

ORIGINAL RESEARCH ARTICLE

Analysis of Physico-Chemical Characteristics and Heavy Metals Content in Water Samples Utilizing Atomic Absorption Spectrophotometry in Kafin Hausa, Jigawa State, Nigeria

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ABSTRACT

Heavy metals contamination of ground, stream, and river water ecosystems is a worldwide environmental problem (Sekabira *et al.*, 2010), and between the wide diversity of contaminants affecting water resources, heavy metals receive particular concern considering their strong toxicity even at low concentrations (Marcovecchio *et al.*, 2007). The levels of heavy metals in surface, ground, and tap water samples collected in Kafin Hausa were determined using Atomic Absorption spectrophotometry, whereas nitrate, ammonium, and phosphate ions were analysed using spectrophotometric methods. The following ranges were obtained for Mn (0.032 ± 0.001 to 0.092 ± 0.001 mg/l), Fe (0.029 ± 0.001 to 0.051 ± 0.001 mg/l), Pb (0.017 ± 0.001 to 0.0095 ± 0.001 mg/l), Cu (0.016 ± 0.001 to 0.032 ± 0.001 mg/l), Cd (0 to 0.0055 ± 0.001 mg/l), Zn (0.016 ± 0.001 to 0.032 ± 0.001 mg/l), $\text{NO}_3\text{-N}$ (1.16 ± 0.261 to 1.55 ± 0.232 mg/l), $\text{PO}_4\text{-P}$ (0.596 ± 0.267 to 1.26 ± 0.502 mg/l) and $\text{NH}_4\text{-N}$ (0.700 ± 0.041 to 0.771 ± 0.094 mg/l), pH (8.2 ± 0.1 to 8.4 ± 0.1), EC (134.7 ± 0.5 to 161.6 ± 0.4 $\mu\text{S/cm}$), Temperature (26.5 ± 0.00 to 27.8 ± 0.00 °C) and TDS (64.9 ± 0.3 to 80.8 ± 0.2 mg/l). The results of this study showed that out of those metals and anions analyzed only that of PO_4 exceeded the permissible limit of good portable water set by NAFDAC/WHO (2011) in all the samples analyzed.

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KEYWORDS

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INTRODUCTION

It has been reported that, by 2030, in some developing regions of the world, water demand will exceed supply by 50% (Es'haghi *et al.*, 2011). Groundwater and surface water are important and major drinking water sources in both urban and rural areas in Kafin-Hausa. Determination of water quality is one of the most important aspects of water studies. Evaluated concentration of heavy metals in water may cause phytotoxicity, direct hazard to human health, indirect effects due to transmission through the food chain, or contamination of groundwater or surface water (Omolaye *et al.*, 2010). Heavy metals are persistent environmental contaminants at least five times denser than water. As such, they cannot be metabolized by the body and are stable and bio-accumulative (Es'haghi *et al.*, 2011). Lead is one such heavy metal and the most common of these elements. It is a general metabolic poison and enzyme inhibitor. It can cause mental retardation and semi-permanent brain damage in young children. Lead can replace calcium in bone to form sites for long-term replacement (Es'haghi *et al.*, 2011). Heavy metals like copper are the essential trace elements but

show toxicity in excess amounts. With increasing industrial use, environmental pollution, and associated toxic exposure, concern has increased about their long-term exposure and potentially toxic effects on human health, especially infants and young children, which are at the peak of growth (Min *et al.*, 1996; Mahajan *et al.*, 2005). Cadmium is extremely toxic even in low concentrations and will bio-accumulate in organisms and ecosystems, and it has a long biological half-life in the human body, ranging from 10 to 33 years (Es'haghi *et al.*, 2011). Long-term exposure to cadmium also induces renal damage (Celik and Oehlenschlager, 2007).

Thus, there is a need to continuously assess the quality of ground and surface water sources. That's why a matter of great importance is to analyze and quantify these toxic agents in the environment. The Atomic Absorption Spectrophotometry (AAS) technique has been widely accepted as the standard technique for metal determination since it offers satisfactory sensitivity and fairly low acquisition cost (Es'haghi *et al.*, 2011; Celik and

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Oehlenschlager, 2007). Heavy metals, including essential and non-essential elements, have a particular significance in ecotoxicology since they are highly persistent and potentially toxic to living organisms. Heavy metals are members of a loosely defined subset of elements that exhibit metallic properties. It mainly includes transition metals, some metalloids, lanthanides, and actinides. Heavy metals occur naturally in the ecosystem with large variations in concentration (Mohsen and Salisu, 2008).

