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International Journal of Research Publication and Reviews

Journal homepage: www.ijrpr.com ISSN 2582-7421

Determination of Some Heavy Metals in Soil and Water Using High Performance Liquid Chromatography (HPLC) along the Banks of River Yadzaram.

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ABSTRACT

The determination of the level of some heavy metals concentrations in soil and water samples using high performance liquid chromatography (HPLC) along the banks of river Yadzaram (Mayobani and Biam) of Adamawa state Nigeria using standard method for the determination of heavy metals with HPLC. Heavy metals are vital and necessary in our daily lives. However if the amount of heavy metals superseded that of the acceptable limits set by (WHO and EPA 2020) in soil and water, indeed they can cause diseases in human bodies, therefore determining, monitoring and measuring the amount of heavy metals is crucial. The corresponding result of the mean concentrations of heavy metals in soil from Mayobani are given as; Fe 4.082, Pd 5.725, Zn 8.381, Cd 2.378, Cr 13.809 and As 8.231mg/kg. Water sample; Fe 0.500, Pd 0.186, Zn 0.287, Cd 0.051, Cr 0.121 and As 0.027mg/L. heavy metals in soil from Biam region given as; Fe 2.732, Pd 9.559, Zn 12.440, Cd 1.292, Cr 20.445 and As 1.221mg/kg. Water sample; Fe 0.341, Pd 0.059, Zn 0.341, Cd 0.039, Cr 0.045 and As 0.023mg/L. Almost all the heavy metals determined in water have exceeded the acceptable limits set by (WHO and EPA 2020) but those found in soil are either within or below the limits.

KEY WORDS: Heavy metals, water, soil, HPLC, Mayobani, Biam.

INTRODUCTION

The detection and quantification of pollutants are critical for environmental monitoring and assessment, High performance liquid chromatography (HPLC) has emerged as a powerful analytical technique for the separation and quantification of organic compounds including heavy metals. HPLC provides high sensitivity and specificity making it suitable for the environmental sample's detection (Ghosh *et al.,* 2021)). Heavy metals are well-known environmental pollutants owing to their toxicity, longevity in the atmosphere, and ability to accumulate in the human body via bio-accumulation. The pollution of terrestrial and aquatic ecosystems with heavy metals is a major environmental concern that has consequences for public health because most heavy metals occur naturally, but few are derived from anthropogenic sources (Saikat *et al.,* 2022). The levels and compositions of heavy metals in soil are often determined and controlled by local activities like farming while those suspended in air is monitored by the metallic properties and some environmental factors such as precipitation, rainfall and wind. (Surface and ground) water pollution by heavy metals is a global issue that requires urgent concern (Iulietto, M. 2023).

Heavy metals are important and natural components of the earth's crust occurring in varied concentrations in the ecosystem and are present in rocks, soils and air in small amounts from geological sources that influence the chemical composition and the nature of airborne particulates and dusts inhaled or ingested environmental contaminants, and their toxicity is a significant environmental issue. (Kumar *et al.,* 2022. Ogbaran and Joseph, 2022). Heavy metals are significant group of chemical pollutants that enter our bodies primarily through food, and some are irreversibly bound to human body tissues, such as cadmium to kidneys and lead to bone (Bocheva *et al.,* 2023). The presence of heavy metals at trace level and essential elements at elevated concentration causes deleterious toxic effects if exposed to human population. Serious complications associated with heavy metals contamination were observed to include damage of the nervous system, kidney disease, heart disease and infertility (Tatah *et al.,* 2020). Wastewater released from industrial process contains many heavy metals. Cadmium (Cd) is a nonessential element and a health hazard, even at very low concentrations in water. Cobalt (Co) can be used to treat anemia, but an excess of it in the human body is harmful to the hematological systems and skin allergies, the ingestion of more than

30.0μg/L uranium (U) from food and drinking water is toxic to the kidneys. The International Agency for Research on Cancer listed several heavy metals as being possibly carcinogenic to humans based on animal data. (Wu, X. 2023)

Materials and reagents: All materials and reagents are of standard analytical grade and these includes: Soil and water samples for the analysis, Nitric acid (HNO_3), Hydrogen peroxide (H_2O_2), Acetonitrile, Distilled water, Methanol, Buffer solutions (e.g., phosphate buffer, ammonium acetate).

