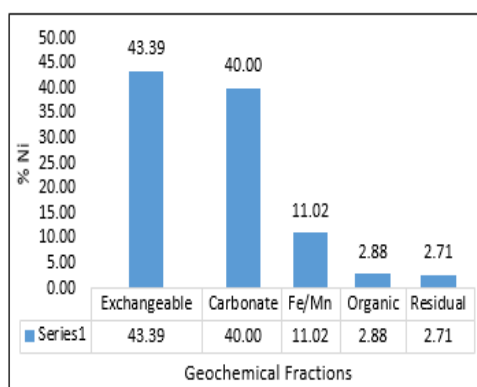


Fig 7. Percent Distribution of Ni at the surface layer of Sand Filter Media [SFM] Waste Dumpsite

Speciation results of Fe distribution from Sample C showed that the percentage concentration of Fe in the exchangeable and Carbonate fractions continued to decrease to 6.40% and 5.20% at 100m away from the dumpsite as compared to 8.29% and 15.28% at the bottom and 21.73% and 18.42% at the top of the dumpsite. Percentages of the inert or less biologically available fraction of the residual, Organic and Fe/Mn fraction recorded increase to 42.40%, 29.60% and 23.60% respectively. The dumpsite has more active HMs than the control sample (Ashraf *et al.*, 2012).

**Nickel Speciation:** The speciation of Ni at the top of the dumpsite [sample A] gave the exchangeable and carbonate fractions as the highest being 43.395 and 40.00% respectively. The trend of the percentage distribution of the other fraction were 31.02% for Fe/Mn, 2.88% for Organic and 2.71% for residual fractions respectively. The results showed that at the surface of the dumpsite, Ni exists more in the fractions that are most available to biological species.

Speciation of Ni at 100cm depth of SFM [sample B] showed that the fraction of Exchangeable and Carbonate bound Ni increased to 46.17% and 41.81% respectively. While there was reduction in the fractions of Fe/Mn to 3.83% from 11.02% at the top. Organic and Residual fractions increased to 3.66% and 4.53% respectively from 2.88% and 2.71% respectively at the top of the dumpsite.

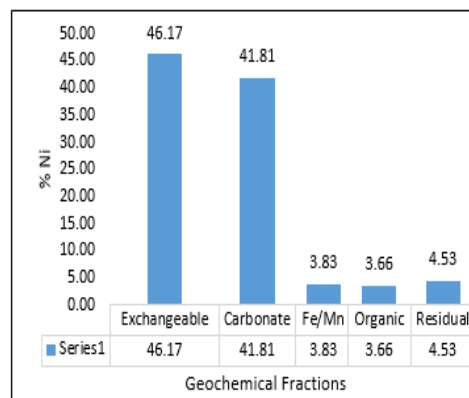


Fig 8. Percent Distribution of Pb at 100 cm depth of the Sand Filter Media [SFM] Waste Dumpsite

The speciation of Ni in the sample collected 100m away [sample C] showed that the Exchangeable and Carbonate fractions were highest with 48.39% and 43.57% while the other fractions of Fe/Mn, Organic and Residual were 5.00%, 1.96% and 1.07% respectively. The trend for speciation of Ni at 100m distance away from the dumpsite surprisingly showed high availability of Ni for biological intake like the samples in the dumpsite.

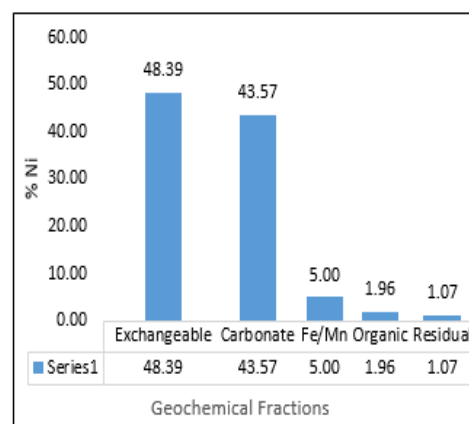


Fig 9. Percent Distribution of Ni at 100 meters away from the Sand Filter Media [SFM] Waste Dumpsite

**Chromium Speciation:** The speciation of Cr at the top of the dumpsite [Sample a] had 46.29% in the Exchangeable fraction, 21.48% in the Carbonate fraction, 15.75% in the Fe/Mn fraction, 15.39% in the Organic fraction and 1.90% in the residual fraction. This showed that Cr was highest in the exchangeable fraction and lowest in the inactive residual fraction.