This study aims to assess physicochemical properties, including anions, and determine the levels of some heavy metals in tap, ground, and surface water sources in Kafin Hausa, Jigawa State, using an Atomic Absorption Spectrophotometer.

Study area

The study was conducted in Kafin Hausa Local Government area of Jigawa state, Nigeria. Its headquarters are in Kafin Hausa town and situated approximately between latitudes 11.00 °N to 12.26 °N and longitudes 9.00 °E to 10.00 °E (Suleiman *et al.*, 2018). It has a projected population of 271,058 in 2016 (NPC, 2006). The people of Kafin Hausa are predominantly farmers growing many varieties of cereals.

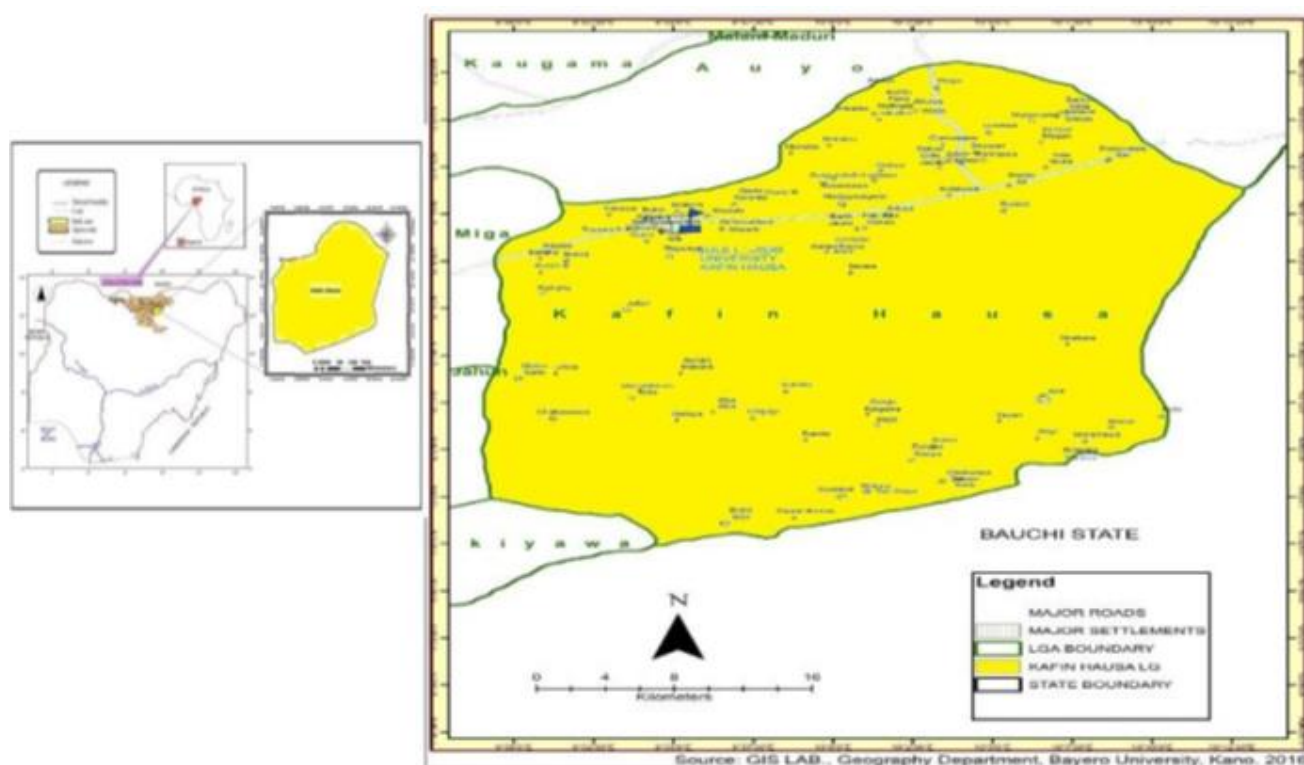


Figure. 1: Location map of Kafin Hausa (Adopted from Suleiman *et al.*, 2018).

