



Nutrient speciation in sediment and water quality assessment of the Circului Lake, Bucharest

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Abstract. This paper brings an extension to the studies regarding the eutrophication process, particularly to those focused on the role of nutrients from the sediment on the eutrophication process. This paper describes the distribution of the nutrient concentration within the water phase and the sediments from the Circului Lake, Bucharest, and the speciation of nutrients, as the latest has been reported to play an important role in the processes that take place within the water body. This paper depicts the evaluation of water and sediment quality of an interior lake that does not communicate with any surface water bodies. Several water quality indicators were monitored during one year, with special emphasis on nutrient levels in water and sediments. Nutrient speciation in sediments was determined and associations were made between the phosphorus and nitrogen contents in water and sediment. Bacterial communities involved in the nitrogen cycle were also studied. This paper is the first to describe the dynamics of the concentrations of nutrients and their speciation in sediments within the Circului Lake, Bucharest. Results showed that most of phosphorus is bonded to calcium, while for the readily adsorbed phosphorus the lowest values were obtained. High densities of ammonifying and nitrifying bacteria in sediments reflected the predominance of protein hydrolysis and ammonia oxidation processes. The variation of other water quality indicators relevant to the eutrophication process is depicted. The results showed that the lake is prone to eutrophication, especially during the warm season, even with the mitigation/remediation measures undertaken by the local authorities.

Key Words: eutrophication, nutrient speciation, Circului Lake, sediment characteristics.

Introduction. One of the major concerns of the human population is water quality assessment as the available water resources are scarce. European regulations provide the criteria for the classification of water into five quality classes (Directive 2000/60/EC).

Natural processes and human activities are the main sources of water pollution (Deak et al 2015). Among water pollution, the eutrophication of surface water bodies is a complex process and has multiple causes. High contents of phosphorus in water bodies are known as the primary cause of eutrophication and lead to an excessive algal growth followed by a decrease of oxygen required for the oxidation of organic material (Cirtina & Mitran 2011).

Eutrophication is the biological response of a water body (Yang et al 2008; Khan & Ansari 2005; Wang & Wang 2009; Baxter 2011; Qin et al 2013). It is the result of the autotrophic production of organic matter. As to the effects of eutrophication, one mentions among others, the development and growth of various algae, the reduction of available light, the increase of organic matter production and the emergence of hypoxic and anoxic conditions (Yang et al 2008; Qin et al 2013).

The role of nutrient concentrations in sediments and the importance of the predominant fractions have been acknowledged by various studies. In the recent years, studies have focused on the role of the content of nutrients from the sediment on the eutrophication process, particularly on the distribution of their concentration within the water phase and the sediments and on the fractionation of nutrients, as the latest has proven to play an important role in the absorption/desorption processes that take place within the water body. Also, research studies were conducted for reducing and/or removing nutrients from water (Kemp & Mudrochova 1972; Nurnberg 1994; Olila et al

1995; House & Warwick 1999; Rydin 2000; Golterman 2001; Hu et al 2001; Carpenter 2005; Fytianos & Kotzakioti 2005; Khan & Ansari 2005; Haggard & Soerens 2006; Moosmann et al 2006; Sen et al 2007; Lukkari et al 2007; Chao et al 2008; Kapanen 2008; da Silva 2009; Levin 2009; Wang & Lee 2010; Gaoa 2012; Sahin et al 2012; Wang 2012; Zhang et al 2012; Zhu et al 2013; Kangur et al 2013; Topçu & Pulatsü 2014; Ionescu et al 2015; Middelburg & Radu et al 2015; Tociu & Diacu 2015).

This paper shows the evaluation of water and sediment quality of an interior lake that does not communicate with any other surface water bodies. One has focused on the assessment of nutrients (i.e. ammonia, nitrates, nitrites, total nitrogen, total phosphorus, phosphates and chlorophyll *a*) as well as on the monitoring of other significant physicochemical indicators, such as temperature, pH, oxygen consumption (quantified as biochemical and chemical oxygen demand) and salinity (chloride, sulphates, magnesium and calcium content).

