

Heavy Metal Contents of Soils and Plants as Influenced By Leached Ash Refuse From Black Soap-Making at Ikere-Ekiti, Nigeria

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Abstract: The effects of ash refuse pile from a black soap-making facility were studied on the surface (0-15 cm) layer of soils and commonest plants at Ori-Ebu, Afao Quarter, Ikere-Ekiti in Nigeria. Soil and plant samples were collected at 0, 15, 30, 60, 75, 105 and 150 m from the ash refuse pile and analyzed for cadmium (Cd), cobalt (Co), chromium (Cr), lead (Pb) and nickel (Ni). The prominent plant species were *Physalis angulatus* at 0 m, *Starchytapheta jamaicensis* at 15 m, *Starchytapheta cayenensis* at 30 m, *Asystasia gagentica* at 60, 75 and 105 m and *Sida scabida* at 150 m. Co, Cd, Pb and Ni contents were lower in the site than the control. Co, Pb and Cd increased with distance from the ash refuse pile while Cr showed an accumulation at 60 m. Heavy metal concentration in plants exceeded the control except Ni and Co at 150 m. Accumulative factors used to access the level of pollution were contamination factor, pollution load index and transfer factor. Co, Cd, Pb and Ni contamination was low ($CF < 1$) while the site has been moderately contaminated with Cr $CF = 1.53-3.10$). The pollution load index was low ($PLI = 0.43-0.96$) such that the site was not contaminated by the heavy metals. The transfer factors at 1.02, 1.1, 1.14, 2.54 and 29.59 for Cd, Pb, Ni, Co and Cr, respectively indicate high levels of heavy metal bio-accumulation which can have implications for livestock and human health through the food chain.

Keywords: Wood ash, heavy metals, accumulative factors, bio-accumulation

I. Introduction

Wood ash is the residue powder left after the burning of wood and contains the main nutrients used by the tree for growth and development: calcium (Ca), phosphorus (P), potassium (K) and magnesium (Mg); micronutrients needed in trace amounts and varying amounts of toxic heavy metals (Mahmood *et al.*, 2000; Ozolinčius and Varnagyritė, 2005). These are primarily oxides (Lickacz, 2002; Hume, 2006) being products of complete oxidation of the wood.

Leaching of wood ash yields lye, a corrosive alkaline solution consisting mainly of potassium hydroxides (Addison, 2013), valued for its use in soap-making. Soap is a salt of fatty acid (Naught and Wilkinson, 1997) obtained by treating vegetable or animal oils and fats with a strongly alkaline solution. The ensuing chemical reaction- saponification- involves hydrolysis of fats to fatty acids that combine with the alkali to form soap and liberating glycerol (glycerine) which is left in or washed out and recovered as a useful by-product (Cavitch, 1997). Different kinds and types of soap are now manufactured in factories through processes, equipment and operations that are under censored regulations on raw materials handling, emission and effluent control, by-products and waste disposal which minimize hazards to the environment and human life. Nevertheless, hand-made or natural soap (Native or African black soap) is still made through processes that involve leaching wood ash to obtain lye which is mixed with vegetable oil and the mixture is continually stirred in metal or earthen pots with moderate heat from firewood until it solidifies and the brown to black soap is allowed to cool and cut to sizes ready for use or sale (Popovitch, 2013).

Black soap-making is a small-scale localized industry without safety regulations in the production process and whose operations can continue to pollute the environment through the leached ash disposed of by dumping on surrounding lands. Afao Quarter, Ikere-Ekiti, Ekiti State is one of the places where black soap is produced in Nigeria and the leached ash residue from over 50 years of operation has piled up and spread on about 0.40 hectares of land. This pile, over a long period of time, can be a source of heavy metal pollutants as the trees from which the wood burnt is derived would have absorbed these elements during their lifetime. Although wood ash contains typically low amounts of heavy metals (Risse and Harris, 2008) of diverse mobility but would be of ecotoxicological significance due to changes they can cause in the soil, ground vegetation and ground water chemistry (Campbell, 1990). Moreover, the Science for Environment Policy (2008) noted that wood ash can contain surprisingly high concentrations of heavy metals, with Pb, Cd and Zn attaining levels high enough for the ash to be regarded as a toxic waste. Land application of wood ash increases the pH of the organic matter-rich surface layer of the soil (Saarsalmi *et al.*, 2001) which reduces the solubility of heavy metals and so would cause their accumulation in soils. Herein lay the interest in heavy metals which at high concentrations form

complex compounds with the organic and inorganic soil colloids, sorb on clay and sesquioxides surfaces or precipitate as hydroxides and carbonates (Ruby, 1999).