Reagents and sample collection

5.5 M H₂SO₄, 2.5 M H₂SO₄, concentrated nitric acid (HNO₃), concentrated HCl, alkaline persulfate, 0.00324 M ammonium molybdate, 0.1 M ascorbic acid, 0.008214 M potassium antimony tartrate, reducing agent, 0.0581 M acidic sulphanilamide, N(1-Naphthyl) ethylenediamine dihydrochloride, phenol solution, sodium nitroprusside solution, oxidizing solution were used throughout the experiments. All solvents were of analytical grade and were used as received.

Water samples from Kafin Hausa were collected in 1000 cm³ low-density polyethylene bottles from four (4) sites spread across five sampling locations 300 m apart. The water samples used for heavy metals analysis were acidified using 3.0 cm³ of 1:1 trinitrate (V) acid (Mitsios and Golia, 2005). This reduces precipitation and

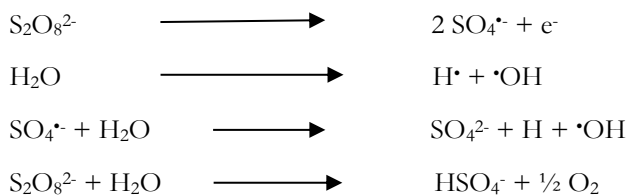
adsorption losses to the container walls. Samplings were carried out during the early part of the wet season. All the samples were appropriately labeled as River water, Openwell water, Tap water, and Borehole water, respectively, stored in large plastic buckets with ice blocks and transported to the laboratory. The water samples were filtered using a filter paper and stored in a fridge at 4 °C.

Experimental procedures

Sample Digestion for the Determination of Total Phosphorus and Total Nitrogen

To 50 cm³ of the water sample in a 125 cm³ Erlenmeyer flask was added 1 cm³ of 2.5M H₂SO₄ solution, and 25 cm³ of alkaline persulfate solution was boiled gently on a preheated hot plate until a final volume of 10 cm³ was

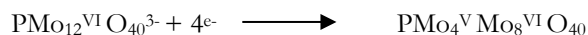
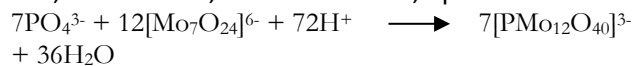
reached. The solution was cooled and diluted to 30 cm³ capacity with distilled water. A drop of phenolphthalein indicator solution was added and neutralized to a faint pink color with NaOH solution (Liang *et al.*, 2007).



Persulfate digestion (Liang *et al.*, 2007)

Phosphate determination

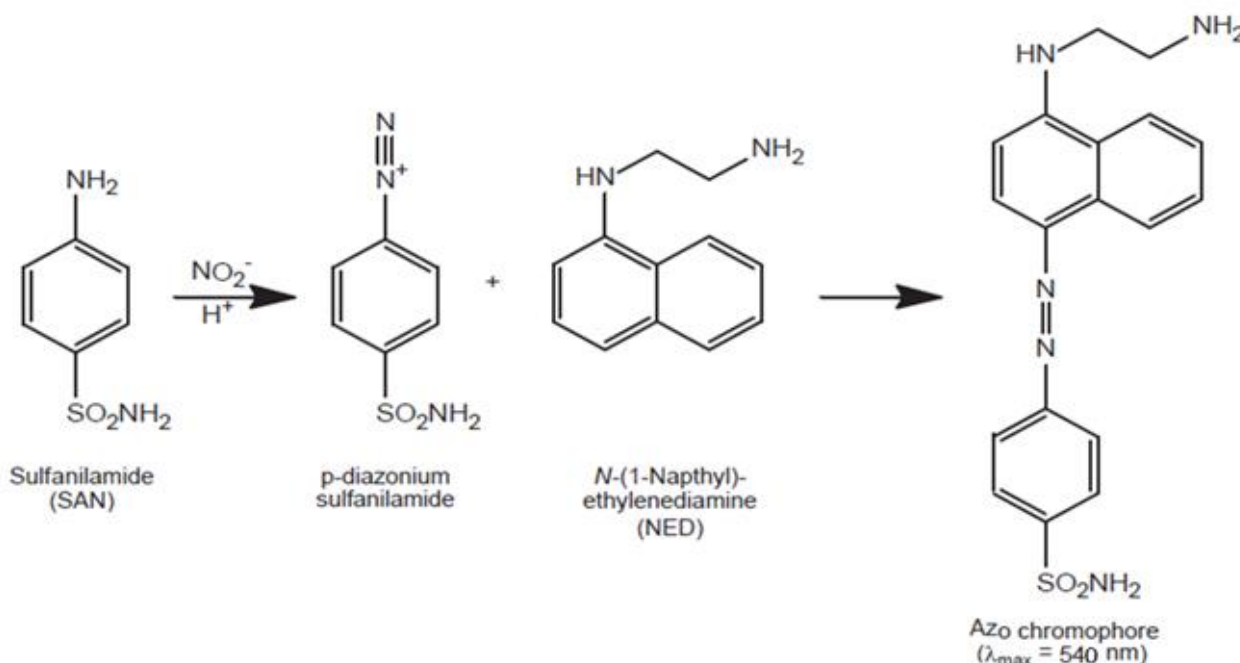
30 cm³ of the sample was pipetted into a 50 cm³ calibrated flask with the addition of 8 cm³ of the mixed reagent and mixed thoroughly. The solution was allowed to stand for 20 minutes for optimum color formation; after that, the absorbance of the sample was measured at 880 nm, using reagent blank as the reference solution (Charles *et al.* 2003).