This paper is the first to describe the dynamics of the concentrations of nutrients and their speciation in sediments within the Circului Lake, Bucharest. The analysed phosphorus fractions from sediments include phosphorus bonded to iron and aluminium (Fe+Al~P), phosphorus bonded to calcium (Ca~P) and soluble and loosely sorbed phosphorus bonded to carbonate (CO₃~P). As to nitrogen fractions, one has analysed ammonium (N-NH₄), nitrite (N-NO₂) and nitrate (N-NO₃).

Material and Method. Circului Lake (44°27'25.752"-44°27'28.867"N; 26°6'41.923"-26°6'46.848"E) is an interior anthropic lake located in the central part of Bucharest, Romania, with a relatively small area (7500 m²). The lake does not communicate with other surface water bodies and is unique among other lakes from Bucharest by that it is the habitat for several plant and animal species, including some protected species such as *Nymphaea lotus* and *Emys orbicularis*. The lake has a maximum depth of 2.5 m, with an average depth of 1.5 m (www.alpa.ro). The lake is aerated during summer by means of fountains. Also, the excessive vegetation is removed periodically to avoid the emergence of hypoxic/anoxic conditions.

Water samples were taken during March, May, July and September 2015 from two sampling points (C1 and C2), as depicted in Figure 1. Sampling point C1 is located close to the promenade, while sampling point C2 was chosen due to its proximity to an aeration fountain employed mainly during summer. The samples were taken from the near-bottom water at a distance of 1.5 m from the lake shore.

Water indicators were analysed using adequate methods (SR EN 1899:2002; SR EN ISO 5814:2013; SR ISO 6060:1996; SR EN ISO 6878:2005; SR ISO 7150:2001; SR ISO 7890:2000; SR EN ISO 7980:2002; SR EN ISO 8467:2001; SR ISO 9297:2001; SR ISO 10260:1996; SR EN ISO 10523:2012; SR EN ISO 11905:2003; SR EN 26777:2002) and validated similar to procedures described elsewhere (Ionescu et al 2014).

From each sampling point, 15-cm sediment core samples were also taken. The sediment showed two distinctive layers, corresponding to the nature of the processes that take place within it, as follows: an upper layer consisting of freshly deposited organic matter (S1) and another layer deposited prior to the occurrence of hypoxic conditions (S2). In some cases, the later had a grey colour due to its deposition prior to eutrophication. Sediment samples were dried at room temperature, milled and sieved through a 90 µm-sieve. Total phosphorus and nitrogen as well as their fractions were analysed for each sediment layer.

Phosphorus fractions were analysed according to the procedure described in Figure 2, starting from the extraction procedures described in literature (Olila et al 1995; Rydin 2000; Kapanen 2008; Sahin et al 2012). A mixture of dried sediment and extraction agent was subjected to magnetic stirring (300 rpm) followed by filtration through a 0.45 µm cellulosic filter prior rinsed with boiled doubly distilled water. The filtrate was further subjected to direct colorimetric analysis at a wavelength of 715 nm, using the ammonium molybdate spectrometric method (SR EN ISO 6878:2005).

