Ethiopian Journal of Environmental Studies & Management 18(3): 272 – 284, 2025
ISSN:1998-0507 doi: https://ejesm.org/doi/v18i3.1
Submitted: January 28, 2025 Accepted: July 18, 2025

PHOSPHATE SPECIATION IN SEDIMENTS OF CAPE TOWN WATERWAYS- AN INSIGHT INTO ANTHROPOGENIC INPUTS

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Abstract
This study investigated the levels and distribution of phosphorus in sediments along the Swart River, Cape Town, and identified the pollution trends along the river. The harmonised protocol developed by the Standards Measurements and Testing Program of the European Commission was used to extract the different fractions of phosphorus in the sediments. The phosphorus levels in the extracts were measured with a spectrophotometer operated at 880 nm. The total phosphorus levels ranged from 797.7 – 6877.1 ug/g with the inorganic phosphorus being the principal constituent of the total extractable phosphorus in most of the sampling sites surveyed. The speciation results showed that HCl-P was the dominant form of inorganic phosphorus, while NaOH-P was the minor non-apatitic constituent in the sediments. Most of the inorganic phosphorus was fixed in the apatitic form. The total phosphorus levels in the sediments are indicative of high pollution along the Swart River, with the deposition majorly from anthropogenic sources (industrial activities, sewage/effluent discharges, and agricultural run-off). Eutrophication problems could arise at some of the sampling points in the future if the prevailing conditions are not mitigated.

Keywords: *Phosphorus, Sediments, Speciation, Swart River, Cape Town*

Introduction

Phosphorus is an essential macronutrient required by all living organisms due to its involvement in many biochemical processes ((Bains *et al*., 2019). It is considered a limiting nutrient for primary production in terrestrial and aquatic ecosystems (Elser, 2012). The occurrence of excess phosphorus in fresh surface waters is undesirable as it accelerates eutrophication (Prüter *et al*., 2020; Heathwaite and Sharpley, 1999). This phenomenon dramatically reduces the quality of water and adversely impacts aquatic ecosystems. Thus, the leaching of nutrients such as phosphorus from anthropogenic and natural sources must be carefully monitored and controlled where possible (Li *et al*., 2020; Paytan and McLaughlin, 2007).

Phosphorus in surface water bodies results from several anthropogenic activities such as domestic waste dumping, industrial discharges, agricultural waste discharges, and storm run-offs (McDonald *et al*., 2019; Li *et al*., 2018; Owa, 2014). Phosphorus speciation in water systems occurs in various chemical forms, establishing a dynamic equilibrium between the dissolved and particulate state ($P_{dissolved} \leftrightarrow P_{particulate}$) (Spivakov *et al*., 1999). The sediment of water bodies acts as a sink for the entrapment of phosphorus (Acharya *et al*., 2016) and usually consists of embedded organic and inorganic forms (Li *et al*., 2020). However, the entrapped phosphorus in sediments may further fuel the eutrophication process or increase phosphorus levels in water bodies as changes in the water chemistry can mobilise the sequestered phosphorus ((Bastami *et al*., 2018; Mainstone and Parr, 2002; Bradford and Peters, 1987). The phosphorus enrichment is favoured under anaerobic conditions and substantial pH increases in water systems ((Mao *et al*., 2021; Spivakov *et al*., 1999). Generally, agitations in water bodies leading to the re-suspension of sediments and chemical conditions such as pH, dissolved oxygen, surface charges, and variation in iron concentrations can accelerate the deposition of phosphorus into the overlying water column (Liu *et al*., 2016; Zhou *et al*., 2016; Meng *et al*., 2014; Coelho *et al*., 2004).

Phosphorus mainly occurs in forms such as the loosely-sorbed-P, Fe-bound-P, authigenic/biogenic-P, detrital apatite-P and organic-P (Bastami *et al*., 2018; Ruttenberg, 1992). The nature of these species (i.e. biological and chemical activity) determines whether the phosphorus is reactive or refractory. This has crucial implications for mobility and bioavailability (Dan *et al*., 2020; Bastami *et al*., 2018). The levels of the different fractionated phosphorus species in water sediments play a critical role in the overall health of water bodies (Yang *et al*., 2019; Li *et al*., 2018). Due to the relationship between sediment phosphorus concentration and surface water quality, it has become pertinent to monitor various phosphorus species, especially in water bodies where such information is scarce.

