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Degradation of Water Resources by Agricultural Pesticides and Nutrients, Weruweru, Tanzania

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Abstract: Agrochemicals nutrients and residues of pesticides in surface water and sediments samples of Weruweru sub-catchment were investigated to determine their concentrations and quality of water for human consumption. Plant nutrients NO_3^-, NO_2^-, NH_3 and PO_4^{-3} concentrations ranging from 0.005 to 0.96 mg/l were detected in surface water samples. Pesticides residues cyanazine, □-chlordane, endosulfan sulphate, *p,p'*-DDT, *p,p'*-DDD, *p,p'*-DDE, lindane and cypermethrin concentrations ranging from below detection limit to 45.7 □g/l and below detection limit to 157 □g/kg dw were detected in surface water samples and sediments samples, respectively. In most sites, nutrients and pesticides residues concentrations were below the maximum limits as per WHO and national limits for drinking water. The agrochemical contaminants were considered to originate from agricultural runoff and weathered agricultural soils. All parameters were measured by standard methods.

Key words: Agriculture · Pesticides · Nutrients · Sediments · Surface water · Agrochemicals

INTRODUCTION

Agrochemical is a common term encompassing various chemical products that are used in agricultural activities. It refers to the wide range of pesticides, synthetic fertilizers, hormones and other chemical growth agents as well as concentrated stores of raw animal manure. The majority of agrochemicals are pesticides and plant nutrients, which are used to control pests' invasion, control of vectors of human and animal diseases and improve soil fertility [1]. The widespread use of these chemicals over the past half-century has led to their detection in many hydrological systems of many countries [2]. Despite the fact that pesticides and nutrients pollution in aquatic environment can also originate from other sectors, agriculture is undoubtedly seen as the most important source of this contamination [3]. The major concerns are the ways in which they are applied and handled that pose threat of diffuse water pollution. Farmers have inadequate knowledge in pesticides and nutrients use and they rely on the directives given by pesticides dealers. Other problems include use of repackaged products, lack of safety equipments, unsafe storage facilities, unlabelled, or labeled in unfamiliar languages and complex instructions that are difficult to apply [4]. Pesticides and nutrients from areas under agriculture activities can therefore reach the aquatic environment through direct runoff, leaching, careless disposal of empty containers and equipment washings.

In addition to reducing the ecological status of river systems, pesticides and nutrients can have significant social and economic costs through polluting drinking and bathing waters, degrading fisheries and potentially increasing food risks [5]. Excess nitrogen and phosphates in rivers, lakes, reservoirs and ponds can lead to massive overgrowth of algae and deplete the oxygen levels that fish, shellfish and other aquatic organisms need to survive. High level of nitrogen in drinking water can pose particular risk to infants and children. For example, the blue baby syndrome is a common health effect in children arising from high levels of nitrogen in drinking water. Galloway and Cowling [6] reported that chemicals used to disinfect drinking water such as chlorine can react with algae in water to form disinfection byproducts that have been associated with reproductive and developmental

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health problems. Pesticides are poisonous by nature and are intended to kill, destroy or control animals and plant species which interfere with agricultural processes or are vectors of human diseases. Environmental contamination of natural waters by pesticide residues is of great concern. Water pollution by pesticides can affect many non-targeted biological systems, such as fish, birds, beneficial insects and plants. They may take very long time to clear and can pose danger of bioaccumulation [7]. Some pesticides are endocrine disruptors that mimic or antagonise natural hormones in the body. Such pesticides are linked to human health effects such as immune suppression, hormone disruption, diminished intelligence, reproductive abnormalities and cancer [8]. Thus, with continuously high influx of agriculture products from increased population in Weruweru sub-catchments the magnitude of the problem cannot be underestimated. Unfortunately, there is lack of information related to pesticide and nutrients contamination levels in the sub-catchment of Pangani river basin, in Tanzania. The study was therefore undertaken in order to ascertain the levels of plants nutrients and pesticides residues in selected surface water and sediments of Weruweru sub-catchment. An understanding of the effects of pesticides and nutrients contaminations is a positive step towards addressing the imminent problems posed to biological organisms and proposes some remedial approaches in Weruweru sub-catchment area.

