

# Phosphorus Flux from the Pelham Bay Wetlands, Bronx, New York

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## Abstract

Phosphorus (P) released and transported from wetlands affects wetland ecosystems and surrounding water systems. Sediment samples from the Pelham Bay wetlands in the Bronx, NY, have been collected and analyzed. Analysis of the sorption characteristics, P compound identification and P mineralization, showed significant correlations between the sorption maximum ( $S_{max}$ ) and Ox-Fe ( $r = 0.894$ ), ash-TP and HCl-Ca ( $r = 0.94$ ), ash-TP and TOC ( $r = 0.96$ ), as well as TOC vs HCl-Ca ( $r = 0.93$ ). These results indicate that mineral content affects the OP content and the sorption process. P sorption maxima  $S_{max}$  ranged from 70.4 to 1667 mg/Kg, and the equilibrium P concentration  $EPC_0$  ranged from 0.09 to 0.2 mg/L. The high  $S_{max}$  in most of the sites, fairly high  $EPC_0$  and high percentages of Pr (>96%), indicate substantial amounts of P could be bioavailable for plant uptake in the water column under changing hydro-climatic conditions. Dominant P compounds are dihydroxyacetone phosphate (DHAP), phosphoenolpyruvates (PEP), inosine monophosphate (IMP), glucose-6-phosphate (G6P), nucleoside monophosphates (NMP), glycerophosphate (GlyP), polynucleotides (PolyN), and pyrophosphates (Pyrop). The active P pool could maintain substantial P bioavailability and potentially cause eutrophication. The mineralization of 7, 15 and 30 days in laboratory experiments indicates a decrease of  $NaHCO_3$  and HCl, and an increase of NaOH.

## Keywords

Pelham Bay Wetlands, Phosphorus, Organic Phosphorus, Sorption,  $^{31}P$ -NMR, P Mineralization, Nonflooding Incubation

## 1. Introduction

Wetland sediments, soils, and pore waters are sources of Phosphorus (P) and are

a factor influencing the eutrophication of water bodies [1] (Massen and Balla, 2010). Wetlands help improve water quality, and artificial wetlands have been built to treat wastewater and seepage, mill waste and animal waste [2] (Nairn and Mitsch, 2000). Goose Creek, the Hutchinson River and the Lagoon feed the three major marshes in the Pelham Bay Park, which has the largest assemblage of tidal wetlands in New York City, outside of Jamaica Bay. Salt marshes trap pollutants/contaminants, acting as a natural filtration system to reduce contamination in bays and oceans. Wetlands have the ability to absorb fertilizer, reduce erosion, and improve water quality [3] (NYC Parks). P dynamics in wetlands are influenced by sediment physicochemical characteristics, and P release from wetland sediment is critical for wetland restoration; P management in wetlands is important to reduce nutrient loading to downstream water bodies [4] (Dunne *et al.*, 2010). Hydrological factors affect water quality in wetlands, and riparian wetlands help improve the water quality in urban watersheds [2] (Nairn and Mitsch, 2000). Wetland is a unique ecosystem providing water resources and protecting biodiversity. Wetland type plays an important role on the P biogeochemical cycle [5] (Hu *et al.*, 2022).

P is an essential nutrient for plants and animals; however, excessive P can be released into water bodies and wetlands from anthropogenic sources, e.g. storm water, fertilizer application, and sewage overflow. These anthropogenic factors cause eutrophication from algae blooming and oxygen depletion [6] (Adam *et al.*, 2007), and degraded water quality in fresh water systems such as rivers, wetlands, and saline waters system in estuary areas. P can be sorbed or precipitated by Fe, Al, Ca, Mg; and these metal ions play important roles on P absorption and desorption. In the Pelham Bay wetlands, Ca is especially dominated by P concentrations and equilibrium phosphorus concentration-EPC<sub>0</sub>. The sediments from the freshwater wetlands contain Ca that precipitates P in a stable form, apatite. Sediment size also influences the sorption process, because fine-grained sediments, including silt and clay, have a large surface area [6] (Adam *et al.*, 2007).

P can be released from sediment/soil pore water into the water column and incoming water bodies in the wetland that contain lower concentrations of P [4] (Dunne *et al.*, 2010). Organic P accumulates in wetlands, and it can be released as inorganic phosphate by microbial decomposition [7] (Newman and Robinson, 1997; [8] Diaz *et al.*, 2006). Physicochemical factors, such as pH and temperature, as well as other hydrological conditions, influence P release and organic matter decomposition [9] (Wright and Reddy, 2001). The internal supply of P in wetlands causes P release from soil/sediment to overlying water. Sediment/soil physicochemical conditions, such as pH, redox, organic matter, nutrient content, calcium/magnesium content in alkaline soil, and iron/aluminum content in acidic soil, as well as overlying floodwater temperature, oxygen content/ionic strength can affect P release [4] (Dunne *et al.*, 2010).

Amorphous acid oxalate-extractable aluminum and iron content in the soil/sediments are naturally associated with P retention capacity in wetlands,

bogs, swamps, and rivers. The extractable aluminum content of the soil predicts the P adsorption potential in wetland ecosystems [10] (Richardson, 1985). Microbial and plant uptake, adsorption and incorporation of organic P (OP) into soil peat can remove dissolved inorganic P (DIP) from the water, consequently determining the P storage in wetlands. P is released after tissue death of the plant [10] (Richardson, 1985).

Anthropogenic sources impact P loading in a nutrient-limited ecosystem (*i.e.*, northern Everglades) [7] (Newman *et al.*, 1997). Reducing P loading is a strategy to help environmental restoration [11] (Daroub *et al.*, 2003). Sediment P plays an important role in eutrophication in shallower water bodies [12] (Bostrom *et al.*, 1982). The physiochemical properties of sediments impact P internal cycling, and P released from sediments has a significant impact on P concentrations in the water column [12] (Bostrom *et al.*, 1982). Sediment could be either sink or source for P depending on the water column and sediment physicochemical properties [13] (Reddy *et al.*, 1995; [10] Richardson, 1985). Analyzing TP concentrations and major P compounds in the sediments is an approach used to estimate the potential impact of sediments on shallow water bodies such as wetlands, streams, and lakes [8] (Diaz *et al.*, 2006).

Organic P can comprise more than 50% of sediment TP in wetland ecosystems and could be as much as 90% of the water column TP. The accumulation and mineralization of OP is important in P cycle [14] (Reddy *et al.*, 1998; [15] Rigler, 1964, [7] Newman and Robinson, 1997). OP, typically identified as the difference between TP and IP, could be either a sink or a source for bioavailable P. The mineralization of labile OP is critical for internal P cycling and productivity of an ecosystem [7] (Newman and Robinson, 1997). Sequential extraction is used to identify OP [16] (Hedley *et al.*, 1982). Chromatographic fractionation,  $^{31}\text{P}$  nuclear magnetic resonance (NMR), and enzymatic hydrolysis are techniques used to isolate and identify specific OP compounds [7] (Newman and Robinson, 1997). There are six common forms of P using  $^{31}\text{P}$ -NMR: phosphonates, inorganic orthophosphate, orthophosphate monoesters, orthophosphate diesters, pyrophosphates, and polyphosphates [17] (Dai *et al.*, 1996). The  $^{31}\text{P}$ -NMR extraction solution that has been commonly used is 0.5 M NaOH [18] (Bowman and Moir, 1993); some studies also used 0.25 M NaOH-0.05 M EDTA [19] (Cade-Menun and Preston, 1996).

The objective of this research is to estimate the nutrient impact on water quality in the wetlands in Pelham Bay Park, estimate internal P loading to the water column and sediments, identify P compounds and relative composition in wetland sediments, and estimate their potential bioavailability. This research sponsored by a grant from the New York Park Division was conducted to analyze the wetland sediments characteristics on sorption, in order to predict the potential bioavailability of phosphorus, the sources of phosphorus, the causes of algae blooms and the toxic algae's deleterious effects on ecosystems and human health. The research also helps form an effective strategy for water quality improvement in Pelham Bay estuary area. An improvement in water quality will

