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Assessment of Heavy Metals and Polycyclic Aromatic Hydrocarbons in Agricultural Soils around Kubanni Bridge area, Zaria, Kaduna State, Nigeria

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The level of heavy metals and polycyclic aromatic hydrocarbons (PAH) were determined from agricultural soil collected in Kubanni area of Zaria using standard methods**.** The result mean±SD of the heavy metals in the five study locations; (KP1- KP3) ranges from 4.05±0.18-1.25±0.32 for Lead, 4.43±0.11-0.03±0.01 for Cadmium,4.30±0.14 - 0.13±0.08 for Chromium, 3.32±0.25-1.03±0.13 for copper. And 1.80±0.01- 0.02±0.01 for Zinc. The results of the persistent organic pollutants in the five study locations KP1- KP3 showed the mean range (±SD) concentrations from7.08±0.02-0.13±0.01 (naphthalene), 7.00±0.08 - 0.02±0.01 (anthracene) and 7.31±0.21 - 0.02±0.01 (phenanthrene). There was appreciable number of heavy metals and PAH in the study area even though it was found to be below the permissible level set by United State Environmental Protection Agency (USEPA) and National Environmental Standards and Regulations Enforcement Agency (NESREA) in most of the locations. There is the tendency of these residues from the soil to accumulate into crops grown in the studied area, which can constitute serious health risk.

Keywords: Heavy Metals; Agricultural Soils; Polycyclic aromatic hydrocarbons; Zaria.

1. Introduction

Since the start of industrialization, a large variety of chemical compounds have been synthesized for countless uses on agricultural soils. Despite the importance of these chemicals or substances, they still cause damage to the environment. Heavy metals and polycyclic aromatic hydrocarbons (PAH) are examples of such substances. According to an estimate, about 20 million hectares of land worldwide is utilizing wastewater for irrigation with 10% of the total world's population being dependent upon food irrigated with wastewater (Mweogoha and Kihamba, 2010). Sources of heavy metal contamination include municipal solid wastes, industrial waste, automobile emissions, mining activity, and agricultural practice (Adeyeye, 1994). polycyclic aromatic hydrocarbons (PAH) are organic substances that, to a varying degree, resist photolytic, biological and chemical degradation. These substances are also known for their ability to biomagnifies and bioconcentrates under typical environmental conditions, thereby potentially achieving toxicologically relevant concentrations (Persson, 2007). Polycyclic Aromatic Hydrocarbons (PAHs) are group of organic compounds containing two or more fused aromatic rings and do not contain heteroatom or carry substituent which can stay on the environment for a very long period (Igwe and Ukaogo, 2015).

Galadima and Garba (2012) have identified illegal mining of gold as one of the major lead poisonings in Zamfara state, Nigeria. The incident was because of some illegal activities by miners. These illegal activities resulted to the contamination of the drinking water as well as the agricultural soil, which lead to the destruction of so many lives leaving so many organisms infected with diseases due to the agricultural soil contamination. Human activities are the major causes of environmental pollution. These activities include emission from automobile, refuse dumping, industrial effluents, agricultural activities (application of pesticides, herbicides etc), quarrying and mining activities. This anthropogenic activity has resulted to the environmental degradation of Kubanni area due to release of the heavy metals, in a similar work reported by Ali (2013) who worked only on heavy metals.

The search for literature on the level of both heavy metals and Polycyclic Aromatic Hydrocarbons on the agricultural soil around Kubanni area of Zaria local Government has yielded little no results. Researchers only account for heavy metals on the study area despite the applications of agricultural inputs such as fertilizers and pesticides in the area which are carcinogenic due their bioaccumulation in fatty tissues. This necessitated the present study to ascertain the effects of these anthropogenic activities on the agricultural soil around Kubanni area for both heavy metals and PAH. The finding of this study will help towards mitigating the adverse effect of these activities on the agricultural soil in the area (Ali, 2013). Edu et al. (2016) identified the potential risks of polycyclic aromatic hydrocarbons in freshwater ecosystem in Ikpa river basin, Niger Delta. In their study, they have quantified some PAHs in various environmental matrices in Ikpa river basin and nearby dumpsites using gas chromatography mass spectrometry. The levels of the pollutants were further subjected to models to estimate possible sources and potential risks. The results gotten discovered the nearby dumpsites as the major source of contamination of the Ikpa River Basin. This water contamination however, resulted to the contamination of the nearby agricultural soils.

