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Full Length Research Paper

Assessment of trace metals in sewage water and sludge from River Kubanni drainage basin

Adamu H[1]*, Uzairu A.[2] and Harrison G. F. S.[2]

[1]Department of Environmental Management, Abubakar Tafawa Balewa University, Bauchi, Nigeria
[2]Department of Chemistry, Ahmadu Bello University, Zaria, Nigeria.

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The concentrations of trace metals in sewage water and sludge samples from River Kubanni drainage basin in Zaria City, Nigeria were investigated in this study. The drainage basin is utilized as a source for irrigation water, during dry seasons. The sewage water quality characteristics in three month sampling periods, that is, February - April, 2008 (peak of dry season and period of intensive usage of the sewage water), the speciation of metals in the sewage sludge from the drainage basin, and the risk to sewage water column contamination were evaluated. The sewage water quality characteristics were mostly beyond the recommended irrigation water standards by the food and agriculture organization (FAO) and United State environmental protection agency (USEPA) except for zinc and nickel. In addition, the average values of Cd, Cu, Pb, Cl^- and NO_3^- in sewage water samples analyzed were higher than the respective reference values for irrigation water. To study the speciation of metals in sewage sludge, five metals (Zn, Ni, Cu, Pb and Cd) in the sludge were subjected to sequential extractions. The metals analyzed were distributed in both the non-residual and residual phases. Total extractable trace metals in sewage sludge were: Zn (403.3 mg/kg dry weight), Ni (184.2 mg/kg dry weight), Cu (303.4 mg/kg dry weight), Pb (129.0 mg/kg dry weight) and Cd (19.7 mg/kg dry weight). However, there was low risk to sewage water contamination based on the calculated individual contamination factors (ICF) obtained for sewage sludge from the trace metal sequential extractions. From the calculated individual contamination factors, Ni and Zn followed by Cd and Pb posed the highest risk to sewage water contamination. Based on this study, the human health is at risk, since sewage water from the drainage basin has been the source for irrigation water during dry seasons, which might lead to trace metal ingestion by soil and subsequently by vegetables. Thus, this might become important pathways of human exposure to metal contamination.

Key words: Trace metals, speciation, contamination factor, sewage water and sludge.

INTRODUCTION

Environmental pollution is one of the most investigated subjects of research at present time. Most industrial and municipal wastes end up in rivers, lakes and sea. Hence, River Kabunni drainage basin, being one of the basins that drain Zaria, through which domestic sewage and refuse find their ways from many settlements nearby the river basin. Trade wastes (from auto-mechanics, hotels, the idea of having continuous survey of water quality receives widespread attention. In recent years, because of industrialization and increasing pollution, water pollution has become an important problem all over the world (IAEA, 1976; USEPA, 1984). Rapid urbanization with improper environmental planning often lead to discharge of municipal waste into lakes and rivers. The lakes and rivers have a complex and fragile ecosystem, as they do not have self-cleaning ability and therefore readily accumulate pollutants. This is not in exception to abattoir and road side carwash operators) are also directly or indirectly drain into the river basin.

River Kubanni is relatively a seasonal river with intermittent flows occurring in flashes mainly during rainy seasons and thus, chiefly becoming sewage-bearing river

*Corresponding author. E-mail: aisonhardo2003@yahoo.com.

in dry seasons (Eigbefo, 1987; KDSG, 2000). During dry season much consideration and attention has been given to this drainage basin because of the numerous irrigation activities that takes place seasonally along its course, as well as its economic importance to the inhabitants. A great number of Zaria peasants have farms along the course of the river basin, from which they obtain their food stock much as maize, sugarcane and vegetables like tomato, pepper, onion, lettuce, spinach, cabbage and carrot. Unfortunately, sewage water of municipal origin contain appreciable amount of heavy metal, which may give rise to accumulation of heavy metals in the top soil when used as irrigation water (Williams et al., 1980) and hence in the tissue of plant grown on them (Day et al., 1979). Besides, the chemical status of sewage water has its influence on the receiving lands, which might possibly reflect on the produce cultivated on such lands and subsequently ends up within the inner system of humans and becomes a danger to life (Furedy and Chowdhury, 1996). As a result, this study was initiated to (i) assess the water quality characteristics of river Kubanni drainage basin and evaluated the risk of Kubanni drainage basin water contamination by sludges, and (ii) Investigate the chemical fractionation of Zn, Ni, Cu, Pb and Cd in the sewage sludge because of their environmental implications and beside that use sludge as a pollution indicator, since sludge can also provide deeper insight into pollution state of water body. This is owing to the fact that sludge is ready sink or reservoir of pollutants including trace metals where they concentrate according to pollution level (Onyari et al., 2003) and indeed, sludges being the sediments of sewage water play a considerable role in the remobilization of trace metals in aquatic systems and the release of trace metals from the sludges into water body depends on the chemical fractionation of metals and other physico-chemical characteristics of the aquatic system (Canavan et al,. 2007).