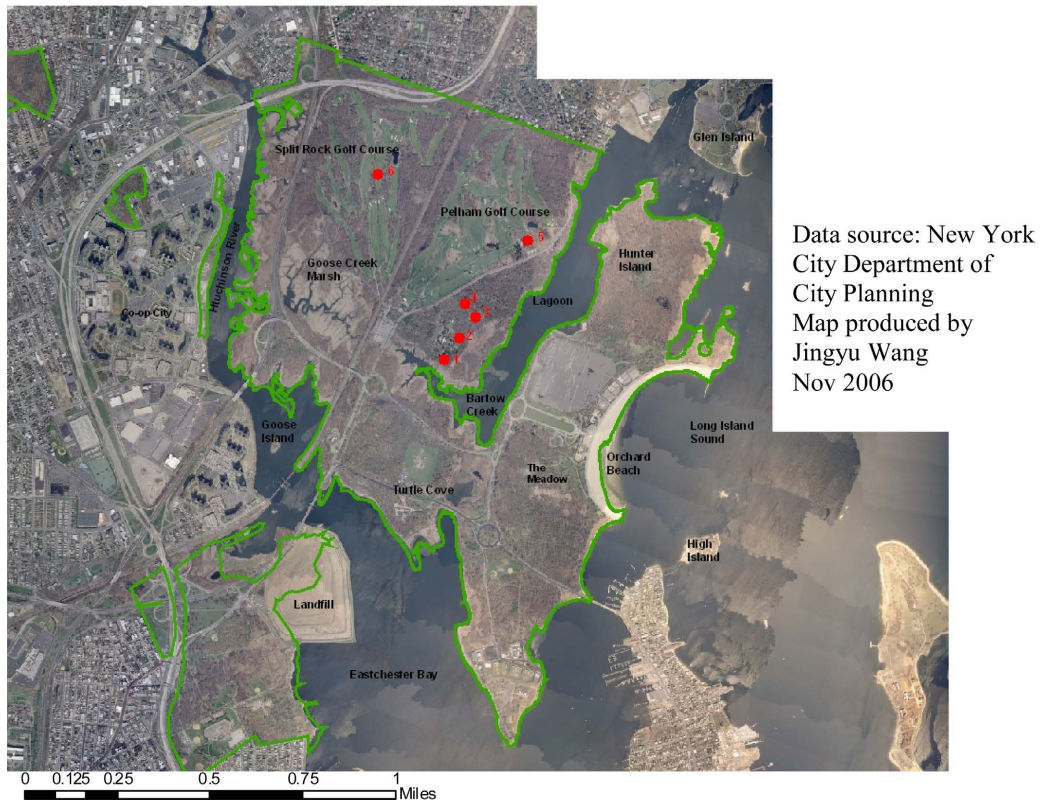
also benefit New Yorkers' health.

## 2. Material and Methods

### 2.1. Study Area

Pelham Bay Park, the largest municipal park in the New York City, is located on Long Island Sound, in the northeastern corner of the Borough of the Bronx. It contains nearly 500 acres of tidal marshes [20] (NYS DOS, 1992), comprised of high marshes, intertidal marshes, salt flats, lagoons, a narrow bay, and a wetland complex. The area is managed by the City of New York Department of Parks and Recreation [20] (NYS DOS, 1992). Wetland and aquatic habitats in Pelham Bay Park have been seriously degraded by discharges of sewage, stormwater runoff, and industrial effluents containing heavy loads of nutrients, sediments, or chemical pollutants into contiguous water bodies; or water pollution caused by oil spills, waste disposal, and excessive turbidity causing by construction/dredging [20] (NYS DOS, 1992).

The Pelham Bay wetlands are located in Pelham Bay Park, Bronx, NY. The Pelham Bay Park Wetlands, one of the largest natural estuarine habitats on the north shore of western Long Island Sound, is the only undisturbed tidal wetlands areas remaining in the Bronx [20] (NY DOS, 1992). For this study we collected samples at six sampling sites within the wetlands area. The six fresh water wetland sites locations were (Figure 1; Table 1): sites 1, 2, 3, and 4, located in the



**Figure 1.** Pelham bay freshwater wetlands study area.

**Table 1.** Geographical locations of the sampling sites and sediment texture in Pelham Bay freshwater wetlands.

Sites #	locations	latitude	longitude	Sediment texture
1-3	Close to Bartow-Pell Mansion Museum	40°52'17.13"N	73°48'15.92"W	sticky, silty, composed most of silt & clay
4	Close to Bartow-Pell Mansion Museum	40°52'17.13"N	73°48'15.92"W	Sandy, much different texture from the first three sites, there are plant roots in the soil.
5	Pelham Golf Course	40°52'17.13"N	73°48'15.90"W	silty, stinky odor
6	Split Rock Golf Course	40°52'45.54"N	73°48'51.53"W	very sticky, composed most of silt & clay, stinky smell

wetlands close to the Bartow-Pell Mansion Museum; site 5 in the wetland at the Pelham Golf Course; and site 6 located in a ditch in the Split Rock Golf Course (Figure 1). The major contents in the sediment are silt and clay. The sediment at sites 1, 2, and 3 were very sticky and silty; site 4 was more sandy, with detritus materials such as plant roots; sites 5 and 6 had a putrid smell, very sticky, composed mostly of silt and clay. The putrid odor came from fertilizer that had applied on the golf courses.

## 2.2. Sediment Sampling in Freshwater Wetlands

Sediment samples were collected from the six wetland sites in July 2006. Each site was located with a Global Positioning System (GPS) unit, and the coordinates are provided in Table 1. A Core Sampler (diameter = 8 cm; length = 17 cm) was used to obtain the bed sediments. The sediment samples were sealed in one gallon zipper bags, and transported to the Environmental Laboratory of the Department of Environmental, Geographic, and Geological Sciences, Lehman College of The City University of New York at the end of each sampling day, and stored at 4°C until further experimentation. The sediment samples were immediately homogenized and saved in waterproof double-track zipper bags (10.2 × 15.2 cm; made by Fisher Scientific Co., USA), and stored at 4°C until they were used for further analysis. A portion of each homogenized sediment sample was dried at 70°C for 72 h, and then finely ground and used for selected physico-chemical analysis.

## 2.3. Physico-Chemical Analysis of the Sediments

The wet sediment samples were oven dried at 105°C for 24 h and then analyzed for their water content; sediment samples were also dried at 70°C for 72 h and



finely ground for total phosphorus (TP) analysis. To determine TP, 0.5 to 1.0 g of finely ground dry sediments were combusted at 550°C in a muffle furnace for 5 h and the ash was dissolved in 6 M HCl. The digestate was filtered to a 100 ml volumetric flask with Fisher filter paper using an automated ascorbic acid method (ESS Method 310.1; USEPA, 1992) [21]. Meanwhile, the total organic matter (TOM) in sediments was determined by combustion in a muffle furnace. The total organic carbon (TOC) is equal to 0.58 times of the amount of TOM (TOC = 0.58\*TOM).

To determine the different poorly crystalline Fe and Al effect on phosphorus sorption, 0.5 g of finely ground dry sediment was extracted with 30 ml 0.175 M ammonium oxalate + 0.1 M oxalic acid at a ratio of 1:60 (0.5 ml/30ml = 1:60) by shaking for 4 h on an end-over-end mechanical shaker [22] (Pant and Reddy, 2001). Then the suspensions were centrifuged at 10,000 rpm for 10 min and filtered through a 0.45 µm membrane filter in the dark. The filtrates were analyzed for oxalate-extractable Al and Fe, plus oxalate-extractable P by an inductively coupled argon plasma spectrometry (ICP).

To analyze Ca and Mg effects on P sorption, a similar procedure as the previous step was used where the finely ground dry sediment of 0.5 g was extracted with 30 ml 1 M HCl at a ratio of 1:60 (0.5 ml/30ml = 1:60) by shaking on an end-over-end mechanical shaker for 4 h [22] (Pant and Reddy, 2001). The suspensions were centrifuged at 10,000 rpm for 5 min and filtered through a 0.45 µm membrane filter in the dark. The filtrates were then analyzed for HCl-extractable Ca, Mg, and P by inductively coupled argon plasma spectrometry (ICP-MS).

## 2.4. Phosphorus Sorption Experiments

A batch incubation was conducted to determine P absorption and desorption properties. All of the sediments were considered under aerobic conditions. Five grams of wet sediments were placed in a 50 ml centrifuge tube and shaken for 24 h in a 10 ml solution with orthophosphate-potassium phosphate monobasic (KH<sub>2</sub>PO<sub>4</sub>) in 0.01 M (mol/L) potassium chloride (KCl) solution with concentrations of 0, 0.1, 0.2, 0.5, 1, 2, 5, 10, 25, 50, and 100 mg/L (ppm) P, using an end-over-end mechanical shaker at 25°C ± 2°C. This was the absorption procedure. Thereafter, the homogenized samples were centrifuged for 20 min at 20°C, 10,000 rpm. Then the supernatants were decanted to 25 ml vials, acidified with one drop of concentrated sulfuric acid H<sub>2</sub>SO<sub>4</sub> and stored at 4°C for P absorption analysis. The sediment residues were then weighted and filled with 10 ml 0.01 M KCl solutions for desorption procedure, shaking for 24 h, using an end-over-end mechanical shaker at 25°C ± 2°C. Then the samples were centrifuged at 10,000 rpm for 20 min, decanted to 25 ml vials, and acidified with concentrated H<sub>2</sub>SO<sub>4</sub>. The concentration of P in the supernatants from both absorption and desorption were measured at 880 nm by UV-2501PC UV-VIS Recording Spectrophotometer using an automated ascorbic method (ESS Method 310.1; USEPA, 1992) [21].