2. Materials and Methods

2.1 Reagents Used

Hydrochloric acid (HCl) (Assay; 37%, V/V, MW; 36.46 g/mol, [purchased from Friendermann Schmidt, (Parkwood, Australia)]); Conc. Nitric acid (HNO_3) (Assay; 70%, V/V, MW; 63 g/mol supplied by Sigma Aldrich, Germany); Lead nitrate ($Pb(NO_3)_2$ (Assay; 99 %, MW; 331.23 g/mol Supplied by Fisher Scientific, Walthman, MA, USA);Cadmium nitrate tetra hydrate [$Cd(NO_3)_2.4(H_2O)$] (Assay; 98 %, MW; 236.42 g/mol Supplied by Fisher Scientific, Walthman, MA, USA); Potassium dichromate $K_2Cr_2O_7$ (Assay; 99 %; MW; 294.18 g/mol Supplied by Fisher Scientific, Walthman, MA, USA); Copper (II) sulphate pentahydrate $CuSO_4.5H_2O$ (Assay; 99.99 %; MW 249.64 g/mol Supplied by Fisher Scientific, Walthman, MA, USA); Sulphuric acid (H_2SO_4) (Assay; 98 %; MW; 98.08 g/mol purchased from friendermann Schmidt, (Parkwood, Australia)]); n-hexane (C_6H_{14}) (Assay;95%; MW; 86.178 g/mol, obtained from Sigma-Aldich, USA) Dichloromethane (CH_2Cl_2) (Assay; 99.5%; MW; 84.93 g/mol, purchased from Fisher (Pittsburgh, USA); Acetone (C_3H_6O) (Assay; 99.5% w/w MW; 58.08 obtained from Sigma-Aldich, USA); Perchloric acid ($HClO_4$) (Assay;70%, MW; 100.454 g/mol, obtained from Sigma-Aldich, USA); and Sodium sulphate (Na_2SO_4) (Assay; not less than 99.0%, MW; 142.04 g/mol) Deionised water.

2.2 Sampling and Study Area

Soil samples were taken at several locations in a Zig-zag pattern from four different locations Kubanni Point 1 (KP1), Kubanni Point 2(KP2), Kubanni Point 3 (KP3) and Kubanni Point 4 (KP4) on the study area. Each of the location is divided into five (5) different areas. In each of the area, ten samples (10 g) each were taken and a composite sample (20 g) was obtained from the ten (10) samples taken using coning and quartering method. A total of twenty (20) composite samples were obtained, five (5) each from the four locations (KP1, KP2, KP3 and KP4) which made agood representation of the entire study area. Another five samples (10 g) each from Kofan Kibo (KP3) where there are no agricultural activities was taken which served as a control, to the study area (Adeola et al., 2015). The sampling state and local government area is as depicted in Figure 1 and 2 respectively.

Figure1: Map of Nigeria, showing Kaduna State and Zaria

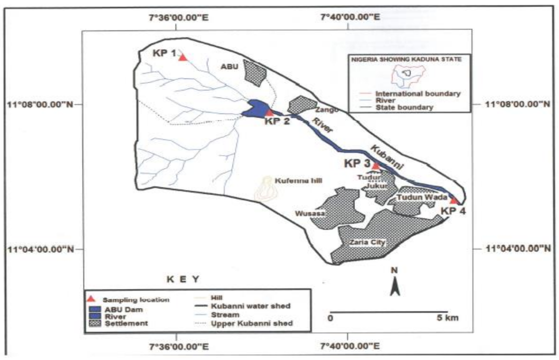

Figure 2: Map of Kubanni area showing the agricultural soils where sampling was conducted. (Source: NCAT Zaria).