Reaction scheme for Phosphorus determination

Nitrate determination

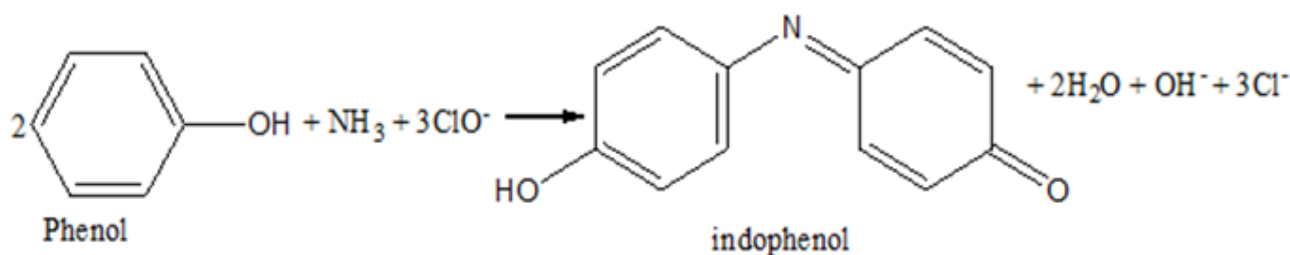
5 cm³ of the digested sample was pipetted into a 50 cm³ volumetric flask, and 10 cm³ of 0.5 M NaOH solution and 10 cm³ of the reducing reagent were added and heated for 15 minutes at 52 °C. 10 cm³ of 0.0581 M acidic sulfanilamide solution was added, shaken thoroughly for 5 minutes for the diazotization reaction to go to completion. After that, 10 cm³ N-(1-Naphthyl) ethylenediamine dihydrochloride solution was added to form an azo dye, and the contents were diluted to 50 cm³ with water. The absorbance of the pink-colored dye solution was measured at 540 nm against the corresponding reagent blank (Jones *et al.* 2016).



NH₄⁺-N determination

25 cm³ of the sample was transferred into a 50 cm³ Erlenmeyer flask, adding 1 cm³ phenol solution, 1 cm³ sodium nitroprusside solution, and 2.5 cm³ oxidizing

solution, the resulting solution was thoroughly mixed after each addition. The samples were covered with plastic wrap and kept in the dark at ambient temperature for 1 hour (Nikolaos *et al.*, 2010). The absorbance was measured at 640 nm against the reagent blank.



Sample digestion for Heavy Metals

500 cm³ of the filtered water sample in a 1000 cm³ was placed on a hot plate and evaporated to about 50 cm³. It was allowed to cool and transferred into a 250 cm³ beaker. 5 cm³ of concentrated HNO₃ was added, and the resulting solution was heated at 85 °C until a clear solution was obtained. The digested sample was allowed to cool, then transferred into a 100 cm³ volumetric flask and made up to mark with deionized water (APHA, 2005).

