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Speciation of phosphorus and nitrogen in sediments of Ogun River in Abeokuta, Southwestern Nigeria

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Sediments in rivers can contribute to the concentration of phosphorus and nitrogen in overlying water which affect aquatic life adversely at excessive concentrations. This study examined the various forms of phosphorus and nitrogen in sediments of Ogun River, the major river in the city of Abeokuta.Total phosphorus and total nitrogen concentrations ranged from 820-2740 and 840-1960 µg N g^{-1} respectively. Inorganic phosphorus was the major component of phosphorus in the sediments. Exchangeable phosphorus, aluminium-bound phosphorus, iron-bound phosphorus, reductant phosphorus, and calcium-bound phosphorus were the inorganic phosphorus fractions obtained by sequential extraction using appropriate extractants. Exchangeable phosphorus was least present, while calcium-bound phosphorus was predominantly present at all the sampling sites; showing that the sediments in the river were apatitic in nature. Inorganic nitrogen forms: nitrate-nitrogen, nitrite-nitrogen and ammonium-nitrogen were obtained and nitrate-nitrogen was found to be the most dominant form of inorganic nitrogen at all the sampling sites. The results from this study suggest that Ogun River, Abeokuta, contained phosphorus and nitrogen which were traceable to anthropogenic activities from residential homes, farmlands, and an abattoir found close to the river. These elements have the potential of increasing algal bloom in the river.

Key words: Sediments contamination, phosphorus speciation, nitrogen speciation, Ogun River.

INTRODUCTION

Phosphorus (P) and nitrogen (N) are both limiting nutrients and important constituents of biogeochemical cycles (Pandey et al., 2023; Awasthi et al., 2022a; Papera et al., 2021; Zhang et al., 2018; Claesson and Ryding, 2013), The excessive accumulation of these macronutrients in water bodies causes eutrophication (Colborne et al., 2019; Davis et al., 2015; Erisman et al., 2013), which has detrimental effects on water bodies and is characterised by the proliferation of algal blooms, excessive phytoplankton growth, and the occurrence of hypoxia and anoxia (Adhikari and Mandal, 2019; Reinl et al., 2021). Eutrophication alters aquatic ecosystems, destroys plants and aquatic organisms (Dar et al., 2021) aggravates the susceptibility of aquatic animals to contagious ailments (Club and Branch, 2013), and causes economic losses (Wurtsbaugh et al., 2019). The severity of eutrophication has witnessed a notable increase in recent decades attributed to increased

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anthropogenic activities (Chen et al., 2020), and its impact is widespread in lakes, rivers, and streams. The capacity of water bodies to assimilate these nutrient inputs is posing substantial challenges to the preservation of water quality and safety (Liu et al., 2020; Schindler et al., 2012; Zamparas and Zacharias, 2014). Phosphorous derives primarily from weathering of rocks and is altered during biogeochemical progressions prior to it becomes available to any natural ecosystem (Sun et al., 2013; Padalia et al., 2022; Manral et al., 2020). Throughout this process of phosphorus transformation, microbial activities serve as the vital modules and continuously supply the bio-available phosphorous to the ecosystem (Awasthi et al., 2022b; Manral et al., 2022, 2023).

The point and non-point sources of P and N involve anthropogenic causes (Mekonnen and Hoekstra, 2018) related to agricultural runoff, industrial pollution, wastewater releases, and metropolitan areas with high population density (Cao et al., 2016; Rabalais et al., 2010), and also include stormwater runoff, soil nitrogen pool and atmospheric deposition of nitrogen (Pandey et al., 2024, Ahmad et al., 2016; Zhang et al., 2022). The effects of P and N in water bodies have proved largely inseparable, as they tend to vary collinearly (Lambert and Davy, 2011). The high influx of these nutrients leads to disturbances in aquatic food webs, consequently compromising the functioning of aquatic ecosystems (Grman et al., 2010).

The availability of P and N in water affects biogeochemical cycles (Ni and Wang, 2015; Zhu et al., 2019). Sediment is considered as an effective ecological filters as it acts as a sink for P and N loaded into water bodies (Lewandowski et al., 2015; Shang et al., 2013) making sedimentcontains substantial pools of the nutrients relative to overlying water. Overlying water and sediment are interdependent and linked by a variety of physical, biological and chemical processes (Shayo and Limbu, 2018) that are responsible for the decreased retention of P and N by sediment and their release into the water column, which increase their concentrations in overlying water (Rodellas et al., 2018; Wang et al., 2020; Wu et al., 2014). The internal P and N in sediments emerge as the main contributor to P and N loading in water, exerting significant influence on eutrophication processes when external P and N inputs are controlled (Barik et al., 2019; Ma et al., 2019; Spears et al., 2012; Wang et al., 2015; Xu et al., 2018). Hence, effectively managing the discharge of internal P and N in sediment plays a pivotal role in the restoration of eutrophic lakes (Wang et al., 2020). Achieving this goal involves enhancing the dissolved oxygen levels at the sediment-water boundary to restrain the release of P and N from sediment to the water column (Wang et al., 2016).

High P concentration is a key factor limiting primary productivity in water (Zhang et al., 2018), and is often the main driver of biodiversity loss, decline in aquatic plants, and impaired ecosystem function (Lambert and Davy, 2011). P species in sediments include exchangeable P, Al-bound P, Fe-bound P, Ca-bound P, reductant P and organic P (Wang, 2012). Sequential fractionation methodologies using different chemical extractants emerge as one of the few viable approaches for determining P species linked to sediment samples, primarily due to the inherently limited concentrations of P found within these substances (Wang et al., 2013).