Findings of this study can be used as baseline information towards management of water quality in the study area and may be replicated other watersheds with similar agricultural activities and population scenarios.

MATERIALS AND METHODS

Study Area: MapWindow GIS version 4.3, software delineated the watershed and drainage network of Weruweru sub-catchment from Enhanced Thematic Mapper (+ETM, 2006) Landsat imagery. The computed watershed was then draped over Shuttle Radar Topographic Mission (srtm, 30 x 30m^2) satellite 3DEM through 3DEM software (Figure 1). The computed watershed covers an area of 250.646 km^2, located at the southern base of Mount Kilimanjaro in the north-western part of Pangani River Basin (PRB), Kilimanjaro region, Tanzania (Figure 3). It is situated at latitudes 3°00' to 3°30'S and longitudes 36° 30' to 37°15'E. The altitude above sea-level ranges between approximately 700 m at the confluence with Kikafu River to over 4,360 m at the snowline of Mount Kilimanjaro. The sub-catchment is characterized by steep slope valleys to gentle slopes, lowlands and in a few areas valley side slopes (Figures 1 and 2). Weruweru sub-catchment is estimated to have a total population of 196,800 people, distributed in upper, middle and lower zones. The upper zone supports production of coffee; middle zone supports maize and vegetables, while the lower zone supports production of vegetables and tropical fruits. The three zones also support livestock rearing that includes dairy and beef cattle, sheep, poultry, goats and pigs. The length of Weruweru River starting from the upper zone to the confluence of Kiladeda is about 17.9 km and about 19.0 km to the sub-catchment. The river is a tributary of the large PRB and it joins the Pangani River through Kiladeda River [9].

Sample Collection: Water and sediments samples were concurrently collected from 6 selected sampling sites (two from each zone) during the dry (February 2013) and rainy (April 2013) seasons. The sites were selected based on the qualitative information obtained from key stakeholders and 3DEM software algorithms. The locations were divided into three zones based on their respective heights above sea-levels (a.s.l.). The zones lower zone (below 800 m), middle zone (between 800 – 1200 m) and upper zone (above 1200 m) were assumed to approximate agro-ecological units in the study area (Figure 2). Details of sampling sites are described in Table 1 and indicated in the sub-catchment map in Figure 3. The indication of sampling sites in the sub-catchment map was enabled by Global Position System (GPS) points that were measured during sampling campaigns.

Water samples were collected by grabbing technique, preserved, kept in cool boxes and later transported to the laboratory for analysis. In the laboratory, water samples for pesticide analysis were stored in temperatures between 0-4°C prior to extraction and analysis, while samples for nutrients were analysed immediately upon arrival. Water samples for pesticides analysis were collected in one litre sampling bottles with Teflon stop cork. After sampling, samples were measured for physico-chemical parameters temperature, pH, DO, TDS and EC using hand-held potable water quality monitor and later preserved by 10% of 1000 g of NaCl salt as described by Akerblom [10]. Water samples for nutrients analysis were collected in 500 mL plastic bottles. The samples were filtered and preserved with concentrate H_2SO_4. The physico-chemical parameters already measured in water samples for pesticide analysis were deemed to be adequate for the nutrient samples.