Heavy metal concentration is one of the indicators used to assess the level of contamination in an environment. Since ash application causes short-term availability of heavy metal through accumulation in the organic matter-rich surface layer of soil, the concentration in plant tissues should be affected (Nilsson and Eriksson, 1998). Nabulo *et al.* (2008) noted that heavy metal uptake from soils differs among plant species and sites, therefore, it is necessary to analyze and understand the effects of long-term presence of ash residue on the soils and dominant weeds and comparing these with soil from an area not impacted by the ash pile and in reference to FAO and United States Environmental Protection Agency (USEPA) critical levels.

II. Materials And Methods

The study area is Ori-Ebu located in Afao Quarter, Ikere-Ekiti in Ekiti State, Nigeria, where ash refuse had piled on about 0.4 hectare land as a result of black soap-making operations for over fifty years. Ikere is located on latitude 7°39'N and longitude 5°33'E and falls into the tropical sub-humid rain forest region with distinct dry and wet seasons which prevail between November to March and April to October respectively. The mean monthly temperature is 28°C, the relative humidity is about 70% and the mean annual rainfall is 1,800 mm. The site slopes down into Odo Aia (a stream) and was prime agricultural land but now consists of residential buildings and vacant plots. Most of the residents maintain home garden crops: tomato, okra, leaf amaranth, water leaf, bitter leaf, plantain, banana, pawpaw, citrus, etc.

Surface (0-15 cm) soil samples were taken at specific distances from the point of ash residue deposit downward the slope to the stream at 0, 15, 30, 60, 75, 105, and 150 m. A control sample was taken from an area where ash has not been deposited. The soil samples were air-dried, slightly crushed and passed through a 2 mm sieve. Plant samples were collected based on species availability. The plants in each location were identified and the most abundant species collected as 2-6 replicate samples within 4 m² and mixed to form a composite, packed in separate envelopes and labelled. The samples were rinsed in water and separated into roots, stems and leaves, oven-dried at 70°C for 48 hours and ground into fine powder with mortar and pestle.

Soil samples were analysed for pH in distilled water, organic carbon, particle size distribution, exchangeable cations and available P using standard laboratory procedures described in IITA (1979). Heavy metals were determined from one gram (1g) samples digested with 100 ml concentrated HNO₃ and HClO₄ in ratio 2:1 on a Tecator Digestion system. The heavy metals were determined on atomic absorption spectrophotometer (AAS) (Buck Scientific 205 Model). 250 mg finely-ground samples of the plant materials from the site were digested with 5 ml concentrated nitric acid and the digests diluted to 60 ml with demineralized distilled water. Co, Cd, Cr, Cu, Fe, Ni, Zn and Pb were determined with AAS.

Simple correlation analysis was carried out between soil properties and contents of heavy metals in the soils and plants. Accumulative factors were used to assess the extent of pollution in the site. The extent of soil pollution was compared to a baseline concentration and indicators of heavy metal enrichment calculated.

(1) Contamination Factor (C_F) = $\frac{C_m \text{ sample}}{C_m \text{ background}}$

Where, C_m (sample) = mean of the concentrations of individual metals from all distances

C_m (background) = background or baseline concentration of individual metal

The interpretation is in four categories as follows: $CF < 1.0$ = low contamination; $1 < CF < 3$ = moderate contamination; $3 < CF < 6$ = considerable contamination and $6 < CF$ = very high contamination

(2) Pollution Load Index (PLI) = $(C_{F1} \times C_{F2} \times C_{F3} \dots C_{Fn})^{1/n}$

Where, n = number of metals investigated

C_F = Concentration factor i.e. ratio of concentration of each metal in sample to the baseline soil