MATERIALS AND METHODS

Description of the study area

Zaria, Nigeria is situated on a plateau at a height of about 570.56 m above sea level in the centre of Northern Nigeria and more than 644 km way from the sea. It is characterized by a tropical climate with two distinct season, a rainy season (May – October) and a dry/harmatan season (November - April). Based on the classification of geographical regions of Nigeria, Zaria falls into Guinea savannah climate (Udo, 1978). The River Kabanni drainage basin is located within this region (Figure 1). The Kubanni drainage basin is one of the most important river basins that drain Zaria and its environ. The Ahmadu Bello University, Zaria, the Institute for agriculture research, Samaru and Nigeria College of Aviation Technology, Zaria are located at the northern side of the basin which flows to form a natural frontier between Tudun Wada on one hand and sabon Gari on the other. It also flows through the back of Nigeria military school, the Nigerian army deport and the advanced teachers college before emptying into the Galma river. Fadama lands (flood plains) is located along the river basin channel, which receive ready and regular water supplies from the rive basin for irrigation during dry seasons.

Quality assurance

The quality assurance for sewage water sample was conducted as described by Awofolu et al. (2005) through the spinking method to evaluate the digestion process and the effectiveness of AAS machines. A chemical analysis protocol through multi-element standard solution was applied. Similarly, the quality assurance for sludge samples was also carried out following the procedure described by Shariadah (1999).

Sewage water collection and analysis

The location of River Kubanni drainage basin in shown in Figure 1 along with the sampling points for sewage water and sludge samples collected. Sampling was carried out in February - April, 2008, for sewage water and sludge. Sewage water samples were collected into acid washed polyethylene plastic containers kept cooled en route to the laboratory and stored at 4°C and determination of some general parameters and trace metals conform to standard methods (APHA, 1992). The pH and conductivity of the sewage water was determined on the site immediately after collection. The other physico-chemical parameters determined include alkalinity, phosphate, sulphate, chloride, nitrate, ammonia and trace metals (Zn, Ni, Cu, Pb and Cd). Digests of sewage water were aspirated into AA 650 atomic absorption spectrophotometer. Triplicate digestions and analysis of the sample together with blank were carried out.

Sewage-sludge collection and analysis

Sewage-sludge samples was collected using pre-cleaned stainless steel Ekman dredge and immediately placed in acid washed plastic containers. The sludge samples were kept cooled en route to the laboratory and kept frozen at -18°C until analyzed. Sludge samples were allowed to deforst, then air-dried in a circulating oven at 30°C. In order to normalize the variation in grain size distributions, the air-dried sludge samples were ground using acid washed porcelain mortar and pestle and then sieved mechanically using a 2 mm sieve (Awofolu et al., 2005). This was then stored in acid washed polyethylene bottles until analyzed. The sludge pH and electrical conductivity (EC) were measured in air-dried sample (1:10, w/v) using digital pH and conductivity meter. Organic matter content was determined by loss on ignition. Exchangeable cations were extracted by 1.0 mol/dm^3 CH_3COONH_4 and subsequently determined by AES and AAS for Na, K and Ca, Mg respectively (Agbenin, 1995). Finally, 3 sludge sub-samples (1 g each) were subjected to a 6 step sequential extraction separately using the method of Sujeet and Banerjee (2004) as following:

Water soluble phase: Each sample was extracted with 15 ml of deionized water for 2 h.

Exchangeable phase: Residue from 1 was shaken at room temperature with 16 ml of 1 mol/dm^3 $Mg(NO_3)_2$ at pH 7.0 for 1 h, centrifuged and the supernatant decanted and made up 40 ml with distilled water prior to analysis.

