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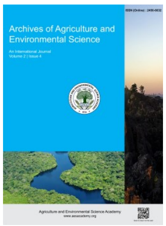

ORIGINAL RESEARCH ARTICLE

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Assessment of nitrate, phosphate and sulphate levels in wastewater from Muhammad Ayuba dam in Kazaure, Jigawa state, Nigeria

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ABSTRACT

In the continuation of the evaluation of the physicochemical parameters of wastewater of Muhammad Ayuba dam in Kazaure, Nigeria, this investigation was carried out for the assessment of certain nutrients parameters *i.e.*, nitrate, phosphate and sulphate that are primarily responsible for the eutrophication. This investigation was conducted during November, 2019 to January, 2020 (Harmattan season) to measure the content of nitrate, phosphate and sulphate ions in the wastewater collected from Muhammad Ayuba dam. During the study, standard techniques were used to determine the content of nitrate, phosphate and sulphate using spectrophotometer at specific wavelengths in determination of parameter of interests. The results obtained indicated that the concentrations were in the range of 78.50 – 88.40 mg/L for nitrate, 55.70 – 62.40 mg/L for phosphate and 91.40 – 100.20 mg/L for sulphate ions, respectively. The contents of nitrate, phosphate and sulphate in the wastewater exceeded the limit set by FAO/WHO for these anions in wastewater with exception of sulphate ions that was below the limit. Pearson correlation (r = 0.484) of the different anions indicated that their level of contamination might be traced to the same source means runoff from fertilizer application. Therefore, the water of the dam is majorly polluted with domestic wastes and absolutely rich in sulphate ions especially detergents from nearby houses. The study also revealed gradual accumulation of anions in the wastewater of the dam suggested more pronounced pollution in it. Thus, regular monitoring of these ions must be required to evaluate their environmental impacts and possible potential risks.

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INTRODUCTION

Sulphur is the 14th most abundant element in the earth's crust. Sulphate is produced in the environment from the oxidation of elemental sulphur, sulphide minerals or organic sulphur. It is naturally occurring compound containing sulphur and oxygen present in various mineral salts that are found in soil forming salts with variety of elements like barium, calcium, magnesium, potassium and sodium (Field, 1972). Sulphate may be leached from the soil into groundwater and commonly found in most water supplies. Magnesium, potassium and sodium sulphate salts are all soluble in water while calcium and barium sulphates are slightly soluble in water. Decaying of plant and animal matters may also release sulphate into water. Numerous chemical products including ammonium sulphate fertilizers contain sulphate in a variety of forms. The treatment of water with aluminium sulphate (alum) or copper sulphate also introduces sulphate into a water supply (US-EPA, 2001). Human activities such as the combustion of fossil fuels and gas processing releases sulphur-oxides to the atmosphere, some of which are converted to sulphate. Sulphate is generally considered to be non-toxic. The consumption of drinking water containing high amounts of magnesium or sodium sulphate may result in intestinal discomfort, diarrhoea and consequently dehydration

(Gomez *et al*., 1995). This laxative effect is observed when drinking water that contains greater than 500 milligrams per litre (mg/L) of sulphate. Diarrhoea and dehydration are often observed with individuals accustomed to drinking water with high concentrations of sulphate (US-EPA, 2001). Soils are thought to have an average of 850 mg/Kg sulphate and sea water has 885 mg/L sulphate. Industrial-sulphate results from the burning of sulphur containing fossil fuels, household wastes (e.g., detergents) and effluents from tanneries, steel mills, sulphate-pulp mills and textile plants (Kumar *et al*., 2020). Sulphuric acid accounts for an estimated 80 percent of commercial sulphur production (NRC, 1989). Additionally, thousands of tons of sulphate compounds are produced each year; annual production of sodium sulphate was estimated at 792 tons in 1987 (US-EPA, 1987). Most public water supplies contain sulphate concentrations of less than 500 mg/L (Oladeji and Saeed, 2015; US-EPA, 2001). Sulphate levels in water is expected to be around 250 mg/L and any values above are detectable due to an offensive odour and taste causing those exposed to water with higher concentrations of sulphates witch to bottled-water source for drinking.