N species in sediments are found to be nitrate, nitrite and ammonium (Akbarzadeh et al., 2018). Nitrate garners significant focus as N pollutants, and the majority of advanced nations enforce stringent standards for the quality of water concerning nitrates (Oenema et al., 2011). Nitrite is formed during the initial stages of nitrification and denitrification, originating from ammonium and nitrate, respectively, and while nitrite levels are anticipated to be insignificant, an accretion of nitrite has been detected in rivers and streams (Akbarzadeh et al., 2018). Nitrite also serves as a reactive intermediate generated and utilized in various redox processes within the N cycle (Mordy et al., 2010).

Ogun River in Nigeria is experiencing numerous anthropogenic discharges and the river transverses through Abeokuta and Owode. Previous studies have shown that the river at these key zones receives nutrient loading from abattoirs, farms and residential buildings (Akpan et al., 2020; Olaniran et al., 2019; Onunkwor et al., 2022). However, the quantification of internal loading of phosphorus and nitrogen as well as their fractions in sediments of the river has not been fully investigated. Thus, the information on various forms of phosphorus and nitrogen is necessary to understand their bioavailability in the river setting. In this study, we investigated the speciation of phosphorus and nitrogen in the sediments of Ogun River. Phosphorus speciation was determined using the sequential extraction method developed by Zhang and Kovar, while nitrogen speciation was determined through colorimetric analysis employing various chemical extractants (Zhang and Kovar, 2009). This work hoped to enhance the understanding of the levels and distributions of phosphorus and nitrogen species in surface sediment of Ogun River. Therefore, the objective of this study was to determine the extent of phosphorus and nitrogen pollution in the sediments of Ogun River and evaluate the contribution of sediments to pollution and algal bloom in Ogun River, Abeokuta.

MATERIALS AND METHODS

Description of study sites and sampling

Ogun River transverses through Abeokuta in Ogun state, Southwest Nigeria. It has a low topography and eventually drains into the Lagos Lagoon (Oke et al., 2015). In Abeokuta, the river flows through Lafenwa Bridge and discharges to Ogun State Water

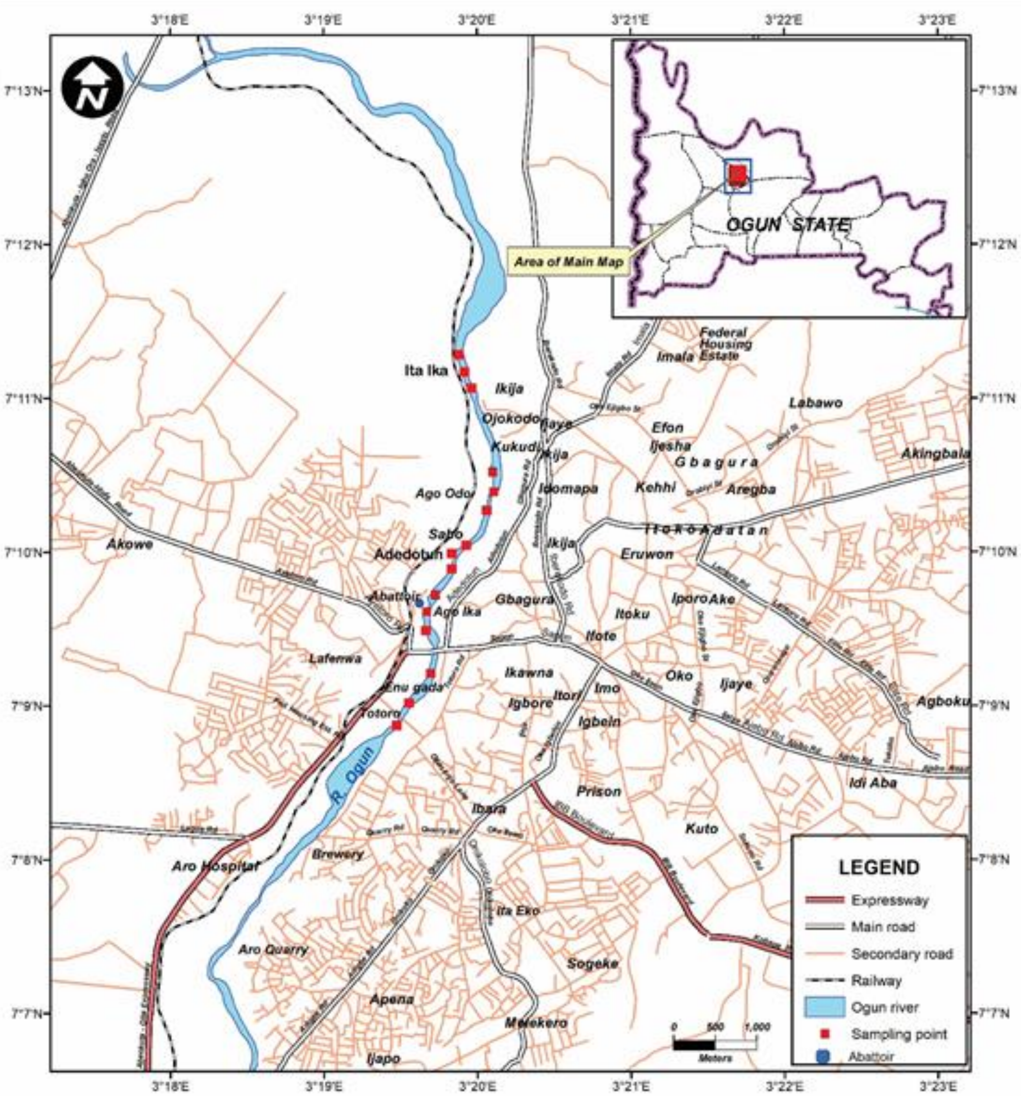

Figure 1. Map of Abeokuta showing sampling points.