Study Area: The study area is Michika and Mubi North LGA of Adamawa state, which is in the northeastern region of Nigeria, it covers an aerial extent of about 188.5Km2. It lies within latitudes 10^0 32'N to 10^0 14'N and latitudes 13^0 19'E to 13^0 25'E. It is bounded to the East by Republic of Cameroon, to the South by Mubi Local Government Area Adamawa State, to the West by Askira Uba Local Government Area of Borno State and to the West by Madagali Local Government Area respectively.

Sampling Location*:* 5 samples were collected from different site along the river Yadzaram banks, including the area with different land uses (e.g. Agricultural and domestic/residential)

Water Sampling: Standard water sampling procedure were adopted as described by Zira *et al.*,2018 using Grab sampling techniques for collection of all the water samples. The water samples were kept in a labelled plastic bottle and immediately preserved after collection in order to minimize degradation of heavy metals on the field. The samples were transported to the laboratory and stored in the freezer prior to analysis.

Method

Standard Analytical procedures for the determination of heavy metals in soil and water using HPLC-UV.The analysis of heavy in environmental samples were performed using high-performance liquid chromatography with ultraviolet detection (HPLC-UV) with appropriate sample preparation and chromatographic conditions (Mendoza *et al.,* 2016). Sample collection and storage followed standardized protocols to prevent degradation. Soil samples were homogenized, sieved (2 mm), and stored at 20°C in amber glass containers. Water samples (1 L) were acidified to pH 2 with hydrochloric acid, and refrigerated at 4°C to inhibit microbial activity (USEPA, 2007). Extraction of target analytes was carried out using organic solvents optimized for HPLC compatibility.

For soil, 10 g of sample was extracted with 20 mL of acetonitrile:water (8:2, v/v) via ultrasonication for 30 min, followed by centrifugation at 3000 rpm for 10 min (Zhang *et al.,* 2019). The supernatant was collected, and the extraction was repeated twice.

For water samples, solid phase extraction (SPE) was performed using C18 cartridges preconditioned with methanol and water. After loading 500 mL of sample (adjusted to pH 7), the cartridges were eluted with 5 mL of acetonitrile, and the extract was concentrated to 1 mL using rotary evaporator (Sánchez-Brunete *et al.,* 2015).

Chromatographic separation was achieved using a reverse-phase C18 column (250 mm × 4.6 mm, 5 μm) with a gradient mobile phase consisting of (A) water and (B) acetonitrile. The gradient program started at 50% B, increased linearly to 95% B over 25 min, and held for 5 min before re equilibration (flow rate: 1.0 mL/min). Detection was performed at 220 nm to maximize sensitivity for chlorinated compounds (Mendoza *et al.,* 2016). Under these conditions the compounds eluted consistent with their increasing hydrophobicity.

Quantification was conducted using external calibration curves (0.1–50 µg/mL) prepared from certified reference materials. Method validation included recovery studies (spiked at 10 µg/kg in soil and 1 µg/L in water), yielding recoveries of 75–105%, meeting acceptable criteria for environmental analysis (Sánchez-Brunete *et al.,* 2015).

RESULTS AND DISCUSION

DISCUSION

The level of heavy metals found in both soil and water samples from Biam and Mayobani region which includes; Iron (Fe), Lead(Ld), Zinc(Zn), Cadmium(Cd), Chromium(Cr) and Arsenic(As) obtained from different locations within the bank of river Yedzaram basin were determined using HPLC-UV, by performing triplicate analysis which involve performing a measurement or analysis three times to ensure accuracy and reliability and calculating the mean of the three result obtained, you can reduce errors, increase precision, detect outliers and improve reliability. (Gruber *et al.,* 2020).

From the results obtained from Mayobani region, the following shows differents mean concentrations of heavy metals determined.

The heavy metals contamination in water are listed below; with the mean concentration of Iron (Fe)0.500mg/L,Lead(Ld)0.186mg/L,Zinc(Zn) 0.287mg/L,Cadmium(Cd)0.051mg/L,Chromium(Cr)0.0121mg/L,Arsenic(As)0.027mgL.

