



## Prevalence of Some Heavy Metals, Physicochemical Parameters and Pesticide Residues in Water and Soil of River Nukkai Jalingo Taraba State, Nigeria

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### ABSTRACT

Pesticides residues and heavy metals contaminants became threats with alarming effects on ecosystem around River Nukkai in Jalingo, Taraba State were human activities discharge pesticides residues and heavy metals within the environment. The study was aimed to ascertain the concentrations of heavy metals in the water and soil using Atomic Absorption Spectrometry (AAS) and the concentrations of pesticide residues in water and soil using GC-MS. The result revealed marked variations of distribution of the physicochemical parameters from all the three sampling sites of the River Nukkai. pH, temperature, EC, TDS, and TSS have values that fall below the WHO and USEPA permissible limit for drinking water with exception of turbidity (119.03 mg/l). The mean concentration of heavy metals examine in water and soil at sampling points revealed are; Zn (0.06 mg/L), Cu (0.15 mg/L), Pb (0.10 mg/kg), Cd (0.07 mg/kg), Zn (22.27 mg/kg), Cu (3.27 mg/kg), and Cr (2.98 mg/kg) determined in water and soil respectively were within international safe limits while Cd (0.03 mg/l), Pb (0.04 mg/l) and Cr (0.39 mg/l) levels in water are beyond (0.003, 0.01 and 0.05mg/l) USEPA safer limits required for clean water. Within the seven sampling points, total of 13 pesticides were analyzed in both water and soil samples at the area. The concentration of pesticides in sampling sites of river Nukkai, ranged from RNI = 0.039649 mg/L to 3.37558 mg/L, RNM = 0.013662 mg/L to 0.92898 mg/L, and RNO = 0.118807 mg/L to 8.27369 mg/L respectively. While the concentration of pesticides in sampling sites of agricultural soil around river Nukkai banks, ranged from RNSN = 0.017585 to 25.5124, RNSE = 0.100216 to 47.1208, RNSS = 0.214391 to 31.6663, and RNSW = 0.029031 to 27.9176 mg/kg respectively. Methoxychlor and P, P' -DDE are the predominant contaminants of water while Aldrin and Alpha-lindane are the major contaminants of soils. It was also observed that all the pesticide residues and heavy metals concentration detected at the area are beyond the Maximum Residue Limits set for drinking water and agricultural soils, due to excess utilization of pesticides by the citizens of the area.

**Keywords:** Water, Soil, Contaminants, Heavy metals, Pesticide residues, concentration

### INTRODUCTION

Pesticide residues in the soil and water caused threats due to human activities, resulting to gradual degradation and deterioration of the natural factors of ecosystem in environment as pollutants (Prajapati, and Meravi, 2014). Effluence of heavy metals contaminate the surface soil arises from industrial revolutions of advancement in economy. Tracing the origin of heavy metals and pesticide residues

they progress towards ecological environment through anthropogenic activities, such as mining, smelting, sewage sludge disposal, application of pesticides and inorganic fertilizers, and atmospheric deposition. Recent economic developments in agriculture and industry have transform natural environmental features with polluted effluences (Ali, *et al.*, 2019).



Heavy metals and pesticides are environmental contaminants destabilizing the habitats of entire ecosystem, by changing physical and functional features (Chin, 2010). Heavy metals are metals with a high atomic weight and a density greater than  $5 \text{ g/cm}^3$  (Zhang, *et al.*, 2019). Researchers investigated that their toxicity level in many polluted environment exceeded permissible limits of residue limits as prescribed by international organizations where Cd, Pb, Cu, Cr and Zn cause a tremendous threats to environment and health risks problems. The menace of heavy metals harmful effect caused threats that needed urgent remediation's to reduce the hazards to a minimal stage. Although heavy metals penetrated several organs of both plants and humans causing harmful effects beyond permissible limits. Many researchers reported that the origin of heavy metals initiated from farming practices, mining, chemicals spraying, and industrial effluence. Xiao *et al.*, (2017) who described that industrial and farming contributed immensely in heavy metals contamination on soil compositions which are absorbed by plants roots, consequently occurring at cement and other electron discharging effluent industries. Top soil constitute the layer that contain humus in association with heavy metals interacting through water absorption into plant tissues. The health risks caused by toxicants increased rapidly due to their penetration and accumulation through the food chain, and their persistence in the ecosystem resulted to alarming loss of life (Verger, and Boobis, 2013). Reports showed that human health risk on heavy metals treats due to constant exposure have drastic effect on human health particularly teenage and infants became victims (Lu *et al.*, 2014).

Advancement in technology have mutilated the ecosystem and human's health by contact of harmful chemicals through application

chemicals and fertilizers (Özkara, *et al.*, 2016). Pesticides spread during farming activities destroyed pest but causes bioaccumulation affected food chains resulted to health risk to the entire ecosystem (Liu, *et al.*, 2016). Pesticides pose hazards to humans and many organisms through polluted food chain, by process of inhalation, ingestion and dermal contact of contaminated air (Kim, *et al.*, 2017). Exposure of many pollutants of pesticides beyond permissible level evolved hazardous effects to the active behavior and physiology of flora and fauna (Mascarelli, 2013). Furthermore, pesticides effect on humans instigated to a numerous diseases and causalities that lead to death (Sharon *et al.*, 2012).

## MATERIALS AND METHODS

### Study Area

River Nukkai is a major river flowing along the north-western outskirts of Jalingo, Taraba State. It originates from the mountains and hills of the Mambilla Plateau and flows through Jalingo before joining the Benue River. The river form as a natural boundary between Jalingo and Lau Local Government Areas. River Nukkai is known for its scenic beauty, with lush vegetation and diverse wildlife manifested along its banks. The service of the river for local communities in providing water source for supporting agricultural activities and providing a habitat for various aquatic species. River basin of Nukkai was constantly polluted with unsustainable human activities. Agricultural run-off, industrial waste, and improper sewage disposal can adversely impact its water quality and ecological balance. It is vital to implement measures such as proper waste management, community education, and regular monitoring of water and soil quality to preserve the integrity of water and farm land around the river banks and ensure their sustainable use (Figure 1).

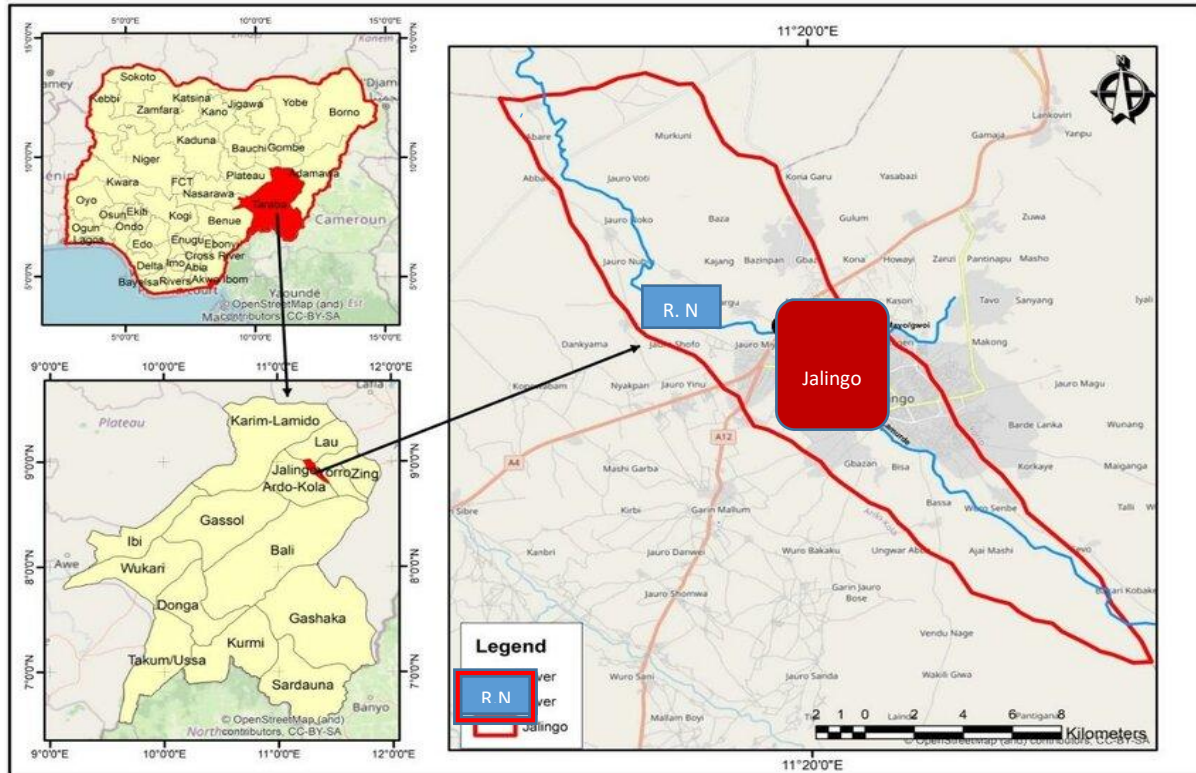


Figure 1: Map Jalingo showing study area

## Sampling and Sample Preparations

### Water sampling

The water samples were collected by at the bottom water depth of one meter in the sampling location using a plastic bottles. The sample was collected from three locations at Inlet (RNI) Middle (RNM) and Outlet (RNO) of the river at three different points which are five meters away from each sample area that formed a composite sample. Every sampling locations, was allocated a separate sampling clean bottles before collection of the sample. The samples were kept upon pouring 2 ml of concentrated  $\text{HNO}_3$  that prevented metal adsorption onto the inner surface of the container. As adopted with slight modifications by Jacob *et al.*, (2015). All the samples were kept in an ice bag that maintain the water quality integrity before transporting to Chemistry Lab. and analysis was carried out immediately.