Corporation dam at Ara Konga, where it is obstructed and its water is utilised for public consumption. Along the river is an abattoir which is a slaughterhouse at the Ago Ika area of Lafenwa and which empties its wastes into the river. The river is also flagged with refuse dumps by the sides and is used for fishing, irrigation, washing of beef, hide and locust beans. Five sampling sites were selected along the river and three sediments samples were collected from each site between April and June, 2021 (Figure 1). Sediment samples were collected using a grab sampler, stored in zip-lock polyethylene bags, transported frozen in the dark to the laboratory where they were air-dried. The samples were ground to a fine powder using mortar and pestle, and sieved with a 2 mm sieve. Prepared sediments were subsequently stored in sealed polyethylene bags for analysis.

Physicochemical analysis: pH, Bulk density, TOC, Total P and N

The pH measurements of prepared sediment samples were carried out using a calibrated pH meter (Kabała et al., 2016).Total organic carbon (TOC) and bulk density were determined by Walkley-Black wet oxidation core sampling methods, respectively (Nelson and Sommers, 1996; Zeng et al., 2014). Particle size analysis was carried following Bouyoucos method (Ashworth et al., 2001). Total phosphorus in sediments was determined using the metavanadate colorimetry method (Bender and Wood, 2000). Sediments (1.0 g) were measured into a 250 ml Erlenmeyer flask placed in a fume hood and 10 ml of concentrated nitric acid was added. The solution was heated to oxidise the sediments. 8 ml of perchloric acid was added and the solution was digested. The mixture was cooled and centrifuged for 10 min at 3500 rpm. The supernatant was decanted into a 50 ml standard flask and made up to mark with distilled water. The solution was analysed with a UV-Visible spectrophotometer at 400 nm to determine the total phosphorus.

Total Nitrogen was determined by the Kjeldahl Digestion Method (Bremner, 1996). 0.5 g of the prepared sediments was weighed and transferred into a 100 ml Kjeldahl digestion flask. The sediment was digested in the flask after the addition of 5 g of Kjeldahl catalyst, 10

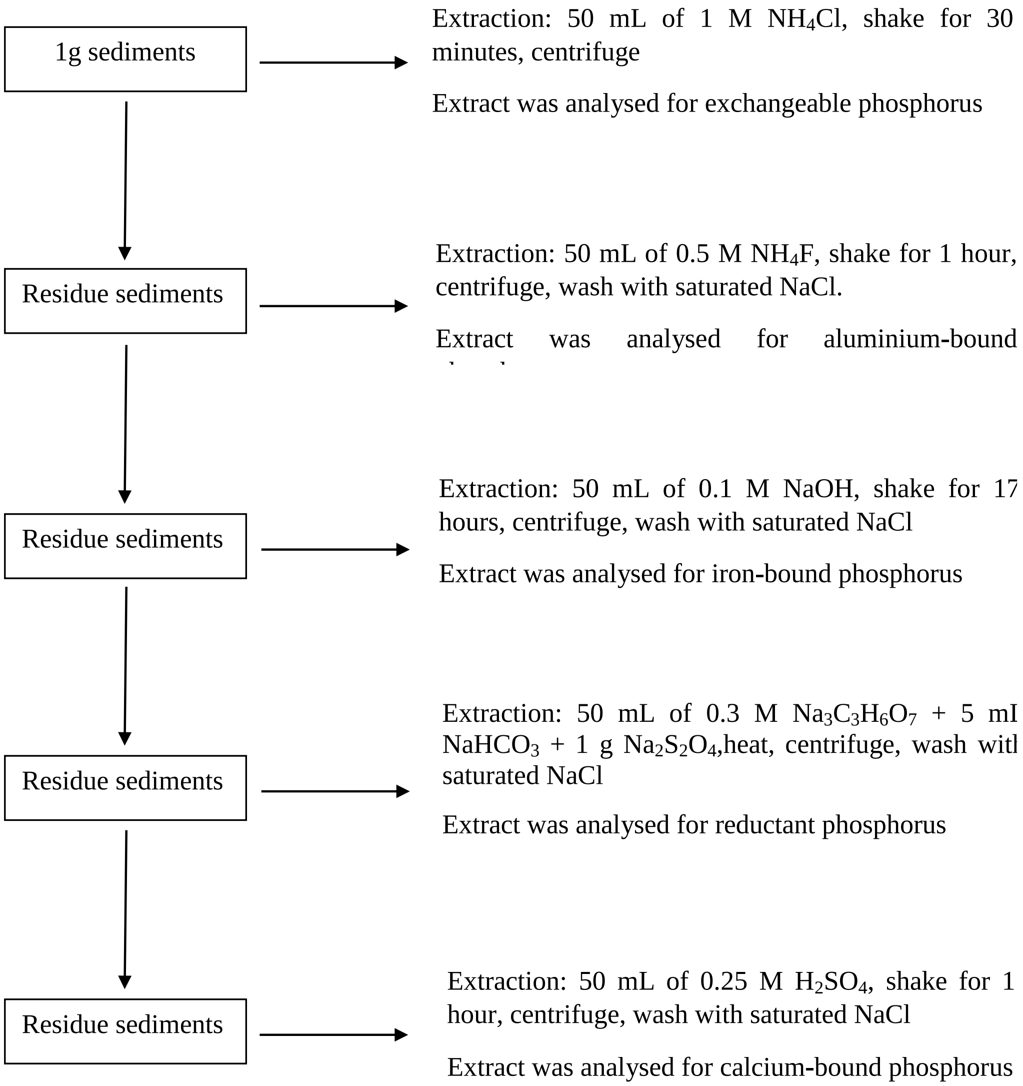

Figure 2. Sequential extraction scheme for inorganic phosphorus fractions.

ml of concentrated H_2SO_4 and anhydrous sodium sulphate.