## 2.5. $^{31}\text{P}$ -NMR Identification

A wet sediment sample of 40 g was extracted twice with 80 ml 0.4 M NaOH, each for 4 h by shaking in an end-over-end shaker at  $25^\circ\text{C} + 2^\circ\text{C}$ , 230 l/min. The suspensions were centrifuged at  $21^\circ\text{C} + 2^\circ\text{C}$ ,  $10,000 \times g$  for 30 min after each extraction. The supernatants were combined and concentrated 10 times from 100 ml to 10 ml using a Vacuum Rotatory Evaporator (R-205, BUCHI) at  $25^\circ\text{C}$ . Then 0.45 ml concentrated extracts were put in a 0.5 mm diameter Wilmad NMR tube, one drop of  $\text{D}_2\text{O}$  was added to lock the signal, and then the solution was scanned at 121.4944448360 MHz on a BRUKER 300 UltraShield  $^{31}\text{P}$  NMR for 15 h, and the spectra were processed using TOPSIN 2.0 software. The solution composition was determined with an external standard of 85% phosphoric acid, and the NMR spectra peaks were identified by comparing the sample peaks with the differences from the reference peaks: 0.6 ml samples were spiked with 0.02 ml  $10,000 \text{ mg}\cdot\text{l}^{-1}$  sodium pyrophosphate ( $\text{Na}_4\text{HP}_2\text{O}_7\cdot 10\text{H}_2\text{O}$ ) as an internal standard [23] (Gadian *et al.*, 1979; [24] Pant *et al.*, 1999).

## 2.6. Phosphorus Mineralization Experiments

Wet sediments (2 g) were incubated under a nonflooding condition at  $37^\circ\text{C}$  for 7, 15, 30, and 45 days (d), and extracted with  $\text{NaHCO}_3$ , NaOH, and HCl sequentially as mentioned previously. The changes in  $\text{NaHCO}_3\text{-P}$ , NaOH-P, HCl-P, residue-P in the sediments during incubations at  $37^\circ\text{C}$  for 7, 15, 30, 45 d from without incubation were to determine the extent of P mineralization at physiological/optimum temperature.

## 2.7. Statistical Analysis

All the P mineralization experiments were carried out in triplicate and analyzed with SAS JMP Version 13.0 [25] (SAS Institute Inc., 2008) using two-way analysis of variance (ANOVA). A Tukey's HSD (honestly significant difference) test was used for statistical differences at  $p < 0.05$  level as described by [26] Tao *et al.* (2008). Similarly, correlation coefficients were tested on sorption parameters using Pearson's correlation ( $r$ ) significant test at the  $p < 0.05$  or  $p < 0.01$  level using SPSS Version.15.0 [27] (SPSS Inc., 2006). All regression analyses were performed using the SPSS 15.0 software.

# 3. Results and Discussion

## 3.1. Physio-Chemical Characteristics of the Wetland Sediments

No significant difference in pH was found among the six freshwater wetland sites in the Pelham Bay (Table 2). EC ranges from 87.9 at site 2 to  $540 \mu\text{S}/\text{cm}$  at site 6 Split Rock Golf Course. Total organic carbon (TOC) ranges from 3.4 to 9.2. Ash-TP ranges from 337.6 to 666.1 mg/Kg (Table 2). The highest TOC%, Ox-P, Ox-Al, HCl-Ca, HCl-P and ash-TP, and the second highest Ox-Fe, HCl-Mg appeared at Site 4 beside Turtle Creek close to the Bartao-Pell Mansion Museum. The four sites close to Bartao-Pell Mansion Museum (sites 1, 2, 3 and

**Table 2.** Pelham fresh water wetlands chemical characteristics.

Site#	location	Oxalate-extractable				HCl-extractable				pH	EC μs/cm	
		Al	Fe	P	Fe:Al	TOC	Ca	Mg	P			ash-P
		mg/Kg				%	mg/Kg					
1	close to Bartao-Pell Mansion Museum	1139.7	14,937	364.5	13.1	6.7	5392	1048.4	360.2	666.1	5.6	185.3
2	close to Bartao-Pell Mansion Museum	960.8	19,166	427.9	19.9	5.6	3257	880.4	413.7	619.2	5.5	87.9
3	close to Bartao-Pell Mansion Museum	1115.7	10,534	389.2	9.4	5.5	2544	770	365.6	437.4	5.4	106.4
4	close to Bartao-Pell Mansion Museum	1162.2	14,712	517.7	12.7	9.2	5811	1135	443.5	874.9	6.4	213
5	Pelham Golf Course	679.6	5474	154.6	8.1	3.4	1497	850	199.8	337.3	5.4	97.1
6	Split Rock Golf Course	1019.3	8018	291.6	7.9	4.5	2523	1569.5	359.3	409.9	5.5	540

OM: total organic carbon, %. ash-P: the total phosphorus in bed sediments from Anderson ignition method, mg·kg<sup>-1</sup>. EC: electrical conductivity, μs·cm<sup>-1</sup>.

4) have relatively high Ox-Fe, Ox-Al (other than site 2, with a value of that was lower than site 6, Ox-P, Fe:Al, TOC, HCl-Ca, and total ash-P, which might be related to the sticky silty and clay type of texture, and OM could be attached to ferric (Fe<sup>3+</sup>) in aerobic conditions.

### 3.2. Sorption

$S_{max}$  ranges from 70.4 to 1666.7 mg/Kg (Table 3), with the highest value at sites 1-3.  $EPC_0$  (ranging from 0.086 to 0.235), were also high indicating a substantial amount of P that remained in the sediments could be bioavailable to the water column under seasonal conditions (Table 3). Pearson's correlation results show that  $S_{max}$  (P sorption maximum) is strongly correlated to Ox-Fe (after removing outlier  $S_{max}$  at site 4) ( $r = 0.89$ , significant probability level  $p < 0.05$ ) (Table 4). The maximum  $S_{max}$  at site 2 was 1666.7 mg/Kg (Figure 2 and Table 3), and the Ox-Fe at site 2 was 19,166 mg/Kg (Table 2), both were highest among the six sites. The strong correlation between  $S_{max}$  and Ox-Fe ( $r = 0.894$ ) indicates that the sediments that had high Fe content tended to have high maximum sorption capacity (Table 4). Site 1 and site 2 were mostly controlled by fine clay and silt; the soil texture was sticky for both sites. Redox has an impact on sorption. A high content of amorphous and poorly crystalline Fe under aerobic conditions results in high  $S_{max}$  values [22] (Pant and Reddy, 2001; [28] Wang and Pant, 2010a). The wetland area was exposed to air and ferric iron as Fe II converted to ferrous Fe as FeIII. The FeIII in both sites bound P tightly and had a high capacity to bind P [28] (Wang and Pant 2010a).  $EPC_0$  at site 2 was 0.086 mg/L, which was the lowest among the six sites (Figure 3 and Table 3), indicating that  $EPC_0$



**Table 3.** Phosphorus sorption characteristics of the Pelham fresh water wetlands.

site#	location	$K_d$	$S_0$	$EPC_0$	$S_{max}$	$k$	$K_f$	$P_r$
		L/Kg	mg/Kg	Mg/L	Mg/Kg	L/mg	L/Kg	% Pad
1	close to Bartao-Pell Mansion Museum	136.4	14.2	0.104	1428.6	0.12	118.4	1
2	close to Bartao-Pell Mansion Museum	745.8	64.2	0.086	1666.7	0.2	247.4	1
3	close to Bartao-Pell Mansion Museum	590.6	56.9	0.096	1111.1	0.69	492.8	99.16
4	close to Bartao-Pell Mansion Museum	141.7	33.2	0.235	454.5	0.42	95.96	95.93
5	Pelham Golf Course	253.4	32.7	0.129	434.8	0.96	151.9	98.59
6	Split Rock Golf Course	605.1	68.0	0.112	70.4	284	457.1	99.65

$K_d$ : distribution coefficient,  $L \cdot kg^{-1}$ .  $S_0$ : P originally sorbed on the solid phase,  $mg \cdot kg^{-1}$ .  $EPC_0$ : equilibrium P concentration,  $mg \cdot L^{-1}$ .  $S_{max}$ : P sorption maximum,  $mg \cdot kg^{-1}$ .  $k$ : a constant related to the bonding strength,  $L \cdot mg^{-1}$ .  $K_f$ : P absorption energy,  $L \cdot kg^{-1}$ .