PLI value below or close to 1 means baseline heavy metal loads while > 1 means heavy metal pollution or accumulation at the site

(3) Transfer or bio-concentration factor (TF or BCF) of the heavy metal into plants was determined using the expression C_p/C_s

Where, C_p = concentration of the metal in plant sample

C_s = concentration of the metal in corresponding soil sample

$TF > 1$ means high level of heavy metal contamination in the plant

III. Results And Discussion

Table 1 shows the most abundant weed species located within quadrants in the study area and the physical and chemical characteristics of the surface layer (0-15 cm) of soils. The most abundant weed species were *Physallis angulatus* at 0 m, *Starchytapheta jamaicensis* at 15 m, *Starchytapheta cayenensis* at 30 m, *Asystasia gangetica* at 60, 70, and 105 m and *Sida scrobida* at 150 m. In the control location, *Chromolaena odorata* was most abundant. This is a widely dispersed neo-tropical shrub which occurs in disturbed areas,

grasslands, fallows and forest re-growths and has become an obnoxious weed in arable fields and commercial tree crop plantations in the humid forest and derived savannah zones of Nigeria (Agunbiade and Fawale, 2009).

The soil at 0 m was a silt loam, being mainly ash material; sandy clay loams at 15-105 m and sandy loams at 150 m and the control. Soil pH was slightly alkaline to moderately alkaline with the highest soil pH obtained at 0 m location (pH=8.3) and decreased with distance from the ash refuse pile but the values were higher than the slightly acid-neutral pH of the control. The high pH values are attributed to the ash deposit whose influence appeared to decrease with distance. Wood ash is rich in basic cations even as the other elements are present primarily in oxide forms (Lickacz, 2002; Hume, 2006) being products of complete oxidation of the wood and so explain the ability to raise soil pH. Soil organic matter varied between 1.68 to 3.43% in the location with the highest value at 150 m. This pattern of distribution can be attributed to removal of organic matter-rich surface layer of soils in the upper slope, transport in runoff and deposition as sediment at the valley bottom. This would also be responsible for low total N at 0 and 15 m (0.11-0.16%) and an increase to the highest value (0.52%) at 150 m. Exchangeable cations were high compared to the control which falls within the range of values indicated for soils in South-west Nigeria (FMANR, 1990). The highest exchangeable values were: Ca and Mg at 0 m, and K and Na at 150 m. The highest values at 0 m are due to the presence of ash which is rich in basic cations (Pitman, 2006; Serafimova *et al.*, 2011). Ash is a liming material whose efficiency relates to the presence of elements, especially Ca, as oxides such that the calcium carbonate efficiency is 55-65% when incorporated into the soil (Lickacz, 2002; Risse and Harris, 2008). High values of the soil fertility indices at 150 m are characteristic of soils in the inland valley systems (Andriesie, 1986) and reflect accumulation of nutrients due to slow movement of erosion water and vegetative cover which cause deposition of sediments eroded from the upper slopes.

Table 2 shows the contents of some heavy metals in the soils. Pb was the most abundant and ranged from 4.8-54.9 mg/kg while Co and Cd had the least ranges of values at 1.85-10.50 and 4.93-8.85mg/kg respectively. Co and Cd increased with distance from the ash refuse pile but the highest values at 75 and 150 m are lower than in the control. Cr level was higher in the location than the control and showed an accumulation at 60 m. Pb was least at 0 m, increased till 30 m and decreased thereafter with the values being higher than the control at 30, 60 and 105 m distances. Soil Ni content was highest at 0, 105 and 150 m distances but exceeded the control soil at 150 m only. The long-term presence of ash reduced the concentrations of Cd, Co, Ni and Pb in the soils, especially at 0 and 15 m distances compared to the control because of the ash-induced increase in soil pH. Saarsalmi *et al.* (2001) noted the ability of ash to increase soil pH and that the liming effect lasted long enough to cause reduction in the solubility of the heavy metal cations. The surface layers of soils usually contain higher organic matter and so would retain heavy metals by cation exchange reactions. The retained metal can be removed by the action of water and get translocated elsewhere (Ghrefat and Yusuf; 2006). Since the direction of water flow is downstream, the heavy metals get dispersed down slope through runoff (Ngole and Ebosse; 2012) such that the soils at 105 and 150 m contain higher concentrations than at 0 and 15 m.