Nitrate-containing compounds in the soil are generally soluble that means they dissolve easily in water (ATSDR, 2011). Thus, nitrates flow easily into groundwater. Microbes break down animal and human organic wastes in soil and water to ammonia. This breakdown process of converting organic wastes into ammonia, which oxidizes into nitrite and nitrate, is referred to as Nitrification process done by *Nitrosomonas* and *Nitrobacter* bacteria (Grubben, 1976). Plants uptake phosphate from soil mostly in the orthophosphate forms. Natural soil phosphate level is often low enough to limit crop production. Both inorganic phosphate fertilizers (treated rock phosphate) and organic phosphate sources (animal manures) are equally adept at supplying the orthophosphate ion and correcting phosphate deficiencies in soil (Oladeji, 2017; Edwards *et al*., 1990). The primary biological importance of phosphates is components of nucleotides which serves as energy storage within cells Adenosine Triphosphate (ATP) or when linked together form the nucleic acids DNA and RNA. The double helix of DNA is only possible because of the phosphate ester bridge that binds the helix, besides making biomolecules. Phosphorus is also found in bones and the enamel of mammalian teeth whose strength is derived from calcium phosphate. It is also found in the exoskeleton of insects and phospholipids (Oladeji and Saeed, 2018; Huttuner *et al*., 2006). In addition, it functions as a buffering agent in maintaining acid base homeostasis in the human body. Muhammad Ayuba dam is very close to Kazaure community making its prone to different degree of contaminations (Figure 1). This research is aimed toward determining nitrate, phosphate and sulphate ions in Muhammad Ayuba dam as to assess its level of contamination with anions.

MATERIALS AND METHODS

Collection of samples

Samples of wastewater from Muhammad Ayuba dam in Kazaure were collected for three-months (November, 2019 – January, 2020) on weekly basis at point of inlet into the stream at five different locations along its channels. Sampling covered the peak period of harmattan season in northern, Nigeria. Composite sampling technique was adopted, collected inside polyethylene plastic containers that were properly cleaned by washing in non-ionic detergent as described by Ademoroti (1996). Samples were collected inside 1000 cm^3 capacity bottles from each of the designated sampling points. Sample bottles used were properly rinsed with sampled water three times and then filled to the brim at a depth of one meter below the wastewater from each sampling point. Sample bottles were labelled accordingly, stored in ice-blocked coolers and transported to the laboratory where they were stored in the refrigerator at about 4°C prior to the analysis (APHA, 1998).

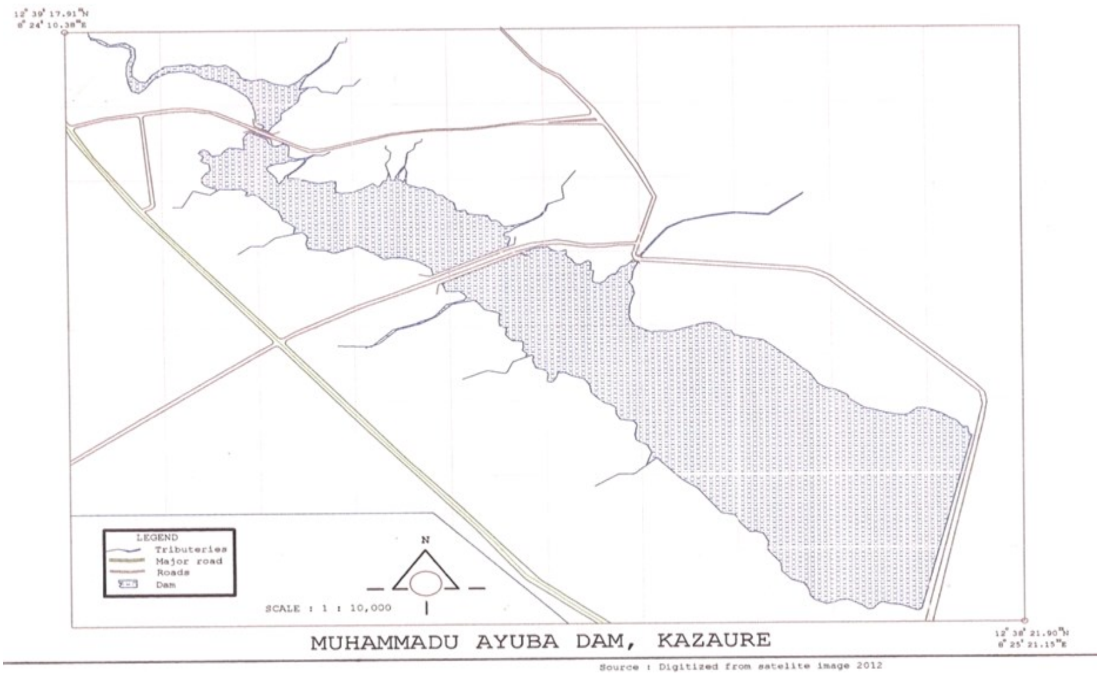

Figure 1. *Muhammad Ayuba dam, Kazaure, Nigeria (Source: Digital from satellite image, 2020).*