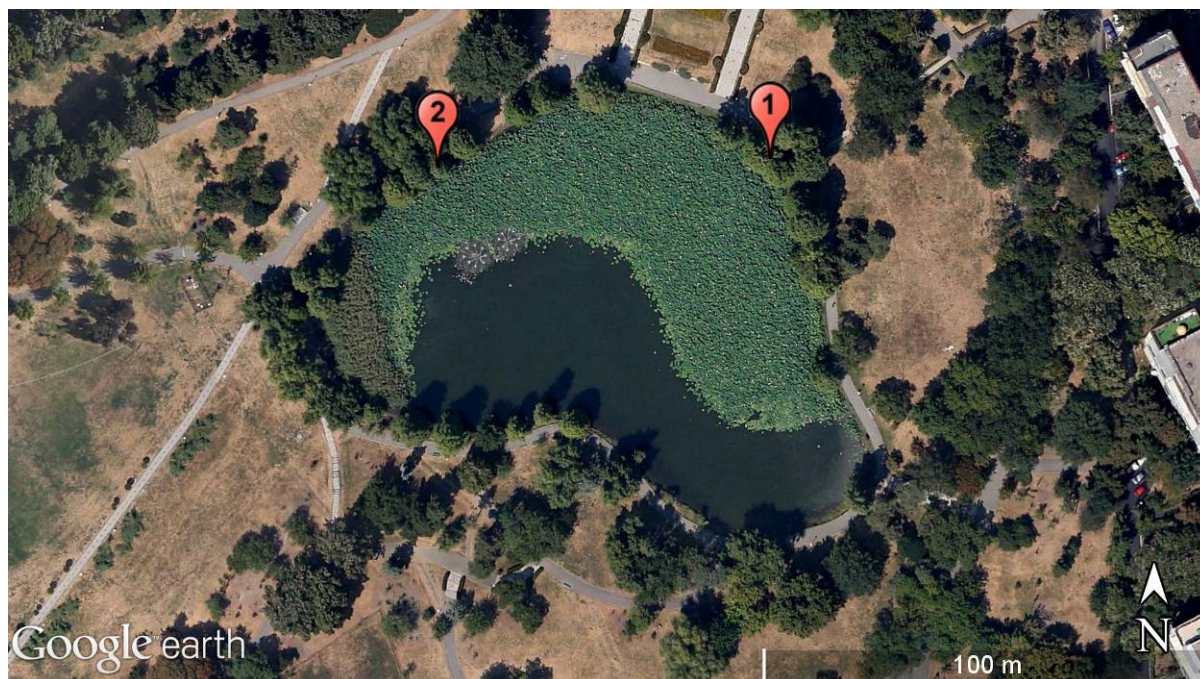


Figure 1. Aerial view of the sampling points location on the Circului Lake (earth.google.com).

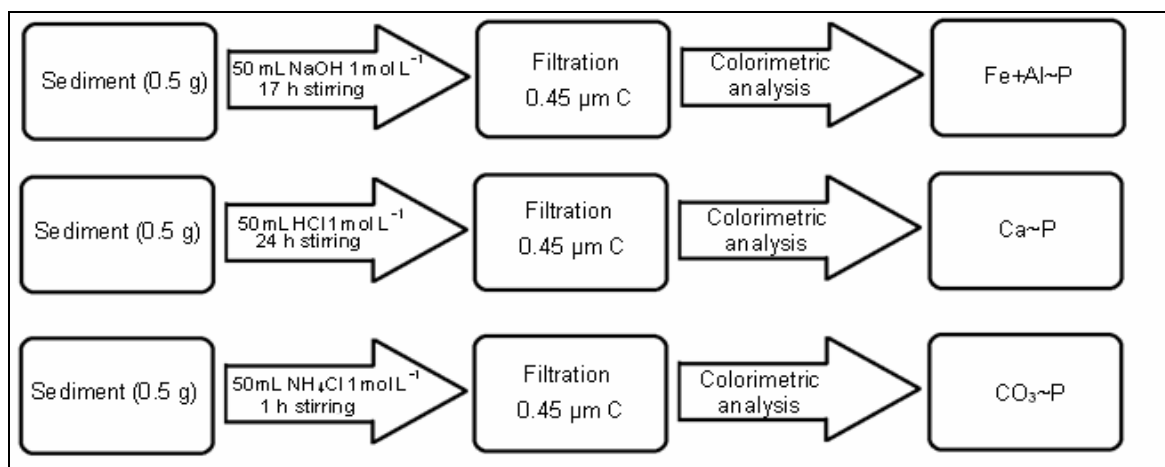


Figure 2. The extraction procedure used for the analysis of phosphorus fractions from the sediment (adapted from Sahin et al 2012).

The nitrogen fractions were analysed according to a standard method used for soil samples (SR ISO 14255:2000), depicted in Figure 3. The sediment sample was extracted with a $0.01 \text{ mol L}^{-1} \text{ CaCl}_2$ solution by stirring the solution for two hours at 1000 rpm, followed by filtration through a cellulosic filter rinsed with boiled doubly distilled water. The nitrogen fractions were analysed from the resulting filtrate, through direct colorimetric analysis, using specific agents and wavelengths.