Table 1: Description of the Sampling Sites in Weruweru Sub-catchment

Site	Sampling Site	Coordinates	Site characteristics
W1&S1	Upper zone (1)	03°19' 8.13''S 37°15' 9.26''E	Human settlements, farming activities (coffee), domestic activities (fetching water and washing)
W2&S2	Upper zone (2)	03°19' 8.32''S 37°15' 9.15''E	Human settlements, farming activities (coffee and livestock) and domestic activities (fetching water and washing). Water flows to middle zone of the river
W3&S3	Middle zone (1)	03°21' 3.37''S 37°17' 5.07''E	Human settlements, farming activities (maize, banana and vegetables such as cucumbers, tomatoes, green vegetables), fetching water and animals drinking
W4&S4	Middle zone (2)	03°21' 0.3''S 37°17' 5.23''E	Human settlements, farming activities (maize, banana and vegetables) and domestic activities (fetching water) and livestock. Water flows to the lower zone of the river
W5&S5	Lower zone (1)	03°23' 10.3''S 37°17'11.33''E	Human settlements, farming activities (tropical fruits, beans and vegetables such as tomatoes, green vegetables, cucumbers, onions) and livestock, domestic activities (fetching water, bathing and washing)
W6&S6	Lower zone (2)	03°23'11.23''S 37°17' 11.7''E	Human settlements, farming activities (beans, green vegetables, tomatoes, onions and millet and tropical fruits) and (poultry and livestock) and domestic activities (fetching water, bathing and washing). Lower zone of Weruweru River discharges water to Kifaru river

W1-W2 = water sampling sites, S1-S2 = sediment sampling sites

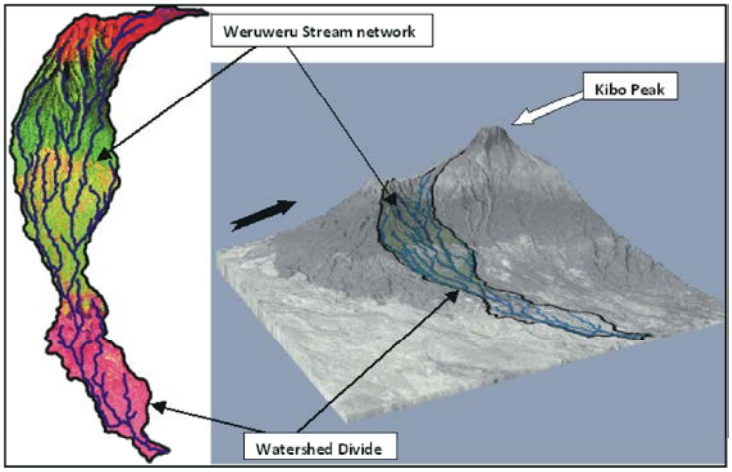

Fig. 1: Delineated watershed and stream network of Weruweru River

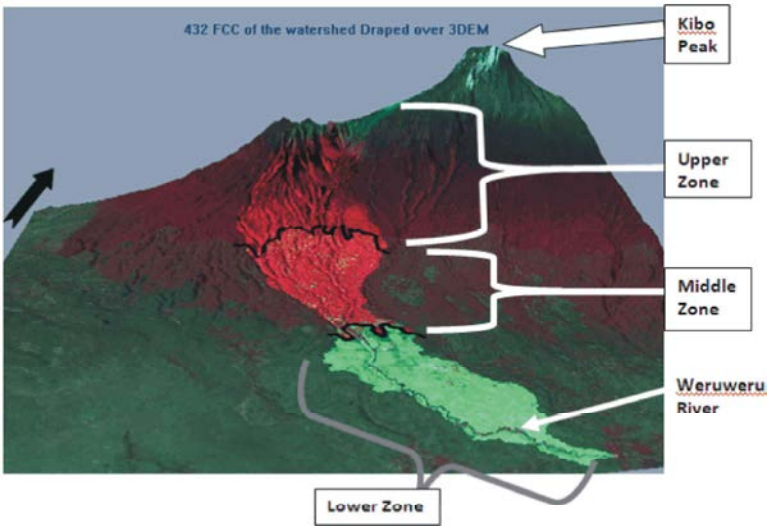

Fig. 2: 3D perspective view of 432 FCC of Weruweru River watersheds showing the 3 zones