Determination of nitrate in wastewater

Ten-centimetre cube of each wastewater samples was injected into sample containers. The content of one (NitraVer 5 Nitrate) reagent powder pillow was added to each wastewater samples. It was stoppered and the cell shaken thoroughly for 1 minute. The sample concentration was read for each wastewater at 500 nm after 5 minutes using a portable Data Logging Spectrophotometer (HACH DR/2010) and multiplied by a conversion factor of 4.427 as stated by APHA (1998).

Determination of phosphate in wastewater

Phosphate in wastewater samples was determined as described by APHA (1998). Orthophosphate ion combines with ammonium molybdate under acidic condition to form a yellow complex compound known as ammonium phosphor-molybdate. It was reduced by tin (II)chloride solution in glycerol, the yellow complex was reduced to a blue-coloured compound due to molybdenum blue as described by APHA (1998).

Determination of sulphate in wastewater

A clean sample container was filled with ten centimetre cube of wastewater samples each. The content of one sulphate reagent powder pillow was added to each wastewater samples and swirled to dissolve. The cell was allowed to stand undisturbed for 5 minutes. The concentration was read at 450 nm using a portable Data Logging Spectrophotometer. The blanks samples were treated in the same way (APHA, 1998).

Statistical analysis

The results of anions in wastewater analysed were expressed in form of bar-charts using Microsoft Excel (Window 7 Professional). Pearson Product Moment Correlations (PPMC) using Statistical Package for the Social Sciences (SPSS) 20.0 version software was used to check if there is significant correlation among the anions analyzed. Statistical decision for Pearson correlation coefficients (r) was taken by following Robert (1992).

i) If $0.05 \le r \le 0.20$ there is negligible relationship
ii) If $0.21 \le r \le 0.40$ there is low relationship
iii) If $0.41 \le r \le 0.60$ there is moderate relationship
iv) If $0.61 \le r \le 0.80$ there is substantial relationship
v) If $0.81 \le r \le 1.00$ there is very high relationship

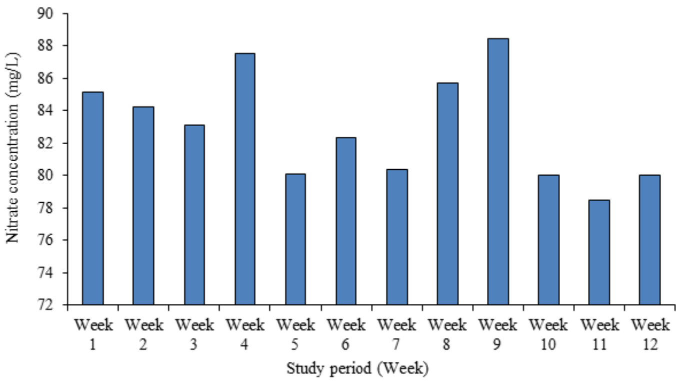

Figure 2. *Mean concentrations of nitrate in wastewater along Muhammad Ayuba dam in Kazaure, Nigeria.*