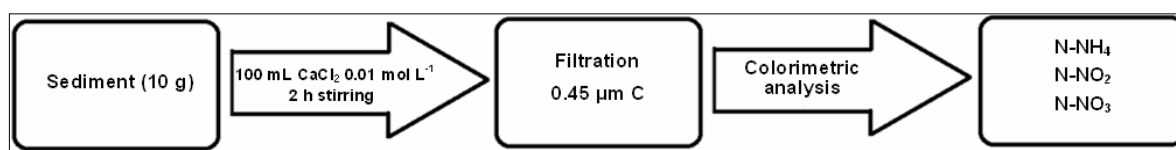


Figure 3. The extraction procedure used for the analysis of nitrogen fractions from the sediment.

Results and Discussion. Tables 1 and 2 show the values of the physicochemical indicators of water quality for the Circului Lake and the classification of water quality according to the Ministerial Order no. 161/2006.

Table 1

Physicochemical indicators monitored within C1 sampling point and the corresponding water quality classification (between brackets)

Physicochemical indicator (unit)	Value (quality class according to the Ministry Order 161/2006)			
	March	May	July	September
T (°C)	7.7	21.0	23.3	20.9
pH	7.36 (I)	7.73 (I)	7.39 (I)	7.33 (I)
DO (mg L ⁻¹)	7.66 (II)	4.45 (I)	3.25 (V)	1.29 (V)
BOD ₅ (mg L ⁻¹)	1.91 (I)	3.28 (II)	2.55 (I)	4.62 (II)
COD-Mn (mg L ⁻¹)	3.25 (I)	4.15 (I)	4.88 (I)	5.85 (II)
COD-Cr (mg L ⁻¹)	11.98 (II)	9.65 (II)	19.10 (II)	21.6 (II)
N-NH ₄ ⁺ (mg L ⁻¹)	0.04 (I)	0.02 (I)	0.04 (I)	0.33 (I)
N-NO ₂ ⁻ (mg L ⁻¹)	0.003 (I)	0.004 (I)	0.007 (I)	0.004 (I)
N-NO ₃ ⁻ (mg L ⁻¹)	0.03 (I)	0.02 (I)	0.04 (I)	0.02 (I)
TN (mg L ⁻¹)	0.47 (I)	1.46 (I)	0.82 (I)	0.76 (I)
P-PO ₄ ³⁻ (mg L ⁻¹)	0.04 (I)	0.01 (I)	0.03 (I)	0.1 (I)
TP (mg L ⁻¹)	0.09 (I)	0.05 (I)	0.11 (I)	0.24 (II)
Chl <i>a</i> (mg L ⁻¹)	8.3 (I)	15.2 (I)	21.4 (I)	50.0 (II)
Chlorides (mg L ⁻¹)	30.49 (II)	29.07 (II)	32.62 (II)	30.84 (II)
SO ₄ ²⁻ (mg L ⁻¹)	15.39 (I)	21.92 (I)	24.01 (I)	43.13 (I)
Mg ²⁺ (mg L ⁻¹)	17.51 (II)	26.27 (II)	48.62 (II)	82.65 (III)
Ca ²⁺ (mg L ⁻¹)	48.70 (I)	57.72 (II)	51.30 (II)	72.14 (II)

T – temperature; DO – dissolved oxygen; BOD₅ – biochemical oxygen demand; COD-Mn, COD-Cr – chemical oxygen demand; N-NH₄⁺ – ammonium; N-NO₂⁻ – nitrites; N-NO₃⁻ – nitrates; TN – total nitrogen; P-PO₄³⁻ – phosphates; TP – total phosphorus; Chl *a* – chlorophyll *a*; SO₄²⁻ – sulphates; Mg²⁺ – magnesium; Ca²⁺ – calcium.

Table 2

Physicochemical indicators monitored within C2 sampling point and the corresponding water quality classification (between brackets)