2.3 Sample Digestion

The samples were digested using Baker and Amacher method. Each of the Kubanni point was considered individually. Two grams (2 g) of the sample was transferred to a Teflon beaker on a hot plate, distilled water (25 cm^3) was added and mixed with 2cm^3 conc. HNO_3 and was allowed to cool. About 3 drops of conc. H_2SO_4 and HF(10 cm^3) was added. The sample were then placed on a sand bath and subjected to heat untill the temperature rose to 100^oC and allowed to evaporate to dryness. Addition of conc. HNO_3 (15 cm^3), H_2SO_4 (2 cm^3) and $HClO_4$ (5 cm^3) was followed under heating until a white dense precipitate was produced. The container was cooled and the resulting solution was transferred to a 50 cm^3 volumetric flask by adding distilled water. The resulting solution was transferred into clean plastic bottles and analysed using Atomic Absorption Spectrophotometer (Chimezie *et al.,* 2013).

2.4 Contamination Factor (CF) and Pollution Load Index (PLI)

The average mean values for each of the heavy metals from all the four locations were calculated and the mean value (MV) and the Reference Standard (NESREA, 2009) of each of the heavy metal were used to calculate the Contamination Factor and Pollution Load Index using equations 1 and 2 respectively:

$$CF = \frac{MV}{RF} \quad \ldots\ldots 1$$

Where, CF = contamination factor; MV = mean value (Average Concentration of the heavy metals) and RF = Reference Value (NESREA Standard).

$$PLI = (CF_1 + CF_2 + CF_3 + \ldots\ldots CF_n)^{1/n} \ldots\ldots\ldots\ldots 2$$

Where, PLI = pollution load index, CF_1 to CF_2 are the contamination factors for the various locations KP1-KP4 and n = number of heavy metals used for this research (Gong *et al*, 2008)

2.5 Extraction of PAH

The dried sample (10 g) was mixed with 50 cm^3 of the acetone in an acetone rinsed beaker. The mixed sample was placed in a sonicator and allowed to sonicate for about 10-15 minutes. Ten grams (10 g) of anhydrous sodium sulphate (Na_2SO_4) was added to the sample so as to remove excess available water from the solution. The solution was poured into a round bottom flask. The same procedure was repeated once with an additional 50 cm^3 of the acetone and the resulting solution was allowed to settle and decanted into the round bottom flask containing the first extract. The sample was fractionated using dichloromethane for the PAHs fractions by using silica gel. The column was packed with 10 g of 100-200-mesh silica and preconditioned at 105^oC overnight and mixed with n-hexane to form slurry (Wloka and Smol, 2014). The extract was used for GC-MS analysis using (GC-MS Agilent Technologies, Santa Clara, CA, USA) and Agilent 7683B Injector (Agilent Technologies, Santa Clara, CA, USA).

2.6 GC/MS Analysis

The extracts were injected in the split less mode at an injection temperature of 300 °C. The transfer line and ion source temperatures were 280 °C and 200 °C. The column temperature was originally held at 40 °C for 1 min, raised to 120 °C at the rate of 25 °C/min, and to 160 °C at the rate of 10 °C/min, and lastly to 300 °C at the rate of 5 °C/min, held at final temperature for 15 min. Detector temperature was kept at 280 °C. The carrier gas was Helium at a constant flow rate of 1 mL/min. Mass spectrometry was acquired using the electron ionization (EI) and selective ion monitoring (SIM) modes (Cheng-Di *et al.*, 2012).

2.7 Data Analysis

The descriptive numerical parameters were calculated using GraphPad Prism Statistical software Version 6.0. Data were summarized using mean and standard deviations as they `were normally distributed following normality tests. One-way ANOVA was performed to test for significance differences in heavy metal and PAHs concentrations in soil samples (Kabir, 2006).

3. Results and Discussion

3.1 Results

3.1.1 Heavy metals concentrations in soil of the study area

The mean (±SD) concentrations of Pb, Cd, Cr, Cu and Zn in study locations labelled KP1- KP4 and KP3 (Control) were presented in Table 1.