This study represents a general perspective of speciated phosphorus forms in sediments of the Swart River in Cape Town, South Africa. Major anthropogenic points and contributors along the course of the river were identified to evaluate phosphorus distribution and thus the contribution of human activities and other sources that may increase the phosphorus in river sediments. The modified William's protocol adopted by the Standards, Measurements and Testing Programme (formerly BCR) of the European Commission to extract phosphorus was utilised in this investigation (Ruban *et al*., 1999). The protocol enabled the extraction of total phosphorus (TP), organic phosphorus (OP), inorganic phosphorus (IP), NaOH phosphorus – NaOH-P (P bound to Al, Fe and Mn oxides and hydroxides) and HCl phosphorus – HCl-P (P associated with Ca) from the river sediments (Ruban *et al*., 2001). The validation of the extraction process was done by using certified reference material (BCR-684). The results obtained for the ten replicates showed no significant difference ($p<0.005$) from the values in the certificate of analysis.

Study Area

The City of Cape Town is the provincial capital of Western Cape in

South Africa, and also the legislative capital of the country (34˚ 0ˈ18˚ 30′E). The Atlantic Ocean flanks the western and southern parts of the city, while the eastern region is flanked by the Indian Ocean (Oputu and Akharame, 2022). One of the rivers that flows through the city and empties into the Atlantic Ocean is the Swart River. The Swart River receives water from Langa (source Epping) and Bridgetown (source Heideveld) and passes through the Athlone water treatment plant, Mowbray golf course, converges with the Liesbeek River and continues its path towards the Atlantic Ocean. Along the route of the river, it is exposed to various forms of pollution such as the indiscriminate dumping of refuse from informal settlements (Langa), industrial waste (Bridgetown), fertiliser run-off (Mowbray golf course), as well as, effluent discharges from Athlone wastewater treatment plant. The location of the sampling points is represented in Figure 1.