Physicochemical indicator (unit)	Value (quality class according to the Ministry Order 161/2006)			
	March	May	July	September
T (°C)	9.1	22.6	24.5	20.8
pH	7.41 (I)	7.73 (I)	7.4 (I)	7.46 (I)
DO (mg L ⁻¹)	9.02 (I)	5.82 (III)	3.02 (V)	3.55 (V)
BOD ₅ (mg L ⁻¹)	1.84 (I)	4.46 (II)	1.93 (I)	5.00 (II)
COD-Mn (mg L ⁻¹)	3.25 (I)	4.73 (I)	4.34 (I)	5.64 (II)
COD-Cr (mg L ⁻¹)	12.06 (II)	14.47 (II)	14.33 (II)	14.4 (II)
N-NH ₄ ⁺ (mg L ⁻¹)	0.04 (I)	0.03 (I)	0.06 (I)	0.17 (I)
N-NO ₂ ⁻ (mg L ⁻¹)	0.003 (I)	0.005 (I)	0.006 (I)	0.003 (I)
N-NO ₃ ⁻ (mg L ⁻¹)	0.04 (I)	0.06 (I)	0.05 (I)	0.02 (I)
TN (mg L ⁻¹)	0.23 (I)	0.91 (I)	0.87 (I)	0.991 (I)
P-PO ₄ ³⁻ (mg L ⁻¹)	0.04 (I)	0.02 (I)	0.05 (I)	0.1 (I)
TP (mg L ⁻¹)	0.08 (I)	0.07 (I)	0.11 (I)	0.22 (II)
Chl <i>a</i> (mg L ⁻¹)	17.6 (I)	21.4 (I)	27.3 (II)	49.3 (II)
Chlorides (mg L ⁻¹)	31.2 (II)	29.78 (II)	29.07 (II)	33.33 (II)
SO ₄ ²⁻ (mg L ⁻¹)	14.84 (I)	22.03 (I)	25.24 (I)	45.04 (I)
Mg ²⁺ (mg L ⁻¹)	9.72 (I)	26.27 (II)	46.19 (II)	75.36 (III)
Ca ²⁺ (mg L ⁻¹)	43.99 (I)	59.32 (II)	48.10 (I)	70.54 (II)

T – temperature; DO – dissolved oxygen; BOD₅ – biochemical oxygen demand; COD-Mn, COD-Cr – chemical oxygen demand; N-NH₄⁺ – ammonium; N-NO₂⁻ – nitrites; N-NO₃⁻ – nitrates; TN – total nitrogen; P-PO₄³⁻ – phosphates; TP – total phosphorus; Chl *a* – chlorophyll *a*; SO₄²⁻ – sulphates; Mg²⁺ – magnesium; Ca²⁺ – calcium.

Water quality falls mainly in the first and second quality class, except for dissolved oxygen during July and September (values lower than 4 mg O₂ L⁻¹), which may lead to the occurrence of hypoxic conditions.

Total phosphorus has slightly lower values in May, followed by an increasing tendency together with a decrease of dissolved oxygen and the occurrence of anoxic conditions. As to total nitrogen content, higher values were observed, with a decreasing tendency towards mid-autumn, due to intense phytoplankton activity.

As to the trophic characterization of the lake, the same Ministry Order was used (Table 3). The results show that the status of the lake shifts from eutrophic during spring to hypertrophic at the end of the summer, despite the remediation solutions undertaken by the local authorities, namely lake cleaning and lake aeration by means of fountains.

Table 3

The trophic status of the Circului Lake

<i>Indicators</i>	<i>Sampling point</i>	<i>Trophic status according to the Ministry Order 161/2006</i>			
		<i>March</i>	<i>May</i>	<i>July</i>	<i>September</i>
Total phosphorus	C1	eutrophic	eutrophic	hypertrophic	hypertrophic
	C2	eutrophic	eutrophic	hypertrophic	hypertrophic
Total nitrogen	C1	mesotrophic	eutrophic	eutrophic	eutrophic
	C2	olygotrophic	eutrophic	eutrophic	eutrophic
Chlorophyll <i>a</i>	C1	eutrophic	eutrophic	eutrophic	hypertrophic
	C2	eutrophic	eutrophic	hypertrophic	hypertrophic

In order to establish the structure of the microbial communities and to quantify the contribution of microorganisms to the nitrogen cycle within the sediment, one has determined the density of some specialised microorganisms (Tables 4 and 5), namely proteolytic and ammonifying microorganisms, nitrate and nitrite bacteria and denitrifying bacteria, corresponding to the four stages of the biochemical cycle of nitrogen (i.e. nitrogen fixation; proteolysis and ammonification; nitrification and denitrification).

The density of proteolytic microorganisms shows an increasing tendency from spring to mid-autumn, which confirms the dependence of this group to the autochthonous organic matter.