Conductivity Measurement

The conductivity of the water samples was measured using a calibrated BDS-11C digital conductivity meter. The meter was standardized using a 0.01 M KCl solution with a conductivity reading of 1412 μS/cm at 25 °C. It was switched on and allowed to warm for about 15 minutes. The electrode was then immersed into the sample, and the reading was taken when a stable value was obtained (APHA, 2005).

pH Measurement

The pH was measured at the sampling sites using a portable Wagtext International Potatest pH meter. The meter was calibrated by dipping the electrode tip in a pH 7.0 buffer (HI 7007P) sample at ambient temperature. The reading was allowed to stabilize, and the pH 7.0 trimmer was adjusted until the reading displayed 7.0. The electrode was rinsed with water and dipped into a pH 10.01 buffer solution (HI 7001P). The reading was allowed to stabilize, and the pH 10 trimmer was adjusted to a reading of 10.01. The standardized meter was switched on and warmed for about 15 minutes. The electrode was then immersed into the water sample, and the measurement was taken when a stable value was obtained. The electrode was rinsed with deionized water before each reading (APHA, 2005).

Instrumental analysis

Metals Determination

The absorbance values of Pb, Cu, Cd, Cr, Zn, and Ni in the water samples were taken using an Atomic Absorption Spectrophotometer at their absorption lines. Instrumental calibration was carried out before metal determinations by taking the absorbance values of the standard solutions prepared for the different metals. Calibration curves for the different metals were plotted using the results obtained. The water samples were aspirated into the spectrophotometer, and the absorbance reading was

recorded. The same procedure was used for the blank and standard solutions. The concentrations of metals were extrapolated from the calibration curves.

Phosphate and Nitrate Determination

A 755S UV-VIS spectrophotometer with Deuterium Lamp and Halogen-Tungsten Lamp was used to determine TN and TP levels. Whereby working standards were prepared by further dilution of 1000 ppm stock solution (of KNO₃ and KH₂PO₄), and a calibration curve was generated by plotting absorbance versus concentration. The concentrations of the TN and TP in samples were determined by interpolation.

Data Analysis

All analyses were performed in triplicates, and the results were expressed as means of ±SD. The difference in metal concentrations among the different sites was treated by correlation coefficient, one-way analysis of variance method, (ANOVA) and Tukey's test to determine pairwise differences among locations. The comparisons were considered statistically significant in an analysis where P<0.05. All statistical calculations were performed with SPSS 20.0 for Windows.

RESULTS AND DISCUSSION

Table 1: physical characteristics of water samples from Kafin Hausa

Sites	TDS (mg/l)	EC (μS/cm)	pH	Temp. (°C)
River	67.3	134.7	8.0	27.7
OW	73.3	142.7	7.6	27.8
TW	80.8	161.6	8.4	27.6
Borehole	64.9	129.9	8.2	26.5

OW = Open Well; TW = Tap Water

The results obtained for the physical properties of the samples collected from the four sampling sites are presented in Table 1. The pH variations for all the water samples were between 7.60 to 8.4, which is within the World Health Organization standards and was not significantly different from one another. It was observed that the Total Dissolved Solid (TDS) value varied from 67.3 to 80.8 mg/l, below the acceptable surface water

limits (300 mg/l). The highest value was obtained in tap water. The concentrations of TDS in all the sites were not significantly different from one another. Tap water has the highest electrical conductivity value among the collected water samples. However, there is no significant difference between the values recorded for all the sites at $p < 0.05$.

Ibrahim *et al.* (2009) studied the seasonal variations in the physicochemical parameters like pH, temperature, electrical conductivity, dissolved oxygen, total dissolved solids, nitrate-nitrogen, and phosphates-phosphorus. They recorded mean values of 7.15, 27.6 °C, 86.4 $\mu\text{s}/\text{cm}$, 4.70 mg/l, 43.2 mg/l, 5.21 mg/l and 6.86 mg/l respectively.