Determination of phosphorus fractions

Sequential extraction procedure is displayed in details in Figure 2, by which inorganic phosphorus is classified as different forms of exchangeable phosphorus, aluminium-bound phosphorus, iron-bound phosphorus, reductant phosphorus, and calcium-bound phosphorus fractions (Zhang and Kovar, 2009). Furthermore, sediment sample used in a stage was applied for the next stage. The extracted solution was filtered through a 0.45 μm filter membrane before phosphorus measurement was done. In each phase and after extraction, samples were centrifuged at 4000 rpm for 20 min and phosphorus content was measured according to the method of molybdenum blue/ascorbic acid at 880 nm wavelength using a UV-VIS spectrophotometer (Murphy and Riley, 1962). The analysis was carried out in triplicates. For determination of total phosphorus concentration, dried sediment of each sample in 50 ml extractant were analysed using standard method of analysis (Ruttenberg, 1992). The concentration of organic phosphorus in the sediments was determined by deducting the inorganic phosphorus concentration from the total phosphorus concentration.

Nitrate-nitrogen, nitrite-nitrogen, and ammonium-nitrogen

Nitrate-nitrogen in sediments was determined by Brucine colorimetric method as described by Donald Nicholas and Nason (Nicholas and Nason, 1957). 1 g of prepared sediments was weighed into a beaker and 30 ml of 0.2 M NH_4Cl solution was added. The solution was shaken for 30 min and centrifuged for 10 min at 3500 rpm. The supernatant solution was decanted and 10 ml of the solution was transferred into a 25 ml volumetric flask. 2 ml of brucine reagent was added followed by quick addition of 2.5 ml of concentrated H_2SO_4. The solution was mixed for about 30 sec and

Table 1. Physicochemical characteristics of soil samples from sampling sites.

Sampling site	Code	pH	Total organic carbon (%)	Bulk density (g/cm^3)	Sand (%)	Silt (%)	Clay (%)
Enu Gada	EG	6.8 ± 0.1^c	0.35 ± 0.01^a	2.80 ± 0.26^a	98.2^d	0.1^a	1.7^a
Ago Ika	AI	6.6 ± 0.1^b	2.03 ± 0.02^e	1.72 ± 0.01^a	96.2^a	1.0^b	2.8^b
Adedotun	AD	7.1 ± 0.1^e	0.82 ± 0.01^b	1.68 ± 0.03^a	94.2^b	3.7^c	2.1^b
Ago Odo	AO	6.5 ± 0.1^a	1.72 ± 0.01^d	1.73 ± 0.01^a	97.2^c	1.0^b	1.8^a
Ita Ika	II	7.0 ± 0.1^d	1.51 ± 0.01^c	1.75 ± 0.02^a	97.2d	1.0^a	1.8^a

Mean values with different superscripts (a,b,c,d,e) in column are significantly different at p = 0.05; % Sand, % Silt and % Clay = mean values.

allowed to stand for 5 min. The volumetric flask was immersed in cold water for about 5 min. The solution was made up to the mark with distilled water and the absorbance was measured on a UV-VIS Spectrophotometer at 470 nm.

Nitrite-nitrogen in sediments was determined by spectrophotometry using sulphanilic acid and N-1-naphthyl-ethylene-diamine dihydrochloride as described by Hatton and Pickering (Hatton and Pickering, 1991). The process involves diazotisation of sulphanilic acid by nitrite with subsequent coupling to N-1-naphthyl-ethylene-diamine dihydrochloride to give a red colour solution. 30 ml of 0.2 M KCl solution was added to 1 g of the prepared sediment measured into a beaker. The mixture was centrifuged at 3500 rpm for 10 min. The solution was filtered using 0.45 µm membrane filters. 15 ml of the filtrate was treated with 0.5 ml of 1% sulphanilic acid solution (in 10% HCl). 0.5 ml of 0.1% N-1-naphthyl-ethylene-diamine dihydrochloride solution was added. The solution turned red and the red solution was shaken and allowed to stand for 20 min. Absorbance was read on UV-Visible spectrophotometer at 513 nm.

Ammonium-nitrogen in sediments was determined by colorimetry (Okalebo et al., 2002). 1 g of sediments was weighed and 10 ml of 0.5 M K_2SO_4 solution was added. The bottle was stoppered and the solution was shaken for 1 h. The solution was filtered through 0.45 µm membrane filters and 0.2 ml of the sample extract was measured into a test tube. 5 ml of sodium salicylate – sodium citrate – sodium tartrate - sodium nitroprusside solution was added into the test tube. The test tube was well shaken. 5 ml of sodium hydroxide – sodium hypochlorite solution was added to the test tube. The solution was allowed to stand for 1 h. A blue colour was observed. Absorbance was read on a UV-VIS Spectrophotometer at 655 nm.

Statistical analysis

Data were analysed using descriptive analysis and one-way ANOVA at $\alpha_{0.05}$. Mean comparison was conducted using Duncan's multiple range test. The relations among measured parameter were investigated using Pearson correlation.

RESULTS AND DISCUSSION

Physicochemical properties of the sediments

The physicochemical characteristics of the sediments sampled from each of the sampling sites are presented in Table 1, showing the average values for pH, percentage organic carbon, and bulk density, respectively. All the sampling sites differ markedly in physicochemical properties of sediments. Generally, physicochemical properties in sediments vary in space and time because of variation in topography, climate, weathering processes, vegetation cover and microbial activities (Paudel and Sah, 2003; Bargali et al*.,* 2018) and several other biotic and abiotic factors (Bargali et al*.,* 2019, Pandey et al*.,* 2024). In the highly dissected landscapes, bioclimatic conditions change rapidly and may vary within short distances resulting in a pronounced heterogeneity in chemical and physical properties (Baumler, 2015; Vibhuti et al., 2020). The pH value of the sediments from all the sampling sites ranged closely from 6.5±0.1 to 7.1±0.1, showing that the sediments sampled from the river were slightly acidic and neutral with a mean pH value of 6.8. The pH values fall within the ideal pH for aquatic organisms, 6.5-8, according to the U.S. EPA standard (EPA, 2012). The pH of sediments of Ogun River indicates a suitable environment for maintaining its water quality and aquatic organisms. The neutral pH profile of the sediments indicates the neutralization capacity that can possibly be demonstrated by bacterial sulphate reduction in sediment (Meier et al*.,* 2004). If there were no neutralization mechanism, the sediments would rapidly be acidified by possible domestic acidic wastes received by the river. The sediments had low percentages of Total Organic Carbon (TOC) ranging from 0.35±0.01 to 2.03±0.01%. The TOC levels were significantly different within the sampling locations (Table1). The total organic carbon contents are similar to what was obtained in Golovita lake (Catianis et al*.,* 2018). The low organic carbon in the sediments may not contribute significantly to the depletion of oxygen, algae growth, and eutrophication in the river. There was a significant correlation between organic carbon content in sediments of Ogun river and total phosphorus (r= -0.750) and nitrogen (r= -0.471), indicating possible eutrophication in the river characterized by excessive algal growth due to increase nutrient availability. The impact of eutrophication has consequences for aquatic organism in the river. The low organic carbon content observed in this study is similar to the report of Yoswaty et al. (2021) in which low organic carbon levels and high sand fraction were observed in Bengkalic island sediments (Yoswaty et al*.,* 2021). The organic carbon in the sediments is as a