Ammonifying bacteria deaminate the resulting products of proteolysis, as well as the resulting products of hydrolysis of nucleic acids and urea. The proteolytic and ammonifying microorganisms are constantly present within the lacustrine ecosystem. The results of this study show high values of their density during the entire year, with higher density of ammonifying bacteria during the warm season and at the beginning of the autumn, due to the accumulation of plant debris.

As to nitrite and nitrate bacteria, the results show higher density of the latter, as a result of the immediate oxidation of the nitrites generated by the nitrite bacteria. Seasonal variation of nitrate bacteria was similar to the variation of ammonifying bacteria, with more intense activity during the summer and at the beginning of the autumn, with maximum values during the autumn.

The results of this study show low density of denitrifying bacteria, which may lead to the increase of nitrogen fractions within the aquatic ecosystem and to a poorer efficiency of the complete oxidation of organic matter and of the release of gaseous nitrogen in the air.

The high densities of ammonifying and nitrifying bacteria show that the processes of hydrolysis and oxidation of ammonia are detrimental to the reduction of organic matter, which may lead to the formation of nitrogen and nitrogen oxides.

Table 4

Density of microorganisms involved in the biochemical cycle of nitrogen within the water from the Circului Lake

Microbial community	Sampling point	Density within water, no L ⁻¹			
		March	May	July	September
Proteolytic microorganisms	C1	161000	172000	221000	54200
	C2	17200	34800	542000	348000
Ammonifying microorganisms	C1	348000	278000	348000	542000
	C2	348000	542000	345000	1610000
Nitrite bacteria	C1	34800	4600	5420	9200
	C2	170	2210	1720	1720
Nitrate bacteria	C1	2600	54200	175000	920000
	C2	220	92000	109000	542000
Denitrifying bacteria	C1	1100	490	3480	5420
	C2	2210	790	1800	3480

Table 5

Density of microorganisms involved in the biochemical cycle of nitrogen within the sediments from the Circului Lake

Microbial community	Sampling point	Density within sediment, no g ⁻¹			
		March	May	July	September
Proteolytic microorganisms	C1	320	1453	4639	4599
	C2	722	933	1880	214480
Ammonifying microorganisms	C1	12530	18795	294593	459885
	C2	18795	22928	722486	1226360
Nitrite bacteria	C1	2293	2946	4639	122636
	C2	3199	3706	7225	122636
Nitrate bacteria	C1	22928	23328	214480	214480
	C2	3706	14530	122636	214480
Denitrifying bacteria	C1	1053	1880	2399	7225
	C2	2293	2946	2399	7225

Among the factors that affect the eutrophication process, nutrients are of a paramount importance. Thus, one has also assessed the content and the dynamics of nutrient in water and sediments.

Figures 4 and 5 depict the total phosphorus content and phosphorus fractions and total nitrogen and nitrogen fractions within the two distinctive layers of sediment taken from the C1 sampling point. Figures 6 and 7 depict the total phosphorus content and phosphorus fractions and total nitrogen and nitrogen fractions within the two distinctive layers of sediment from the C2 sampling point.

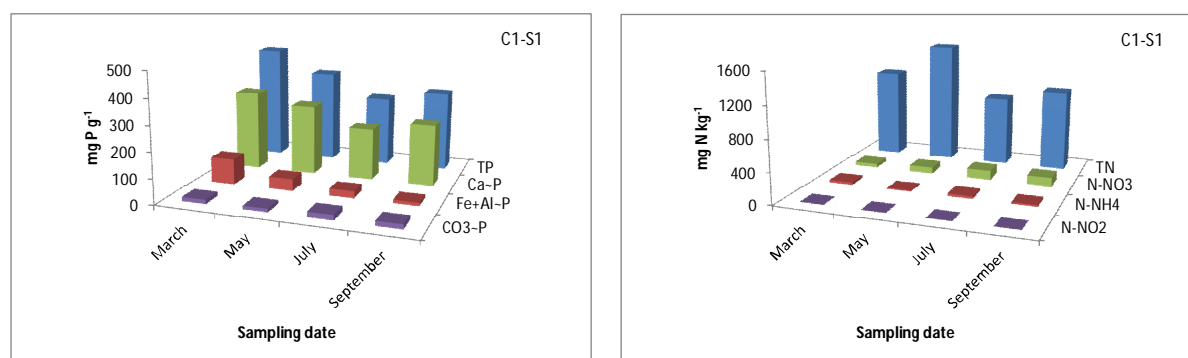


Figure 4. Total phosphorus and phosphorus fractions and total nitrogen and nitrogen fractions in the upper (S1) layer of sediment samples from the C1 sampling point.