3.1.2 Polycyclic aromatic hydrocarbons (PAH) Concentrations in the study area

The mean (±SD) concentrations of Napthalene, Anthracene and Phenanthrene in the study areas [KP1-KP4 and KP3 (Control)] are presented in Table 2.

3.1.3 Contamination Factors in the study locations

The contamination factors for the various heavy metals studied in each of the Kubanni point, were calculated and presented in Table 3.

3.1.4 Pollution Load Indexes of each of the Kubanni Point (KP1-KP4)

Pollution load index of each of the kubanni point was determined from the contamination factors calculated. Table 4 showed the pollution load index of each of the Kubanni Point (KP).

Table 1: Heavy metals concentration in soil of the study area

Heavy metal	KP1	KP2	KP3	KP4	Control	p-value*
Pb (mg/Kg)	3.36±0.08	4.05±0.18	3.34±0.20	2.56±0.22	1.25±0.32	P<0.0001
Cd (mg/Kg)	2.87±0.05	2.32±0.04	3.11±0.01	4.43±0.11	0.03±0.01	P<0.0001
Cr (mg/Kg)	2.85±0.04	2.29±0.11	3.13±0.11	4.30±0.14	0.13±0.08	P<0.0001
Cu (mg/Kg)	3.32±0.25	2.92±0.04	2.39±0.10	2.42±0.15	1.03±0.13	P<0.0001
Zn (mg/Kg)	1.62±0.01	1.80±0.01	0.80±0.01	0.93±0.02	0.02±0.01	P<0.0001

Key: *obtained using One-way ANOVA. KP: Kubanni point.

Table 2: Polycyclic aromatic hydrocarbons (PAH) concentration in the study area.

PAH	KP1	KP2	KP3	KP4	Control	p-value*
Naphthalene (mg/Kg)	6.37±0.09	5.16±0.04	7.08±0.02	4.21±0.02	0.13±0.01	P<0.0001
Anthracene (mg/Kg)	4.28±0.05	4.92±0.10	5.01±0.08	7.00±0.08	0.02±0.01	P<0.0001
Phenanthrene (mg/Kg)	7.03±0.02	5.03±0.02	7.31±0.21	6.31±0.18	0.02±0.01	P<0.0001

Key: *obtained using One-way ANOVA. KP: Kubanni point.

Table 3: Contamination Factor for the various sampling points

	Reference value (mg/kg)*	**KP1 Mean value (mg/kg)**	**CF**	**KP2 Mean value (mg/kg)**	**CF**	**KP3 Mean value (mg/kg)**	**CF**	**KP4 Mean value (mg/kg)**	**CF**
Pb	164	3.36	0.02	4.05	0.03	3.34	0.02	2.56	0.02
Cr	100	2.85	0.03	2.29	0.02	3.13	0.03	4.30	0.04
Cd	3	2.87	0.96	2.32	0.78	3.11	1.04	4.43	1.48
Cu	100	3.32	0.03	2.92	0.03	2.39	0.02	2.42	0.02
Zn	421	1.62	0.004	1.80	0.004	0.80	0.002	0.93	0.002

Key: *National Environmental (Food Bever1ages and Tobacco) Regulations, 2009; CF: Contamination Factor; KP1-4: denote locations

Table 4: Pollution Load Index from KP1-KP4

	CF_1 x CF2 …CF_n	**PLI=(CF_1 x CF2…CF_n)$^{1/n*}$**	**Inference#**
KP1	7.151×10^{-08}	0	PLI Class 1=0; Moderately contaminated
KP2	5.474×10^{-08}	0	PLI Class 1=0; Moderately contaminated
KP3	3.009×10^{-08}	0	PLI Class 1=0; Moderately contaminated
KP4	5.303×10^{-08}	0	PLI Class 1=0; Moderately contaminated

*n=number of heavy metals considered for the research (n=5); #Chakravarty and Patgiri (2008); CF: Contamination Factor;KP1-4: PLI: Pollution Load Index[PLI=(CF_1 x CF2…CF_n)$^{1/n*}$].