Table 2. Concentrations of total phosphorus (µg P g^{-1}) and total nitrogen (µg Ng^{-1}) for all. sampling sites.

Sampling site	Code	Total phosphorus	Total nitrogen	N:P ratio
Enu Gada	EG	1293 ± 16^{b}	1960 ± 16^{e}	1.52
Ago Ika	AI	820 ± 5^{a}	1124 ± 13^{b}	1.37
Adedotun	AD	2740 ± 21^{c}	1400 ± 29^{c}	0.51
Ago Odo	AO	1138 ± 10^{b}	1677 ± 17^{d}	1.47
Ita Ika	II	902 ± 7^{ab}	840 ± 10^{a}	0.93

Mean values with different superscripts (a,b,c,d,e) in column are significantly different at p = 0.05.

result of animal and plant decomposition (Avramidis and Bekiari, 2021). The sediments obtained from all the sampling sites are sandy as indicated in Table 1, with composition ranging from 94.2 to 98.2%. The sand fraction was the most abundant, followed by the clay fraction, and the silt fraction was the least abundant in sediments. High bulk density is associated to sandy sediments owing to its low porosity. The porosity of sandy sediments is known to be low compared to those of silt and clay (Dutilleul et al., 2020). The bulk density of the sediments in Ogun River ranged from 1.68±0.03 to 2.80±0.26 g/cm^3.More compact sandy sediments often have a bulk density of around 1.0 to 2.0 g/cm^3 (Shankar et al., 2021), and this range is around the bulk densities obtained for Ogun River. These high bulk densities are associated to low porosity, as limited pore space in the sediment results to higher mass sediment per unit volume. According to USDA National Resources Conservation Service (NRCS), average bulk density is at 1.33 g/cm^3; bulk density less than 1.60 g/cm^3 is suitable for the growth of plants while bulk density greater than 1.80 g/cm^3 limits the growth of plants (NRCS, 2008). The study revealed that aquatic plant growth would be inhibited at Enu Gada as the bulk density of the sediments greatly exceeded the limit stated by USDA-NRCS.

Total phosphorus and total nitrogen

The concentrations of total phosphorus and total nitrogen are shown in Table 2. The total phosphorus and total nitrogen concentrations in the sediments ranged from 820±5 to 2740±21 µg P g^{-1} and 840±10 to 1960±16µg N g^{-1}, respectively, with a mean concentration of 1380±780 µg P g^{-1} and 1400±441 µg N g^{-1}. This is an indication that the sediment was rich in nitrogen relative to phosphorus, and this is similar to the report on sediment of Lake Tahoe in California that possess enormous pools of nitrogen compared to phosphorus level (Beutel and Horne, 2018). The mean total phosphorus concentration in Ogun River is more than that of intertidal zone (88.80-227.56 µg P g^{-1}) in the Yellow River Delta (Liang et al., 2021), and the total nitrogen is less than that of Baiyangdian Lake in China, containing 1809 µg N g^{-1} (Zhu et al., 2019).Utah lake was identified as a phosphorus sink with a concentration range of 600-800 µg/g (Abu-Hmeidan et al., 2018). Even with Utah Lake acting as a large phosphorus sink, the total phosphorus levels in Ogun River sediment were higher than levels of Utah lake sediment. The concentrations of total phosphorus exceeded the lowest permissible level of 600 µg/g (Persaud et al., 1993). High total nitrogen and phosphorus contents might result from anthropogenic activities including washing, bathing, and dumping of refuse particularly from the abattoir situated close to the river. There was an evident growth of algae on the surface of Ogun River, and it was very high at locations EG and AD, with AD locations having the highest spread of algal growth. This justifies the levels of total phosphorus shown in Table 2. The total concentration of phosphorus at location AD (2740±21 µg/g/) surpasses the concentration limit of 2000 µg/g, being the severe effect level of phosphorus that will significantly affect the use of sediment by benthic organism (Persaud et al., 1993). The high concentration of total phosphorus at location AD was due to the refuse dumpsite, which was highly dense at the location compared to other sampling locations. Sediments at sampling location EG were enriched with total nitrogen of 1960±16 µg/g, which exceeded the lowest acceptable limit of 550 µg/g (Persaud et al., 1993). This lowest acceptable limit indicates a level of sediment contamination that can be tolerated by the majority of aquatic organisms, and the level of pollution is regarded as considerably mild. None of the sampling locations had a total nitrogen concentration that was above what is considered as severe effect level (4800 µg/g) by Ontario Sediment Guideline (Persaud et al., 1993). At severe effect level of nitrogen, the sediments are regarded to be highly polluted with excess of nitrogen that could pose threat to the health of aquatic organisms. High concentration of total nitrogen signifies a high level of nitrification, which is one of the processes contributing to the fixed nitrogen pool of the river. Nitrification is an aerobic process occurring in sediments by which ammonia oxidizes to nitrite and then to nitrate (Supajaruwong et al., 2021; Wu et al., 2023). High levels of total phosphorus and total

Table 3. Concentrations of inorganic and organic phosphorus (μg P g^{-1}) forms for all sampling sites.