### Soil sampling

The soil samples were collected at the river basin farm lands of Nukkai river banks. About 1 kg of the soil samples was collected from four different points; North (RNSN), East(RNSE), South (RNSS), and West (RNSW) which are approximately five meters away from each other, they were then mixed together that emanated represented sample. The top soil layer was extracted during sampling using dried garden hoe by scooping soils and then stored in a pre-cleaned 1000ml polyethylene container, labelled and transported Laboratory for further treatment and analysis. Samples of soil crumbs from sampling sites were dried in air at the laboratory then sieved through a 2 mm sieve to remove coarse particles and other traces of heavy metal impurities as reported with slight modifications by Momohshuaibu *et al.*, (2022).



## Physicochemical Properties

### *pH, TDS and EC*

The pH, of the water samples were recorded using HANNA HI 9810 pH meter. The meter was standardized with a buffer solution (i.e. buffer 7 and 9). The buffer tablet was immersed with 100 cm<sup>3</sup> distilled water in a 250 ml beaker. The probe of the meter of any of the instrument mention above was inserted in the solution and was adjusted to read 000 to standardize it. The electrode response was checked by measuring the pH. TDS and Electrical conductivity (EC) of the water samples were tested first with distilled water and then with the samples using the standard instruments. Analyzed samples always settled, then accurate reading was recorded as adopted with little modifications by Jacob *et al.*, (2015)

### *Turbidity of water*

Samples of water collected was shaken vigorously then air bubbles are removed using Nephelometric method. Exactly 100 cm<sup>3</sup> of shaken water sample are poured into a cell and the turbidity meter which immediately displayed the recorded values. The turbidity meter (HACH 2100P) was calibrated using a turbidity standard reagent before any sample reading was taken in according to Jacob *et al.*, (2015).

### *Total Suspended Solids of water*

Gravimetric method was adopted that measured the TSS of the samples, exactly 100 cm<sup>3</sup> of the water sample was filtered using pre weighed filter paper. The filter paper used was dried between 103°C and 105°C. TSS was calculated using the formula: Total Suspended Solids (TSS) mg/l = (A-B) × 10<sup>3</sup>/C

Where A=weight of filter plus solids (g); B=weight of filter (g); C=volume of sample filtered (ml) as reported by Jacob *et al.*, (2015).

## Analysis of Heavy Metals using AAS

About 20 cm<sup>3</sup> of water sample was mixed thoroughly with 20 cm<sup>3</sup> concentrated HNO<sub>3</sub> acid and transferred into beaker (250 cm<sup>3</sup>). The mixture was heated while addition of concentrated HNO<sub>3</sub> was incorporated until the solution evaporated to a minimal 20 cm<sup>3</sup> of the content, where digestion was completed at that point a light color signal showed clear solution. The content was allowed to cool at room temperature. The content was transferred to pre cleaned plastic bottle with capacity of 20 ml, which was diluted with distilled water up to its peak mark. Portions of this solution was used for heavy metals analysis using Atomic Absorption Spectroscopy (AAS) as adopted by Jacob *et al.*, (2015).

## Pesticide Residues Extractions from Water Samples

Solvent Extraction Method was used in which the water sample was collected and filter using a suitable filter to remove any particulate matter. About 20 cm<sup>3</sup> of the filtered water sample was poured into a beaker 250 cm<sup>3</sup>. Pesticide residues from the water sample was extracted by adding about 20 cm<sup>3</sup> of a suitable organic solvent such as acetone, hexane etc. The content was mixed thoroughly by shaking the container for a defined period. The settled mixture separated into two layers where the organic solvent phase was carefully poured into a clean sample vials using separation funnel, then extracted organic solvent phase was analyzed to assess the concentration of pesticide residues using GC-MS as adopted with slight modification by Usman *et al.*, (2021)

## Digestion of Soil Samples for Heavy Metals Analysis

Soil samples collected are kept for air dried, then constant weight attended and sieved using a 2 mm mesh (Cai, 2019). 1g of samples were weighed using a weighing balance and place



in 250 cm<sup>3</sup> beakers separately and 15 cm<sup>3</sup> of aqua regia (HCl and H<sub>2</sub>SO<sub>4</sub>) in ratio of 3:1 was added. The mixture was digested at 70 °C till the solution become transparent as adopted by Jacob *et al.*, (2015). The resulting solution was filtered through filter paper and poured into a sample bottle and filled with distilled water to the mark level. The sample solutions were analyzed for concentration of Cr, Zn, Cd, Cu, and Pb using an atomic absorption spectrophotometer.

### Extraction of Pesticide Residues from Soil Samples

A mixture of 2g of soil sample and 4g granular sodium sulphate was ground into a powder with a mortar and pestle. The ground sample was extracted with 20 cm<sup>3</sup> of a mixture of n-Hexane and acetone (1:2). The extract was poured into a round bottom flask and about 20 cm<sup>3</sup> and heated on a water bath at constant temperature of 50 °C - 55 °C. The concentrated solvent extract were evaporated with a rotary evaporator to least of 5 cm<sup>3</sup>. The concentrated solvent extract was accurately poured to a centrifuge tube, concentrated with nitrogen evaporator to 0.5 cm<sup>3</sup> and diluted with 2 cm<sup>3</sup> of n-hexane which before taken to GC-MS for analysis as described by Momohshuaibu *et al.*, (2022).

## RESULTS AND DISCUSSION

### Physiochemical Analysis

Physicochemical analysis of water samples analyzed are; pH, Electrical conductivity, Temperature, Turbidity, Total Suspended Solids (TSS), and Total Dissolved Solids (TDS). The results obtained are expressed in respect to the specific units of measurement which displayed Mean and Standard Deviations compared with recommended permissible limits.

### pH Values Sample Water

The mean pH values recorded for sampling points are; RNI= 4.61, RNM=6.73 and RNO=5.33 as represented on Figure 2. Sample RNM (pH= 6.73) from the middle point of the river was the most acidic among all the analyzed water samples. The acidic pH of the water bodies arose from dissolved substances, originated rock surfaces, soil and other waste water. The maximum pH value recorded was lower than 8.22, and 8.1 as reported in similar research of water bodies (Arimieri, *et al.*, 2014). Values of pH recorded ranged between 4.61 and 6.73 which are almost synonymous to the work of Jacob *et al.*, (2015) with a pH range between 4.90 and 7.30 in water. Thus, pH of all the locations investigated were within the 6.5-8.5 WHO, (2016) standard limits for drinking water.

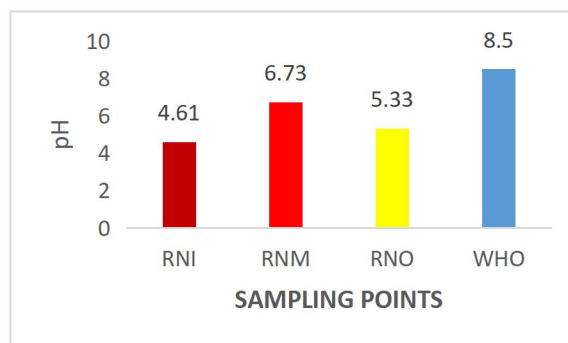
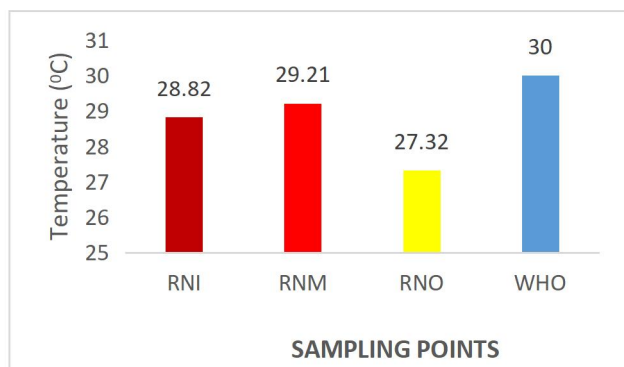


Figure 2: Mean pH values of surface water.