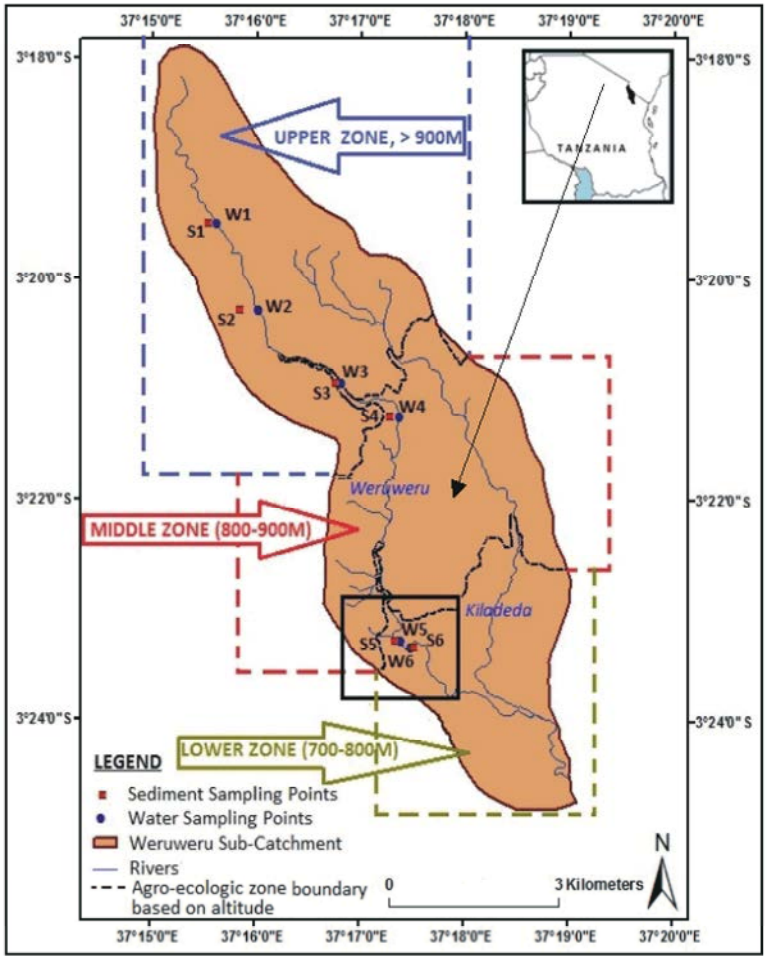

Fig. 3: Map of Weruweru sub-catchment showing sampling points in the three zones

Bed sediment samples were taken by using a stainless spoon, wrapped in aluminium foil, labeled, placed in air-tight bags, kept in ice-coolers and transported to the laboratory where they were kept at 20°C before extraction and analysis. Two field points were selected randomly at 0–20 cm depths per sampling site in each zone because sediments at this depth level are expected to be the most contaminated and have the greatest potential for exchange with the water column [11].

Pesticides Analysis

Sediment Samples Extraction and Clean up: Three sub-samples (20 g, 10 g and 5 g) were measured in analytical balance. The first sub-sample of 5 g was used for measuring sediment pH values. The sediment sample was mixed with 5 ml of de-ionised water (pH 7.0) in a small clean container and gently stirred by a scoop into slurry. The mixture was then left to settle for approximately 15 min before recording the pH value using a calibrated pH meter. Another sub-sample of 10 g was taken in a pre-weighed petri dish for dry weight and organic matter determinations. It was later dried in an oven at 105°C for 12 hours and 400°C for 3 hours for determination of moisture and organic contents, respectively.

The third sub-sample (20 g) was grinded with 30 g anhydrous sodium sulphate in the mortar and more sodium sulphate were added to make the sample free floating powder to bind the water. The mixture was poured into a column and eluted with 120 ml dichloromethane in a beaker while shaking the beaker and left to settle for 15 min. The contents were then decanted through a plug of glasswool into an evaporation flask. The remaining sodium sulphate was rinsed with 20-30 ml dichlorome thane mixture and decanted through the same glasswool. The resulting extract was concentrated in a rotary evaporator and the solvent was changed to cyclohexane and concentrated to 2 ml ready for clean up.