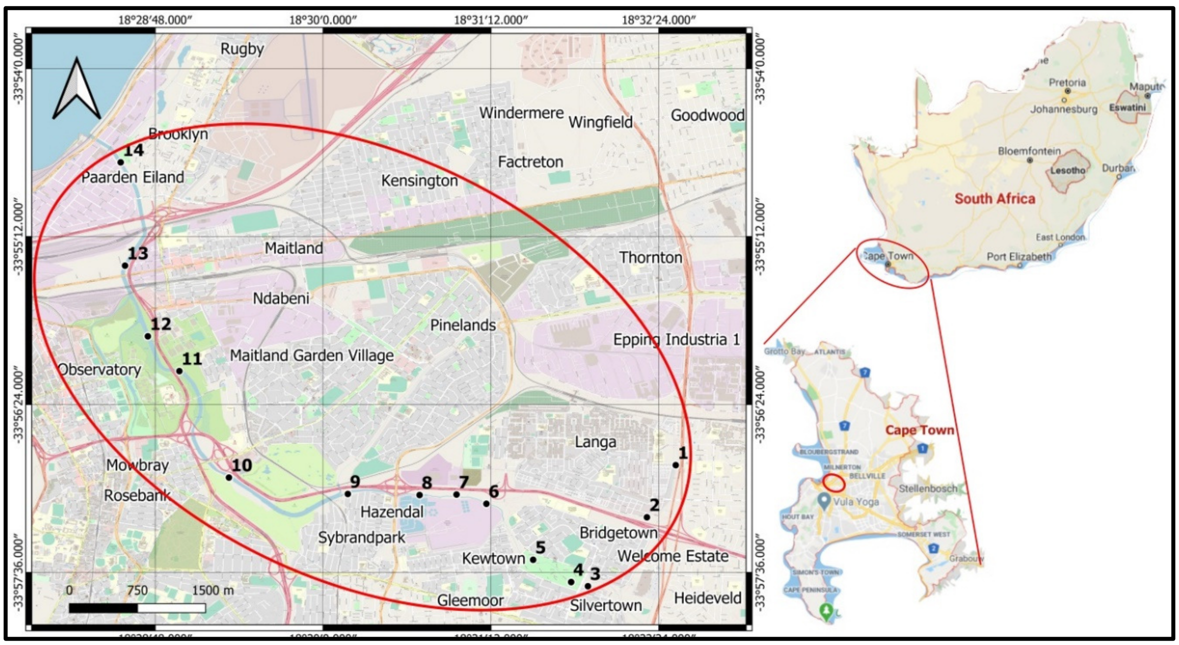

Fig. 1: Map showing sampling locations (insert: Map of Cape Town, South Africa)

Sample Collection and Extraction

A description of the 14 sampling points in the Swart River is presented in Table 1. The river sediment was collected from each of the sampling points with a PVC grab sampler (10 cm diameter x 5 cm). The different sediment scoops from each location were pooled together to cater to the within-site variability. This was followed by storing them in pre-labelled clean zip-lock bags. Thereafter, the sediments were transported to the laboratory and air-dried at room temperature, and dried sediments were sieved (0.15 mm sieve). Phosphate-free soap was utilised for the washing of all glassware used in the analysis before rinsing with copious amounts of distilled water (Onianwa *et al*., 2013).

Table 1: Site description

Sampling point	Location number	Site activity (human)	Site activity (industry)	Sediment type
Langa	1	High	Low	Sandy
	2	High	Low	Sandy
Bridgetown	3	Low	High	Clay
	4	Low	High	Clay
	5	Low	Low	Sandy
	6	Low	High	Sandy
Athlone WWTP and conjunctions	7	Low	High	Sandy/clay
	8	Moderate	High	Sandy
	9	Moderate	High	Sandy/clay
	10	High	Low	Sandy
Mowbray golf course	11	High	Low	Sandy/clay
	12	High	Low	Clay
Along Swart River toward the ocean	13	Low	Moderate	Sandy
	14	Low	Moderate	Sandy

The extraction of the different fractions of phosphorus in the sediments was done using the harmonised protocol developed by the Standards Measurements and Testing Program of the European Commission, SMT protocol (Ruban et al., 2001). The fractions of phosphorus extracted using the protocol include TP, OP, IP, NaOH-P, and HCl-P. The extraction process involves contacting the sediment with the appropriate extractant, shaking for a specified period, and separating the extractant and sediment by centrifuging the mixture (Figure 2). Briefly, the sediment (0.2 g) was calcined at 450˚C for three hours for TP extraction. The residue was then mixed with 20 mL of 3.5 M HCl and shaken for 16 h. TP was obtained from the extract. For organic and inorganic extraction of phosphorus, the sediment (0.2 g) was mixed with 20 mL of 1 M HCl and shaken for 16 h and IP was obtained from the extract. For OP, the residue obtained from the extract was washed (saturated NaCl solution), dried (80°C), and then calcined (450°C for 3 h). After that, the residue was mixed with 20 mL of 1 M HCl and shaken for 16 h and the OP fraction was obtained from the extract. For NaOH-P, the sediment (0.2 g) was mixed with 20 mL of 1 M NaOH and shaken for 16 h. The extract was mixed with 3.5 M HCl (4 mL) and then left to stand for 16 h. The NaOH-P was obtained from the extract. For HCl-P, the sediment (0.2 g) was mixed with 20 mL of 1 M NaOH and shaken for 16 h. The residue obtained from the extract was washed with NaCl, then mixed with 1 M HCl (20 mL) and shaken for 16 h. HCl-phosphate was obtained from the extract.