**Table 4.** P sorption parameters and selected chemical characteristics coefficient r-values.

parameter	$S_{max}$	$EPC_0$	ash-P	TOC	$P_r$	$K_f$
Ox-Fe	0.894*	-0.195	0.754	0.619	-0.764	-0.311
Ox-Al	0.413	0.392	0.653	0.774	-0.168	0.124
HCl-Ca	0.626	0.557	0.940*	0.928*	-0.387	-0.561
HCl-Mg	-0.604	0.122	0.059	0.075	0.210	0.186
TOC	0.765	0.723	0.957*	1	-0.144	-0.431

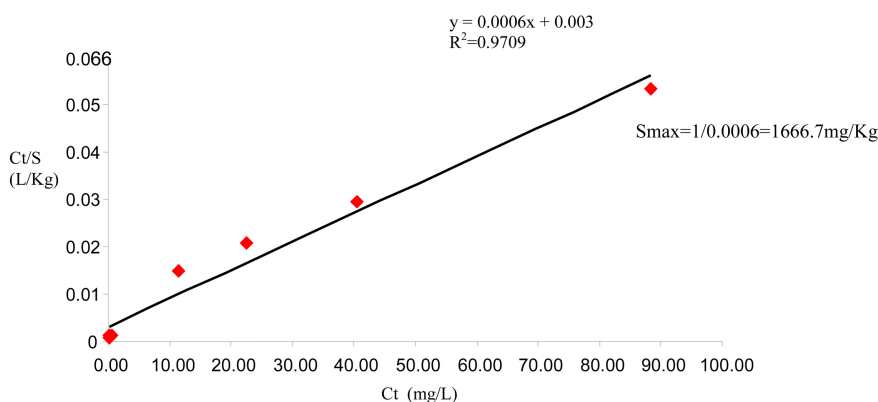
\*Correlation is significant at the 0.05 level (2-tailed),  $p < 0.05$ . \*\*Correlation is significant at the 0.01 level,  $p \leq 0.01$ .

was not impacted by Ox-Fe [28] (Wang and Pant, 2010a). The higher  $EPC_0$  is mostly associated with anaerobic conditions, and ferrous FeIII is reduced to ferrous Fe II [22] (Pant and Reddy, 2001). Sediment would absorb P if the P concentration in the water column was more than  $EPC_0$  in the wetland sediment; sediment would desorb P if the P concentration in the water column was less than sediment's  $EPC_0$  ([29] Kunishi *et al.*, 1972; [30] Wang *et al.*, 2009; [28] Wang and Pant, 2010a).  $S_0$  (P originally sorbed on the solid phase) is strongly correlated to  $K_d$  (adsorption energy), and the Pearson correlation coefficient  $r$  was 0.936 (Table 5).  $P_r\%$  at the six sites ranges from 96% to 100% (Table 3), indicating that most adsorbed P was retained at a higher level than desorbed P [28] (Wang and Pant, 2010a).  $EPC_0$  is not significantly correlated to  $P_r\%$  ( $P_r\% = \frac{P_r}{P_{ad}} \times 100\%$ ), however, site 2 has the lowest  $EPC_0$  but the highest  $P_r\%$  and

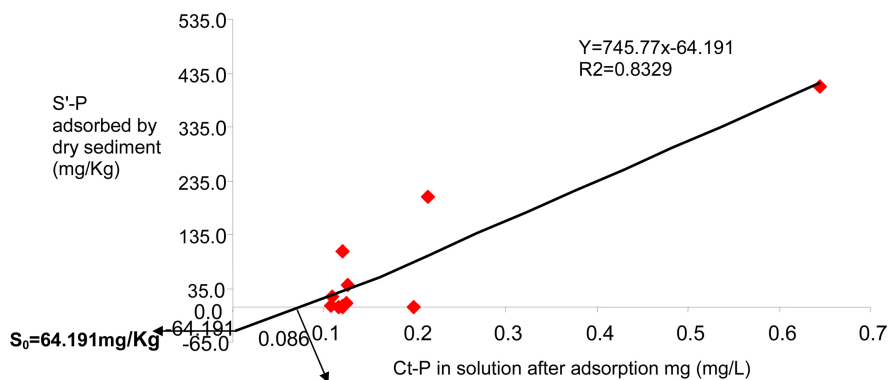
**Table 5.** Pearson’s correlation matrix (r) of sorption parameters.

parameter	K <sub>d</sub>	S <sub>0</sub>	EPC <sub>0</sub>	S <sub>max</sub>	k	K <sub>f</sub>	P <sub>r</sub>
K <sub>d</sub>	1	0.936**	-0.620	0.216	0.355	0.758	0.427
S <sub>0</sub>	0.936**	1	-0.357	-0.106	0.531	0.790	0.223
EPC <sub>0</sub>	-0.620	-0.357	1	-0.500	-0.132	-0.508	-0.922**
S <sub>max</sub>	0.216	-0.106	-0.106	1	-0.613	-0.136	0.255
k	0.355	0.531	0.531	-0.613	1	0.553	0.361
K <sub>f</sub>	0.758	0.790	0.790	-0.136	0.553	1	0.516
P <sub>r</sub>	0.427	0.223	0.223	0.255	0.361	0.516	1

\*Correlation is significant at the 0.05 level (2-tailed),  $p < 0.05$ . \*\*Correlation is significant at the 0.01 level,  $p \leq 0.01$ .



**Figure 2.** Pelham wetland 2 (close to Bartao-Pell mansion museum) S<sub>max</sub> calculation.



**Figure 3.** Pelham wetland 2 (close to Bartao-Pell Mansion Museum). The langmuir isotherms batch experiment for EPC<sub>0</sub> and S<sub>0</sub> calculation.

S<sub>max</sub>. The high retention ratio in the sediment made it saturated with P, therefore the equilibrium P concentration decreased when S<sub>max</sub> and Pr% increased. Changes of water chemistry in sediments, such as pH and redox, could potentially cause massive P release to the water column ([30] Wang and Pant, 2009; [28] 2010; [31] 2010; [32] 2011; [33] 2011; [34] 2012).

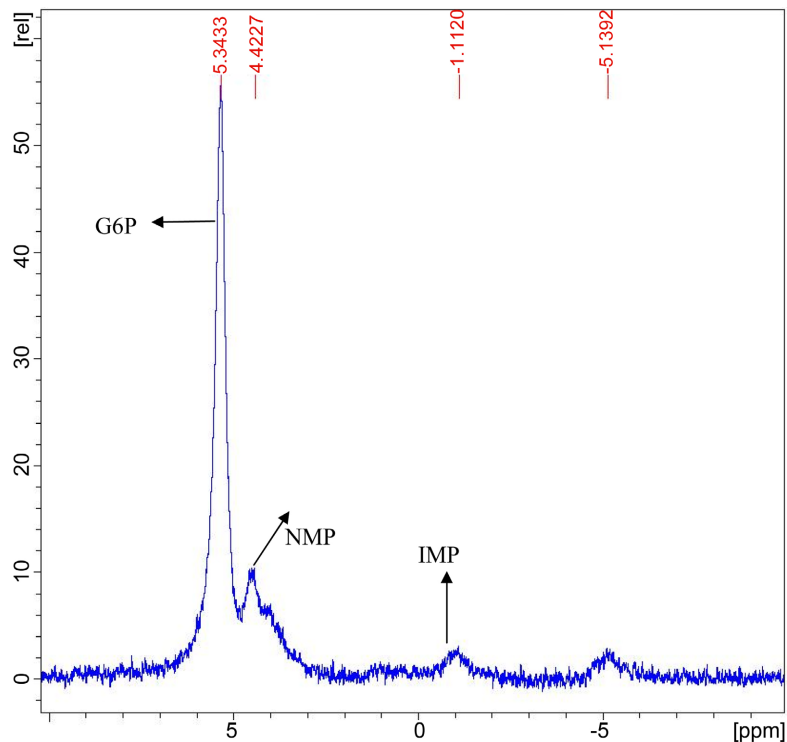
The significant correlations at the 0.01 level (2-tailed) between TOC (total or-

ganic carbon) and HCl-Ca, HCl-Ca vs ash-TP and TOC vs ash-TP (**Table 4**) indicate that Ca and OM bounded P could become a potential source of P in the water column under different redox conditions [8] (Diaz *et al.*, 2006). HCl-Pi (HCl extracted IP) could range from 46% - 64% of TP in water conservation area canals in the Everglades [8] (Diaz *et al.*, 2006). HCl extracted SRP (comparatively equal to HCl-Pi) ranged from 12% to 60%, and concentrations ranged from 73.9 to 255.9 mg/Kg in Pelham Bay wetland sediments. HCl-Pi, Ca, and Mg bound P is relatively stable and unavailable for biological assimilation [35] (Sonzogni *et al.*, 1982). Organic P extracted by NaOH can be hydrolyzed to bioavailable P, which is associated with humic and fulvic acid ([36] Bowman and Cole, 1978; [37] Ivanoff *et al.*, 1998).

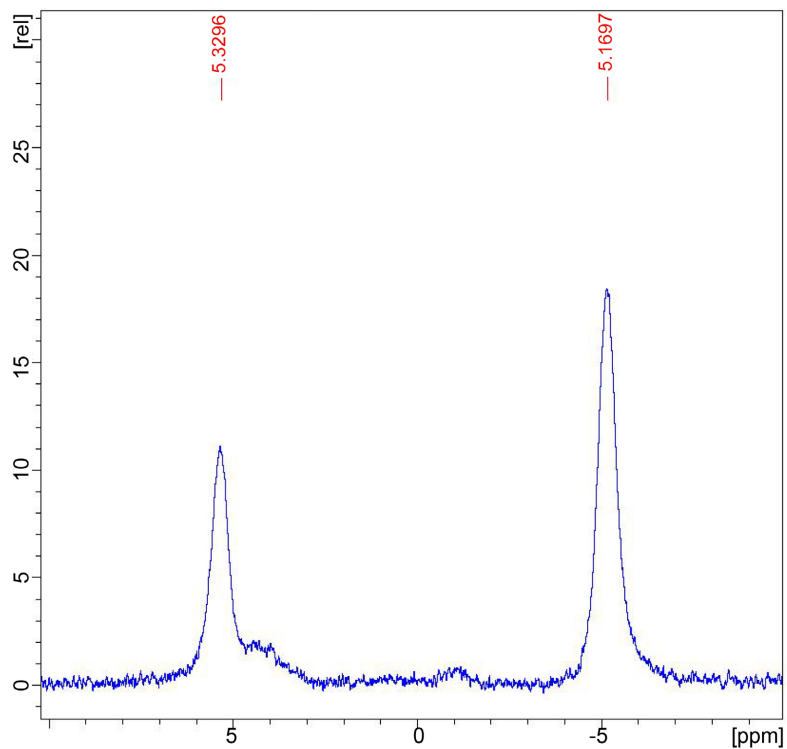
### 3.3. P Compound Identification-31P-NMR