Table 3 shows the correlation coefficients of the relationships between some soil properties and the heavy metals. The pairs with strong positive correlations are Co/sand, Cd/sand, Cr/pH, Pb/clay and Cd/ECEC while pairs with negative correlations are Co/silt, Co/pH, Co/ECEC, Cd/pH, Pb/silt and Ni/clay. The low correlations with clay and organic matter are not expected because they dominate the colloidal fraction and exchange complex of soils and should have high retention capacity for the heavy metals and their contamination would increase so long as the nearby sources are active (Ghrefat and Yusuf, 2006).

Table 4 shows the heavy metal concentration in the plant species. The heavy metal concentrations in *Chromolaena odorata* as the control sample was lower than the values in the different plant species taken at specific distances from the ash refuse pile except Ni and Co at 150 m. Differences in the pattern of heavy metal distribution occur because the uptake by plants depends on plant specific factors which include plant species and variety, age and growth rate, size, depth and distribution of the root system, transpiration coefficient and nutrient requirements of the plant (Macnicol and Beckett, 1985). Cd and Co increased to the highest values at 30 m and thereafter reduced slightly with distance. Cr was lower in the plant samples taken at 0-105 m than the control but the high value at 0 m decreased to 75 m and increased to the highest value of 2,657.80 mg/kg at 150 m. Ni increased with distance to the highest value at 105 m. Pb increased to the highest value at 75 m and thereafter decreased to 22.60 mg/kg at 150 m. Thus, the plant species at 150 m contained the least concentrations of the heavy metals except Cr. The mean heavy metal concentrations in the plants over the distance from the ash refuse pile exceeded the concentration in the control plant sample and were in the order Cr>Pb>Ni>Co>Co>Cd.

The correlation coefficients of the relationships between soil properties and heavy metal contents with the concentrations of heavy metals in plants are shown in Table 5. Soil pH had significant correlations with Cd, Co and Pb. The pairs with significant correlations are Cd/Cr and Ni/Cr. Only the Cr/Pb pair showed a positive correlation but which is not significant.

The contamination factors of heavy metals at specific distances are shown in Table 6. Co and Cd gave values of CF=<1 but which increased with distance in the study area indicating low levels of contamination. Pb

contamination was low at 0, 15, 75 and 150 m while CF exceeded 1.0 at other distances. Ni contamination was low except at 150 m. The CF values for Cr indicated that contamination was moderate at all distances and considerable at 60 m such that the average at 1.93 showed that the site was moderately contaminated. The contamination factors were used to calculate Pollution Load Index (PLI) as basis for interpreting the state of contamination that had occurred in the environment. The PLI values were less than 1.0 at all the distances which shows that the environment has not been contaminated by heavy metals.

Transfer factors calculated as the ratio of concentrations of heavy metals in plant tissues and in the soil would indicate whether bio-accumulation and bio-magnification have taken place and so the extent of potential human exposure through the food chain. Table 7 shows that heavy metal accumulation was high in the plants as indicated by $TF > 1$ for Cd except at 60, 105, and 150 m; Pb at 30, 60 and 105 m and Ni at 0 m. Kalfakakour and Akridu-Demerti (2000) had noted that TF determines the relative difference in bio-availability of metals. TF was highest at 0 m for Cd, Pb, and Co while TF decreased from 0-60 m for Cr and rose steadily to the highest value at 150 m.