3.2 Discussion

3.2.1 Lead (Pb)

From the results obtained for the soil analysis depicted in Table 1, the highest concentration value of Lead was obtained in KP2 (4.05 mg/kg) which was lower than the permissible level of 164mg/kg set by NESREA (2009) and 100mg/kg set by USEPA (2011). The Pb concentration is significantly lower than the mean range of 351-457mg/kg reported by Adefemi and Awokunmi (2010) in a similar work conducted around a municipal dump site and agricultural farms in Iree, Osun, South west of Nigeria. On the other hand, Pb concentration in the studied area (Kubanni point) differs with the finding of Opaluwa *et al.,* (2012) who reported a lower level of Pb concentration (0.47-0.55) mg/kg in another study in Lafia, North Central Nigeria. It is significant to note that the study areas were different and this account for the wide disparity in recorded concentrations. However, the sources of Pb in soil could be as a result of discharged of dry cell batteries, run-off of waste and atmospheric depositions.

3.2.2 Cadmium (Cd)

From the results obtained of the soil analysis depicted in Table 1, the highest concentration of Cadmium was obtained in KP4 and the lowest is at KP2. However the results obtained varies with concentration of the Cadmium of (0.48 – 84)mg/kg reported by Ezeudo (2014) who obtained higher values of cadmium in a similar work in Nsukka Nigeria. Cadmium concentration was also low at KP3 (control) as compared to the other locations, but KP3 and KP4 showed higher values more than the permissible limit of 3mg/kg set by NESREA (2009). However, the high concentration of Cd in this study may be due to the deposition of atmospheric contaminant or leaching and the use of agro-based chemicals and waste containing dumped PVC plastics, Ni – Cd batteries, printer ink, and photocopying machine parts are the likely sources of cadmium in the studied area.(Nwaedozie*et al.,* 2015).

3.2.3 Chromium (Cr)

There was substantial difference in the concentration of chromium at various sampling points. The highest concentration was detected at sampling area KP4 where drainages from abattoir, runoff from mechanical and motor workshops flows into the environment at Kubanni. Maximum concentrations of chromium may be attributed to the inflowing channel from Tudun Wada which consists of various wastes from industrial facilities and domestic discharges, including automobile garages and car wash, which release mixtures of oil and car washing into the soil matrixes. Although, the value of Cr obtained in this study was below the acceptable limits of 100mg/kg (USEPA, 2011; NESREA, 2009). More so, the values of Cr obtained in this study were lower than the 75mg/kg recorded in a similar study within the Ibadan town in South – West Nigeria by Adelekan and Abegunde (2011).

3.2.4 Copper (Cu)

The maximum concentration of copper was obtained at sampling location KP1. Lowest concentration at KP3. This location (KP1) features plumbing works and manufacture of electroplating materials. Furthermore, there was substantial difference in the concentration of copper at various sampling points, ranging from KP1-KP3. The concentrations of copper in soil throughout the Kubanni area are below the specified level of 100 mg/kg set by USEPA (2011) and NESREA (2009). As stated above, the highest concentration was detected at sampling point KP1 which also has to do with the drainages from abattoir and runoff from mechanical and motor workshops which flows into the agricultural soil at Kubanni and the lowest concentration recorded at sampling location KP3 where there was no any agricultural activity. Also, the high concentration of copper may be attributed to the inflowing channel from Tudun Wada which consists of various wastes from industrial facilities and domestic discharges, including automobile garages and car wash, which release mixtures of oil and car washing into the soil matrixes (Abdulmojeed and Abdulrahman, 2011).

3.2.5 Zinc (Zn)

There was substantial difference in the concentration of Zinc at various sampling points, ranging from KP1-KP3. The highest and lowest concentrations were detected at sampling points KP2 and KP3 respectively. The concentrations of Zinc in soil throughout the Kubanni area is within the acceptable limits of 200mg/kg and 421mg/kg specified levels set by USEPA (2011) and NESREA (2009) respectively. The highest concentration obtained (KP2) can be attributed to runoff from mechanical and motor workshops which flow into the agricultural soil at Kubanni. Maximum concentrations of Zinc may also be attributed to the inflowing channel from Tudun Jukun (KP3) and (KP1) behind ABU, which consists of various wastes from industrial facilities and domestic discharges, including automobile garages and car wash, which release mixtures of oil and car washing into the soil matrixes which must have accumulated in point KP2(Adefemi and Awokunmi 2010).