Organic P compounds were found in the 6 sampling sites, which could be related to the OM attached to  $Fe^{3+}$  [30] (Wang and Pant, 2009). The dominant P compounds G6P and DHAP are organic P compounds in sites 1, 2, 3, and 4, and those four sites also have the highest Ox-Fe, Ox-Al, Ox-P, TOC, HCl-Ca, HCl-P and ashP, indicating organic P is dominant in those fine sediments [30] (Wang and Pant, 2009). G6P (glucose-6-phosphate, major peak at 5.34 ppm, 81.5%), NMP (nucleoside monophosphates, peak at 4.42 ppm, 7.2%), IMP (inosine monophosphate, peak at -1.11, trace amount of 3.8%) and trace amount of PyroP (pyrophosphate) were found in site 1 (**Figure 4** & **Figure 5**). The percentage proportion was determined by the peak area %. The major peak at site 1 was G6P (**Table 6**), and it had the highest TOC among the six sites. G6P appeared in all sites. DHAP (dihydroxyacetone phosphate) was the dominant compound (peak at 5.59 ppm, nearly 100%), and trace amounts of PEP (phosphoenopyruvates, peak at 0.11, 0.1%) and IMP (peak at -0.23, 0.1%) at site 2 (**Figure 6** & **Figure 7**) were also present. There is around 100% DHAP (99.97%) at site 3 (peak at 5.16 ppm), plus trace amounts of orthophosphate (peak at 8.01 ppm, 0.01%), GPEA (glycerophosphoethanolamine, peak at 0.47 ppm, 0.01%) and PEP (phosphoenolpyruvates, peak at -0.10 ppm, 0.01%) (**Figure 8** & **Figure 9**). Major P compound at site 4 was G6P (4.47 ppm, 97.40%) (**Figure 10** & **Figure 11**), trace peaks are NMP (3.57 and 3.23 ppm, 2.4%), IMP (-1.86 ppm, 0.1%), and PyroP (-5.99 ppm, 0.1%). G6P is about 100% at site 5 (**Figure 12** & **Figure 13**). The spectra at site 6 showed an interesting pattern, with diverse P compounds (**Figure 14** & **Figure 15**): trace amount DHAP (peak at 6.49 ppm, 0.1%), PolyN (peak at 3.65 ppm, 0.1%), and major P compounds are GlyP (peak at 5.19 ppm, 26.3%), NMP (peak at 4.25 ppm, 26.3%) and PyroP (peak at -5.14, 25.6% and peak at -6.07, 21.5%).

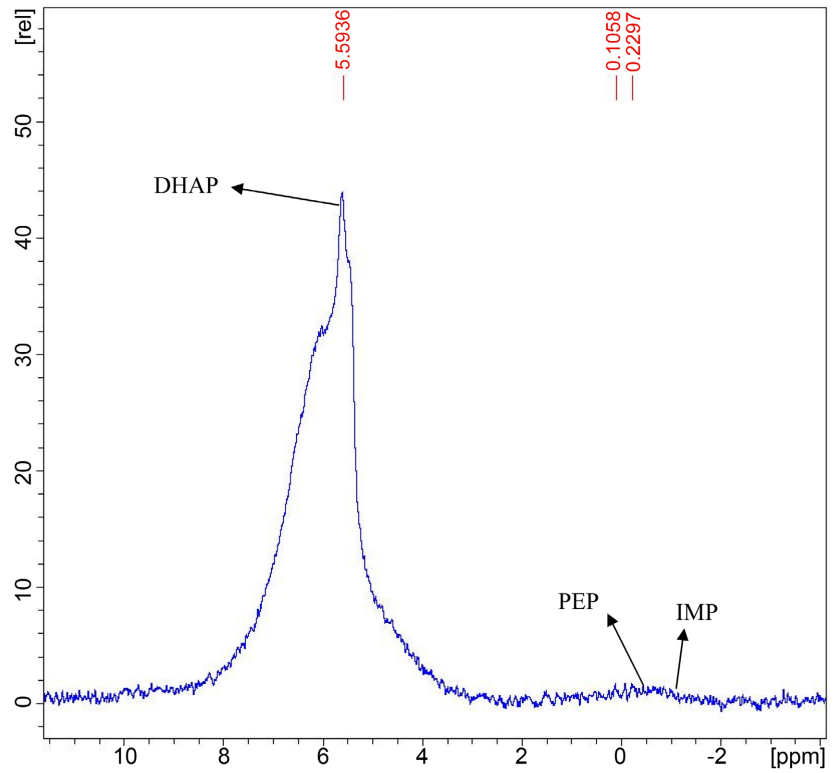
Compared to the Bronx River, P compounds in the Pelham Bay freshwater wetlands are quite different. The P compounds in the river bed sediments are mostly GlyP, NMP and PolyN, few DHAP and IMP. Major OP compounds in wetlands are G6P, DHAP, and small amounts of PEP [30] (Wang and Pant,



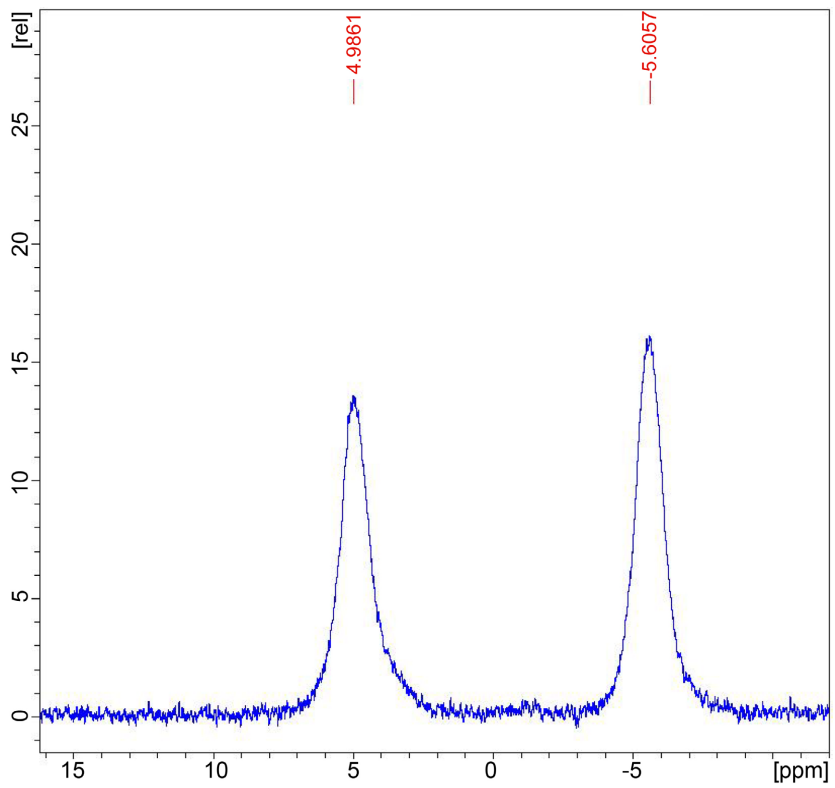
**Figure 4.** PW 1 concentrated sample. Site 1 PW1-Pelham Bay wetland close to Bartow-Pell Mansion Museum. There were three peaks in this sites concentrated sample, the major peak at 5.34 was G6P, the one at 4.42 was NMP, and the one at -1.11 was IMP. They represented G6P-81.5%, NMP-7.2%, and IMP-3.8% of relative composition respectively.



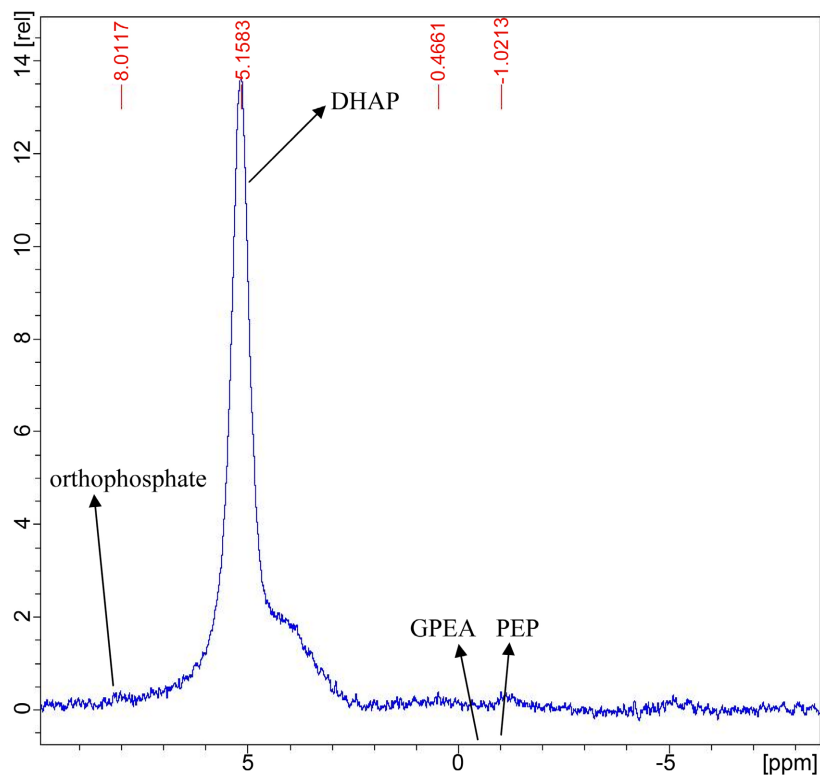
**Figure 5.** PW1 spiked sample, Pyrop spike: -5.1697.