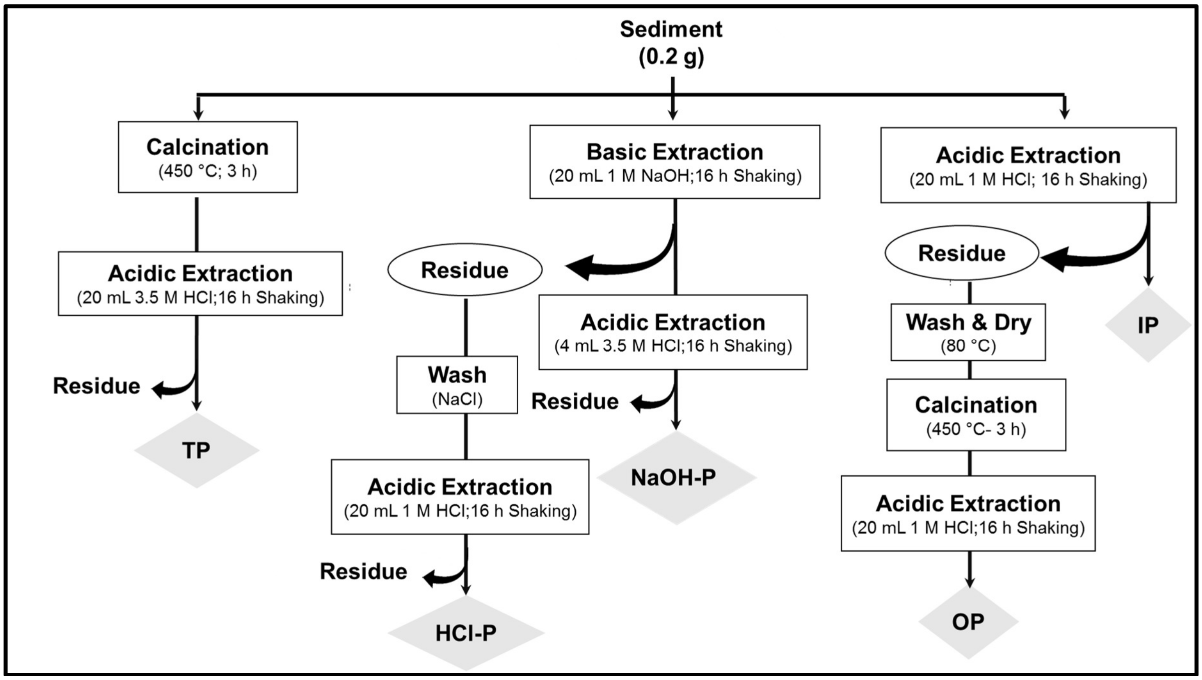

Fig. 2: Various extraction methods of phosphorus species

Phosphorus Analysis

To analyse the phosphorus content of the sediments, the Murphy and Riley Molybdenum Blue reagent was employed (Kovar and Pierzynski, 2009; Ruban et al., 2001; Murphy and Riley, 1962). 2 mL aliquots of supernatants were transferred into a 50 mL volumetric flask, and the pH of all samples adjusted to neutral, using 5 M NaOH. *P*-nitrophenol was used as an indicator (colour change from yellow to colourless), and 10 mL of the mixed reagent was added to develop a blue colour. Each solution was mixed, and the volume was brought to the mark with distilled water. The colour developed within 10 min, and the absorbance was measured immediately using a Cary UV-Vis spectrophotometer (Agilent Technologies, Santa Clara, USA) operated at 880 nm.

Quality Control and Quality Assurance

The validity of the P extraction and analysis method was established by assaying the certified reference material – BCR-684 purchased from Sigma Aldrich (South Africa). The extraction method and the results obtained from the analysis were compared statistically. A t-test was done on the results at a 95% confidential level to ascertain the significant difference between the certified results and the results obtained from the analysis.

Results and Discussion

Quality Control and Quality Assurance Result

The results obtained from the analysis and the BCR-certified results are presented in Table 2. The t-test report showed that all the t-critical values were less than the values recorded for t-stat (Table 3). Therefore, there is no significant difference between the results from the analysis and the certified results. The methods used for analysis were validated and proven to be reproducible.