RESULTS AND DISCUSSION

The results of anions analyzed in wastewaters from Muhammad Ayuba dam in Kazaure is presented in form of bar charts. PPMC was used to check the level of correlation among the anions and presented in Table 1. Figure 2 shows mean concentrations of nitrate in wastewater across the sampling site of Muhammad Ayuba dam. Nitrate had mean concentration in the range of 78.50 – 88.40 mg/L. Highest mean level of 88.40 mg/L was obtained in week 9 followed by 87.50 mg/L in week 4 and this was closely followed by 85.70 mg/L in week 8 whereas the least mean concentration of 78.50 mg/L was recorded in week 11 as indicated in Figure 2. High levels of nitrate could be related to indiscriminate disposal of animal and human faeces from nearby houses into sampling site coupled with fertilizer runoff from agricultural farmland as suggested by Oladeji and Saeed (2018a); Ikemoto *et al.* (2002) and McCall and Willumsen (1998). WHO recommends 45 mg/L as maximum limit for nitrate in wastewater before it could be discharged into surface water and this indicates that concentrations obtained in this study exceeds the limit. Previously, Oladeji (2017) reported 23.05 – 283.54 mg/L while Akan *et al.* (2008) reported 211.43 – 284.33 mg/L as nitrate levels in wastewater which was higher than concentrations obtained in this present study.

Phosphate had mean concentrations in the range of 55.70 – 62.40 mg/L as indicated in Figure 3. Highest mean level of 62.40 mg/L was recorded in week 11 followed by 61.10 mg/L in week 6 and this was closely followed by 60.20 mg/L in week 5 while the least mean level of 55.70 mg/L was obtained in week 1. Elevated level of phosphate could be as a result of runoff from agricultural land, sewage and detergents as suggested by Oladeji and Saeed (2018b); Edwards *et al.* (1990). Phosphate concentration was generally high in wastewater which might be due to heavy discharged of effluents from municipal areas as noticed by Oladeji and Saeed (2018b). WHO (1985) has set 5 mg/L as maximum contaminant level for phosphate in wastewater before it could be discharged on river and this indicates wastewaters analyzed are polluted with phosphate ions. In their previous studies, Oladeji and Saeed (2018) reported 3.85 – 72.80 mg/L while Akan *et al.* (2008) reported 103.23 – 164.22 mg/L as phosphate levels in wastewater which was above concentrations obtained from this study.

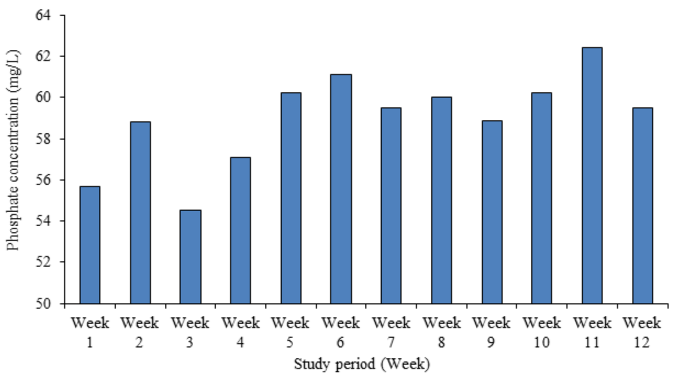

Figure 3. *Mean concentrations of phosphate in wastewater along Muhammad Ayuba dam in Kazaure, Nigeria.*

Table 1. Summary of Pearson product moment correlation for nitrate, phosphate and sulphate in wastewater.

Variables	N	x̄	SD	r	df	Significance
Nitrate	12	82.942	1.612	0.484	11	0.067
Phosphate	12	58.988	1.447			
Sulphate	12	96.020	1.345			

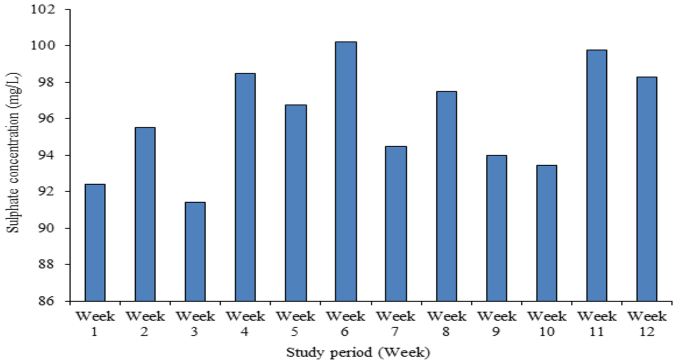

Figure 4. *Mean concentrations of sulphate in wastewater along Muhammad Ayuba dam in Kazaure, Nigeria.*