Table 2: Concentration of heavy metals (mg/l) in water sample

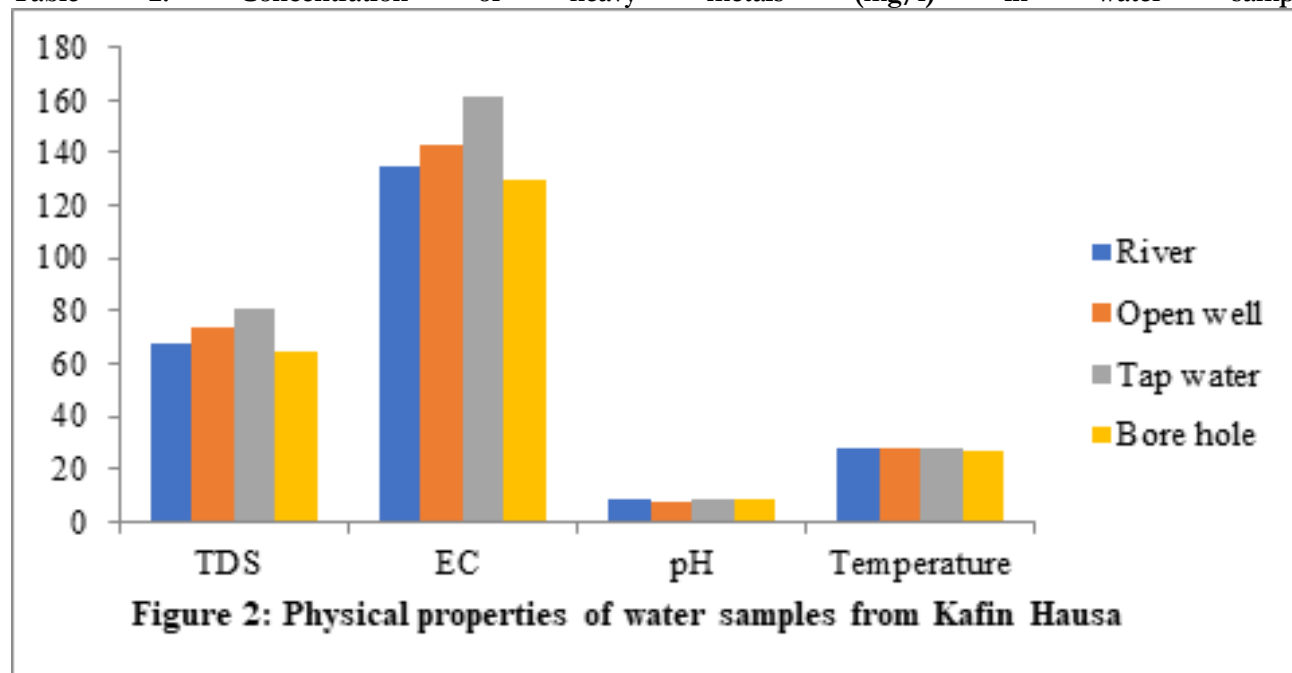


Figure 2: Physical properties of water samples from Kafin Hausa

from Kafin Hausa

Sites	Pb	Cd	Cu	Mn	Zn	Fe
River	0.0095±0.0001	0.0055±0.0001	0.032±0.006	0.048±0.001	0.021±0.002	0.051±0.001
Open well	0.0044±0.0001	0.0036±0.0001	0.025±0.001	0.081±0.002	0.016±0.001	0.044±0.001
Tap water	0.0017±0.0001	0.0018±0.0001	0.029±0.001	0.097±0.001	0.027±0.001	0.036±0.001
Borehole	0.0025±0.0001	ND	0.016±0.001	0.032±0.001	0.032±0.001	0.029±0.001