Table 2: Table showing experimental results versus BCR-analysis

	NaOH-P in ug/g		HCl-P in ug/g		Inorganic-P in ug/g		Organic-P in ug/g		Total-P in ug/g	
Replicate	**Analysis**	**BCR**	**Analysis**	**BCR**	**Analysis**	**BCR**	**Analysis**	**BCR**	**Analysis**	**BCR**
1	530	576	593	486	1201	1110	168	201	1574	1339
2	625	527	557	569	1001	1126	178	211	1295	1391
3	670	527	575	457	1125	1044	216	190	1522	1423
4	500	568	443	527	1959	1072	211	221	1464	1264
5	482	489	459	528	1059	1161	222	188	1334	1386
6	562	544	429	445	1324	1111	213	193	1398	1314
7	508	582	525	562	1158	1095	226	193	1206	1268
8	549	517	451	515	1025	1125	193	214	1130	1407
9	521	587	601	573	1120	1130	203	221	1295	1382
10	493	588	435	545	1158	1146	236	229	1273	1427

Table 3: The t-test results at 95% confidential level.

P-fractionation	T-critical	T-stat	Comment
NaOH-P	1.761	0.3776	No significant difference between the two means
HCl-P	1.724	0.057	No significant difference between the two means
Inorganic-P	1.761	0.1507	No significant difference between the two means
Organic-P	1.73	0.4554	No significant difference between the two means
Total-P	1.717	0.1116	No significant difference between the two means

Total Phosphorus

Table 4 shows the concentration of TP, OP, IP, NaOH-P, and HCl-P. Total phosphate (TP) varied significantly in all the samples, and this could be attributed to the different sampling points taken along the Swart River. The TP levels in the samples were 1014 - 1186 ug/g (Langa), 797 - 3833 ug/g (Bridgetown), 1645 - 3468 ug/g (Athlone water treatment plant), 2308 - 2475 ug/g (Mowbray golf course), and 1034 - 6877 ug/g (towards the ocean). The TP concentrations obtained from the various sites were primarily influenced by external factors (McDonald *et al*., 2019; Li *et al*., 2018). In Langa, an informal township settlement with inadequate sanitary/toiletry facilities (Philander, 2015), the waterway is used as a dumping site for domestic waste. As such, the anthropogenic activity could account for the high TP. Bridgetown and the Athlone water treatment plant had TP in the range of 797-3833 ug/g and 1645-3468 ug/g, respectively. Both sampling sites are located in industrial areas with minimal human activity, and their waterway is characterised by clayey sediment. Industrial seepage and clayey sediment contribute immensely to TP as the latter absorbs more phosphate than sandy sediments.

Mowbray golf course had TP in the range of 2308 - 2475 ug/g, and the presence of TP may be hugely attributed to the fertiliser residues that were washed off the golf course (Thin *et al*., 2020). Fertilisers are composed of nitrogen, potassium and phosphorus. The phosphorus component contributed immensely to the TP in the samples. Furthermore, the sediment in this sampling point was clayey and hence could retain more phosphorus. Along the Swart River, towards the ocean, the TP ranged from 1034 to 6877 ug/g. This sampling site contained the highest concentration of TP. This could be because the sampling point was at the meeting of the Swart River (Langa with high anthropogenic activity) and the Liesbeek River (high industrial activity). The high TP observed was possibly a combination of the phosphate from both rivers.

Generally, anthropogenic activities contribute to the elevation of phosphorus in sediments and the overlying waters. Typically, anthropogenic phosphorus is deposited in the aquatic environment via industrial activities (phosphoric acid/fertiliser production, metal production/plating), sewage/effluent discharges, agricultural run-off (farms and lawns/golf courses) and run-offs from animal farms (dairy and piggery) (DFFE, 2022). The sampling locations in this study have one of the above anthropogenic activities taking place which may be responsible for the elevated levels of phosphorus recorded in the sediments.