Oxidizable phase (Bound to organic matter): residue from 2 + 10 ml H_2O_2 8.8 mol/dm^3 + 6 ml HNO_3 0.02 mol/dm^3 was shaken for 5 + 1 h at 98°C. 10 ml CH_3COONH_4 3.5 mol/dm^3 was added as an extracting agent, centrifuged and supernatant decanted and made up to 40 ml with distilled water prior to analysis.

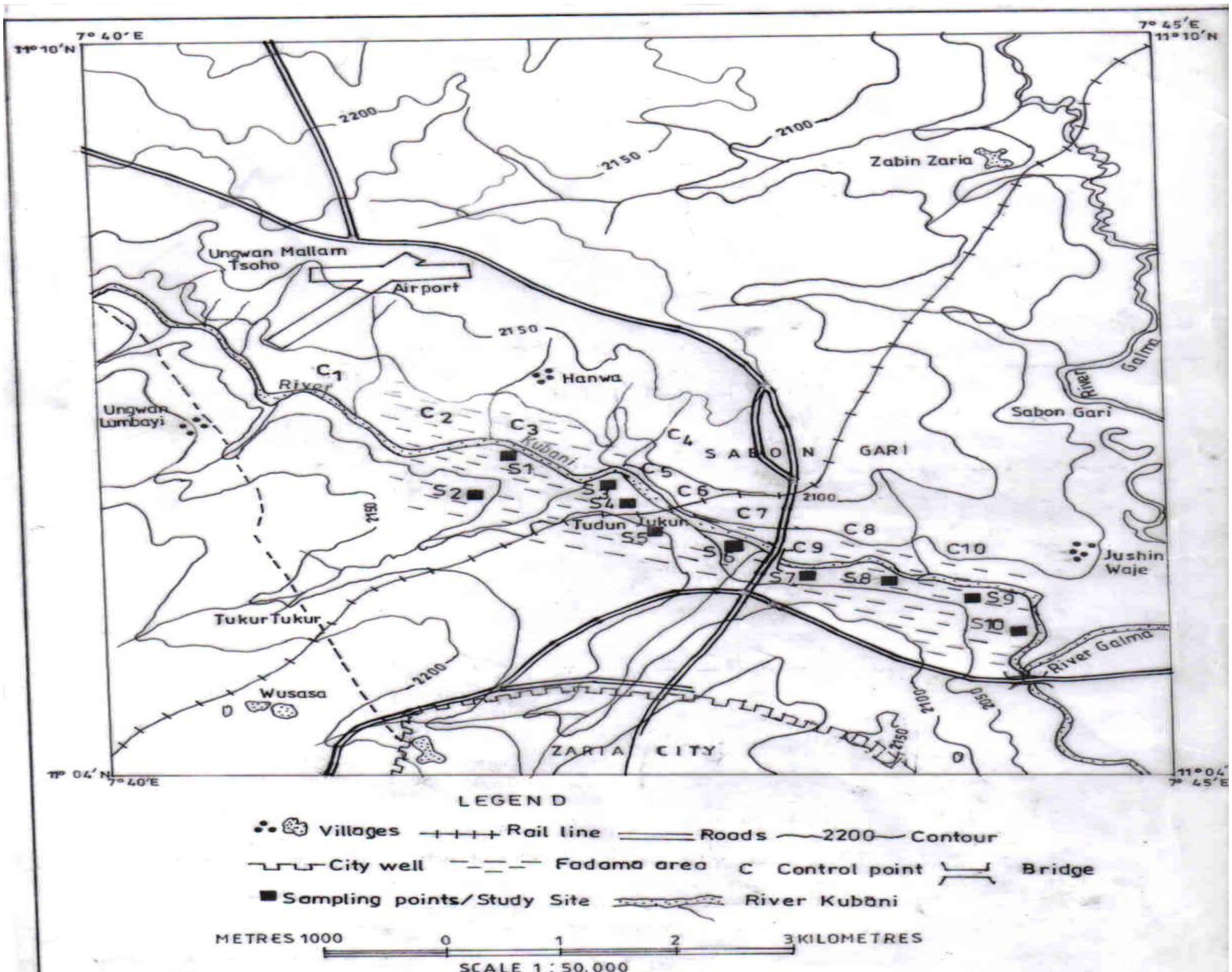

FIGURE 1 : STUDY SITE ALONG KUBANI RIVER

Acid soluble phase (Bound to carbonate): 25 ml of 0.05 mol/dm^3 Na_2EDTA was added to the residue from 3, shaken for 6 h and centrifuged. The supernatant was decanted and made up to 40 ml with distilled water prior to analysis.