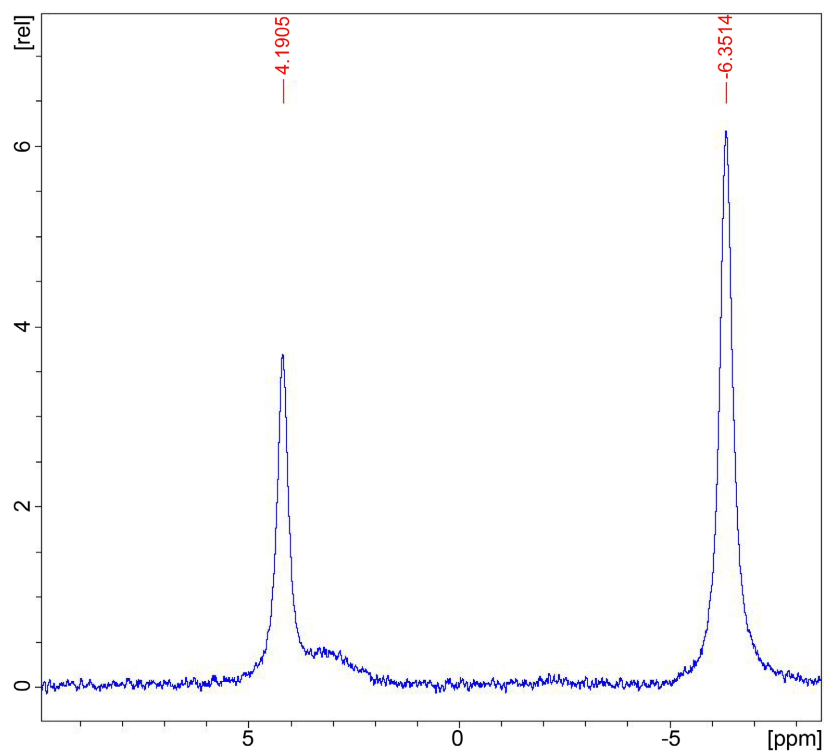
**Figure 6.** PW2 concentrated sample. Site 2 PW2-Pelham Bay wetland close to Bartow-Pell Mansion Museum. PW2 had DHAP for 99.8%, PEP 0.1%, and IMP 0.1% showing in **Figure 6**.



**Figure 7.** PW 2 spiked sample, pyrop spike: -5.6057.

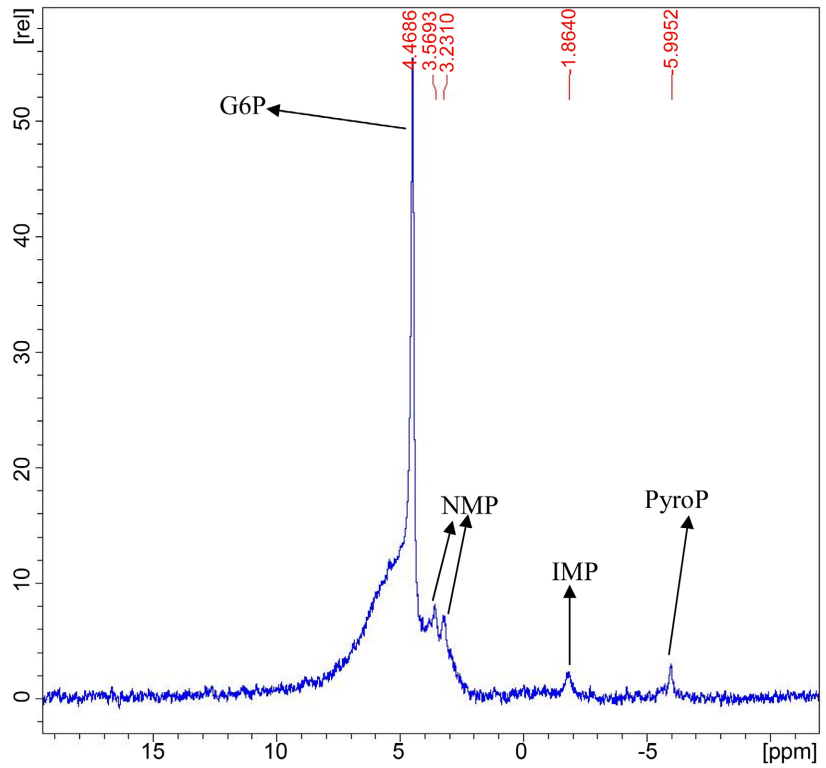


**Figure 8.** PW3 concentrated sample. Site 3. PW3-Pelham Bay wetland close to Bartow-Pell Mansion Museum. The major peak at 5.16 ppm was DHAP 99.97%. The rest of three peaks have not been identified yet, peak at 8.01 ppm orthophosphate (0.01%), 0.47 ppm GPEA (0.01%) and -1.02 ppm PEP (0.01%).