Table 4: Concentrations in ug/g for total-P, inorganic-P, organic-P, NaOH-P, and HCl-P

Site	Total-P	Inorganic-P	Organic-P	NaOH-P	HCl-P
LANGA					
1	1014.7±13.14[b]	553.0±27.52[c]	366.0±26.83[a]	113.5±15.59[a]	1074.0±53.76[de]
2	1186.2±21.82[c]	418.0±31.99[b]	526.0±36.67[c]	222.9±33.76[b]	679.5±37.16[b]
BRIDGETOWN					
3	3833.8±35.09[j]	2583.8±61.76[i]	514.8±24.63[bc]	351.5±35.75[cd]	1924.3±66.43[g]
4	3795.0±39.01[j]	3187.0±65.42[k]	535.2±41.92[c]	212.3±28.56[b]	574.9±28.27[a]
5	797.2±26.48[a]	105.1±11.17[a]	680.9±46.70[d]	506.0±42.00[e]	2233.3±92.53[i]
ATHLONE WATER TREATMENT PLANT AND CONJUNCTION					
6	1882.7±43.57[e]	1222.8±36.78[f]	612.4±22.17[d]	289.1±27.41[c]	800.9±39.70[c]
7	3243.4±48.25[h]	2479.4±66.90[h]	734.1±47.94[e]	378.5±50.26[d]	2097.7±77.94[h]
8	3468.2±38.29[i]	2815.8±34.49[j]	320.4±31.26[a]	609.0±38.04[f]	1320.4±39.94[f]
9	1645.7±28.09[d]	801.7±28.82[d]	507.9±37.47[bc]	112.6±13.16[a]	2128.0±38.98[h]
MOWBRAY GOLF COURSE					
10	2308.6±40.80[f]	843.0±33.14[d]	1413.6±47.94[f]	382.9±41.73[d]	543.2±32.30[a]
11	2475.2±60.99[g]	1915.1±36.99[g]	513.7±40.43[bc]	353.2±36.78[cd]	986.7±55.26[d]
ALONG SWART RIVER TOWARD THE OCEAN					
12	6877.1±70.37[k]	3420.7±44.93[l]	3418.7±88.16[g]	1012.4±73.79[g]	2390.0±73.54[j]
13	1034.8±23.34[b]	404.9±22.32[b]	534.8±49.10[c]	350.0±18.50[cd]	1161.2±37.31[e]
14	1579.6±27.68[d]	1131.5±39.95[e]	442.1±37.23[b]	307.7±34.92[c]	801.0±24.26[c]

Values are means± deviation, Different superscripts in the same column indicate Significant differences at $p < 0.05$ according to the Duncan Multiple Range Test (DMRT).

The phosphorus levels recorded in this study align with those reported in similar studies by various scholars. A comparison of the phosphorus speciation levels recorded in the literature for some rivers, lakes, and coastal sediments and the present study is presented in Table 5. Phosphorus levels reported for oceanic continental sediments range from 248 to 3345 µg P/g (Filippelli, 1997). The Chinese Environmental Dredging Standards stipulate that TP >500 µg P/g in sediment is considered heavily polluted (Liu *et al.*, 1999). Also, the province of Ontario in Canada benchmarked a phosphorus level of 600 µg P/g for marginally clean or unpolluted sediments, while contaminated sediments are > 600 µg/g (Persaud *et al.*, 1993. Currently, there is no background level set by the South African government for assessing phosphorus contamination in sediments. As such, by using the Chinese and Canadian guidelines, it can be inferred that the Swart River is heavily polluted.