Code	Exchangeable phosphorus	Aluminium-bound phosphorus	Iron-bound phosphorus	Reductant phosphorus	Calcium-bound phosphorus	Inorganic phosphorus	Organic phosphorus
EG	8.8 ± 0.5^c	42.9 ± 3.6^d	162 ± 5^c	71.8 ± 3.5^b	360 ± 9^a	646	647
AI	9.7 ± 0.6^d	ND	115 ± 4^a	97.1 ± 7.5^d	544 ± 12^c	767	53
AD	0.6 ± 0.3^a	12.1 ± 2.0^a	135 ± 5^b	103 ± 6^e	909 ± 15^e	1160	1580
AO	ND	28.4 ± 2.6^b	137 ± 5^d	53.8 ± 3.5^a	511 ± 10^b	730	408
II	3.4 ± 0.3^b	15.8 ± 3.0^c	119 ± 5^a	84.4 ± 3.6^c	658 ± 8^d	880	22

ND: Not Detected; Mean values with different superscripts (a,b,c,d,e) in the column are significantly different at p = 0.05.

Table 4. Percentage of Inorganic Phosphorus and Organic Phosphorus for all sampling sites.

Sampling site	IP/TP (%)	OP/TP (%)
EG	50	50
AI	93.5	6.5
AD	42.3	57.7
AO	64.1	35.9
II	97.6	2.4

IP: Inorganic phosphorus; OP: Organic phosphorus; TP: Total phosphorus.

nitrogen above permissible limits in the sediments can have adverse impact on the Ogun River by the growth of algae and the depletion of dissolved oxygen as phosphorus and nitrogen are released from surficial sediment to overlaying water. Internal loading of phosphorus and nitrogen from the sediment to overlaying water will not only impair the quality of the Ogun River but delay water quality recovery in the river. The nitrogen to phosphorus ratio often provides insights into nutrient limitation for assessing sediment quality and understanding the potential for eutrophication in water bodies (Savic et al., 2022). The N: P ratios for all the sampling locations follow the trend EG > AO > AI > II > AD (Table 2).

Inorganic phosphorus

Inorganic phosphorus concentration was determined by adding the values of inorganic phosphorus fractions obtained via sequential extraction. The inorganic phosphorus fractions were exchangeable phosphorus, aluminium-bound phosphorus, iron-bound phosphorus, reductant phosphorus and calcium-bound phosphorus. The concentrations of the inorganic phosphorus fractions at the five sampling sites are presented in Table 3. The total concentration of inorganic phosphorus in the sediments of the river range from 646-1160 μg P g^{-1}, and the mean inorganic phosphorus concentration is 837±199μg P g^{-1}. These high values of inorganic phosphorus imply that the sediments in the river have phosphorus adsorbed on aluminium, iron, and calcium (Tang et al., 2014).The lowest and highest concentrations of inorganic phosphorus were in the sediments from EG and AD locations, respectively. Inorganic phosphorus (IP) makes up 60.7% of the total phosphorus (TP) concentration in the sediments, and the IP/TP ratio were in the range of 42.3-97.6% for all the sampling locations (Table 4). Inorganic phosphorus being the major portion of total phosphorus in the sediments implies that organic phosphorus in the sediments has been highly mineralized (Onianwa et al., 2013). Organic Phosphorus/Total Phosphorus ratios were in the range of 2.4-57.7% (Table 4). Calcium-bound phosphorus (Ca-P) has the highest concentrations while exchangeable phosphorus (E-P) has the lowest concentrations in all of the sampling sites. The Ca-P concentrations ranged from 360±9 to 909±15 μg/g, which accounted for 55.7-74.8% of total phosphorus (TP) (Figure 3). The highest Ca-P concentration observed in this study is similar to the report of Onianwa et al. (2013). This is an indication that Ogun River has most of its inorganic phosphorus in an apatitic state. The dominance of Ca-P is traceable to calcium apatite, which is one of the major forms of inorganic phosphorus in natural water. The Ca-P/IP ratios in Ogun River sediments are about the proportion of Ca-P (67.0-92.0% of TP) reported for the northern gulf of Mexico sediments (Adhikari et al., 2015).

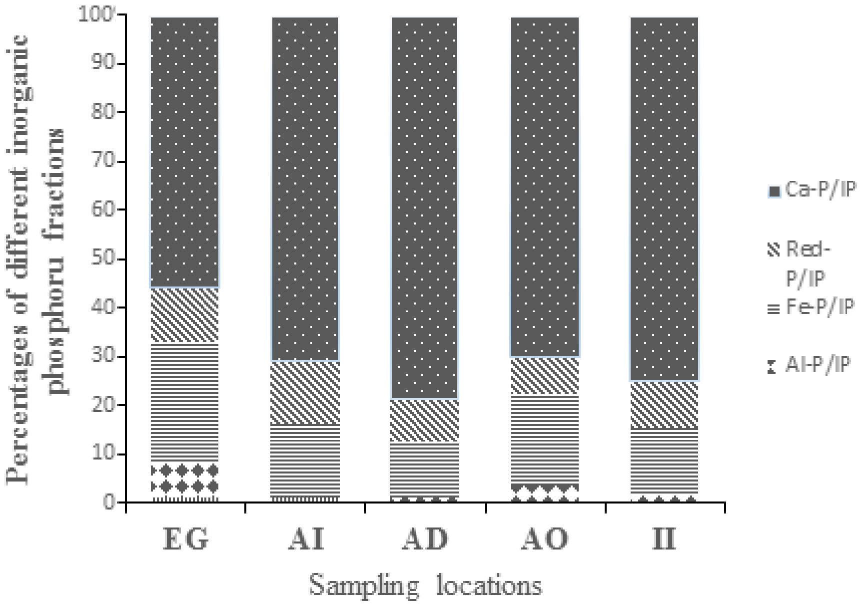

Figure 3. Distribution of percentages of different inorganic phosphorus fractions for all sampling locations in Ogun River sediments. (E-P/IP: Exchangeable Phosphorus/Inorganic Phosphorus; Al-P/IP: Aluminium-bound Phosphorus/Inorganic Phosphorus; Fe-P/IP: Iron-bound Phosphorus/Inorganic Phosphorus; Red-P/IP: Reductant Phosphorus/Inorganic Phosphorus; Ca-P/IP: Calcium-bound Phosphorus/Inorganic Phosphorus)

Table 5. Comparison of concentrations of phosphorus speciation in literature and this study.