The heavy metals contamination in soil are listed below; with the mean concentration of Iron(Fe)4.082mg/L Lead(Ld)5.727mg/L, Zinc(Zn)8.381mg/L, Cadmium(Cd)2.378mg/L, Chromium(Cr) 13,809mg/L, and Arsenic(As) 8.231mg/L.

.Iron(Fe): shows the mean concentration of soil sample is 4.082mg/L which is within the acceptable limits set by (WHO and EPA 2020), Fe has no specific limit in soil but when is in excessive level it can cause environmental issues, whereas in water Fe has a mean concentration of 0.005mg/L which exceed the acceptable limit set by (WHO and EPA 2020), and thereby causes potential health risk. Excessive Fe intake can cause health issues such as gastrointestinal problems, hemochromatosis, organ damage, (liver and heart pancreas). The environmental impact elevated level of Fe in water can affect aquatic life and alter ecosystem balance.

Lead (Pd): has a mean concentration of 5.727mg/L in soil sample, which is within the acceptable limits set by (WHO and EPA 2018) as such Pd will have no harmful effects on the environment.in the case of water sample the mean concentration is 0.186mg/L which is above the acceptable limits set, Pd with 0.186mg/L concentration in water is a great concern because it can damage (brain development, leads to reduced IQ, attention deficits and behavioral problems). Developmental issues in children and fetuses are particularly vulnerable to Pd, and also prolong exposure of Pd can harm kidney function.

Zinc (Zn): shows a mean concentration of 8.381mg/L in soil and 0.287mg/L in water sample from Mayobani region which they falls within the acceptable limits set by (WHO and EPA 2020).

Cadmium (Cd): has a mean concentration of 2.378mg/L in soil and 0.051mg/L in water, both samples are within the acceptable limit.

Chromium (Cr): shows a mean concentration of 13.809mg/L in soil which is the highest concentration from all the samples within Mayobani region and is within the acceptable limit set therefore will have no health effects human and its environment. In the case of water sample Cr has a mean concentration of 0.121mg/L, this shows a slightly high concentration of Cr in water with 0.021mg/L, perhaps this may be attributed to post harvest applications in the region.

Arsenic (As): as shown from the table below, As show a mean concentration of 8.231mg/L in soil and 0.027mg/L in water respectively. Both As in soil and water are within the acceptable limits set by (WHO and EPA 2020).