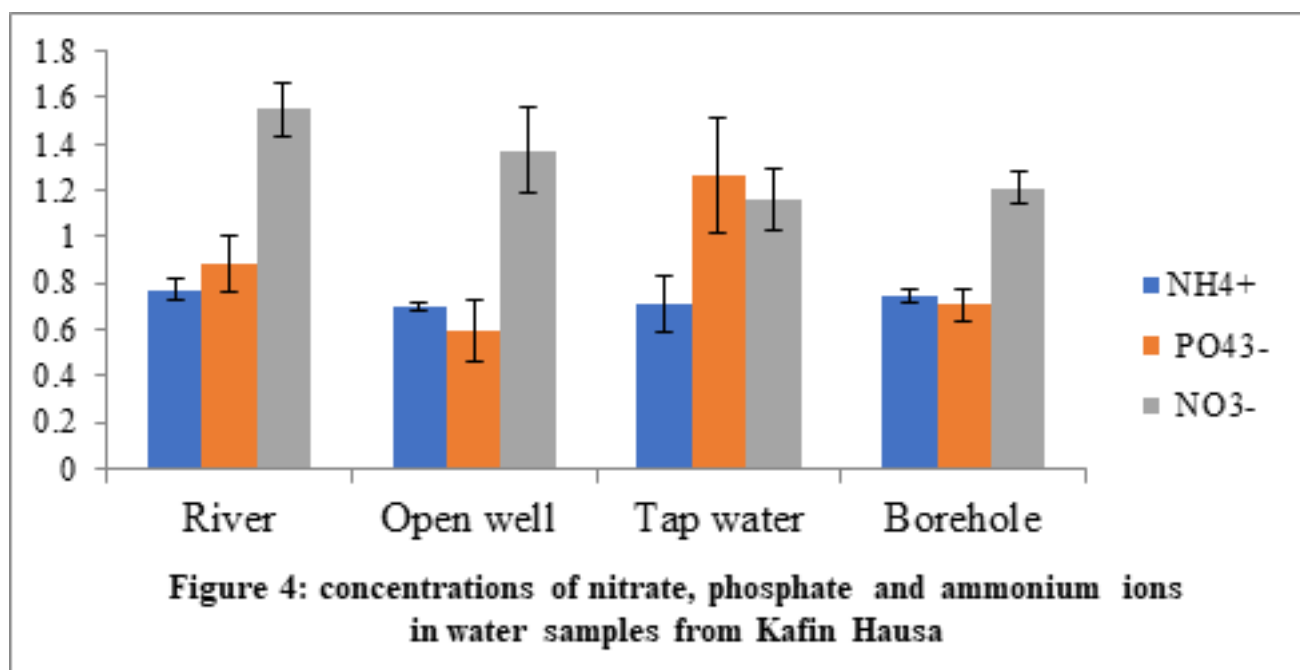
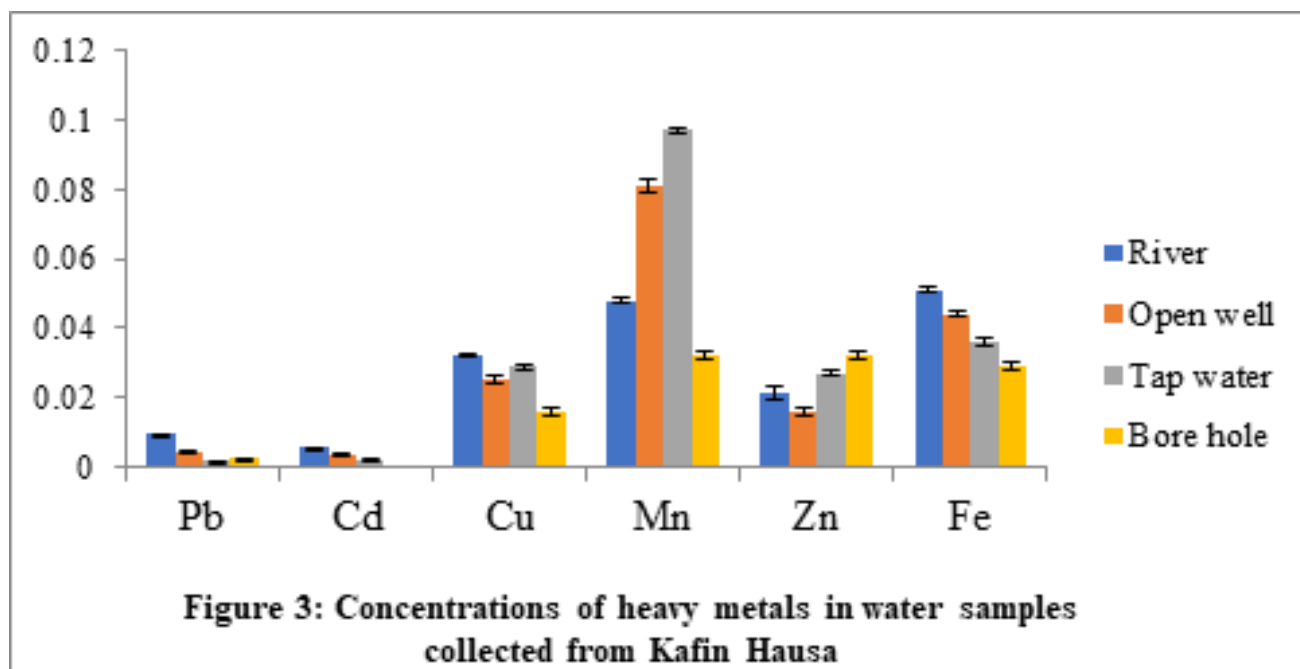
*ND: not detected

Table 2 shows that the mean values of heavy metals in water samples range from 0.0016 mg/l to 0.097 mg/l, with a higher value of Pb in the river, while the lowest level of Zn was observed in the open well water sample. The statistical analysis showed a significant difference among the heavy metal concentrations from the various sites. The Least Significant Difference (LSD) value for heavy metals is 5.22×10^{-4} . The statistical analysis showed that there is no significant difference among the sites. Pearson correlation analysis was conducted between heavy metal levels in water samples to assess if there are similarities in the sources of these heavy metals. Cd shows strong positive correlations with Pb, Cu, and Fe at a significance level of 0.01 (2-tailed), while Positive correlations were also recorded for Cu with Fe at a significance level of 0.01 (2-tailed). The possible source of these heavy metals

displaying positive correlations was considered similar, which was confirmed by the presence of a relation between levels of these metals.