### Temperature of surface water

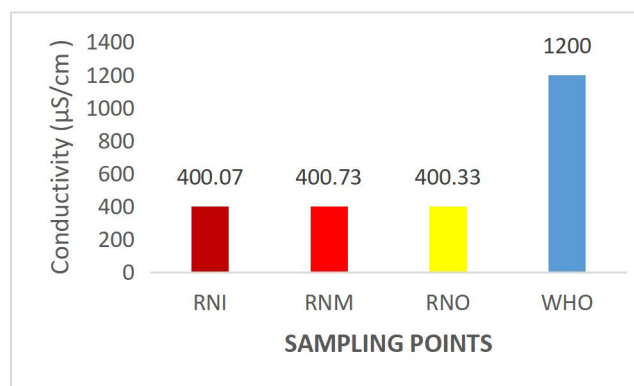
The temperature values recorded was presented on Figure 3 where temperature of the water at inlet= 28.82<sup>0C</sup>, middle =29.21<sup>0C</sup> and outlet= 27.32<sup>0C</sup> sampling points fell within mean values. The temperature values obtained agreed with the results of (Benjamin, *et al.*, 2023) which shows temperature values between 27<sup>0C</sup> to 32<sup>0C</sup>. The result showed that water temperatures increase, decrease or fluctuate at certain locations, hence metabolic activities of microorganisms subsequently malfunction or stop altogether.



**Figure 3:** Mean Temperature values of surface water.

### Electrical Conductivity of Water

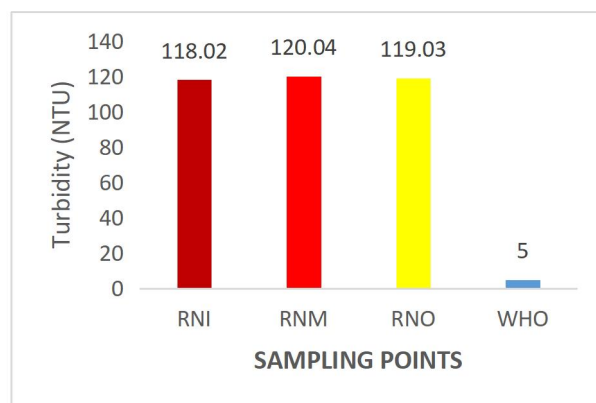
The mean conductivity values recorded obtain are presented in Figure 4. The total mean conductivity values recorded are; inlet = 400.07, middle = 400.73, and outlet= 400.33 ( $\mu\text{S}/\text{cm}$ ) sampling points of the river respectively. Conductivity values obtain revealed at sampling point of RNM (400.73  $\mu\text{S}/\text{cm}$ ) of the river contain the highest conductivity while RNI (400.33  $\mu\text{S}/\text{cm}$ ) was lowest conductivity with appreciable amounts of dissolved ions than the other sampling points. This may be due to increase or decrease of human and other biological activities in the water bodies. The EC of all the locations investigated were within 1200  $\mu\text{S}/\text{cm}$  the permissible limits for drinking water as reported by Benjamin *et al.*, (2023).



**Figure 4:** mean conductivity values ( $\mu\text{S}/\text{cm}$ ) of surface water.

### Turbidity (NTU) of Water

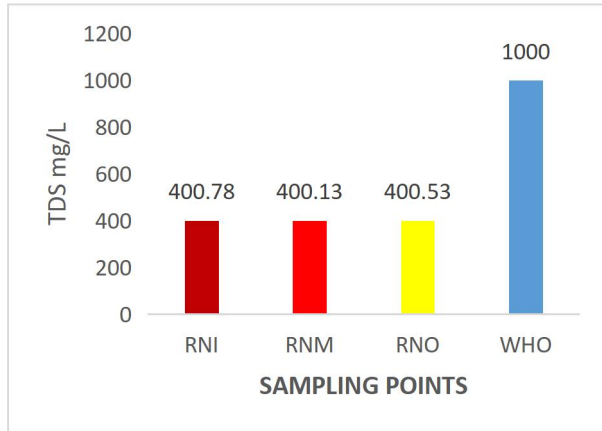
The mean turbidity values recorded as indicated on Figure 5 are; the inlet=118.02, middle=120.04 and outlet=119.03(NTU) of the river sampling points respectively. Results obtained revealed that RNM (120.04  $\mu\text{S}/\text{cm}$ ) as the highest turbid water while RNI (118.02  $\mu\text{S}/\text{cm}$ ) showed the lowest turbid water samples. Analyzed waters are quite turbid when compared to the value of NTU reported by Arimieri *et al.* (2014) in Warri River. Mean turbidity values of the water samples recorded are higher than the value of 5.0 NTU on guideline for drinking water reported by (WHO, 2008).



**Figure 5:** Mean Turbidity values (NTU) of surface water.

### Total Dissolved Solids of Water

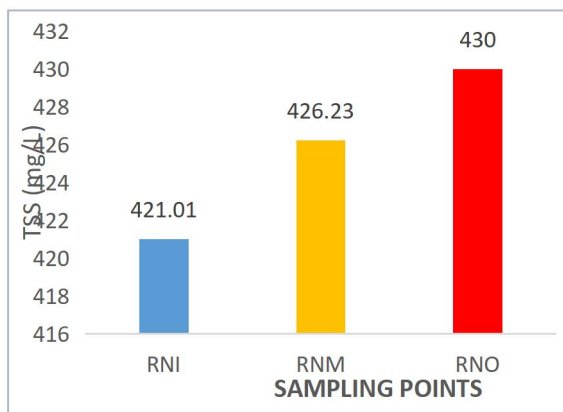
The mean Total Dissolved Solids (TDS) values are recorded as illustrated on Figure 6 were inlet= 400.78, middle= 400.13, and outlet = 400.53 (mg/L as TDS) of the river respectively. The higher TDS values observed accredited to dilution effect and pH derived from contact effects on the solubility of substances in the river as reported by Arimieri *et al.*, (2014). TDS values measured are below the WHO (2008) values of 100 mg/L as reported on guideline of domestic water supply for safety of organism in aquatic life .



**Figure 6:** Mean TDS values (mg/L) of surface water.

### Total Suspended Solids (mg/L)

The mean Total Suspended Solids (TSS) values obtained are inlet = 421.01 mg/L, middle = 426.23 mg/L, and outlet = 430.00 mg/L across the sections of the river respectively as represented on Figure 7. TSS recorded values obtained varied from 421.01 mg/L to 430.00 mg/L. Higher TSS values recorded are initiated to runoffs and soil erosion from cultivated fields arose by human activities as reported by Benjamin, *et al.*, (2023). Results obtained on values of TSS in the entire sampling locations surpassed 50 mg/L as reported on the WHO (2008) for guidelines on domestic water supply for the safety of aquatic biomass.

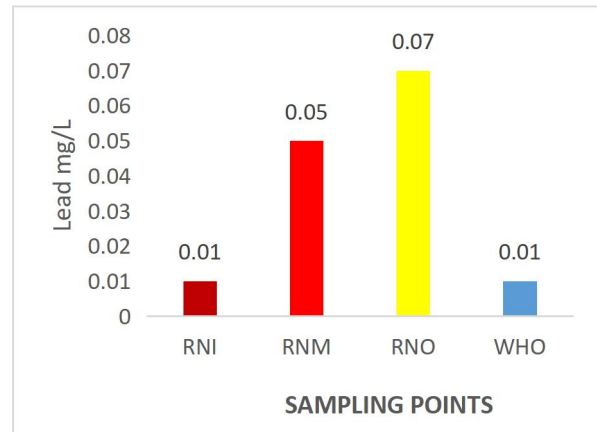


**Figure 7:** Mean TSS values (mg/L) of water.

## Concentration of Heavy Metals in Water Samples

### Lead

The mean lead fixation recorded for analyzed water samples showed  $RNI=0.01\pm0.00 < RNM=0.05\pm0.00 < RNO=0.07\pm0.00$  (mg/L) in increasing order of the sampling sites as illustrated in figure 8. Mean value of Pb obtain on the sampling locations ranged between  $0.01\pm0.00$  mg/L to  $0.07\pm0.00$  mg/L. Observed values of Pb=0.07 mg/L at the sampling point of RNI sustain within the permissible limit set up by WHO (2008) which was indicated on Figure 8 showed low level of Pb contamination in that sampling point of the river. However, at sampling points of RNM and RNO the observed values of Pb investigated was found beyond the permissible limit set up by WHO (2008) which indicated adverse pollution. The high values of Pb recorded at RNM and RNO sampling points in the study area manifested from waste dumpsites close associated at these sampling points.