Study area	pH	Total Phosphorus (µg P g^{-1})	Inorganic Phosphorus (µg P g^{-1})	Organic Phosphorus (µg P g^{-1})	References
Ogun River, Nigeria	6.5 - 7.1	820 - 1293	646 - 1160	22 - 1580	This study
West Holland River, Canada	7.2	1100 - 1600	770 - 1240	-	Audette et al. (2018)
Lake Simcoe, Canada	6.9	900 - 1100	-	-	Audette et al. (2018)
Dongping Lake, China	-	425.9 - 729.6	270.6 - 513.0	113.9 - 229.5	Chen et al. (2014)
Ona River, Nigeria	-	316 - 466	107-314	-	Onianwa et al. (2013)
Kudeti River, Nigeria	-	647 - 754	406 - 606	-	Onianwa et al. (2013)
Ogunpa River, Nigeria	-	355 - 1068	264 - 595	-	Onianwa et al. (2013)
Utah Lake, USA	-	600 - 800	-	-	Abu-Hmeidan et al. (2018)
Intertidal Zone of Yellow River Delta, China	-	88.80 - 227.56	84.21 - 224.26	-	Liang et al. (2021)
Northern Gulf of Mexico, USA	-	474 - 1035	-	-	Adhikari et al. (2015)

The total phosphorus (TP) in northern gulf of Mexico sediment was reported to be 474-1035 µg P g^{-1} compare to 820 – 1293 µg P g^{-1} observed in Ogun River sediments (Table 5). The inorganic phosphorus in Ogun River was mainly composed of 78.7-89.7% of the total phosphorus. These proportions of inorganic phosphorus were slightly above 74.2% of phosphorus observed in Dongping Lake in China, where industrial waste water and domestic sewage discharges were major sources of phosphorus input (Chen et al., 2014). The discharges contributed 270.6 - 513.0 and 113.9 - 229.5 µg/g of inorganic and organic phosphorus, respectively to the sediments of Dongping Lake (Table 5).

Organic phosphorus

Organic phosphorus concentrations of the sampling sites

Table 6. Concentration of inorganic nitrogen fraction and proportion of in total nitrogen for all sampling sites.

Code	Nitrate-Nitrogen (μg N g^{-1})	Nitrite-Nitrogen	Ammonium-Nitrogen	Inorganic Nitrogen	Inorganic Nitrogen/Total Nitrogen (%)
EG	302 ± 4^b	7.10 ± 0.10^a	67.0 ± 5.0^b	376	19.2
AI	509 ± 6^d	132 ± 3^d	41.0 ± 3.0^a	682	60.7
AD	351 ± 9^c	69.0 ± 4.0^b	261 ± 11^e	681	48.6
AO	265 ± 2^a	206 ± 8^e	38.0 ± 6.0^a	509	30.4
II	531 ± 11^e	96.0 ± 6.1^c	79.0 ± 4.1^c	706	84.0

Mean values with different superscripts (a,b,c,d,e) in column are significantly different at p = 0.05.

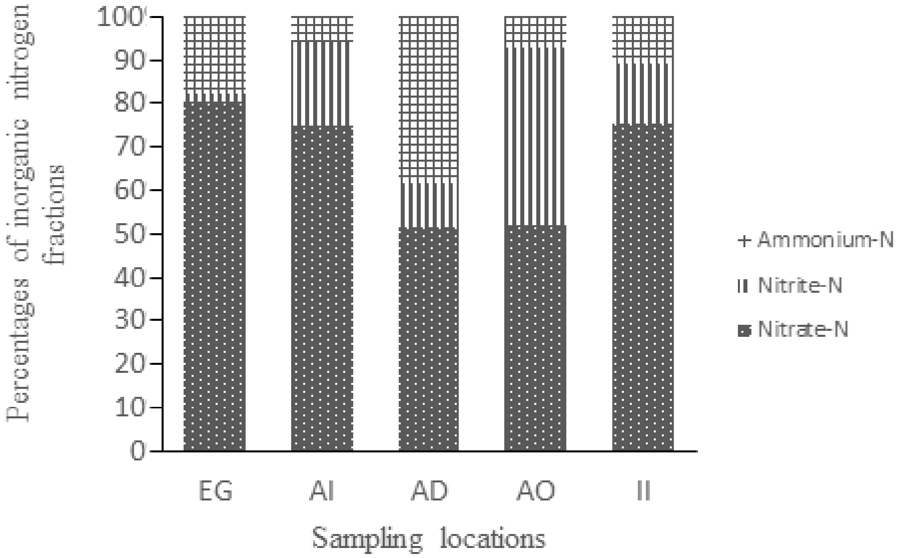

Figure 4. Distribution of percentages of different inorganic nitrogen fractions for all sampling locations in Ogun River sediments.

ranged from 22 to 1580 μg P g^{-1} (Table 3). The mean organic phosphorus concentration is 542 μg P g^{-1} and it forms 39.3% of total phosphorus concentration in the sediments of the river. The high concentration of organic phosphorus (1580 μg/g) for location AD suggests that there is a significant amount of organic matter, which can contribute to low dissolved oxygen and bacteria presence in the river (Ojekunle et al., 2014). Organic phosphorus can be retained in sediments due to a restriction in the movement of exchangeable phosphorus by bacteria and algae in an aquatic ecosystem, leading to settling and degradation of phosphorus in sediments (Audette et al., 2018).