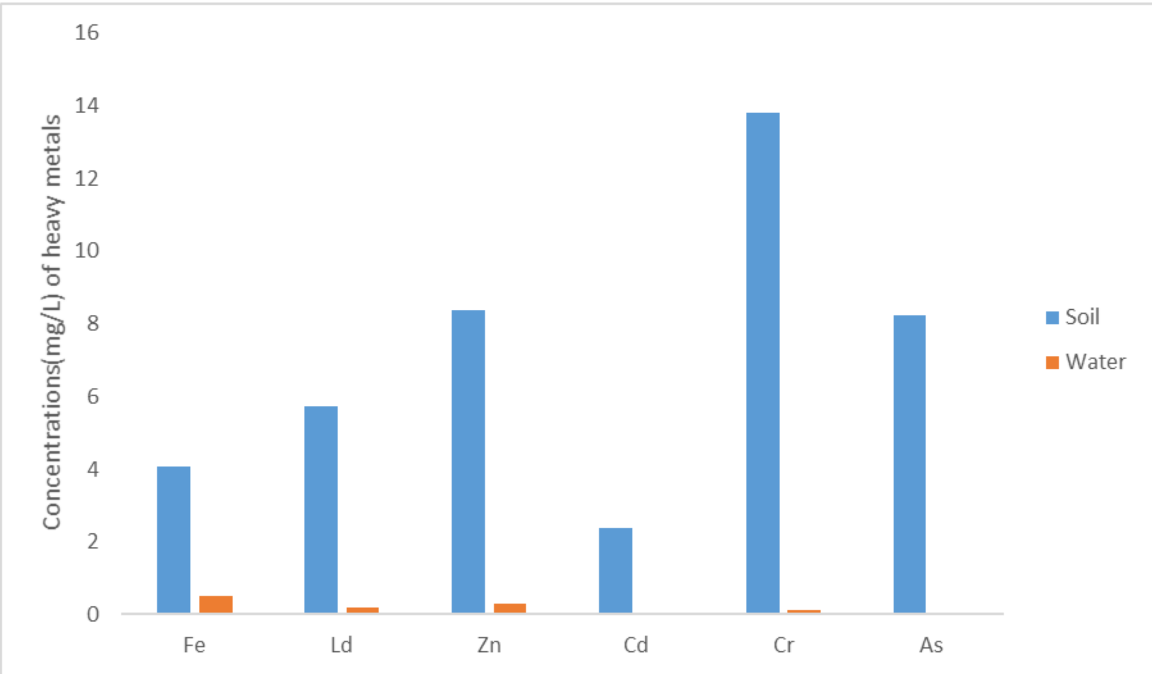

Fig 4.1: Mean concentrations (mg/L) of heavy metal from Mayobani region.

From Figure 4.1 above, the result shows that Cadmium has the lowest concentration of 2.378mg/L, while chromium happened to have the highest level of concentration of 13.809mg/L found in soil sample frm Mayobani region. Subsequently in water sample, Iron has the highest concentration of 0.500mg/L and Arsenic have the lowest concentration of 0.027mg/L respectively.

Results from Figure 4.2 shows the mean concentrations of water sample from Biam region Iron(Fe)0.341mg/L,Lead(Ld)0.059mg/L,Zinc(Zn) 0.341mg/L,Cadmium(Cd)0.039mg/L,Chromium(Cr)0.045mg/L, and Arsenic(As)0.023mg/L respectively.

Also the corresponding soil sample analysis results from Biam region detects the following heavy metals with their mean concentrations. Iron(Fe) 2.732mg/L, lead(Ld) 9.559 mg/L, Zinc(Zn) 12.440mg/L, Cadmium(Cd) 1.292mg/L, Chromium(Cr) 20.445mg/L and lastly Arsenic(As) 1.221mg/L from the result of the analysis of heavy metals obtained it can be seen that Chromium in soil sample have the highest concentration of 20.445mg/L and this concentration is within the acceptable limits according to the maximum allowable toxicant concentration (Avijit. B and Ashish K. P. 2022.).