**Figure 8:** Mean lead values (mg/L) of surface water.

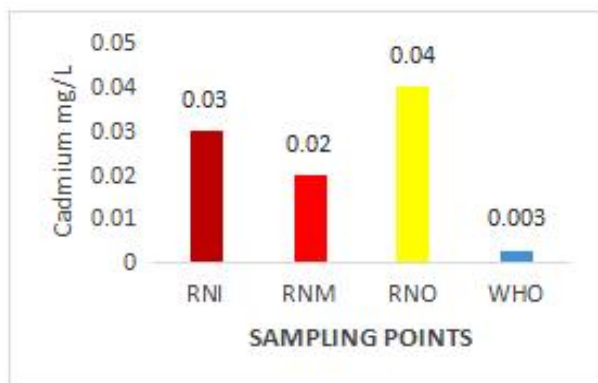
### Chromium

The mean concentration values of chromium in the water samples from sampling points in increasing order showed  $RNI=0.22\pm0.01$  mg/L  $< RNM=0.56\pm0.01$  mg/L  $< RNO=0.93\pm0.01$

mg/L are illustrated on Figure 8. Chromium values obtained are contrary to the results of Cr of 0.01 and mg/l, 0.01 mg/l as reported by Oyatayo *et al.* (2015). Also the mean concentration values of chromium obtained are far below the results obtained by Benjamin, *et al.* (2023) in Assessment of Agro Allied Chemical Pollution on Surface Water Quality of River Lamurde and Mayo-Gwoi.

### Cadmium

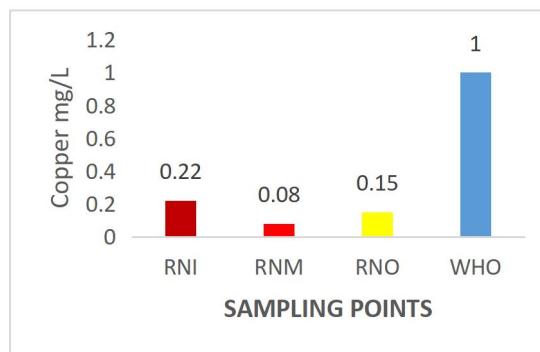
The level of cadmium concentration investigated was tagged at sampling stations in increasing order RNM =  $0.02 \pm 0.00$  mg/L < RNI =  $0.03 \pm 0.00$  mg/L < RNO =  $0.04 \pm 0.00$  mg/L respectively as shown in Figure 9, whereas the highest value of cadmium was recorded at RNO which was  $0.04 \pm 0.00$  mg/L followed by RNI =  $0.03 \pm 0.00$  mg/L and the least value of Cd was shown by RNM =  $0.02 \pm 0.00$  mg/L. The measured estimated concentration of Cd at various destinations of the River Nukkai ranged between  $0.02 \pm 0.00$  mg/L to  $0.04 \pm 0.00$  mg/L for all the sampling sites which exceeded the standard values 0.003 mg/L set by WHO (2008), indicated that Nukkai river water was highly contamination with heavy metal of Cd that could be hazardous to inhabitants of the area.



**Figure 9:** Mean Cadmium values (mg/L) of surface water.

### Copper

The average concentration of copper investigated in River Nukkai was recorded in decreasing order of RNI=  $0.22 \pm 0.02$  mg/L > RNO=  $0.15 \pm 0.02$  mg/L > RNM=  $0.08 \pm 0.02$  mg/L respectively as illustrated in Figure 10. Sample site of RNI recorded the highest value of Cu  $0.22 \pm 0.02$  mg/L followed by RNO ( $0.15 \pm 0.02$  mg/L) and least values goes to with RNM ( $0.08 \pm 0.01$  mg/L). The results obtained was contrary to World Health Organization (WHO), required permissible value for Cu of 1.0 mg/L in drinking water. The observed value for Cu obtained was found below the permissible limit as reported by WHO, thus inhabitants within the vicinity of river Nukkai cannot be at health risk of Cu pollution by drinking of water at that river.



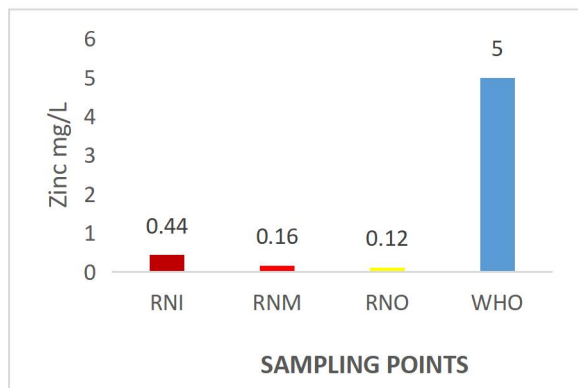
**Figure 10:** Mean Copper values (mg/L) of surface water.

### Zinc

The mean concentration of Zn obtained from the analyzed water samples are shown in decreasing order of RNI =  $0.44 \pm 0.02$  mg/L < RNM =  $0.16 \pm 0.01$  mg/L < RNO =  $0.06 \pm 0.01$  mg/L respectively as illustrated in Figure 11. The mean concentration values of zinc recorded varied between  $0.16 \pm 0.01$  mg/L to  $0.44 \pm 0.02$  mg/L. Concentrations of zinc investigated are slightly beyond the range of 0.00 mg/L, to 0.001 mg/L as reported by Umoh and Etim (2023). Thus Zn metal



investigated have significant pollution in river Nakkai water.

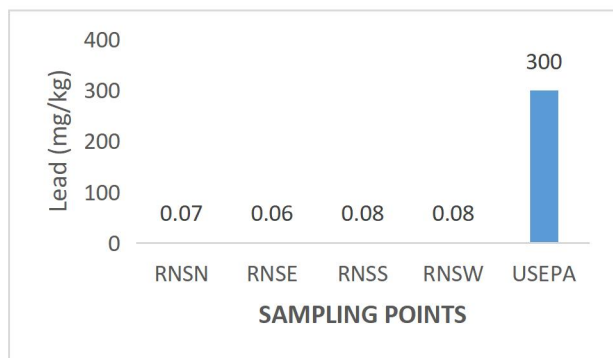


**Figure 11:** Mean zinc values (mg/L) of surface water.

### Concentrations of Heavy Metals in Soil Samples

#### Lead

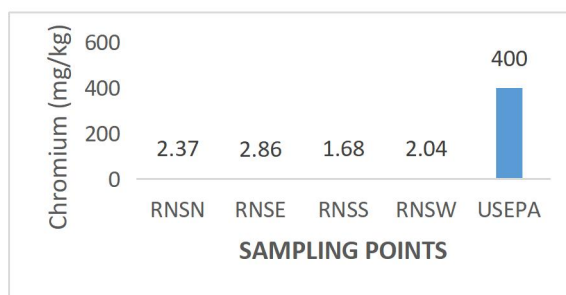
The total mean values of Pb in the soil samples are presented in decreasing order of  $RNSE = 0.06 \pm 0.05 \text{ mg/kg} > RNSN = 0.07 \pm 0.01 \text{ mg/kg} > RNSS = 0.08 \pm 0.01 \text{ mg/kg} > RNSW = 0.08 \pm 0.02 \text{ mg/kg}$ , at the sampling sites respectively illustrated in Figure 12. Results obtain showed that values of Pb in sample soils investigated was far below the permissible limit set by USEPA, (2012) of (300 mg/kg). The results obtained are also synonymous to the results investigated soil with  $Pb = 0.05 \text{ mg/L}$  as reported by Umoh and Etim (2013).



**Figure 12:** Mean Lead values (mg/kg) of Soils.

#### Chromium

The total mean values of Cr in the soil samples are shown in decreasing order for  $RNSE = 2.86 \pm 0.02 \text{ mg/kg} > RNSN = 2.37 \pm 0.01 \text{ mg/kg} > RNSW = 2.04 \pm 0.02 \text{ mg/kg} > RNSS = 1.68 \pm 0.02 \text{ mg/kg}$ , at the sampling sites respectively as represented on Figure 13. Results obtain in the soil samples investigated showed values of Cr was far below the permissible limit as reported by USEPA, (2012) of (400 mg/kg). Negligible amount of Cr stimulates the growth of agricultural crops while an excess concentration of Cr beyond the permissible limits in soil promoted various diseases at the designated environment was unfavorable for humans and animals. Also the result obtained was contrary to  $Cr = 0.002 \text{ mg/kg}$  as reported by Umoh and Etim (2023). Contaminated soil with lead-chromium batteries, coloured polythene bags, discarded plastic materials and empty paint containers are major source of Cr in the soils as reported by (Jung *et al.*, 2006).