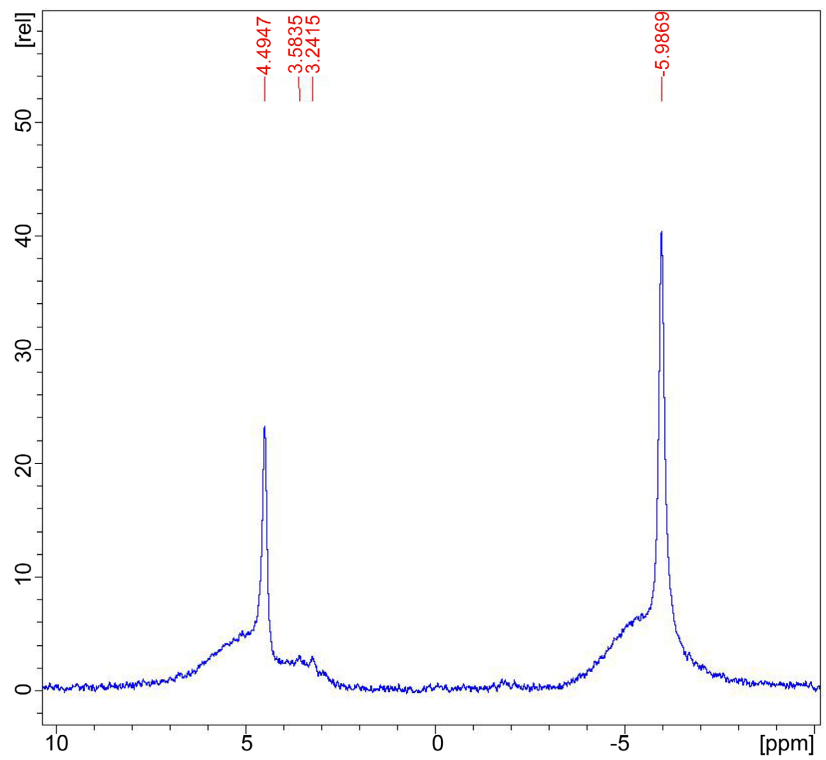


**Figure 9.** PW3 spiked sample, pyrop spike: -6.3514.

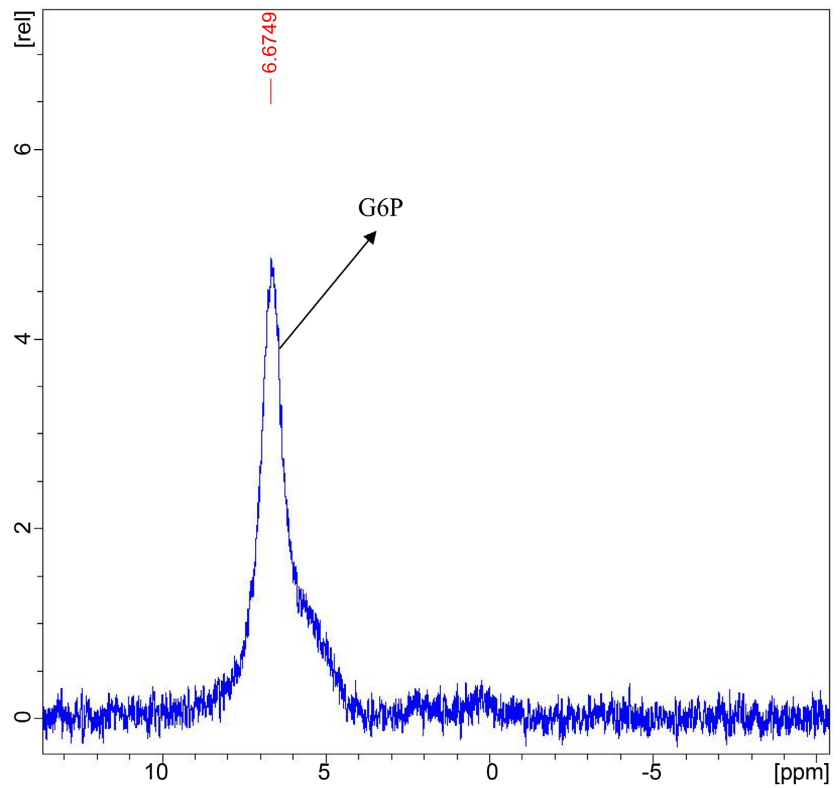




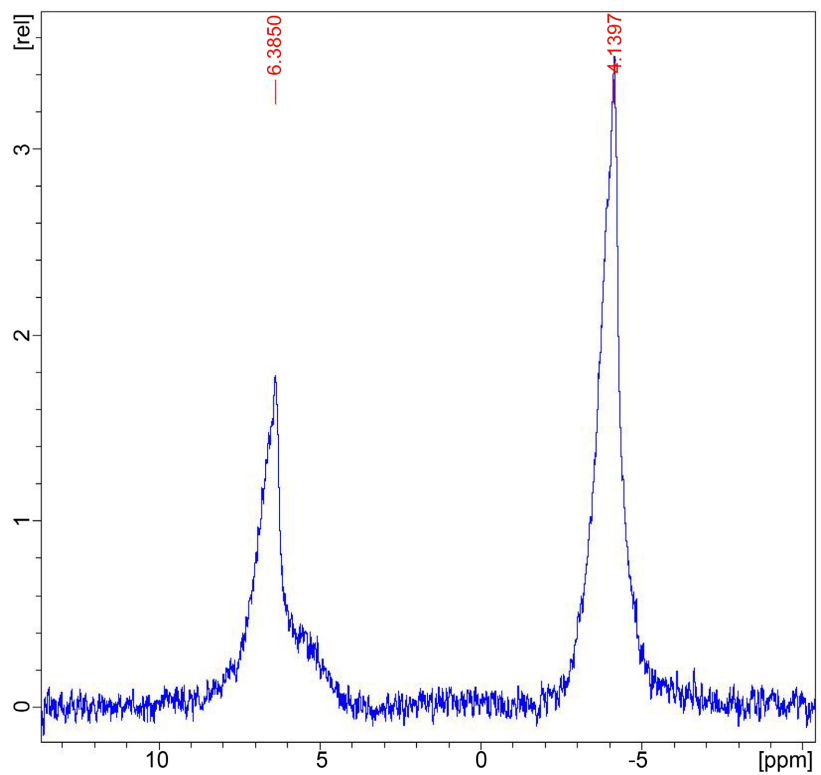
**Figure 10.** PW4 concentrated sample. Site 4. PW4-Pelham bay wetland close to Bartow-Pell mansion museum. W4 had five different P compounds: peak at 4.47 ppm G6P of 97.4%, peaks of 3.57 and 3.23 were both NMP of 1.2% each, -1.86 ppm was IMP of 0.1%, and -5.99 ppm was PyroP of 0.1%.



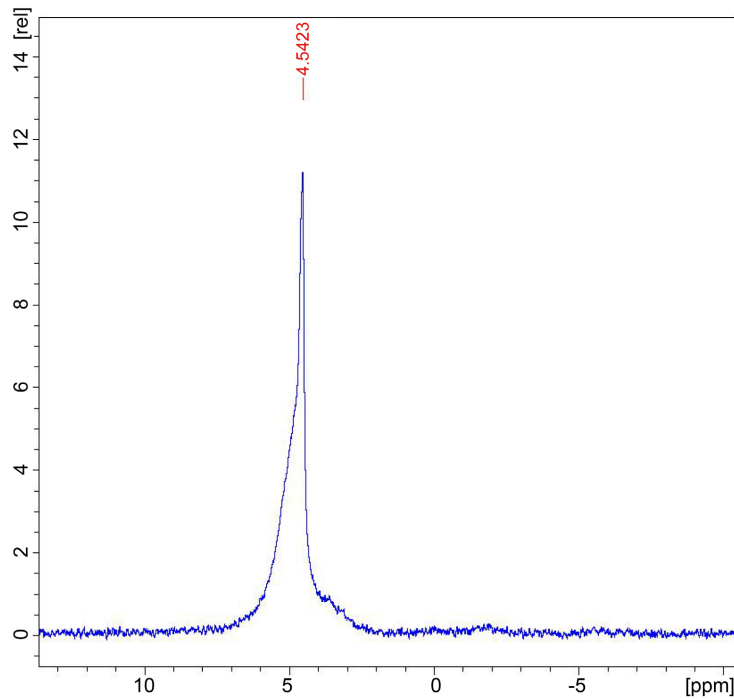
**Figure 11.** PW4 spiked sample, pyrop spike: -5.9869.



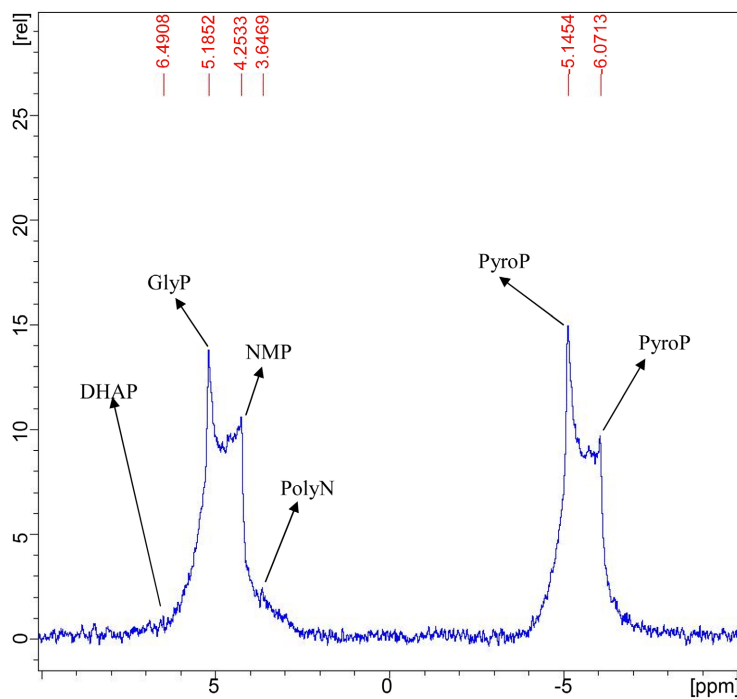
**Figure 12.** Site 5 concentrated sample. Site 5. Pelham golf course. Site 5 had one major peak at 6.67 ppm, which was G6P, accounted for 100% of relative composition.



**Figure 13.** Site 5 spiked sample, pyrop spike: -4.1397.



**Figure 14.** Site 6 concentrated sample. Site 6. Split rock golf course. Site 6 spiked sample showed a very interesting pattern, the first double peaks include peak at 5.19 ppm of GlyP for 26.3% and peak at 4.25 ppm of NMP for 26.3%. Peak at further left 6.49 ppm of DHAP for 0.1%, and further right 3.65 ppm was PolyN for 0.1%; the second double peaks were composed of the Pyrop spike at  $-5.15$  ppm, around 25.6%, and another Pyrop from the sample at  $-6.07$  ppm of 21.5%.



**Figure 15.** Site 6 spiked sample. DHAP 6.49 ppm: 0.1%, PolyN 3.65 ppm: 0.1%, GlyP 5.18 ppm: 26.3%, NMP 4.25 ppm: 26.3%, PyroP  $-5.14$  ppm: 25.6%, PyroP  $-6.07$  ppm: 21.5%.

**Table 6.** NMR results.

Site#	Location	Peak (ppm)	P compound	Relative composition (%)
1	PW1	5.34	G6P	81.5
		4.42	NMP	7.2
		-1.11	IMP	3.8
		-5.14	Pyrop	3.8
2	PW2	5.59	DHAP	99.8
		0.11	PEP	0.1
		-0.23	IMP	0.1
3	PW3	8.01	orthophosphate	0.01
		5.16	DHAP	99.97
		0.47	GPEA	0.01
		-1.02	PEP	0.01
4	PW4	4.47	G6P	97.4
		3.57	NMP	1.2
		3.23	NMP	1.2
		-1.86	IMP	0.1
		-5.99	PyroP	0.1
5	PW5	6.67	G6P	100
6	PW6	6.49	DHAP	0.1
		5.19	GlyP	26.3
		4.25	NMP	26.3
		3.65	PolyN	0.1
		-5.15	Pyrop	25.6
		-6.0713	Pyrop	21.5

G6P: glucose-6-phosphate; NMP: nucleoside monophosphates; IMP: inosine monophosphate; Pyrop: pyrophosphate; DHAP: dihydroxyacetone phosphate; PEP: phosphoenopyruvate; GPEA: glycerophosphoethanolamine; GlyP: glycerophosphate.