Different plants found at specific distances from the ash refuse pile except at 60-105 m which contained the same prominent weeds mean variable levels of the heavy metal. The roots of plants absorb heavy metal ions in the soil solution and translocate amounts which accumulate in the above-ground parts (stem, leaves and fruits) and so used to distinguish plants as excluder, indicator, accumulator or hyper-accumulator (Whiting, 2000; Hogan, 2010). The transfer into the shoot is negligible in excluders and as such the heavy metal concentration is low over a wide range of soil levels, the concentration in the shoot of indicators clearly indicates the level of soil pollution while accumulators and particularly hyper-accumulators extract and concentrate large quantities of metals far beyond the threshold values in the shoot (Nathalie and Sylvie, 2002). The elevated levels in the plants and transfer factors at all distances from the ash refuse pile show that heavy metal pollution had occurred in the study location. Since heavy metals which have contaminated the soil can hardly be completely eliminated, immobilization through the action of some plants can mitigate the effects. Thus, *Sida scrobida* is an accumulator of Cr but excluder of Cd, Co, Ni and Pb while *Chromolaena odorata* is an accumulator of Cr. Also, despite the low soil contents at 0 m, the *Physallis angulatus* extracted and accumulated Cd, Co and Pb. Although these plants are weeds, the bio-accumulation and bio-magnification of the heavy metals should indicate the extent of potential exposure through the food chain and the implications for human health.

IV. Conclusion

Studies were carried out to assess the influence of a 50-year old ash refuse pile on soils and plants in a black soap-making facility at Ikere-Ekiti, Nigeria. The ash pile increased the soil pH to alkaline range due to higher values of Ca, Mg, K and Na but reduced the content of Co, Cd, Pb and Ni especially at 0-15 m distance. Indices of accumulation showed low contamination for the heavy metals except Cr with moderate to considerable level of contamination. The Pollution Load Index was less than 1.00 such that the location was not contaminated with heavy metals. The different weed species found at specific distances from the ash refuse pile contained more Cd, Co, Ni and Pb than the plant in the control location. Cr content was lower in the plants at 0-105 m than those in the control and at 150 m. The transfer factors at greater than 1.00 are an indication of heavy metals pollution. The high Cr content of *Chromolaena odorata* and *Sida scrobida* means that they can be used for mitigation as accumulators while *Sida scrobida* would be an excluder of Cd, Co, Ni and Pb.

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Table 1: Physical and chemical characteristics of top soil (0-15 cm) samples at specific distances (m) from ash refuse pile

Property	Control	0 m	15 m	30 m	60 m	75 m	105 m	150 m
Weeds	<i>Chromolaena odorata</i>	<i>Phyllanthus angulatus</i>	<i>Starchytapheta jamaicensis</i>	<i>Starchytapheta cayenensis</i>	<i>Asystasia gagentica</i>	<i>Asystasia gagentica</i>	<i>Asystasia gagentica</i>	<i>Sida scrobida</i>
Sand	60	30	53	46	48	56	50	55
Silt	22	56	18	22	30	16	21	25
Clay	18	14	29	32	22	26	29	20
pH	6.3	8.3	7.6	7.7	7.8	7.5	7.8	7.1
O. M. %	0.42	2.31	1.23	2.41	1.63	1.95	1.80	3.43
%N	0.02	0.16	0.11	0.32	0.22	0.24	0.26	0.52
P, mg/kg	8.12	24.35	10.32	30.40	13.40	22.54	20.52	36.85
Exch. K	0.13	0.82	0.30	1.16	0.40	0.84	0.68	2.01
Ca	1.50	13.20	7.10	7.60	8.20	6.30	9.10	7.80
Mg	0.82	9.10	6.03	6.20	6.70	4.95	6.80	5.20
Na	0.13	0.49	0.36	0.55	0.40	0.51	0.49	0.94
EA	0.20	0.00	0.00	0.00	0.00	0.00	0.00	0.00
ECEC*	2.75	23.61	13.79	15.15	15.70	12.61	17.07	15.95

*ECEC= Effective Cation Exchange Capacity

SL=Sandy loam, SCL=Sandy clay loam, SiL= Silty loam.