Reducible phase (Bound to Fe/Mn oxides and hydroxides): Residue from 4 + 17 ml NH_2 OH. HCl 0.1 mil/dm^3 + 17 ml CH_3COONH_4 3.5 mol/dm^3 was shaken for 4 + 1 h at 98°C. The residue was extracted with 10 ml CH_3COONH_4 3.5 mol/md^3, shaken for 1 h, centrifuged, the supernatant was decanted and made up to 40 ml with distilled water prior to analysis.

Residual phase (Bound to silicates and detrital materials): Residue from 5 was digested using aqua regia/hydrofluoric acid ($HCl/HNO_3/HF$) (0.35:12 w/v sludge/ solution ratio) in acid digestion teflon cup. It was dry ashed for 2 h and evaporated to dryness. The residue was diluted to 40 ml with distilled water prior to analysis. After each successive extraction, the samples were centrifuged at 4500 rpm for 15 min (legret et al., 1988). The supernatants were removed with pipette and filtered with whatman No. 42 filter paper. The residue was washed with deionized water followed by vigorous hand shaking and then followed by 15 min of centrifugation before next extraction. The volume of rinsed water was kept to minimum to avoid excessive solubilization of solid materials. Finally, the extracts collected were analyzed using AAS to determine the concentration of Zn, Ni, Cu, Pb and Cd metals.

RESULTS AND DISCUSSION

Quality assurance

The percentage recoveries of spiked sewage water sample with metal standards using open beaker digestion method is presented in Table 1. Percentage recoveries from spiked sewage water sample for metals ranged from 87.1 ± 0.2 to 95.7 ± 0.5. The digestion method and the AAS machine gave a good consistent recovery; hence this was applied to total metal content determination. Similarly, the mean percentage recoveries for spiked pre-digested sludge sample is also presented in Table 1. The result of the mean percentage recoveries ranged from 88.7 ± 0.5 to 96.7 ± 04. Acceptable recoveries were obtained and thus, the AAS machine was adopted and

Table 1. Mean % recoveries (± SD) of trace metal standard added to pre-digested sewage water and sludge.

Trace metal	Spiked conc. (mg/l)	Sewage water	Sewage-sludge
Zn	2.0	91.7 ± 0.4	88.7 ± 0.5
Ni	2.0	93.2 ± 0.3	96.7 ± 0.4
Cu	2.0	87.1 ± 0.2	89.9 ± 0.3
Pb	0.5	95.7 ± 0.5	92.9 ± 0.5
Cd	0.5	88.6 ± 0.4	90.3 ± 0.4

use for total metal content determination in various phases of the sequential extraction.