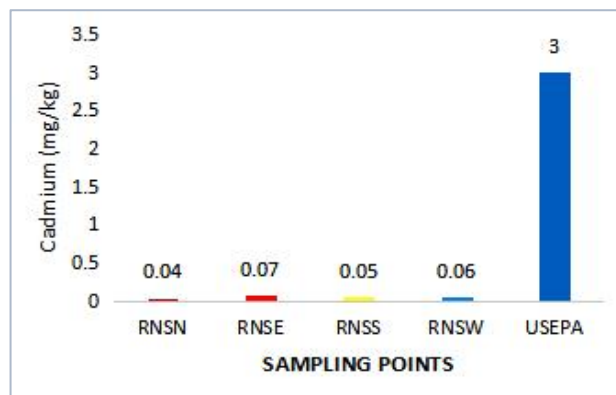


**Figure 13:** Mean Chromium values (mg/kg) of Soils.

#### Cadmium

The total mean concentration of Cd in the soil samples investigated are shown in increasing order of  $RNSN = 0.04 \pm 0.01 \text{ mg/kg} < RNSS = 0.05 \pm 0.01 \text{ mg/kg} < RNSW = 0.06 \pm 0.01 \text{ mg/kg} < RNSE = 0.07 \pm 0.01 \text{ mg/kg}$  at sample sites respectively as illustrated on Figure 14. The mean concentration of cadmium in the analyzed samples varied between 0.04 mg/kg to 0.07 mg/kg. The result

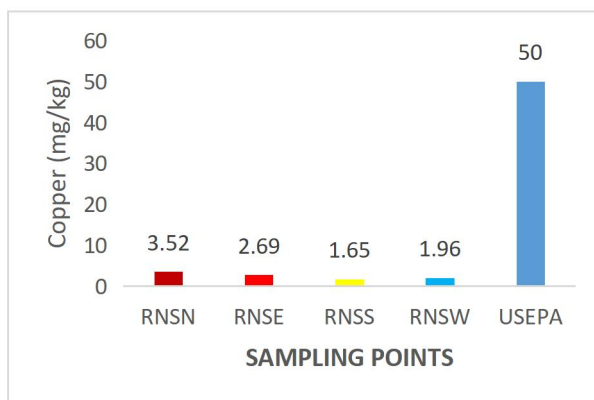
obtained is in agreement with the findings of Umoh and Etim (2023). Also the result obtain was far below the permissible of Cd of 3.0 mg/L thus Cd metal does not contaminated River Nakkai water.



**Figure 14:** Mean Cadmium values (mg/kg) of Soils.

### Copper

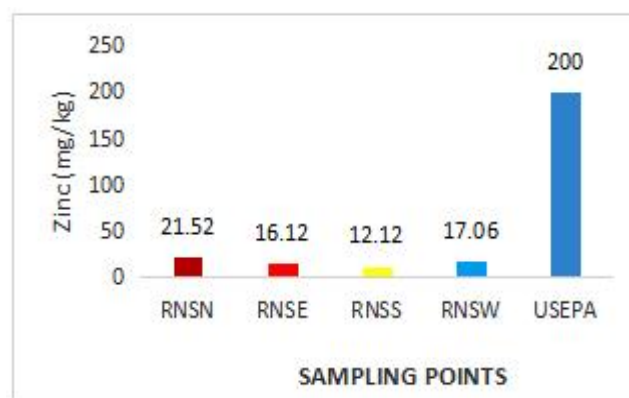
The mean concentration of Cu in the soil samples analyzed at sampling points are in decreasing order of RNSN=3.52±0.02 mg/kg > RNSE=2.69±0.02 mg/kg > RNSW=1.96±0.02 mg/kg > RNSS=1.65±0.03 mg/kg respectively as presented on Figure 15. Results obtain showed values of copper in the soil samples at sampling point RNSN (3.52±0.02) and RNSE (2.69±0.02) contained appreciable amount of copper than the concentration of copper at sampling point RNSS (1.65±0.03) and RNSW (1.96±0.02). This may be due to agricultural and anthropogenic activities carried out around RNSN and RNSE sampling sites. The result obtained showed the concentration of Cu in all the sampling soil sites of River Nukkai are far below 50 mg/kg recommended value of Cu in soil. Thus concentration Cu obtained does not contaminate river Nukkai soil.



**Figure 15:** Mean Copper values (mg/kg) of Soils.

### Zinc

The mean values of Zn analyzed in soil are in decreasing order of RNSN = 21.52±0.01 mg/kg > RNSW=17.06±0.01 mg/kg > RNSE=16.12±0.01 mg/kg > RNSS=12.12±0.02 mg/kg at sampling points respectively as represented on Figure 16. The mean values of zinc in the analyzed soil samples varied between 12.12 mg/kg to 21.52 mg/kg. Result obtain showed that values of Zn investigated in the soils are far below the permissible limit as reported by USEPA of 400 mg/kg. Thus concentration Zn obtained does not contaminated river Nukkai soil.



**Figure 16:** Mean Zinc values (mg/kg) of Soils.



## Concentration of Pesticide Residues in Water Samples from River Nukkai

### *Concentration of pesticide residues in water sample from RNI sampling point.*

The mean values of possible pesticide residues found in RNI sampling point of river Nukkai Jalingo, are presented in Table 1 where thirteen pesticide residues were detected in sample RNI, which include Delta-Lindane, Alpha-Lindane, Gamma-Lindane, Heptachlor, Aldrin, Heptachlor epoxide, Endosulfan I, P,P'-DDE, Endrin, Endosulfan II, P,P'-DDD, P,P'-DDT and methoxychlor. However, HCH possessed eight isomers, but the mutual isomers exhibited insecticides are  $\alpha$ -,  $\beta$ -,  $\gamma$ - and  $\delta$ -HCH. They are generally known as Lindane which are partially soluble in water that bonded with organic matter of the soil and are usually absorbed by the plant. The concentration of Delta, Alpha, and Gamma-lindane in RNI sampling site ranges between 0.039649 mg/L to 0.711809 mg/L respectively as shown in table 1. In addition, the values of Delta, Alpha, and Gamma-lindane recorded was higher than the

permissible limit of 0.002 mg/L as reported by WHO for drinking water. Heptachlor and Heptachlor epoxide have broad-spectrum of components that play role in termites control when in contact with subsurface injected into soil. Heptachlor is quite persistent in soil, where it is mainly transformed to its epoxide, the use of which has been banned or restricted in many countries (WHO, 2006). The concentration values of Heptachlor and Heptachlor epoxide obtained in RNI sampling point was 1.95881 mg/L and 0.315401 mg/L respectively as indicated in table 1. The values of Heptachlor and Heptachlor epoxide obtained from the sampling points are beyond maximum permissible limits of 0.00003 mg/L in water as reported by WHO, (2006). Aldrin chlorinated pesticide that is used against soil-dwelling pests, the level of Aldrin detected at sampling site of RNI was 0.088752 mg/L which was contrary to the level of Aldrin of 0.997 mg/L as reported by Usman *et al.*, (2021). The value of Aldrin obtained was beyond the permissible limit of 0.00003 mg/L in water as reported by WHO, (2006).

**Table 1:** Pesticide Residues Concentration (mg/L) of Water at RNI Sampling point.

Pesticide Residues	Types of Residue	Molecular Formula	Molecular Mass (g/Mol)	Rt (Min)	Concentration In (mg/L)
Delta-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.336	1.02007
Alpha-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.862	1.74483
Gamma-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	6.52	0.711809
Heptachlor	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub>	373	6.738	1.95881
Aldrin	Insecticide	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub>	365	7.287	0.088752
Heptachlor Epoxide	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub> O	389	8.008	0.315401
Endosulfan I	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	407	8.683	0.315401
P,P,-DDE	Insecticide	C <sub>14</sub> H <sub>8</sub> Cl <sub>4</sub>	318	9.049	3.37558
Endrin	Insecticide	C <sub>12</sub> H <sub>16</sub> N <sub>3</sub> O <sub>3</sub> PS	313	9.455	0.201384
Endosulfan II	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>2</sub> S	407	9.782	0.130742
P,P-DDD	Insecticide	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	354	9.942	0.05015
P,-DDT	Insecticide	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	354	10.503	0.039649
Methoxychlor	Insecticide	C <sub>16</sub> H <sub>15</sub> Cl <sub>3</sub> O <sub>2</sub>	346	11.492	0.137684

RT= Retention Time

Other pesticides concentrations obtain at sampling point RNI are shown in decreasing order are; P,P'-DDE=3.37558 mg/L > Endosulfan I=0.315401 mg/L >

Endrin=0.201384 mg/L > Methoxychlor=0.137684 mg/L > Endosulfan II=0.130742 mg/L > P,P'-DDD=0.05015 mg/L > P,P'-DDT=0.039649 mg/L respectively.