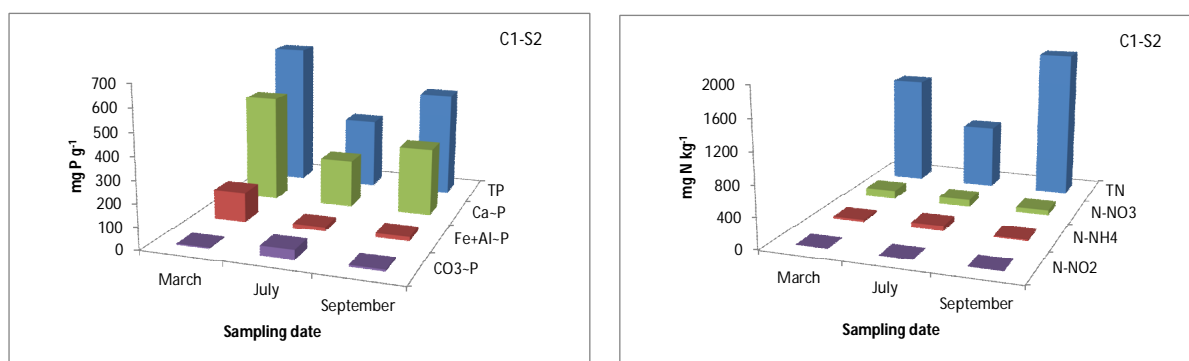


Figure 5. Total phosphorus and phosphorus fractions and total nitrogen and nitrogen fractions in the lower (S2) layer of sediment samples from the C1 sampling point.

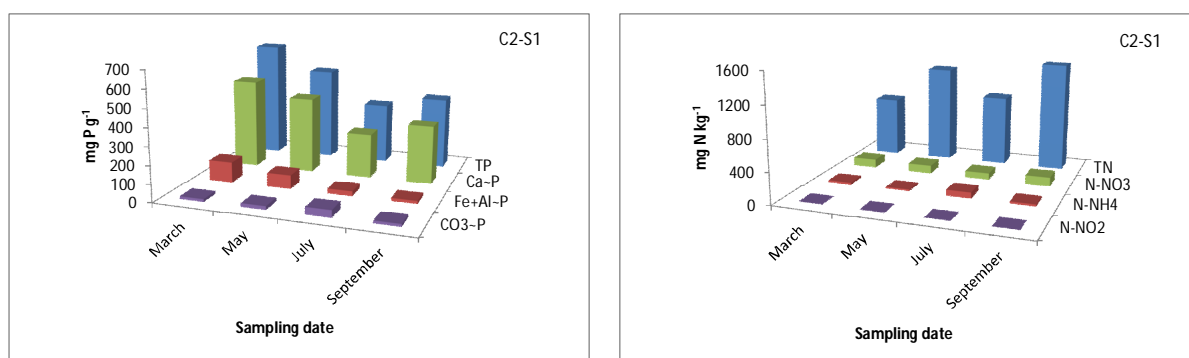


Figure 6. Total phosphorus and phosphorus fractions and total nitrogen and nitrogen fractions in the upper (S1) layer of sediment samples from the C2 sampling point.