It was observed that the difference between the mean concentrations of Pb in sites tap water and borehole, open well and tap water were all above the LSD value, Cu, Mn, Zn, Fe in the river and tap water, open well and tap water, borehole, and tap water were all above the LSD value, too, which indicate that the level of Pb in tap water is significantly lower than the level of Pb in the borehole, open well, and river, it also signifies that the level of Cu in the borehole is lower than the levels of Cu in tap water, open well and river; similarly, Mn in the same site, Zn in open well and finally that of Fe is in borehole. Conversely, the value of Pb in open wells and tap water shows a

significant difference at $p < 0.05$. In addition, a significant variation was observed in the level of Cd among the



various sites. The SLD value (5.22×10^{-4}) of heavy metals is lower than the mean concentrations of the heavy metals in each site, which shows that the level of heavy metals is significantly higher than those recorded. On the other hand, there is no significant difference at $p < 0.05$ between the level of Pb in open wells and tap water. The highest mean concentration of Pb was observed in the river, and the lowest concentration of Cu was observed in the borehole.

Oyeku *et al.* (2010) reported from a study carried out at Ojota in Lagos State that groundwater in the area was

generally alkaline (8.3 ± 2.77) and contained Cu (0.02 ± 0.04 mg/l), Fe (4.23 ± 6.4 mg/l), Pb (2.4 ± 3.3 mg/l) and Co (1.03 ± 1.1 mg/l) concentrations that are higher than the permissible limits recommended by the World Health Organization. The concentrations of Cr, Zn, and Pb in water and fish species of Zobe Dam were reported by Batagarawa and Uli (2000). They observed that the concentrations of Cr and Pb, which were 0.188 mg/l and 0.933 mg/l, respectively, exceeded the permissible limit for drinking water but were within the limit for irrigation.

Table 3: Concentration of anions (mg/l) in water samples from Kafin Hausa

Sites	NO ₃ ⁻	PO ₄ ³⁻	NH ₄ ⁺
River	0.771±0.004	0.887±0.001	1.553±0.005
OW	0.700±0.001	0.596±0.001	1.371±0.001
TW	0.710±0.009	1.261±0.001	1.159±0.001
Borehole	0.747±0.001	0.705±0.001	1.207±0.002

OW = Open Well; TW = Tap Water

Moreover, in a study carried out in River Jakara in Kano State, it was reported that the concentrations of nitrate ranged from 3.25 mg/l to 11.5 mg/l, and a lower mean value of 3.80 mg/l was recorded for phosphate [Dike et al., \(2010\)](#). World Health Organization (WHO, 2011) standards and permissible levels for the heavy metals of interest (Lead, Cadmium, Zinc, Manganese, Copper, and Iron) set up in 2011 are 0.01 mg/l, 0.03 mg/l, 3 mg/l, 0.4 mg/l, 2 mg/l and 3 mg/l and the anion standard such as (NO₃⁻, PO₄³⁻ and NH₄⁺) are 10 mg/l, 0.03 mg/l and 10

mg/l respectively. The result of this project indicated that all the analyzed heavy metals and anions are below the WHO and NAFDAC standard permissible level for safe drinking water except that of PO₄³⁻ and it is therefore a good portable, safe, for drinking and other activities.

CONCLUSION

This project has determined the various concentrations of the heavy metals and physicochemical properties of water samples. It has shown that out of those metals and anions analyzed, only PO₄³⁻ exceeded the permissible limit of good portable water set by NAFDAC and [WHO \(2011\)](#) in all the samples analyzed. Therefore, it is concluded that these water samples in the study area, Kafin Hausa local government, Jigawa state, Nigeria, are not too well portable for consumption due to the high level of PO₄³⁻ while they are safe for other activities such as irrigation and will support aquatic life.

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