2009), IMP, besides GlyP, PolyN and NMP. Here, G6P might not exist in the water system because it was soluble, and it only could exist in wetland or soil environment. There are many other organic P compounds in wetlands rather than in the river. The P bioavailability prediction in wetlands is quite meaningful; because under certain conditions, such as favorable pH, temperature, redox, the organic P (OP) could be mineralized to inorganic P (IP) and become bioavailable to plants [34] (Wang and Pant, 2012) and potentially cause eutrophication. The active P pool in the wetland sediments could maintain substantial bioavaila-

bility of P for seasonal algal growth under changing hydro-climatic conditions.

### 3.4. P Mineralization

The phosphorus pool changed during the mineralization for 7, 15, 30, and 45 days under flooding incubation.  $\text{NaHCO}_3$  increased from 43 to 57 mg/Kg during the 7, 15, 30, and 45 day flooding incubation (**Table 7**, **Table 8**). The high content of NaOH-P in sites 1 - 4 is associated with the high organic matter in those sites, correspondingly dominant OP compounds, and those sticky, silt, and clay, and sandy sediments that are associated with Fe, Al, and under changing redox conditions, could have resulted in high  $S_{\text{max}}$  or high  $\text{EPC}_0$  ([30] Wang and Pant, 2009; [32] 2011).

#### 3.4.1. Spatial Variation of 7, 15, 30, 45 Day Mineralization

The 7-day nonflooding mineralization after sequential extraction by  $\text{NaHCO}_3$ , NaOH and HCl showed that site 3 had the highest average value of 66.26 mg/Kg (**Table 6**) for  $\text{NaCO}_3$ -P, and it was significantly higher than sites 1, 5, and 6. Site 5 had a significantly lower NaOH-P value than the other sites. HCl-P was the highest on site 6 and it was significantly higher than sites 1, 3 and 5. Total cumulative P was highest at site 4 (542 mg/Kg), and that is significantly different from site 5. The ashTP was significantly different among the six sites, and site 4 had the highest one of 875 mg/Kg that is significantly higher than the other five sites. Site 5 had the lowest of 337 mg/Kg (**Table 6**). Residue P had the highest value at site 4 of 333 mg/Kg, which was significantly higher than sites 2, 3, 5 and 6. The negative values at sites 3 and 6 were because ashTP was lower than total cumulative P. Combined sewer overflows (CSOs) in the discharge of untreated sewage into NYC rivers during rainstorms, elevate nutrients and bacteria levels and contaminate the waterways, which could impact P composition, P concentration, P pool, and P mineralization [38] (Wang, 2014) differently from river bed sediments.

The 15 day nonflooding incubation showed variation among the six sites. Sites 1-4 are significantly higher than sites 5 and 6 for  $\text{NaHCO}_3$ -P (**Table 6**). Site 2 had the highest NaOH-P of 378 mg/Kg that is significantly higher than other five sites. Site 6 again had the highest HCl-P of 220 mg/Kg, and it was significantly higher than other sites (similar as 7 day incubation). Cumulative P appeared highest at site 2, but ashTP was the highest at site 4, which had the highest residue P of 412 mg/Kg.

The 30 day nonflooding incubation showed similar patterns as 7 and 15 days. Site 4 had all the highest values other than HCl-P (**Table 6**). The 45-day flooding incubation also showed similar patterns. The highest values of  $\text{NaHCO}_3$ -P, TP-added up, ashTP, and residue occur at site 4, whereas the highest NaOH-P was at site 2 (344 mg/Kg, which was higher than the 7, 15, and 30 day flooding incubation). HCl-P of 206 mg/Kg was highest at site 6, which was lower than the 7, 15, and 30 day flooding incubation.

**Table 7.** Mineralization of 7, 15, 30 and 45 days.

7 day site	NaHCO <sub>3</sub> -P	NaOH-P	HCl-P	TP add up	ashTP	Residue-P
1	43 bcd	261 a	120 bc	424 ab	666 b	242 ab
2	47 abc	260 a	169 abc	476 a	619 c	143 bc
3	66 a	269 a	148 bc	484 a	437 d	-46 d
4	60 ab	294 a	187 ab	542 a	875 a	333 a
5	26 cd	149 b	102 c	277 b	337 f	61 cd
6	22 d	231 ab	236 a	489 a	410 e	-79 d
15 day site						
1	45 a	272 b	130 bc	446 b	666 b	220 b
2	52 a	378 a	164 b	594 a	619 c	26 c
3	41 a	271 b	128 bc	440 b	437 d	-2 c
4	52 a	267 b	144 b	463 ab	875 a	412 a
5	21 b	130 c	86 c	237 c	337 f	100 bc
6	16 b	185 bc	220 a	422 b	410 e	-12 c
30 day site						
1	48 a	188 bc	100 c	337 bc	666 b	329 a
2	50 a	249 ab	169 b	468 a	619 c	151 b
3	53 a	246 ab	125 bc	424 ab	437 d	14 cd
4	66 a	262 a	166 b	495 a	875 a	380 a
5	25 b	133 c	93 c	252 c	337 f	8 bc
6	23 b	201 ab	238 a	462 a	410 e	-52 d
45 day site						
1	57 bc	243 abc	137 abc	437 ab	666 b	229 ab
2	75 ab	344 a	177 ab	596 a	619 c	23 bc
3	53 c	245 abc	131 bc	429 ab	437 d	8 c
4	80 a	303 ab	191 ab	574 a	875 a	301 a
5	20 d	131 c	90 c	242 b	337 f	96 abc
6	24 d	201 bc	206 a	431 ab	410 e	-21 c

Values in the same column with different letters are significantly different (Tukey's HSD test,  $p \leq 0.5$ ).

**Table 8.** All sites 7, 15, 30 and 45 days.

Incubation days	NaHCO <sub>3</sub>	NaOH	HCl	TP add up	ashTP	residue
7	44 a	244 a	160 a	448 a	557 a	109 a
15	38 a	250 a	145 a	433 a	557 a	124 a
30	44 a	213 a	148 a	406 a	557 a	151 a
45	52 a	244 a	155 a	451 a	557 a	106 a

Values in the same column with the same letters are not significantly different (Tukey's HSD test,  $p \leq 0.5$ ).



### 3.4.2. Temporal Variation of 7, 15, 30, 45 Day Incubation

No significant variation exists among 7, 15, 30 and 45 day incubation (Table 7) even though there is a slight difference in concentrations of  $\text{NaHCO}_3\text{-P}$ ,  $\text{NaOH-P}$ ,  $\text{HCl-P}$ , total cumulative P, ash TP, and residue P. It showed a different pattern of mineralization from river bed sediments in the Bronx River [31] (Wang and Pant, 2010). For all of the six sites,  $\text{NaHCO}_3\text{-P}$  increased from day 7 to day 15, 30, and 45. For site 1,  $\text{NaOH-P}$  increased from day 7 to day 15, then decreased on day 30 and increased again on day 45; all other sites had similar patterns other than site 5 at the Pelham golf course and site 6 at the Split Rock Golf Course. The  $\text{NaOH-P}$  indicates there is organic matter in the sediments, and high Al and Fe content in humus matter [32] (Wang and Pant, 2011). For  $\text{HCl-P}$  no significant variation was found at the different sites. Site 1 increased from day 7 to day 15, decreased on day 30, and increased on day 45, and a similar temporal pattern occurred at other sites. Residue-P increased on day 30 for most of the sites.

There is no significant difference for all sites on flooding incubation for 7, 15, 30 and 45 days on  $\text{NaHCO}_3\text{-P}$ ,  $\text{NaOH-P}$ ,  $\text{HCl-P}$ , cumulative TP, ashTP, and residue P (Table 8).

## 4. Conclusion

Phosphorus flux in the Pelham Bay wetlands showed a significant sorption capacity in wetland sediments, showing potential bioavailability and nutrient enrichment. Sites 1, 2, and 3 had significantly higher values of  $S_{\text{max}}$ , Ox-Fe, and Pr%, and there was a significant correlation between  $S_{\text{max}}$  and Ox-Fe that indicated that ferrous Fe sediments in the wetlands have a large capacity for sorption of P. G6P and DHAP are the dominant P compounds at sites 1, 2, and 3, and  $\text{NaOH-P}$  values are highest in these three sites on 7 day incubation, which showed that organic P played a significant role in the sediment in these sites. Site 4 had the highest values of  $\text{EPC}_0$ , Ox-Al, Ox-Fe, Ox-P, TOC, HCl-Ca, HCl-Mg, HCl-P, and ashP. The dominant P compound is G6P, indicating organic matter is highest at this site, and organic matter associated with amorphous and poorly crystalline Fe and Al formed suitable conditions for P sorption. The OM and Ca bounded P could release bioavailable P under different redox conditions. However, the high  $\text{EPC}_0$  produced the lower  $S_{\text{max}}$  value. The changing hydro-climatic conditions such as temperature rise could potentially release more bioavailable P in the wetlands and water system and cause eutrophication that degrades water quality. Organic matter bounded tightly with fine and sticky wetland sediments could have a higher potential to release bioavailable P under different redox conditions. P composition in these wetland sites are dominated by OP and P compounds that could be distinguished from the river bed sediments.

## Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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