Sewage water quality

Table 2 shows some sewage water quality parameters of River Kubanni drainage basin and reference irrigation water values. The pH of the sewage water was found to be in the range of 7.71 ± 0.17 to 8.31 ± 0.11, suggesting that the sewage water from the basin was alkaline. This has agreed with what was reported by Khaiwal et al. (2000) that sewage waters of municipal origin are usually alkaline in nature due to presence of ammoniacal compounds. The pH values obtained in this study fell within the recommended permissible limits as recommended by FAO (1985) and thus, rendered the water fit for irrigation and could not cause nutrient imbalances, as it was reported that abnormal pH in irrigation water may cause nutrient imbalance (Ayers and Westcot, 1985). The mean conductivity of the sewage water was varied between 1291.5 ± 136.1 to 1887.9 ± 136.4 µS cm^{-1}, which indicates that there was greater solubility of ions in the water. The values of EC found also rendered the water suitable for irrigation, as it might not affect soil infiltration rate due to the fact that irrigation water with EC > 200 µS cm^{-1} would not almost invariably results in water infiltration problem (Ayers an Westcot, 1985). However, prolonged usage of irrigation water with greater solubility of ions should be discouraged, as soluble salts from irrigation water accumulated in the soil profile and may deteriorate properties of soil (Agassi et al., 1981). The total alkalinity of the water ranged from 122.3 ± 21.2 to 141.3 ± 17.5 mg $CaCO_3$/l. Alkalinity serves as pH reservoir for inorganic carbon and it is usually taken as an index of productive potential of water (Manahan, 1994). The high values of alkalinity obtained in this study revealed that the water had ability to support alga growth and thus the water was quite polluted. The trend of occurrence of the major anions studied (mg/l) in the sewage water followed the order, sulphate > chloride > nitrate > phosphate. The sulphate and phosphate values of the water fell within the safe limits, respectively ranged from 259 ± 44.6 to 329.1 ± 31.1 mg/l and 0.05 ± 0.02 to 0.16 ± 0.07 mg/l. However, in contrast, the chloride and nitrate contents were not within the safe limits and therefore, the water was quite unfit for irrigation to this regard. The chloride content was quite high, which revealed that the water was not clearer water due excess of chloride and is usually taken as index of pollution (Khaiwal et al., 2000). Regarding its nitrate content, the high values obtained in this work revealed that the water had ability of promoting primary productivity and excess of it in surface water is taken as a warning for algal blooms. Similarly, another pollution indicator in surface water is the content of ammonia. The presence of ammonia in the water was quite a sign of pollution due to sewage intrusion. The chloride, nitrate and ammonia contents found in this work ranged from 38.5 ± 6.3 to 55.7 ± 9.6 mg/l, 9.08 ± 3.2 to 13.7 ± 2.8 mg/l and 0.08 ± 0.03 to 0.11 ± 0.04 mg/l respectively.

The average values of the metals analyzed in the water (Table 2) were higher than the respective reference values (Zn, 2.0; Ni 1.4; Cu, 0.017; Pb, 0.065 and Cd, 0.01 mg/l) for irrigation water (Ayers and Wescot, 1985; USEPA, 2001), with the exception of Zn and Ni contents. Therefore, Cd, Pb and Cu contents in the water were not within the permissible limits. However, total amount of Cd, Pb and Cu permitted in irrigation water that is 0.01, 0.065 and 0.017 mg/l respectively, was violated by the water during the period of investigation. This had rendered the water unfit for irrigation, but with regard to Zn and Ni contents the water was suitable for irrigation. The Zn, Ni, Cu, Pb and Cd contents obtained in this investigation varied between 0.890 ± 0.28 to 0.980 ± 0.21, 1.095 ± 0.28 to 1.129 ± 0.34, 0.125 ± 0.08 to 0.276 ± 0.09, 0.099 ± 0.03 to 0.143 ± 0.04 and 0.081 + 0.01 to 0.086 ± 0.02 mg/l, respectively.

Chemical fractionation of trace metals in sewage sludge

The result of the chemical fractionation of trace metals in sewage-sludge from the drainage basin and some physico-chemical characteristics of the sludge are shown in Table 3. The study revealed that the sludges were slightly acidic in nature with average values ranged from 6.70 ± 0.18 to 6.19 ± 0.14. The organic matter and electrical conductivity were found to be at the range of 37.2 ± 8.3 to 37.9 ± 7.6% and 5.14 ± 0.51 to 1.86 ± 0.33 dSm^{-1}, respectively. Also, the cation exchange capacity (CEC) values determined by the sum of 1.0 mol/dm^3 CH_3COONH_4 exchangeable cations were varied between 73.82 to 79.71 cmol/kg and Ca was the dominant exchangeable cation 41.04 ± 14.0 to 43.41 ± 7.03 cmol/kg.

Table 2 . Mean (± SD) of physico - chemical parameters of sewage water and standard permissible limits for irrigation water.