Clean up of sediment samples were done by column chromatography using florisil (magnesium silicate). A glass column of 5 g (60 cm x 22 mm) packed with florisil, glasswool and anhydrous sodium sulphate was used. 50 ml of cyclohexane was added into the glass column and allowed to pass through drop by drop until very little was left on the upper part of the column. The sample concentrate was poured into the column and drained to make 2 ml of the sample, then transferred into Teflon cork vial and stored at 4°C until analysis was done.

Water Samples Extraction and Clean up: Unfiltered water samples, previously preserved with 10% sodium chloride, were extracted by Liquid-Liquid Extraction (LLE) method. Each water sample 1000 ml was quantitatively transferred to a 1 Litre separating funnel and the sampling bottle was rinsed with 30 ml dichloromethane, which was then transferred to the separating funnel containing the water sample. The combined contents were then successively extracted with dichloromethane (3 x 50 ml). The organic layer was filtered through plugwool containing anhydrous sodium sulphate (30 g) for drying. Sodium sulphate was later rinsed with dichloromethane (2 x 3 ml) and the combined extract concentrated in *vacuo* at 30°C and the solvent changed to cyclohexane. The volume was adjusted in a stream of air to 2 ml in 9:1 cyclohexane: acetone (v/v) in vials ready for analysis. The water extract appeared clean and were not subjected to further clean up [10].

Analytical Quality Assurance

Analytical Quality Assurance for Sediment Samples: A 100 ml aliquot of each n-hexane, dichloromethane, cyclohexane, ethyl acetate and acetone was concentrated to 2 ml and used to check contamination from the solvents used. Two matrices blank from bed sediments were obtained from a virgin land where the water passes and the same procedure of extraction and analysis as that of sediment samples was performed. The result showed that no significant peaks appeared in the chromatograms of the blanks. Recoveries were estimated by spiking the matrix blank with four OCls pesticides standard at concentrations ranging from 0.01-1.1 µg/ml of each analyte. The average of percentage recoveries ± SD (n=4) were α-chlordane 81.7±3.6%, *p,p'*-DDE 89±1.5%, *p,p'*-DDD 80±1.7%, *p,p'*-DDT 95±2.7% and endosulfan sulphate 87±0.4%. The results were not corrected for recoveries since all were with the normal acceptable range of 70-120% [11-12].

Analytical Quality Assurance for Water Samples: A 100 ml aliquot of each n-hexane, dichloromethane, cyclohexane, ethyl acetate and acetone was concentrated to 2ml and used to check contamination from the solvents used. 1 L of distilled water was extracted the same way as water sample. The results showed no significant peaks appeared in the chromatograms of the blanks. Recoveries were estimated by spiking the matrix blank with four OCls pesticides standards at concentration ranging from 0.01-1.1µg/ml of each analyte. The average of percentage recoveries ± SD (n= 4) were α-chlordane 87±0.3%, *p,p'*-DDE 98±0.2%, *p,p'*-DDD 99±0.7%, *p,p'*-DDT 99±1.2% and endosulfan sulphate 97±0.9%. The results were not corrected for recoveries since all were with the normal acceptable range of 70-120% [12]. ill, 2000).

Gas Chromatography Analysis: Pesticide residue analyses were determined as described by Akerblom [10]. GC-Varian CP-3800 gas chromatography equipped with ^{63}Ni Electron Capture (EC) detector was used for analysis. The GC capillary column WCOT FUSED SILICA 30 mm x 0.32 mm, coated with CP-SIL 8CB DF 0.2 µg/ml was used. Nitrogen was used as both a carrier and make up gas in the Electron Capture Detector (ECD) at a flow rate of 30 ± 1 ml/min. The temperature programme was held at 70°C for 1min, 15°C/min to 180°C, 4°C/min to 230°C for 15 min. The injection and detector temperatures were 240°C and 250°C, respectively. Identification of residues was effected by running samples and external reference standards in GC and then comparing the chromatograms [13].