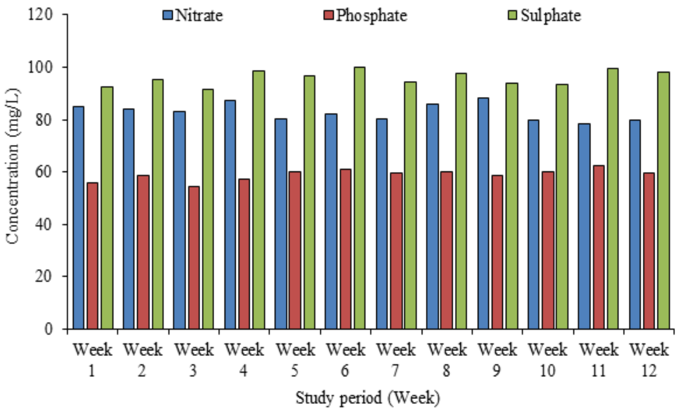

Figure 5. *Mean concentrations of nitrate, phosphate and sulphate in wastewater along Muhammad Ayuba dam in Kazaure, Nigeria.*

Sulphate had mean concentrations in the range of 91.40 – 100.20 mg/L as shown in Figure 4. Highest mean level of 100.20 mg/L was recorded in week 6 followed by 99.74 mg/L in week 11 and this was closely followed by 98.50 mg/L in week 4 whereas the lowest mean level of 91.40 mg/L was obtained in week 3. High level of sulphate ions might be related to its proximity to Kazaure town thereby generated domestic wastes rich in sulphate ions could be easily leached into dam as it was noticed by Butu (2013). WHO (1985) recommends 250 mg/L as Maximum Contaminant Level (MCL) for sulphate in wastewater and this indicates that sampling site is not polluted with sulphate ions. Similarly, Akan *et al.* (2008) reported 154.33 – 252.21 mg/L as sulphate levels in wastewater which was higher than results obtained in this study. Comparison of the three anions analyzed in wastewater samples is as shown in Figure 5. Highest concentration was noticed in sulphate ions followed by nitrate while the least was observed in phosphate ion. This showed that Muhammad Ayuba dam is majorly polluted with domestic wastes rich in sulphate ions especially detergents from nearby houses. The results also showed that agriculture wastes especially runoff from fertilizer application contributed to elevate concentrations of nitrate and phosphate in the dam.

Pearson Product Moment Correlation (PPMC) was conducted to establish the relationship among nitrate, phosphate and sulphate ions in wastewater as shown in Table 1. Statistical analysis showed the mean with standard deviation of 82.942±1.612 for nitrate, 58.988±1.447 for phosphate while 96.020±1.345 for sulphate. Statistical analysis also revealed Pearson correlation (r) = 0.484, degree of freedom (df) = 11 and p = 0.067 > 0.050 this means that there is moderate relationship among nitrate, phosphate and sulphate level in wastewater (Table 1). This could be as a result of sampling site is within the same vicinity therefore, they experienced similar level of contamination, similar geological soil formation and similar anthropogenic activities as stated by Oladeji and Saeed (2015) and Chiroma *et al.* (2014). Irrigating farmland with wastewater at Muhammad Ayuba dam increases the availability of carbonates and hydroxides ions due to precipitation making the soils of this site to be alkaline in nature and this reduces the mobility of heavy metals as they are more mobile at pH < 7 as suggested by Smith and Giller (1992) and Wei *et al.* (2005). According to Nyle and Ray (1999) soil samples with high sand and low clay contents have high pollutant leaching potentials thereby sandy nature of the soil at Kazaure town promote the leaching of heavy metals and other chemicals beneath the earth crust as suggested by Chiroma *et al.* (2014).

Conclusion

This investigation concluded that the contents of nitrate, phosphate and sulphate were exceeded the limit prescribed by FAO/WHO with exception of sulphate ions that was below the limit. This was attributed due to the heavy applications of manure, fertilizers, pesticides, insecticides, fungicides and other chemicals as it was being practiced by the farmers of this area which might contribute significantly to the elevated levels of these anions with time. Pearson Product Moment Correlation (PPMC) results showed moderate relationship among the nitrate, phosphate and sulphate ions of the wastewater analyzed. This was traced to sampling sites are within the same vicinity therefore they experienced similar level of contamination, similar geological soil formation and similar anthropogenic activities. The results per analyte were similar in concentrations which might be traced corresponding to the sampling sites and season and, these sites experienced similar level of contamination and it was due to the anthropogenic activities. Thus, periodical monitoring of these anions in the dam water should be nedded to evaluate their environmental impacts in order to assess their possible potential risks.

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