Table 2: Heavy metals in the top layer (0 – 15 cm) of soils at specific locations from ash refuse pile in the vicinity of a black soap-making facility

Location	Co mg/kg	Cr mg/kg	Cd mg/kg	Pb mg/kg	Ni mg/kg
Control	11.60	14.60	9.13	37.43	44.93
0 m	1.85	27.80	4.93	4.83	35.48
15 m	6.65	20.15	5.58	26.28	8.13
30 m	6.15	22.35	6.43	54.90	13.65
60 m	6.90	45.40	6.48	46.25	17.90
75 m	10.50	30.60	6.73	34.63	11.18
105 m	7.15	27.90	7.63	41.85	33.35
150 m	6.60	23.40	8.85	12.50	46.13
Mean	6.34	28.23	6.66	31.60	23.69

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Table 3: Correlation coefficients of the relationships between topsoil properties and heavy metal concentrations in the soils

Heavy metals	Sand	Silt	Clay	pH	OM	ECEC
Co	0.91**	-0.77*	0.20	-0.79*	-0.52	-0.91**
Cr	-0.28	0.23	0.10	0.56	0.20	0.45
Cd	0.77*	-0.46	-0.12	-0.83**	-0.06	0.65
Pb	0.35	-0.59	0.59	-0.13	-0.36	-0.39
Ni	-0.01	0.33	-0.67	-0.43	0.13	-0.11

*, ** Correlation coefficients are significant at 5% and 1% respectively

Table 4: The heavy metal concentration in plant samples collected at specific distances from the ash refuse pile

Location	Cd mg/kg	Pb mg/kg	Ni mg/kg	Co mg/kg	Cr mg/kg
Control	4.64	24.88	22.55	9.93	1508.92
0 m	7.18	28.33	27.03	18.45	821.38
15 m	6.98	29.39	25.90	19.99	399.11
30 m	8.36	26.96	29.48	22.80	458.01
60 m	6.25	32.16	26.56	13.89	478.95
75 m	6.83	35.84	32.54	17.56	430.81
105 m	6.90	31.36	33.58	17.01	600.63
150 m	5.06	22.60	14.46	6.68	2657.80
Mean	6.79	34.61	27.08	16.63	835.24

Table 5: Correlation coefficients of the relationships between topsoil properties and heavy metal concentration in plant samples

Soil characteristics	Co	Cr	Cd	Pb	Ni
pH	0.65	-0.56	0.76*	0.82**	0.50
OM	-0.11	0.43	0.22	-0.22	-0.31
Sand	-0.44	0.26	-0.56	-0.01	-0.19
Silt	-0.04	0.05	0.11	-0.16	-0.11
Clay	0.36	-0.53	0.58	0.33	0.49
CEC	0.56	-0.27	0.55	0.12	0.18
Co	-0.13	-0.22	-0.18	0.32	0.08
Cr	-0.09	-0.19	-0.18	0.54	0.24
Cd	-0.71*	0.55	-0.62	-0.31	0.01
Pb	0.30	-0.58	0.30	0.43	0.45
Ni	-0.62	0.80**	-0.50	-0.51	-0.48

*, ** Correlation coefficients are significant at 5% and 1% respectively

Table 6: Contamination factor of heavy metals in the top soil at specific distances (m) from the ash refuse pile

Heavy metal	0 m	15 m	30 m	60 m	75 m	105 m	150 m	Mean
Co	0.15	0.57	0.53	0.59	0.91	0.62	0.57	0.56
Cr	1.90	1.38	1.53	3.10	2.09	1.91	1.60	1.93
Cd	0.54	0.61	0.70	0.73	0.73	0.83	0.96	0.73
Pb	0.12	0.72	1.47	1.23	0.92	1.11	0.33	0.84
Ni	0.78	0.18	0.30	0.39	0.25	0.74	1.02	0.52
PLI*	0.43	0.57	0.76	0.92	0.80	0.96	0.78	-

PLI* = Pollution Load Index

Table 7: Transfer factors of heavy metals from the soil into the plants

Heavy metal	0 m	15 m	30 m	60 m	75 m	105 m	150 m	Mean
Cd	1.46	1.25	1.30	0.96	1.01	0.90	0.57	1.06
Pb	5.87	1.12	0.49	0.70	1.03	0.75	1.81	1.68
Ni	0.76	3.19	2.16	1.48	2.91	1.01	0.31	1.69
Co	9.97	3.00	3.71	2.01	1.67	2.38	1.01	3.39
Cr	29.55	19.81	20.49	10.55	14.08	21.53	113.58	32.80