The recorded concentrations of studied pesticides at sampling point RNI were above maximum permissible limit of drinking water set by WHO. Different concentrations found suggested that the sources of the water pollution emanate from possible application of agrochemicals on the farmlands of the study area and wastewater detected is similar to the findings of Ademola, & Gideon (2012).

### **Concentration of Pesticide Residues in Water from RNM Sampling Point.**

The concentration of pesticide residues recorded in the water sample collected at sampling point RNM in river Nukkai. Thirteen pesticide residues were detected in sample RNM as indicated in Table 2.

The pesticide residues concentrations recorded in sampling point RNM are shown in decreasing order of Endosulfan I (0.92898 mg/L) > Aldrin (0.63677 mg/L) > Endrin (0.550129 mg/L) > Alpha-

Linden(0.488709) > Endosulfan II (0.142175 mg/L) > Gamma-Lindane (0.019684 mg/L) > P,P,-DDE (0.014361 mg/L) > Heptachlor (0.013662 mg/L) > Heptachlor epoxide (0.022366 mg/L) >P,P-DDD (0.022681 mg/L) >P,-DDT (0.032523 mg/L) > Methoxychlor (0.035245 mg/L) > Delta-Lindane (0.077852mg/L), respectively. The result obtained contradicted the result of pesticides as reported by Usman *et al.*, (2021). All the thirteen pesticide residues found in RNM water sample exceeded permissible safe limits of ( $\leq 0.001$ mg/L for any pesticide or  $\leq 0.005$ mg /L for total pesticides). This indicated that pesticides degraded very slowly and accumulate in the soil that subsequently leached out into the aquatic system of the surrounding area (Puazi, 2017). This result showed that pesticides contaminated farmlands around river Nukkai which are used in crop cultivation because of their low cost, effectiveness and availability (Zang, 2019).

**Table 2:** Pesticide Residues concentration (mg/L) of Water at RNM Sampling Point

Pesticide Residues	Types of Residue	Molecular Formula	Molecular Mass (g/mol)	Rt (Min)	Concentration In (mg/L)
Delta-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.376	0.077852
Alpha-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.851	0.488709
Gamma-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	6.577	0.019684
Heptachlor	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub>	373	6.795	0.013662
Aldrin	Insecticide	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub>	365	7.241	0.63677
Heptachlor Epoxide	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub> O	389	7.996	0.022366
Endosulfan I	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	407	8.586	0.92898
P,P,-DDE	Insecticide	C <sub>14</sub> H <sub>8</sub> Cl <sub>4</sub>	318	9.112	0.014361
Endrin	Insecticide	C <sub>12</sub> H <sub>16</sub> N <sub>3</sub> O <sub>3</sub> PS	313	9.456	0.550129
Endosulfan II	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>2</sub> S	407	9.782	0.142175
P,P-DDD	Insecticide	C <sub>14</sub> H <sub>10</sub> Cl <sub>4</sub>	320	9.942	0.022681
P,P'-DDT	Insecticide	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	354	10.474	0.032523
Methoxychlor	Insecticide	C <sub>16</sub> H <sub>15</sub> Cl <sub>3</sub> O <sub>2</sub>	346	11.521	0.035245

RT= Retention Time

### **Concentration of Pesticide residues in Water from RNO sampling point.**

The concentrations of pesticide residues in water from RNO sample are shown in Table 3. The concentration of pesticide residues detected at RNO sampling point are shown in decreasing order; Methoxychlor (8.27369

mg/L > P,P,-DDE (7.87936 mg/L) > Aldrin (4.05286 mg/L) > Alpha-Lindane (3.79215 mg/L) > Heptachlor (2.57881 mg/L) > Gamma-Lindane (1.75059 mg/L) > Delta-Lindane (0.703441mg/L) > Endosulfan I (0.637494 mg/L) > P,P'-DDT (0.169229 mg/L) > Endrin (0.160084 mg/L) P,P-DDD



(0.208084 mg/L) > Endrin (0.160084 mg/L) > Heptachlor Epoxide (0.118807 mg/L) respectively. Water from RNO sampling point exceeded the permissible safe limit of  $\leq 0.001\text{mg/L}$  for any pesticide or  $\leq 0.005\text{mg/L}$

for total pesticides. Result obtain showed that pesticides have contaminated farmlands around River Nukkai which eventually polluted river Nukkai water.

**Table 3:** Pesticide Residues Concentration (mg/L) of Water at RNO Sampling Point.

Pesticide Residues	Types of Residue	Molecular Formula	Molecular Mass (g/mol)	Rt (min)	Concentration in (mg/L)
Delta-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.393	0.703441
Alpha-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.862	3.79215
Gamma-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	6.64	1.75059
Heptachlor	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub>	373	6.755	2.57881
Aldrin	Insecticide	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub>	365	7.413	4.05286
Heptachlor Epoxide	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub> O	389	8.002	0.118807
Endosulfan I	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	407	8.631	0.637494
P,P,-DDE	Insecticide	C <sub>14</sub> H <sub>8</sub> Cl <sub>4</sub>	318	9.038	7.87936
Endrin	Insecticide	C <sub>12</sub> H <sub>16</sub> N <sub>3</sub> O <sub>3</sub> PS	313	9.432	0.160084
Endosulfan II	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>2</sub> S	407	9.793	0.341409
P,P-DDD	Insecticide	C <sub>14</sub> H <sub>10</sub> Cl <sub>4</sub>	320	9.919	0.208084
P,-DDT	Insecticide	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	354	10.457	0.169229
Methoxychlor	Insecticide	C <sub>16</sub> H <sub>15</sub> Cl <sub>3</sub> O <sub>2</sub>	346	11.447	8.27369

RT= Retention Time

### Concentration of Pesticide Residues in Soil around Nukkai River

#### *Concentration of pesticide residues in Soil from RNSN sampling point*

The analyzed soil samples from RNSN sampling location are presented in Tables 5. The pesticide residues detected at this sampling location are shown in decreasing order of Aldrin(65.257 mg/kg) > Alpha-Lindane (25.5124 mg/kg) > Gamma-Lindane (15.161 mg/kg) > P,P'-DDE (6.57728 mg/kg) > methoxychlor (4.3941 mg/kg) > Delta-Lindane (3.50859 mg/kg) > Heptachlor (2.91993 mg/kg) > P,P'-DDT (1.56033 mg/kg) > Endosulfan I (1.34088 mg/kg) > Heptachlor epoxide (0.322326 mg/kg) > Endosulfan II (0.309288 mg/kg) > Endrin (0.09533 mg/kg) > P,P'DDD (0.017585 mg/kg) respectively. Soil from RNSN sampling point exceeded the European Economic Commission (EEC) safe limit of  $\leq 0.001\text{mg/L}$  for any pesticide or  $\leq 0.005\text{mg/L}$  for total pesticides. Result obtain showed that pesticides have contaminated farmlands

around River Nukkai which eventually polluted the soil that became inhabitable to microorganisms that distorted the natural composition of the surrounding.

#### *Concentration of pesticide residues in Soil from RNSE sampling point*

Soil sampling from the eastern location (RNSE) of the river have shown concentration of pesticide residues in decreasing order as illustrated in Table 6 whereby Gamma-Lindane (58.721 mg/kg) > Aldrin(47.1208 mg/kg) > Alpha-Lindane (31.5352 mg/kg) > Endosulfan I (11.3787 mg/kg) > P,P'-DDE (8.82693 mg/kg) > P,P'-DDT (6.78577 mg/kg) > methoxychlor (5.70304 mg/kg) > P,P'DDD (2.94629 mg/kg) >

Heptachlor (2.88069 mg/kg) > Heptachlor epoxide (0.614381 mg/kg) > Endosulfan II (0.520643 mg/kg) > Delta-Lindane (0.499677 mg/kg) > Endrin (0.100216 mg/kg) respectively. Soil from RNSE sampling point exceeded permissible safe limit of  $\leq$

0.001mg/L for pesticide are  $\leq 0.005$ mg /L for total pesticides. Result obtain showed that pesticides have contaminated farmlands as

well as the environment at River Nukkai that turned the soil into toxic which became threat to humans, plants and ecosystem in general.