3.2.6 PAH Concentrations in the Soil of the study area

The concentrations for Naphthalene, Anthracene and Phenantrene detected in the soil samples were shown in Table 2. All the soil samples exhibited detectable levels of the three species of PAHs, Naphthalene, Anthracene and Phenantrene. The levels of PAHs detected varied accordingly as follows; Naphthalene ranged from (0.13-7.08) mg/kg, Antharacene, ranged from (0.02-7.00) mg/kg, Phenantrene ranged from (0.02-7.31) mg/kg. The concentrations derived from the soil analysis are indicative of anthropogenic and agricultural activities experienced in the Kubanni bridge area. KP1(behind ABU Zaria) is known for dumpsites, agricultural/farming activities these include tilling of the soil and planting of viable seed and chemical applications such as fertilizer applications, and applications of herbicides to mention a few. All these activities may be responsible for the higher level of PAH found in the soil sample. More so, KP2 (Zango) area accommodates both residential effluents discharges, house hold waste disposal, herbicides/insecticides application and mechanic workshops may wholesomely responsible for both increasing concentration of persistence organic pollutants.

Results derived from KP3 (TudunJukun) shows remarkable amount of Naphthalene, this site is known for Mechanic workshop, dumpsites, irrigation activities and effluent from industries around the site could be responsible for this residual concentrations as recorded. More so, KP4 (Tudun Wada) area is known for agricultural activities, dumpsites and mechanic workshops etc. these activities may be responsible for residual Anthracene recorded which is un healthy for Man other living things found within the studied sites. KP3 recorded remarkable amount of Phenantrene. KP3 is located at Tudun Jukun. The area is known to have an illegal dumpsite where scavengers/vendors gather heap of metal scraps for marketing. KP3 which is the control sample was gotten from Kofar Kibo; this site has little or no agricultural activities and its said to be less in anthropogenic activities. The analysis of the PAH conducted in this research is however related with Edu *et al.* (2016) in a similar work in Ikpa river basin, Niger-Delta Nigeria in a nearby dumpsite. They analysed all the three PAHs considered in this research among other PAH. The results recorded for KP3 Kofar Kibo serves as experimental control/blank which has helped us to attain well-defined results. However the three PAH considered for this research (i.e. Naphthalene, Anthracene and Phenantrene) were all below the permissible limits of 9mg/kg, 13.4mg/kg and 85mg/kg respectively set by UESEPA (2011).

3.2.7 Heavy Metal Concentration and Contamination Assessment Contamination Factor (CF) and Pollution Load Index (PLI)

Contamination factor is considered to be a simple and effective tool in monitoring heavy metals contamination and is assessing pollution load index. According to Chakarvati and Patgiri (2008), PLI = 0 indicates moderately contamination of the study area which is similar to the findings of this research. In view of what Chakarvati and Patgiri (2008) have obtained and what is obtained in this research (Table 4), the studied area is moderately polluted by heavy metals.

4. Conclusion

It is obvious from the present study that significant amount of metal ions were found in the soil samples from Kubani area of Zaria city. More so, PAHs residues are present in the soil around the Kubani area of Zaria. Even though, the Pollution Load Index of the heavy metals indicates moderate contamination of the studied area and the levels of these toxicants were below the permissible limits set by NESREA, other than KP3 and KP4 which showed higher values of Cadmium more than the permissible limit of 3mg/kg set by NESREA. They still pose a threat since they are known to be bio-accumulative. In view of what this research has uncovered and what has been obtained from review of related literatures, indiscriminate dumping of waste within the studied area should legislatively be discouraged to avoid littering of waste into the agricultural soil; and government should educate people from industrial facilities and around the studied area on Extended Producer Responsibility (EPR). This will tremendously help in mitigating the environmental effects of the refuse discharged as well as increase the recycling and reuse of solid wastes using the best available technology.

Conflict of Interest

The authors declare that there is no conflict of interest.

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