A. Physico-chemical Parameters

Sampling dates	pH	EC (dSm^{-1})	Organic Matter (%)	Ex-Na (cmol/kg) dry weight	Ex-K (cmol/kg) dry weight	Ex-Mg (cmol/kg) dry weight	Ex-Ca (cmol/kg) dry weight	(cations (cmol/kg) dry weight
19/02/08	6.74 ± 0.22	1.54 ± 0.15	37.6 ± 8.6	7.15 ± 2.74	10.10 ± 2.24	15.78 ± 2.28	41.04 ± 14.0	74.07
24/03/08	6.70 ± 0.18	1.65 ± 0.4	37.2 ± 8.3	3.80 ± 1.43	10.06 ± 1.82	16.79 ± 3.62	43.17 ± 8.41	73.82
21/04/08	6.91 ± 0.14	1.86 ± 0.33	37.9 ± 7.6	7.57 ± 2.75	10.84 ± 2.94	17.89 ± 2.60	43.41 ± 7.03	79.71

B. standard permissible limits for irrigation water

Trace metal fraction (mg/kg, dry weight)	Water Soluble	Exchangeable	Oxidizable (Bound to Organic Matter)	Acid Soluble (Bound to carbonate)	Reducible (Bound to Fe/Mn oxides and hydroxides)	Residual (Bound to Silicates)	%	Non-Residual	%	Total Extractible ($\sum_T$)
Zn	4.1 ± 1.5	9.4 ± 3.9	22 ± 11.4	28.6 ± 17.3	39.0 ± 15.2	299.9 ± 41.4	74.4	103.4	25.6	403.3
Ni	2.0 ± 0.8	4.7 ± 2.0	12.0 ± 4.9	8.4 ± 3.1	23.1 ± 9.1	134.0 ± 33.7	72.7	50.2	27.3	184.2
Cu	3.0 ± 0.9	4.5 ± 1.7	10.4 ± 3.1	8.2 ± 1.9	7.9 ± 2.5	269.4 ± 31.1	88.8	34.0	11.2	303.4
Pb	1.0 ± 0.3	1.6 ± 0.4	6.4 ± 1.6	3.5 ± 1.1	9.6 ± 2.6	106.9 ± 19.8	82.9	22.1	17.1	129.0
Cd	0.3 ± 0.09	0.4 ± 0.1	0.5 ± 0.	0.8 ± 0.3	0.6 ± 0.2	17.1 ± 3.0	86.8	2.6	13.2	19.7

Table 3. Chemical fractionation of trace metals and some physico-chemical parameters of sewage - sludge.

A. Parameter

Sampling date	pH	Conductivity (μScm^{-1})	Total alkalinity (mg $CaCO_3$/l)	Chloride (mg/l)	Sulphate (mg/l)	Phosphate (mg/l)	Nitrate (mg/l)	Ammonia (mg/l)
19/02/08	7.71± 0.17	1291.5 ± 136.1	122.3 ± 21.2	38.5 ± 6.3	259.0 ± 44.6	0.05 ± 0.02	9.08 ± 3.2	0.08 ± 0.03
24/03/08	8.19 ± 0.1	1646.1 ± 224.5	133.2 ± 17.7	43.7 ± 8.5	295.5 ± 28.8	0.13 ± 0.05	11.99 ± 3.1	0.09 ± 0.04
21/04/08	8.31± 0.11	1887.9 ± 136.4	141.3 ± 17.5	55.7 ± 9.6	329.1 ± 31.1	0.16 ± 0.07	13.70 ± 2.8	0.11 ± 0.04
FAO limits	6.5 - 8.4	> 200		4.0	300	2	5	

B. Trace metal

Sampling date	Zn (mg/l)	Ni (mg/l)	Cu (mg/l)	Pb (mg/l)	Cd (mg/l)
19/02/08	0.890 ± 0.28	1.109 ± 0.38	0.274 ± 0.09	0.099 ± 0.03	0.085 ± 0.02
24/03/08	0.974 ± 0.22	1.095 ± 0.28	0.276 ± 0.09	0.103 ± 0.03	0.086 ± 0.02
21/04/08	0.980 ± 0.21	1.129 ± 0.34	0.251 ± 0.08	0.143 ± 0.04	0.081 ± 0.01
Permissible limits	2.0[b]	1.4[a]	0.017[a]	0.065[a]	0.01[b]

(i) Non-residual is the sum of water soluble, exchangeable, oxidizable, acid soluble and reducible fractions.
(ii) Total extractable is the sum of all six fractions for the metal.
(iii) All data reported as mean with one standard deviation (SD).
Source: USEPA, 2001; [b] Source: Ayers and Westcot FAO, 1985.

Table 4. Total extractable trace metals and their individual contamination factors (ICF) in the river basin.