**Table 4:** Pesticide Residues Concentration (mg/kg) of Soil at RNSN Sampling Point.

Pesticide Residues	Types of Residue	Molecular Formula	Molecular Mass (g/mol)	Rt (min)	Concentration In (mg/kg)
Delta-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.416	3.50859
Alpha-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.868	25.5124
Gamma-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	6.652	15.161
Heptachlor	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub>	373	6.766	2.91993
Aldrin	Insecticide	C <sub>12</sub> H <sub>8</sub> C <sub>16</sub>	365	7.424	65.257
Heptachlor Epoxide	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub> O	389	7.996	0.322326
Endosulfan I	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	407	8.637	1.34088
P,P,-DDE	Insecticide	C <sub>14</sub> H <sub>8</sub> Cl <sub>4</sub>	318	9.055	6.57728
Endrin	Insecticide	C <sub>12</sub> H <sub>16</sub> N <sub>3</sub> O <sub>3</sub> PS	313	9.438	0.09533
Endosulfan II	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>2</sub> S	407	9.77	0.309288
P,P-DDD	Insecticide	C <sub>14</sub> H <sub>10</sub> Cl <sub>4</sub>	320	9.982	0.017585
P,-DDT	Insecticide	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	354	10.377	1.56033
Methoxychlor	Insecticide	C <sub>16</sub> H <sub>15</sub> Cl <sub>3</sub> O <sub>2</sub>	346	11.469	4.3941

RT= Retention Time

**Table 5:** Pesticide Residues Concentration (mg/kg) of Soil at RNSE Sampling Point.

Pesticide Residues	Types of Residue	Molecular Formula	Molecular Mass (g/mol)	Rt (min)	Concentration In (mg/kg)
Delta-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.341	0.499677
Alpha-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.856	31.5352
Gamma-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	6.651	58.721
Heptachlor	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub>	373	6.84	2.88069
Aldrin	Insecticide	C <sub>12</sub> H <sub>8</sub> C <sub>16</sub>	365	7.412	47.1208
Heptachlor Epoxide	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub> O	389	8.002	0.614381
Endosulfan I	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	407	8.683	11.3787
P,P,-DDE	Insecticide	C <sub>14</sub> H <sub>8</sub> Cl <sub>4</sub>	318	9.037	8.82693
Endrin	Insecticide	C <sub>12</sub> H <sub>16</sub> N <sub>3</sub> O <sub>3</sub> PS	313	9.472	0.100216
Endosulfan II	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>2</sub> S	407	9.77	0.520643
P,P-DDD	Insecticide	C <sub>14</sub> H <sub>10</sub> Cl <sub>4</sub>	320	9.861	2.94629
P,-DDT	Insecticide	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	354	10.371	6.78577
Methoxychlor	Insecticide	C <sub>16</sub> H <sub>15</sub> Cl <sub>3</sub> O <sub>2</sub>	346	11.452	5.70304

RT= Retention Time

**Concentration of pesticide residues in Soil from RNSS sampling point**

The analyzed soil sample concentration from the western site (RNSS) of the river was represented on Table 7 and the pesticides residues investigated are shown in decreasing order: Alpha-Lindane (27.9176 mg/kg) > P,P'-DDE (7.87569 mg/kg) > Heptachlor (2.59463 mg/kg) > methoxychlor (1.57707 mg/kg) >

Gamma-Lindane (1.3635 mg/kg) > Endosulfan I (0.814628 mg/kg), > Endosulfan II (0.606558 mg/kg) > P,P'DDD (0.479205 mg/kg) > Endrin (0.324359 mg/kg) > Delta-Lindane (0.246711 mg/kg) > Heptachlor epoxide (0.248977 mg/kg) > Aldrin (0.132528 mg/kg) > P,P'-DDT (0.029031 mg/kg) respectively. Result obtain on RNSS soil showed that pesticides have contaminated farmlands soil as well as the environment at

River Nukkai that turned the soil into toxic which became threat to humans, plants and ecosystem in general.

**Table 6:** Pesticide Residues Concentration (mg/kg) of Soil at RNSS Sampling Point.

Pesticide Residues	Types of Residue	Molecular Formula	Molecular Mass (g/mol)	Rt (min)	Concentration In (mg/kg)
Delta-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.324	0.633013
Alpha-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.856	31.6663
Gamma-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	6.566	0.214391
Heptachlor	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub>	373	6.835	2.73384
Aldrin	Insecticide	C <sub>12</sub> H <sub>8</sub> C <sub>16</sub>	365	7.413	74.3179
Heptachlor Epoxide	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub> O	389	8.013	0.91415
Endosulfan I	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	407	8.62	1.24949
P,P,-DDE	Insecticide	C <sub>14</sub> H <sub>8</sub> Cl <sub>4</sub>	318	9.043	8.73738
Endrin	Insecticide	C <sub>12</sub> H <sub>16</sub> N <sub>3</sub> O <sub>3</sub> PS	313	9.495	0.320759
Endosulfan II	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>2</sub> S	407	9.741	1.02833
P,P-DDD	Insecticide	C <sub>14</sub> H <sub>10</sub> Cl <sub>4</sub>	320	9.873	2.91488
PP,-DDT	Insecticide	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	354	10.371	6.60201
Methoxychlor	Insecticide	C <sub>16</sub> H <sub>15</sub> Cl <sub>3</sub> O <sub>2</sub>	346	11.447	6.132

RT= Retention Time

**Concentration of pesticide residues in Soil from RNSW sampling point**

The analyzed soil sample from the western site (RNSS) of the river was represented on Table 8 and the pesticides residues investigated are shown in decreasing order: Alpha - Lindane(27.9176 mg/kg) > P,P,-DDE (27.9176 mg/kg) > Heptachlor(2.59463 mg/kg) > Methoxychlor(1.57707 mg/kg) > Gamma -Lindane(1.3635 mg/kg)> Endosulfan I (0.814628 mg/kg) > Endosulfan II (0.606558 mg/kg) >P,P- DDD(0.479205 mg/kg) > Endrin(0.324359 mg/kg) > Heptachlor-Epoxide(0.324359 mg/kg) > Delta- Lindane (0.324359 mg/kg) > Aldrin(0.132528 mg/kg) > PP- DDT (0.029031 ng/kg) respectively.

The highest concentration of Alpha - Lindane(27.9176 mg/kg) could be attributed to the constant application of Alpha -Lindane insecticide around the study area which contaminated the soil. The result obtained was contrary to the pesticides residues value reported by Kim *et al.*,(2017). The

concentration of Alpha -Lindane recorded was higher than the mean value of 0.197 mg/kg as reported by (Ademola and Gideon, 2012).

Generally, the presence of these pesticide residues in all the soil samples of farmlands around River Nukkai, Jalingo indicated absolute contamination of the soil through anthropogenic activities especially agricultural activities, such as application of agrochemicals which was observed during collection of samples. It was also observed that all the pesticide residues concentration detected in this study were beyond the Maximum Residue Limits (MSLs) set on agricultural soils.

The presence of Lindane in all the soil samples attributed to wrongs usage or illegal idea of technical HCH mixtures since Lindane have been officially restricted chemical for use on farmlands (Liu *et al.*, 2016) The concentration Lindane values recorded was higher than the mean values of 0.002 and 0.0001 mg/kg recorded in soil samples beyond permissible limits.

**Table 7:** Pesticide Residues Concentration (mg/kg) of Soil at RNSW Sampling Point.

Pesticide Residues	Types of Residue	molecular formula	molecular mass (g/mol)	rt (min)	concentration in (mg/kg)
Delta-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.313	0.246711
Alpha-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	5.862	27.9176
Gamma-Lindane	Insecticide	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	291	6.634	1.3635
Heptachlor	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub>	373	6.76	2.59463
Aldrin	Insecticide	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub>	365	7.298	0.132528
Heptachlor Epoxide	Insecticide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub> O	389	7.996	0.248977
Endosulfan I	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	407	8.626	0.814628
P,P,-DDE	Insecticide	C <sub>14</sub> H <sub>8</sub> Cl <sub>4</sub>	318	9.043	7.87569
Endrin	Insecticide	C <sub>12</sub> H <sub>16</sub> N <sub>3</sub> O <sub>3</sub> PS	313	9.49	0.324359
Endosulfan II	Insecticide	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>2</sub> S	407	9.77	0.606558
P,P-DDD	Insecticide	C <sub>14</sub> H <sub>10</sub> Cl <sub>4</sub>	320	9.93	0.479205
P,P'-DDT	Insecticide	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	354	10.503	0.029031
Methoxychlor	Insecticide	C <sub>16</sub> H <sub>15</sub> Cl <sub>3</sub> O <sub>2</sub>	346	11.441	1.57707