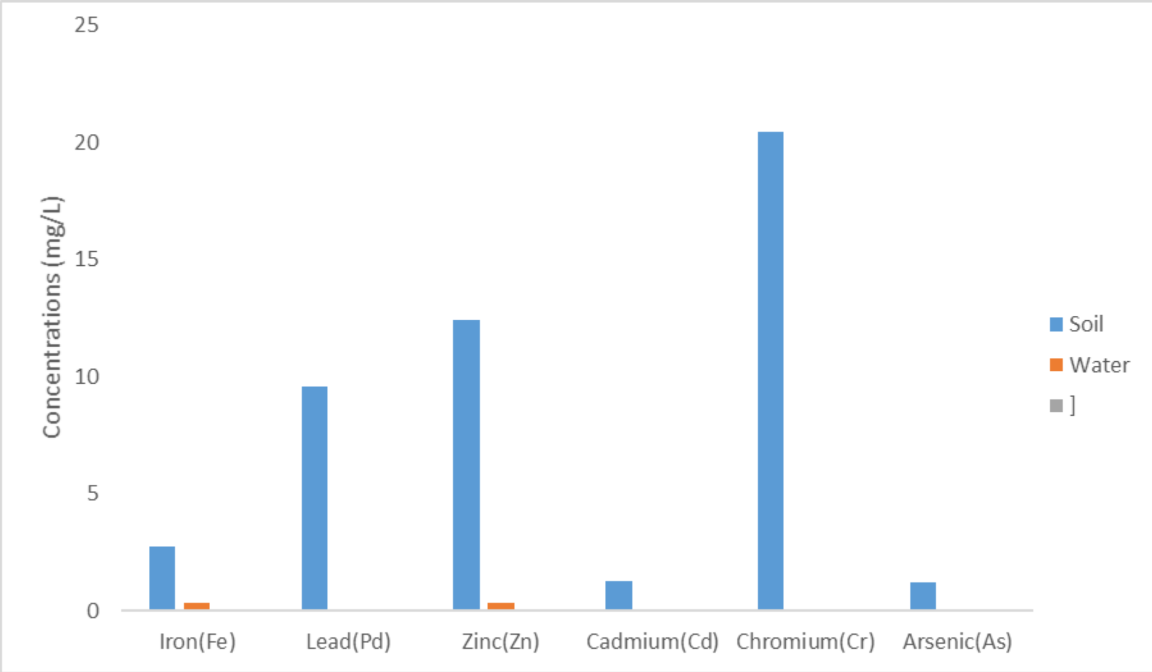

Fig 4.2Mean concentrations (mg/L) of heavy metal from Biam region

From the results of the analysis obtained from Biam region (soil and water samples);

Iron (Fe): shows the mean concentration of 2.732mg/L in soil and 0.341mg/L in water sample, in the case of Fe in soil has no specific limit but when in excess it can cause environmental issues. The concentration of Fe in water has slightly exceeds that of the acceptable limits st by (WHO and EPA 2020), with 0.041mg/L which in turn become harmful to the enviroment.

Lead (Pd): in soil Pd has a mean concentration of 9.559mg/L which is within the acceptable limits, in the case of Pd in water the concentration shows is 0.059mg/L which is a great concern comparing to the acceptable limits set by (WHO and EPA 2020) the maximum limit is 0.015mg/L, therefore this can cause neurological damage, developmental issues, cardiovascular problems, kidney damage, children and fetuses are particularly vulnerable to Pd toxic effects.

Zinc (Zn): shows a mean concentration of 12.440mg/L in soil and 0.341mg/L in water sample they are within the acceptable limits set by (WHO and EPA 2020). This implies both are safe for consumption.

Cadmium (Cd): has a mean concentration of 1.292mg/L which falls within the acceptable limits set by (WHO and EPA 2020), while in water sample it shows a mean concentration of 0.039mg/L which exceeds the acceptable limits thereby causing kidney damage, bone damage (itai-itai or pain-pain disease) and increases risk of cancer.long term exposure to cadmium can leads to serious health problems, therefore it is essential to take action to reduce cadmium exposure by using water filter that removes cadmium, identifying the source of cadmium and consulting local authorities or expert.

Chromium (Cr): shows the mean concentration of 20.445mg/L in soil which possess the high concentration of the sample from within Biam region and 0.045mg/L in water sample, both falls within the acceptable limits set by (WHO and EPA 2020) respectively.

Arsenic (As): shows a mean concentration of 1.221mg/L in soil which is within the acceptable limit, while the concentration shows in water sample is 0.023mg/L that slightly exceed the acceptable limits, therefore it is essential to take action to reduce the Arsenic exposure in water from Biam region.

CONCLUSION

The study has been able to determined different heavy metals in soil and water, from the banks of river Yadzaram, North East part of Adamawa State Nigeria. The study also provided preliminary concentrations using Standard analytical procedure for the determination of heavy metals in soil and water using HPLC-UV to carry out the analysis and the corresponding result of the mean concentrations (ppm) of heavy metals in soil from Mayobani are given as; Fe 4.082, Pd 5.725, Zn 8.381, Cd 2.378, Cr 13.809 and As 8.231. Water sample; Fe 0.500, Pd 0.186, Zn 0.287, Cd 0.051, Cr 0.121 and As 0.027. heavy metals in soil from Biam region given as; Fe 2.732, Pd 9.559, Zn 12.440, Cd 1.292, Cr 20.445 and As 1.221. Water sample; Fe 0.341, Pd 0.059, Zn 0.341, Cd 0.039, Cr 0.045 and As 0.023.

The study has shown that there are considerable amount of heavy metals in soil and Water samples, in which some of them fall within while some are above the acceptable limit set by WHO and EPA. In the case of Fe in soil and water samples from Mayobani region, the level in water is high than the acceptable limit whereas that of soil sample is within the limit set. subsequently Pd, Zn, Cd, Cr, and As. Have exceeded the acceptable limit in water but are within the acceptable limit in soil set by WHO and EPA in soil.

The mean concentration of the analysed heavy metal in water sample from both region shows a significant difference compare to the acceptable limit set by WHO and EPA. Therefore, there is need for urgent intervention from local regulatory monitory of water complain to take action.

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