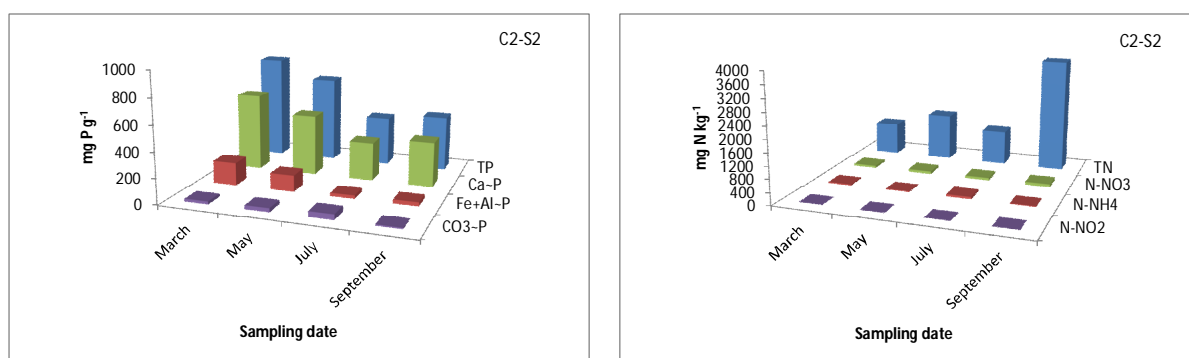


Figure 7. Total phosphorus and phosphorus fractions and total nitrogen and nitrogen fractions in the lower (S2) layer of sediment samples from the C2 sampling point.

Sediment samples exhibited a significant spatial and seasonal variability, with the lowest nutrients concentrations during July due to abundant vegetation and to a decrease of dissolved oxygen content, as the occurrence of hypoxic conditions may cause the release of nutrients in the near-bottom water.

Total phosphorus and nitrogen content is higher in the lower layer of the sediment core as a result of microbial processes that take place mainly in the upper layer. Moreover, the upper layer consists mainly of freshly deposited plant debris.

Results show that sediments are characterized by high contents of phosphorus bonded to calcium (Ca~P), which represents as much as up to 81% of the total phosphorus content of the sediments, regardless of the core depth. According to literature data, this fraction consists mainly of apatite and it is sensitive to low pH values (Kapanen 2008; Sahin et al 2012). The content of phosphorus bonded to calcium is highly dependent to the dynamics of the vegetation and to the dissolved oxygen regime, with high values during the spring and lower values during the summer.

The lowest concentrations were observed for the phosphorus fraction bonded to carbonates ($\text{CO}_3\sim\text{P}$), which represents only up to 13.1% of the total phosphorus content of the sediments. This is the loosely adsorbed phosphorus and is prone to desorption. Its low values may be attributed to a continuous dissolution process that takes place at the sediment-water interface. Results show an increase of the content of loosely adsorbed phosphorus during the warm season (July) followed by a decrease in the cold season.

The results of our study also highlighted a slight difference between the total phosphorus and phosphorus fractions, which may be attributed to organic phosphorus.

As to nitrogen, the results revealed a higher difference between total nitrogen and its fractions that may be attributed to the higher solubility of nitrogen fractions compared to phosphorus fractions and to their continuous generation due to microbial processes that take place within the sediment. These observations are in agreement with the analysis of the microbial communities within the sediment, with high densities of nitrate bacteria, which revealed the occurrence of hydrolysis and oxidation processes, with the formation of nitrogen and nitrogen oxides.

The organic forms of nitrogen proved to be the most abundant nitrogen fraction within sediments, regardless of the core depth. As to inorganic forms, nitrates represent as much as up to 14.4% of the total nitrogen content.

Conclusions. This study shows the assessment of water and sediment quality of the Circului Lake, Bucharest, and is the first to describe the dynamics of nutrients within the sediment layers.

The results show that water quality indicators fall within the first and second quality class, except for dissolved oxygen during July and September, which falls in the fifth quality class. As to the trophic status, the lake shifts from eutrophic during spring to hypertrophic in the autumn.

Sediment samples exhibited a significant spatial and seasonal variability, with the lowest nutrients concentrations during July due to abundant vegetation and to a decrease of dissolved oxygen content, as the occurrence of hypoxic conditions may cause the release of nutrients in the near-bottom water.

The analysis of nutrients fractions in sediment as well as total phosphorus and nitrogen content showed a significant spatial and seasonal variability of the sediment samples, with the lowest nutrients concentrations during July. As to phosphorus fractions, the lowest concentrations were recorded for the phosphorus fraction bonded to calcium (up to 81% of the total phosphorus content).

The analysis of bacterial communities present in water and sediment showed that the processes of hydrolysis and oxidation of ammonia are detrimental to the reduction of organic matter, which may lead to the formation of nitrogen and nitrogen oxides.

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