The high percentage of Cr in the exchangeable fraction indicates that there is availability of Cr for biota in that environment.

There is bioavailability of the Cr in the sample for uptake by biota possible causing bioaccumulation.

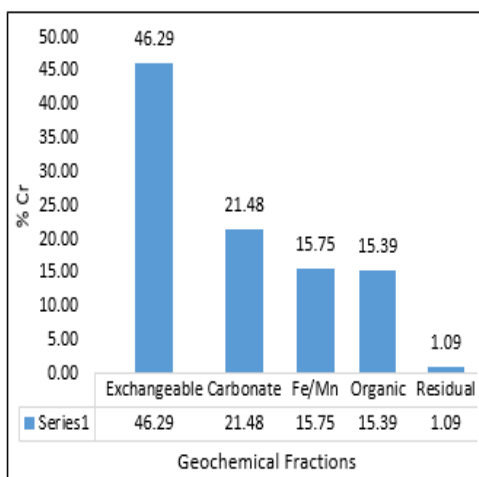


Fig 10. Percent Distribution of Cr at the surface layer of Sand Filter Media [SFM] Waste Dumpsite

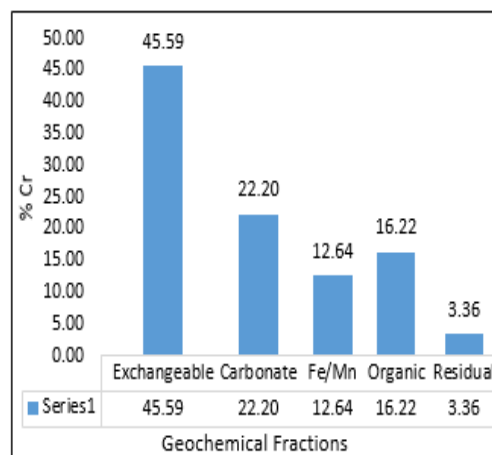


Fig 12. Percent Distribution of Cr at 100meters away from the Sand Filter Media [SFM] Waste Dumpsite

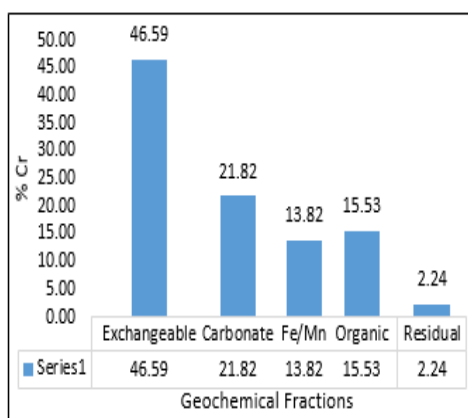


Fig 11. Percent Distribution of Cr at 100 cm depth of the Sand Filter Media [SFM] Waste Dumpsite

**Manganese Speciation:** The speciation of Mn in the sample collected from the top of the dumpsite had exchangeable fraction of 37.30%, carbonate fraction of 14.69%, much higher Fe/Mn fraction of 29.84%, which is the highest of the Fe/Mn in all the samples collected. Organic and residual fractions were 9.79% and 8.39% respectively. Biological available exchangeable fraction of Mn was highest in percentage about a third of the others. This means that Mn is readily available for biota intake and bioaccumulation processes.

Consequently, the availability of Cr in the sand filter media was Exch >> Carb. > Fe/Mn > Org. > Res. The speciation of Cr at 100cm below the surface of the dumpsite [Sample B] followed the same trend for the top surface. Results showed that the Exchangeable fraction was highest with 46.59%, just a little higher than the one from the top of the dumpsite. This was followed by Carbonate fraction that was 21.82%, which was also only slightly higher than 21.48% from the sampled top. Other fractions also trended the same with the lowest being the residual fraction which is 2.24% and slightly lower than 1.09% from the sampled top. The trend revealed mobility of HMs down the dumpsite. This implies that at the depth of 100cm below the Cr in the dumpsite was biologically available and will be bio- accumulated, if taken. The speciation pattern of Cr 100m away from SFM [sample C] relatively showed the same trend wherein the Exchangeable fraction as the highest being 49.99% while the Residual fraction was the lowest 3.36%.