RT= Retention Time

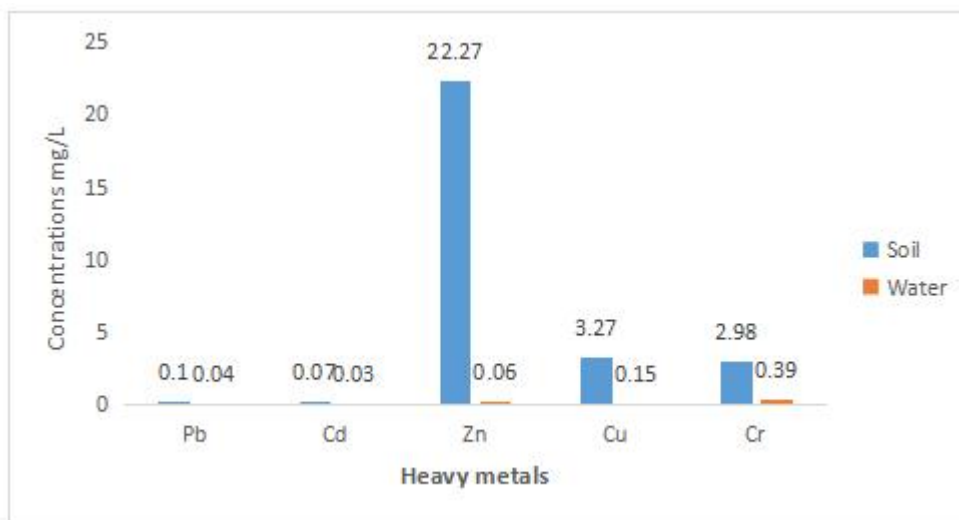
### Comparative Concentrations of Heavy Metals in Water and Soil Around River Nukkai

Comparison in the total mean concentrations of some analyzed heavy metals between water and soil samples around river Nukkai banks, Jalingo was illustrated in Figure 17. Among the studied metals in soil samples, Zn = 22.27 mg/kg was the highest metal detected in the entire soil samples followed by Cu = 3.27 mg/kg, Cr = 2.98 mg/kg, Pb = 0.10 mg/kg and Cd = 0.07mg/kg respectively. In all the studied metals, the concentrations recorded was below the permissible limit set by USEPA for agricultural soils.

The total mean concentrations of Pb (0.10 mg/L) and Cr (0.39 mg/L) in the water samples were above the permissible limits for drinking water of 0.01 mg/L and 0.05mg/L set by USEPA (2012) respectively. Their higher concentrations are beyond the permissible

limits could be carcinogenic to humans and can cause lethality to some aquatic species in the river system (USEPA, 2012). Cadmium (0.003 mg/L) mean concentration fell exactly at USEPA standard of 0.003 mg/L for drinking water, while Cu (0.15 mg/L) and Zn (0.06 mg/L) mean concentrations are detected below USEPA standard of 1 mg/L and 5 mg/L respectively for drinking water. This implies that the recorded values of Cd, Cu and Zn from river Nukkai, Jalingo have no metabolic disorders on aquatic organisms and humans. The comparison of the results of heavy metals in water and soil samples revealed highest concentrations found in soil samples, although, they are all below maximum permissible limits set by USEPA for agricultural soil while water had lower concentrations of these metals. However, some of them are above maximum permissible limits set by USEPA for drinking water. The concentrations were in the order of soil > water.





**Figure 17:** Comparative weight concentrations of Heavy metals in Soil and Water (mg/kg and mg/L).

The low concentrations of these metals in water could be attributed to the fact that sediment acts as a natural sink for heavy metals and other pollutants in surface water, leading to lower concentrations of heavy metals in water bodies (Arimieri, *et al.*, 2014). pH and temperature affect the solubility and mobility of heavy metals which lead to low concentration in the water. The low concentrations of heavy metals in water samples may be due to rainfall which diluted many heavy metals which are observed during samples collection. Higher levels of heavy metals in soil samples, are due to waste water disposal on the soil and other anthropogenic activities. Thus reducing distance and depth of physical dilution and increased level of their movement (Sebiawu, *et al.*, 2020).

### Comparative Concentrations Pesticide Residues in Water and Soil Around River Nukkai

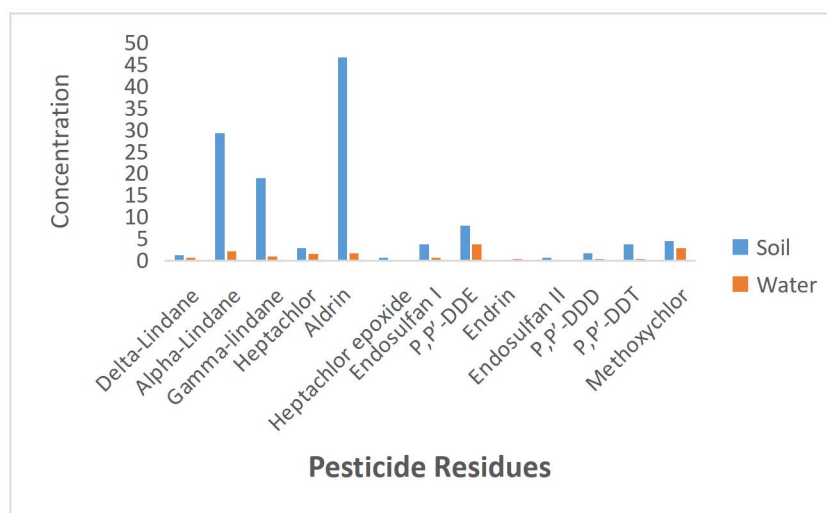
Figure 18 showed the comparison of mean concentrations of analyzed pesticide residues of water and soil samples from river Nukkai. The studied compounds were, Delta-Lindane, Alpha-Lindane, Gamma-Lindane, Heptachlor, Aldrin, Heptachlor epoxide, Endosulfan I,

P,P'-DDE, Endrin, Endosulfan II, P,P'DDD, P,P'-DDT and methoxychlor. Aldrin (74.3179 mg/kg) (65.257mg/kg), Alpha-Lindane (31.6663 mg/kg) and Gamma-Lindane (58.721 mg/kg) were the highest compounds detected in the entire soil samples investigated. Their concentrations are shown decreasing order of; P,P'-DDE(8.00432 mg/kg) > Methoxychlor(4.451552 mg/kg) > P,P'-DDT(3.744285 mg/kg) > Endosulfan I > (3.695924mg/kg) > Heptachlor(2.782272 mg/kg) > P,P'-DDD(1.58949mg/kg) > Delta-Lindane(0.616204 mg/kg) > Endosulfan II(0.524958mg/kg) > Heptachlor epoxide(0.2489 mg/kg) > Endrin(0.210166 mg/kg) respectively. Aldrin concentration exhibited higher concentration than other studied compounds due to its extremely persistent pollutant which enters the food chain. It also remain in the soil for decade and accumulated in crops that became menace to humans (Momohshaibu *et al.*, 2022).

The concentration of Endosulfan I in soil samples was higher than endosulfan II due to the composition of soil contained about 67% endosulfan I by mass of Edosulfan content. Endosulfan I are more thermally stable than

endosulfan II, which is more persistent in the environment (Ademola and Gideon, 2012). Investigations showed that, all the thirteen pesticide residues found in soil samples are beyond permissible limit of  $\leq 0.001\text{mg/kg}$  to  $\leq 0.005\text{mg /kg}$  for, which are higher than the

default maximum residues limits level of pesticide (0.01 mg/kg) for agricultural soil. All these compounds pose health hazards to both humans and animals when found above permissible limits as stated by Usman *et al.*, (2021).



**Figure 18:** Comparative Concentration of Pesticides Residues in Soil and Water s (mg/kg and mg/L).

The concentrations of pesticide residues found in water analyzed are lower than the concentrations found in soil samples as represented on Figure 18. The result showed that the input of pesticides in water was related to suspended particulate concentrations, as impurities residues being absorbed and transported, which vary by seasons and activities of soil erosion. Physiochemical parameters such as pH may have direct effect on the solubility of these pesticides in river water. Thus, these thirteen pesticide residues investigated in water samples are beyond permissible limits of  $\leq 0.001\text{mg/kg}$  to  $\leq 0.005\text{mg /L}$  which posed threat to health risks.

### CONCLUSION

This study revealed physical parameters levels of river water; pH, temperature, Electrical conductivity, TDS and TSS and are below the permissible limits with exception of turbidity. The concentration of Heavy metals in the

water bodies particularly Cd, Pb, and Cr were generally high with exception of Zn and Cu which fell within the permissible limits. They pose treat to both aquatic lives and human health that require remediations. Heavy metals investigated are within the permissible limits in soil samples. Pesticide residues concentrations obtained in water and soil showed that thirteen pesticides (Delta-Lindane, Alpha-Lindane, Gamma-Lindane, Heptachlor, Aldrin, Heptachlor epoxide, Endosulfan I, P,P'-DDE, Endrin, Endosulfan II, P,P'DDD, P,P'-DDT and methoxychlor) were detected and higher than the maximum residue limits, where Aldrin, Alpha-Lindane and Gama-Lindane are among the highest contrated residue observed. These pesticide residues are associated to unselective refuge dumping where constant monitoring of pesticide residues concentrations is paramount for prevention, control, and remediation of environ-mental pollution of health risk to



humans. Adequate sensitization to the farmers on safety measures will reduce the level of pesticide residues hazards in the soil and river.

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