Nutrients Analysis: Nutrients in water samples were analysed following the methods outlined by APHA/AWWA/WEF [14]. For ammonia-nitrogen determination, colorimetric method using Nessler's reagent was used. The detection limit for this method was 0.02 mg/l. Nitrate-nitrogen and phosphoros were determined by the cadmium reduction method and ascorbic acid method, respectively. Methods detection limit for both methods were 0.01 mg/l. The quality of the data was assured by the analysis of blank and duplicate samples according to the standard operating procedures of the analytical laboratory at the Department of Chemistry, University of Dar es Salaam.

Data Analysis: The analyses of data were performed using descriptive statistics including frequencies, percentages, mean and histograms. These were computed through the use of Microsoft excel computer software.

RESULT AND DISCUSSION

Nutrient Concentrations in Weruweru River: Nitrate (NO_3^-), nitrite (NO_2^-), ammonia (NH_3) and orthophosphate (PO_4^{-3}) compounds were analysed in water samples and results tabulated as shown in Figure 4. These compounds are the most significant inorganic forms of two elements; nitrogen and phosphorus that commonly limit the productivity of plants. The concentration of NH_3-N was low during the dry season (0.15–0.22 mg/l, range) than during the rainy season (0.37–0.96 mg/l, range). NH_3-N is pH and temperature dependent, such that at high pH and temperatures high values of NH_3-N are expected. Results from the study indicate that the pH and temperature were high during the dry season than rainy season. This means that pH and temperature cannot be the cause of high NH_3-N during the rainy season. A possible source of high NH_3-N contamination during rainy season is expected to be runoffs containing fertilizers from upstream. This was attributed to the fact that both sampling stations are located close to farmlands where extensive agriculture is conducted.

The concentrations of NO_2^--N during the dry and rainy seasons range from 0.005 to 0.008 mg/l and from 0.08 to 0.13 mg/l, respectively. Both concentrations were below the maximum limits in drinking water quality of 1 mg/l as per Tanzania standards [15]. The concentration for NO_3^--N during the dry season ranged from 0.15 to 0.454 mg/l and rainy season range from 0.17 to 0.37 mg/l. The high range of NO_3^--N than NO_2^--N is normally expected because nitrite is the unstable form of nitrogen and is easily oxidized into nitrate [6]. Exposure to higher levels of nitrites or nitrates has been associated with cancer, brain tumors, leukemia and asopharyngeal. In infants and children, nitrate and nitrite bind to hemoglobin and cause chemically altered hemoglobin (methemoglobinemia) that impairs oxygen delivery to tissues, resulting in the blue colour of the skin "blue baby syndrome". The normal background range of nitrate concentration in natural waters is normally below 5 mg/l, any value above this level is an indication of manmade nitrate pollution [16]. The World Health Organization has set limits of 50 mg/l for NO_3^--N concentration in potable water [17]. In this study concentrations of NO_3^--N in all sampling stations and in both seasons were within the allowable limits for WHO potable water quality and normal range of natural water.

Concentration of PO_4^{3-}-P during the dry season range from 0.05 to 0.14 mg/l, while during the rainy season it ranges from 0.44 to 0.52 mg/l. These concentrations were higher than the natural background levels of PO_4^{3-}-P in river waters which usually range from 0.005 to 0.02 mg/l [18]. High values of phosphorous in rivers can speed up eutrophication and reduction of dissolved oxygen of the river water due to increased minerals and organic nutrients [19]. Therefore, low level of DO observed during the rainy season could partly be associated with high level of phosphorous.

Pesticide Residue in Water and Sediment Samples: Concentration of eight different types of organochlorine pesticides residues cyanazine, □-chlordane, endosulfan sulphate, *p,p'*-DDT, *p,p'*-DDD, *p,p'*-DDE, lindane and cypermethrin were measured in water and sediment samples collected from six different sites in Weruweru sub-catchment. Results of the various concentrations are indicated in Table 2. The computation reveals that most pesticide residues in water and sediment samples were below detection limits. Concentration range for both dry and rainy seasons in water samples was bdl to 4.7 □g/l and in sediment samples ranges from bdl to 157 □g/kg dw.