Nitrogen speciation

Nitrate-nitrogen, nitrite-nitrogen, and ammonium-nitrogen are inorganic forms of nitrogen that make up about 42.0% of the total nitrogen concentration of the sediments. Inorganic nitrogen is determined by the sum of nitrate-nitrogen, nitrite-nitrogen, and ammonium-nitrogen. The order of concentrations of the inorganic nitrogen forms in the sediments was ammonium-nitrogen < nitrite-nitrogen < nitrate-nitrogen for AI, AO and II sampling locations, while nitrite-nitrogen < ammonium-nitrogen < nitrate-nitrogen (Table 6 and Figure 4) for EG and AD sampling locations. The range of inorganic nitrogen concentration was 376-706 μg/g (Table 6). The percentage of inorganic nitrogen in total nitrogen for each of the sampling sites is as displayed in Figure 4, with location II having the highest percentage of 84.0% and EG having the lowest percentage of 19.2%. The concentration of nitrate-nitrogen, nitrite-nitrogen, and ammonium-nitrogen ranged from 265±2 to 531±11, 7.10±0.10 to 206±8, and 38.0±6.0 to 261±11 μg/g, respectively. The concentration of nitrate-nitrogen which was the highest fraction of nitrogen in this study is less compared to the concentration of 13-

Table 7. Comparison of concentrations of nitrogen speciation in literature and this study.

Study area	pH	Total nitrogen (µg/g)	NO_3^--N (µg/g)	NO_2^--N (µg/g)	NH_4^+-N (µg/g)	References
Ogun River	6.5-7.1	560-1677	265-531	7-206	38-261	This study
Baiyangdian Lake, China		445-3499	13-24		48-235	Zhu et al. (2019)

Table 8. Correlation coefficient (r) for the pairs of sediment quality characteristics of Ogun River.

Correlation	Total P	E-P	Al-P	Fe-P	Red-P	Ca-P	Total N	NO_3^--N	NO_2^--N	NH_4^+-N	Organic C	Sand	Silt	Clay
Total P	1.000										-0.750*	0.195	0.314	-0.220
E-P	-0.473*	1.000									0.122	-0.157	-0.332	0.441
Al-P	0.448*	-0.284	1.000								-0.570*	0.779**	-0.503*	-0.857**
Fe-P	0.754**	-0.262	0.799**	1.000							-0.679**	0.428	-0.082	-0.488*
Red-P	0.082	0.294	-0.746**	-0.366	1.000						-0.007	-0.547*	0.505*	0.769**
Ca-P	0.093	-0.204	-0.681**	-0.486*	0.779*	1.000					0.089	-0.352	0.494*	0.439
Total N	0.583**	-0.183	0.706**	0.843**	-0.504*	-0.693**	1.000				-0.471*	0.195	0.022	-0.329
NO_3^--N	-0.466*	0.495*	-0.585*	-0.672**	0.577*	0.586*	-0.879**	1.000			0.216	-0.50	-0.202	0.330
NO_2^--N	-0.584*	-0.299	-0.309	-0.509*	-0.305	-0.018	-0.269	-0.118	1.000			-0.403	0.254	0.110
NH_4^+-N	0.550*	-0.129	-0.068	0.161	0.584*	0.642**	-0.231	0.378	-0.658**	1.000			0.101	-0.018

E-P: Exchangeable Phosphorus; Al-P: Aluminium-bound Phosphorus; Fe-P: Iron-bound Phosphorus; Red-P: Reductant Phosphorus; Ca-P: Calcium-bound Phosphorus. * and ** Correlation is significant at the 0.05 and 0.01 level respectively.

24 µg/g that was found in Baiyangdian Lake, China (Zhu et al., 2019) (Table 7). The concentration of ammonium-nitrogen are higher than the concentrations reported for sediments of Ebrie Lagoon situated around Akouedo landfill in Cote d'Ivoire (N'Goran et al., 2019).Several factors contribute to high nitrate-nitrogen fractions in sediments including human activities and soil properties. The highest proportion of nitrate-nitrogen in Ogun River sediments is associated with minimal agricultural land use of the bank of the river. Low ammonium-nitrogen concentrations could be due to the release of ammonium-nitrogen from the sediments to overlaying water, volatilization of ammonia, and nitrification of ammonium in the sediments (Chen et al., 2020; Hou et al., 2013). Ammonium-nitrogen is produced in sediments by ammonification of organic matter in the sediments (N'Goran et al., 2019). All the correlation coefficients are shown in Table 8. Ammonium-nitrogen and nitrite-nitrogen showed significant correlations with organic matter with r = -0.617 and r = 0.860, respectively. This suggests that these fractions of nitrogen concentrated in organic matter (Avramidis and Bekiari, 2021).

Conclusion

The concentrations and speciation of phosphorus and nitrogen were examined. High concentrations of total phosphorus and total nitrogen in the sediments revealed that the Ogun River was stressed by phosphorus and nitrogen loads. The loads were of anthropogenic sources traceable to residential homes, farmlands, and an abattoir found close to the river, and is responsible for the growth of algae on the river surface causing affected regions in the river to be anoxic and leading to eutrophication. The inorganic phosphorus was mostly apatitic and constituted a larger percentage of total phosphorus than organic phosphorus in the sediments. The proportion of phosphorus and nitrogen fractions varied among the sampling locations with highest abundance of Calcium-P and nitrate-N. This study on the inorganic phosphorus and nitrogen largely reflects the current status of the Ogun River with respect to nutrient loads that enhances algae growth in the overlaying water. Further work exploring the speciation of organic phosphorus is required.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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