Table 5: Levels of phosphorus speciation found in the literature for some river/coastal sediments and the present study

Study area	Total-P (µg P/g)	Inorganic-P (µg P/g)	References
Seto Island Sea, Japan	525 – 1005		Yamada and Kayama (1987)
St. Lawrence estuary, Gulf of St. Laurent	1627		Sundby *et al*. (1992)
Aarhus Bay, Denmark	930 – 1550		Jensen and Thamdrup (1993)
Onondaga Lake, New York, USA	2500 – 3090		Penn and Auer (1997)
Chang Jiang River (Yangtze River), China	170 – 705	135 - 619	Rao and Berner (1997)
Southern and Eastern North Sea	93 – 806		Slomp *et al*. (1998)
Huanghe River, China	600	486	Yue and Ji-Jin (1999)
Huanghe Estuary, China	806	538	
Huanghe Shelf, China	580	512	
Bohai sea, China	322 – 617	252 – 524	
Yellow Sea, China	233 – 512	174 – 481	Liu *et al*. (2004)
Ratones River, Southern Brazil	4650 – 11780		Pagliosa *et al*. (2005)
Tavares River, Souther Brazil	9300 – 14880		
Verissimo River, Southern Brazil	3100 – 6200		
Aririu River, Southern Brazil	20150 – 20460		
Itacorubi River, Southern Brazil	4340 – 21080		
Maruim River, Southern Brazil	2945 – 20150		
Yellow River, China		1187 – 1468	Li and Guo (2006)
Louros River, NW Greece	240 – 620	80 – 380	Katsaounos *et al*. (2007)
Burclar Bay, South Anatolia, Turkey	210 – 206	202 – 205	Aydin *et al*. (2009)
Yangtze River, China	452 – 1140	355 – 956	Hou *et al*. (2009)
NE Mediterranean Sea, Antalya, Turkey	152 – 275	150 – 261	Gunduz et al. (2011)
Poyang Lake, China	688 – 825	582 – 691	Xiang and Zhou (2011)
Alalubosa River, Ibadan, Nigeria	307 – 559	140 – 343	Onianwa *et al*. (2013)
Gege River, Ibadan, Nigeria	1072 – 1240	832 – 943	
Kudeti River, Ibadan, Nigeria	647 – 754	406 – 606	
Ona River, Ibadan, Nigeria	316 – 466	107 – 314	
Ogbere River, Ibadan, Nigeria	249 – 780	230 – 410	
Ogunpa River, Ibadan, Nigeria	355 – 1068	264 – 595	
Onireke River, Ibadan, Nigeria	426 – 647	189 – 603	
Dongping Lake, North China	426 – 730	271 – 513	Chen *et al*. (2014)
Swart River, Cape Town South Africa	798 – 6877	105 – 2816	present study

Inorganic and Organic Phosphorus in Sediment

The IP component ranged from 13% to 84% (Figure 3) of the TP concentrations in the sediment samples. In 9 of the 14 sampled sediments, IP was the major constituent of TP. The high content of IP for almost all the sampling sites indicated a high rate of mineralisation of OP (Onianwa *et al*., 2013). Similarly, the HCl-P component of the IP was the predominant form with percentages ranging from 36% to 84%, while the NaOH-P was in the range of 13% to 53%. The concentration of OP ranged from 320 to 3418 ug/g, with an average of 592 ± 270 ug/g, excluding the highest concentration (3418 ug/g). The OP at site 12 (along Swart River) was at least five times that recorded at any other site. Site 12 is confluent, receiving water from the Liesbeek. The Liesbeek River may have contributed to the phosphorus load in the Swart River, as the highest IP was also recorded at this site. Organic-P was the major component of TP in four sites, namely Lange (site 2; 44%), Bridgetown (site 5; 85%), Mowbray golf course (site 10; 61%) and confluence along Swart River towards the ocean (site 13; 51%).