Endosulfan sulphate, a major degradation of endosulfan was the most detected pesticide residues. It was detected in about 33% of the samples analysed during the dry and rainy season. Its concentration in water and sediment samples ranged from bdl to 12.7 □g/l and bdl to 13 □g/kg dw, respectively during the dry season and rainy season. Although endosulfan sulphate is susceptible to photolysis in the environment, it is expected to have a high occurrence in cultivated areas [20]. Dem and coworkers [21] also reported high occurrences of endosulfan sulphate in soil samples, which were 74% of the sample analysed. Endosulphate is known to be toxic as parent compound endosulfan [22]. Because of their high toxicity, technical endosulfan was restricted in many countries including Tanzania. In the study area however, it was found to be in use under trade name thionex/thiodan.

Percentage detection of DDT metabolites in water samples were 33.4% during the dry season and 16.7% during rainy season. In the lower zone of Weruweru river, *p.p'*-DDD was detected for both seasons and the concentrations ranged bdl-0.506 □g/l. Then, *p,p'*-DDE was only detected in the lower zone of the river during the dry season with concentrations ranging from bdl to 0.81 □g/l. The concentrations of the DDT metabolites were within the acceptable limits in water 2 □g/l as per WHO guidelines [23]. *p,p'*-DDT was detected during the dry season in the sediment samples analysed with concentrations ranging from bdl to 19.0 □g/kg dw. DDT can be degraded into DDD under anaerobic conditions

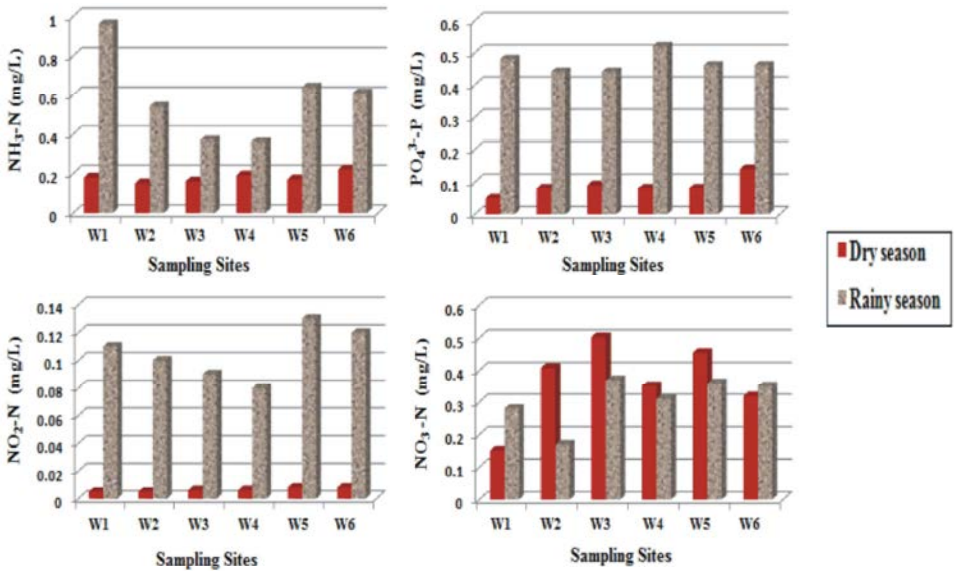

Fig. 4: Concentration of the Nutrients in Weruweru water samples measured at six sampling sites

Table 2: Types and Concentrations of Pesticides Residues Detected in Water (□g/l) and Sediment (□g/kg dw) Samples in Dry and Rainy Seasons