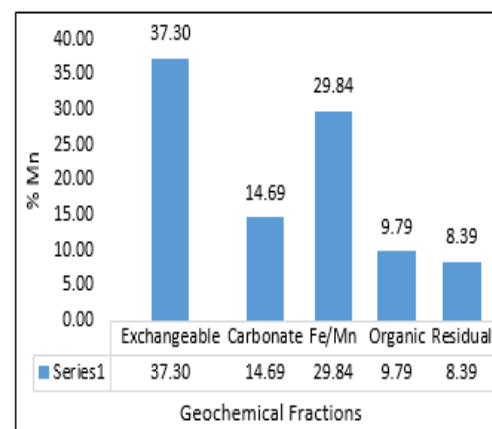


Fig 13. Percent Distribution of Mn at the surface layer of Sand Filter Media [SFM] Waste Dumpsite

100cm below the SFM dumpsite [sample B], speciation results for Mn revealed that the exchangeable fraction increased to 42.26% from 37.30%, Carbonate fraction also increased to 25.60% from 14.69% while Fe/Mn fraction reduced to 12.80% from 29.84% This showed mobility of HMs downward. Other fraction like Organic and Residual fractions still remained in the vicinity of 9.52% and 9.82% respectively. The trend again showed that Mn



is biologically available and infact its availability slightly increased down the SFM dumpsite.

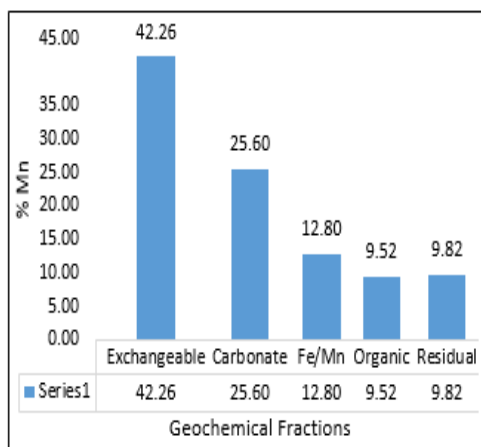


Fig 14. Percent Distribution of Mn at 100 cm depth of the Sand Filter Media [SFM] Waste Dumpsite

The distribution of Cr at 100m away from the SFM, gave exchangeable fractions as highest with 34.29%, Fe/Mn fraction as 23.45%, Carbonate fraction as 19.69%, Residual as 13.50% and Organic fraction as lowest with 9.07%. Results still revealed the exchangeable fraction as highest even at the control sample.

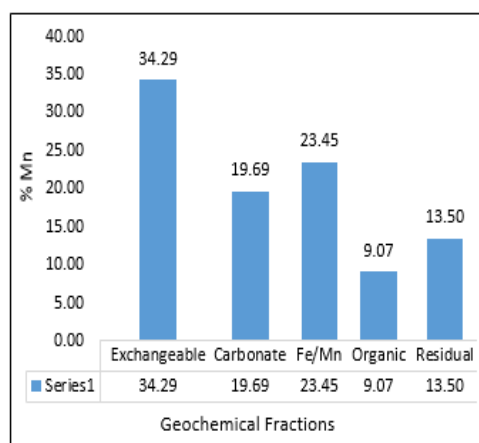


Fig 15. Percent Distribution of Pb at 100 meters away from the Sand Filter Media [SFM] Waste Dumpsite

The investigation of distribution and speciation of five Heavy Metals [HMs] in spent Sand Filter Media [SFM] dumpsite investigated with a view to ascertain the total heavy metals in the SFM and the chemical speciation of the heavy metals concluded that Fe had highest concentration while Pb had the lowest concentration in the order; Fe >>> Cr > Mn > Ni > Pb

The investigation of the chemical speciation of the metals concluded that the trend of geochemical fractions follows the order;

Pb Speciation: Exchangeable >>> Carbonate > Organic = Residual > Fe/Mn  
 Fe Speciation: Residual > Organic > Fe/Mn > Carbonate = Exchangeable  
 Ni Speciation: Exchangeable > Carbonate >> Fe/Mn > Residual > Organic  
 Cr Speciation: Exchangeable >> Carbonate > Organic > Fe/Mn > Residual  
 Mn Speciation: Exchangeable > Fe/Mn > Carbonate > Residual > Organic

It is concluded that the concentration of the five heavy metals studied had Fe as having the highest concentration but this was spread evenly amongst the geochemical fractions while the other four metals (Pb, Cr, Ni and Mn) had more of their geochemical fractions in the exchangeable, carbonate, Fe/Mn and Organic fractions which means that these HMs are of high mobility and thus biochemically available for interaction and intake by biota. Therefore, these metals portend contamination of the ecosystem in the sand filter media [SFM] dumpsite possibly due to bioaccumulation.

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