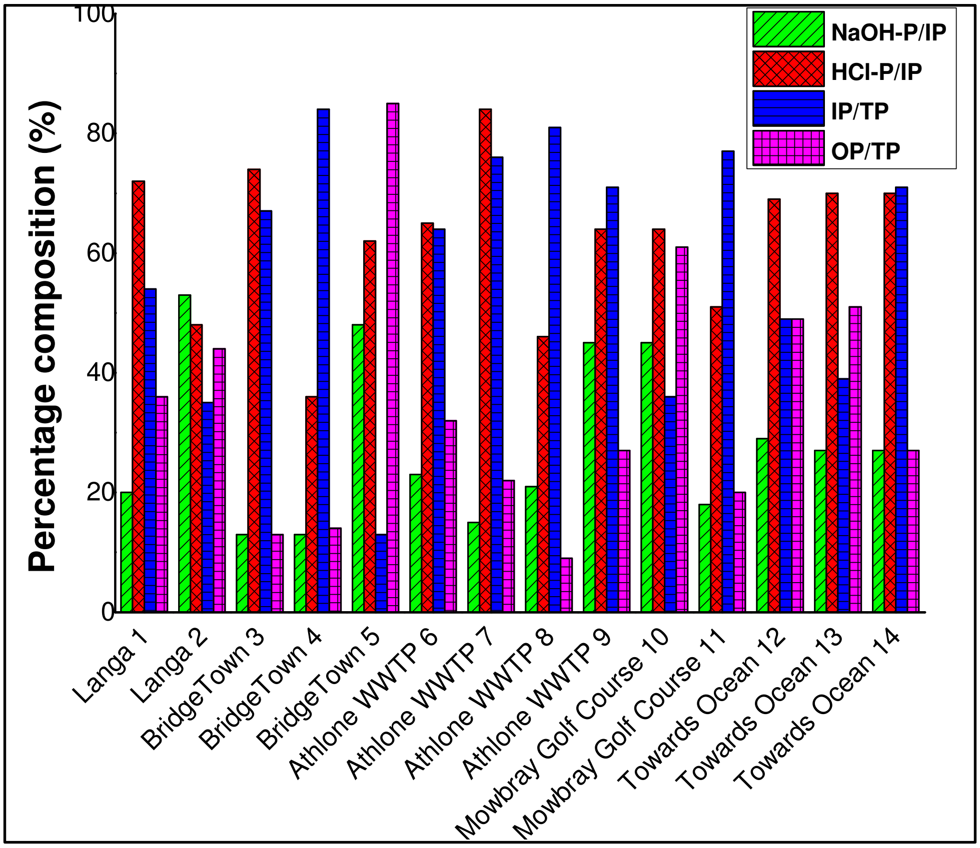

Fig. 3: Percentage composition of the different phosphorus species

Phosphorus Speciation

Inorganic-P was speciated into non-apatite and apatite, those bound to Fe-P and Al-P (NaOH-P) and those bound to Ca-P (HCl-P), respectively. The non-apatite ranged from 112 - 1012 ug/g with a deviation of 322 ± 140 ug/g, while apatite ranged from 574 - 3390 ug/g with an average of 371 ug/g. The speciation results show that HCl-P (Ca-P) was the major component of IP in 12 of the 14 sampling sites. According to studies on the speciation of phosphorus, it has been found that Ca-P and Fe-P can interconvert (Li and Guo, 2006). The extent of the damage done to the river cannot be established without reviewing the sorption capabilities of the sediments from the river. However, if the prevailing conditions persist, in the near future the sediment beds will be a ready source of biologically available phosphorus under favourable conditions. The mobilisation and re-solubility of phosphorus may lead to eutrophication in the aquatic ecosystem; and the resulting anoxic conditions could further increase the re-solubilisation of phosphorus (especially inorganic phosphorus) from the sediment bed (DFFE, 2022).

Conclusion

The speciated forms of phosphorus in the sediments collected along the Swart River were evaluated to identify pollution trends and sources. High levels of the different fractions of phosphorus (TP, OP, IP, HCl-P, and NaOH-P) were recorded. The study revealed that the river was polluted from anthropogenic inputs such as industrial activities, sewage/effluent discharges, and agricultural run-off (use of fertilisers). Speciation results indicate that most of the phosphorus obtained was in the inorganic form, which suggests the mineralisation of OP. The primary composition of IP was bound to Ca-P. The high levels of phosphorus in the sediments of the river can pose a threat if the prevailing conditions are not mitigated. The phosphorus levels may get elevated to severely affect the buffering capacity of the river. Consequently, the water system could experience eutrophication due to high phosphorus levels in the overlying waters.

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