Season	Site	Cyanazine	□-Chlordane	Endosulfan Sulphate	*p,p'*-DDT	*p,p'*-DDD	*p,p'*-DDE	Lindane	Cypermethrin
Dry	W1-W3 (S1-S3)	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
	W4 (S4)	bdl	0.084 (bdl)	bdl	bdl	bdl	bdl	bdl	bdl
	W5 (S5)	bdl	bdl	12.7 (bdl)	bdl (19.0)	bdl	bdl	3.66 (bdl)	bdl (157)
	W6 (S6)	45.7 (0.115)	0.816 (64)	12 (bdl)	bdl (5.63)	0.481(bdl)	0.74 (bdl)	bdl	bdl
Rainy	W1-W2 (S1-S2)	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
	W3 (S3)	bdl	bdl	bdl (13)	bdl	bdl	bdl	bdl	bdl (5.2)
	W4 (S4)	bdl	0.081 (bdl)	bdl	bdl	bdl	bdl	bdl	bdl
	W5 (S5)	bdl	0.028 (bdl)	bdl	bdl	bdl	bdl (81)	bdl	bdl
	W6 (S6)	bdl	0.23 (bdl)	bdl (0.433)	bdl	0.506(bdl)	bdl	bdl	bdl

bdl = below detection limit, w1-w6 = water samples, s1-s6 = sediment samples, values in brackets represent sediment concentrations where there is no brackets all water and sediment concentrations were bdl

and into DDE in aerobic environments. Normally, ratios of (*p,p'*-DDT)/(DDT metabolite) greater than 0.5 indicate recent DDT usage [24]. In the current research study, ratios of *p,p'*-DDT to *p,p'*-DDE was 0.23, suggesting previous inputs of DDT into Weruweru river sediments. The ratio also agrees with the results from farmer's survey that indicated use of DDT up to the late 1990s. DDE may last in the sediment for a very long time, potentially for hundreds of years sticking strongly to sediments and thus may remain in the sediment surfaces for a long span of time. The persistence in the environment of this organochlorine has also been reported in others parts of the world. For instance DDE have been reported to be widely distributed in soils in China despite the fact that its use has been discontinued since 1983 [25].

Lindane was only detected in water samples from one site (W5) during the dry season with concentration of 3.66 □g/l. This concentration is above the acceptable limits for drinking water 2 □g/l as per WHO guidelines [23]. The small percentage of detection indicates the possibility of past usage. Use of lindane was restricted by the United State Environmental Protection Agency (U.S EPA) due to concerns raised over its potential to causing cancer and birth defects in animals [26]. It is highly persistent in soils and sediments, with a half life of approximately 15 months.

Cyanazine was detected in the lower zone of the river during the dry season with concentrations ranging from bdl to 45.7 □g/l. This is higher than acceptable limit in drinking water of 10 □g/l as per WHO guidelines [23]. Concentrations of cyanazine during the rainy season were below detection limits. This could be due to the fact that cyanazine takes 2 to 14 weeks to disappear completely, although it could also have been washed away by runoff from one point to another. High concentrations of cyanazine during the dry season in the study area correlate with a similar study in USA where cyanazine was detected in surface water and groundwater at maximum

concentrations of 1300 and 3500 µg/l, respectively. Cyanazine is restricted by U.S EPA because of its persistence and teratogenicity effects [27].

Alfa-chlordane was detected in 33% of water samples analysed during the dry season and 50% of samples for the rainy season. The concentrations ranged from bdl to 0.82 □g/l. □-chlordane does not biodegrade and is highly persistent in soils with a half-life of about 4 years. Chlordane has been detected in surface water, groundwater and sediments. Concentrations detected in surface water have been very low, while those found in suspended solids and sediments are always higher [27]. In this study, □-chlordane was detected only during the dry season in lower zones of the River, with concentrations ranging from bdl to 64 □g/kg dw. Such values are above the fresh water sediment quality assessment guidelines for alfa-chlordane 4.5 □g/kg dw [28].

CONCLUSION

Results emanating from the current research study reveal that surface water and sediments in Weruweru sub-catchment are contaminated with pesticides and nutrients. The concentrations of contaminants in most of the sites were within the recommended international and national limits for drinking waters. However if such contaminations are not controlled can lead to heavy contamination of the sediments. Sediments normally act as a sink of pollutants that eventually releases into water reserves. It can hence be concluded that, if taken up through plant roots and bioaccumulate in the food chain beyond acceptable limits may cause chronic adverse health effects to consumers.

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