Trace metal	Total extractable	ICF
Zn	403.3	0.3
Ni	184.2	0.4
Cu	303.4	0.1
Pb	129.0	0.2
Cd	19.9	0.2

ICF = Sum of water soluble, exchangeable, oxidizable, acid soluble and reducible/residual.

The data obtained by the sequential extraction procedure indicated the following trace metal distribution pattern.

Zn: residual > reducible > acid soluble > oxidizable > exchangeable > water soluble.
Ni: residual > reducible > oxidizable > acid soluble > exchangeable > water soluble.
Cu: residual > oxidizable > acid soluble > reducible > exchangeable > water soluble.
Pb: residual > reducible > oxidizable > acid soluble > exchangeable > water soluble.
Cd: residual > acid soluble > reducible > oxidizable > exchangeable > water soluble.

The non residual fraction, for all the metals contained more than 11% of the total extractable metals. All the trace metals were mostly concentrated in the residual fraction and this reflected the greater tendency of the metals to become less available to sewage water column. The study also further showed that the sludges contained less significant fractions in non-residual fraction. The residual fraction represents metals that largely embedded in the crystal lattice of the sludges and should not be available for remobilization except under very harsh conditions. Zn, Ni and Pb were largely associated with residual and reducible phase in the sludges. The Fe/Mn oxides (reducible phase) exist as concretions, cement between particles, or as coating on particle and are excellent trace metal scanvengers (Jenne, 1968). On the other hand, Cu existed mostly as residual and oxidizable, while Cd largely associated with residual and acid soluble phases. The oxidizable fraction of Cu was in agreement with the fact that Cu forms stable complexes with organic matters due to high formation constable (Stumm and Morgan, 1981). The much presence of Cd in acid soluble among the non – residual fractions was not surprising, as the acid soluble fraction is influenced by pH and the observed pH of the sludges were ranged from 6.70 to 6.91, which could favour the release of Cd into the sewage water column from this fraction. In a nutshell, removal of all the metals requires strong acid condition and might therefore, be little availability for remobilization into sewage water column since the sludges were slightly acidic.

Total extractable trace metals from the drainage basin sludges and their individual contamination factors (ICF) are presented in Table 4. The data obtained shows that the trend of occurrence of the trace metals in the sludges was: Zn > Cu > Ni > Pb > Cd >. The Zn and Cu values got in this study were less than the corresponding values reportd by Sujeet and Banerjee (2004), whereas the values of Ni, Pb and Cd were higher than what reported by them. The total extractable of Zn, Ni, Cu, Pb and Cd were 403.3, 184.2, 303.4, 129.0 and 19.7 mg/kg dry weight, respectively. The ICF for each metal in the drainage basin was calculated from the result of the fractionation study by dividing the sum of the first 5 fractions (that is, the water soluble, exchangeable, oxidizable, acid soluble and reducible forms) by the residual fraction for the basin and reflects the risk of contamination of a water body by a pollutant. The higher the levels of the mobilizetion fractions, the first 5 fractions in the sludges, the higher the potential risk to sewage water contamination by the drainage basin sludges. The ICF values found were relatively significant, with Ni posed the highest risk to sewage water contamination, followed by Zn, Cd, Pb and Cu posed the lowest risk to sewage water contamination.

Conclusion

The health status of river Kubanni drainage basin with regard to sewage water quality and risk to water column contamination by trace metals in the sewage-sludges from the drainage basin were evaluated in this study. The elevated levels of Cl^-, NO_3^-, Cu, Pb and Cd found in the sewage water rendered the water unsuitable for irrigation, as Cl^- and NO_3^- might deteriorate the properties of soil, whereas Cd, Cu and Pb could be directly detrimental to the vegetables and indirectly to man since the waters are used to irrigate vegetable. Therefore, continual monitoring and assessment are highly essential. Regarding the risk to sewage water contamination by metals was, however, relatively significant based on the calculated individual contamination factors (ICF) obtained for the sludges from the trace metal fractionation study. As a result, periodic check up is strongly recommended, as this work may provide valuable baseline data for future research on the River Kubanni drainage basin, being the only main source for irrigation water during dry seasons for irrigating the biggest sewage water-irrigated vegetation in Zaria city.

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