

# Estimates of benthic fluxes of nutrients across the sediment–water interface (Guarapiranga reservoir, São Paulo, Brazil)

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## Abstract

Concentration profiles of nutrients (dissolved organic carbon, nitrate, nitrite, ammonium and soluble reactive phosphorus) were determined in pore waters from sediment from the Guarapiranga reservoir (São Paulo, Brazil). Redox potential and acid volatile sulfide measurements on bulk sediment samples were determined in the field and laboratory, respectively. The sediment redox potential ranged from  $-170$  to  $-220$  mV at 0–1 cm and increased to somewhat higher values at 20 cm. The acid volatile sulfide (AVS) profile had a bimodal pattern with concentration peaks at 3 cm ( $27\text{--}55$  mg kg $^{-1}$ ) and 14 cm ( $70\text{--}110$  mg kg $^{-1}$ ). Dissolved organic carbon (DOC) concentrations increased from the surface ( $4.7\text{--}5.6$  mg l $^{-1}$ ) to 20 cm (values up to  $12$  mg l $^{-1}$ ). The concentration of ammonium increased significantly with depth, with maximum concentrations occurring at 15 cm; nitrate–nitrite concentrations only increased appreciably at 10 cm. The SRP profiles increased in concentration from the surface to approximately 10-cm depth, with a maximum value of  $1200$   $\mu\text{g H}_2\text{PO}_4^-$  l $^{-1}$ . Benthic fluxes from the sediment into the pore water ranged from  $278$  to  $339$  mg cm $^{-2}$  year $^{-1}$  for ammonium ions and from  $8$  to  $18$  mg cm $^{-2}$  year $^{-1}$  for SRP. These upward diffusive fluxes correspond to 47–70% and to 10–24% of the total deposition of N and P measured in the reservoir, respectively. The burial rates for N and P in these sediments are 30–54% and 76–89%, respectively. © 2001 Elsevier Science B.V. All rights reserved.

**Keywords:** Benthic flux; Sediment–water interface; Redox potential; Dissolved organic carbon (DOC)

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## 1. Introduction

Reservoirs located in highly populated and industrialized regions of the world receive discharges of organic xenobiotics, heavy metals and nutrients that have great potential to impair water quality and threaten aquatic life. Guarapiranga reservoir is located in the metropolitan area of São Paulo city, São Paulo State, Brazil (23° 41' S and 46° 43' W) and is an important drinking water reservoir for the city, providing 25% of its water needs. The reservoir was built in 1906–08 and has an area of 3618 ha with a total water volume of  $194 \times 10^6 \text{ m}^3$  and a mean depth of 7 m (Takino and Maier, 1981). The reservoir has been reported as eutrophic since ca. 1960 based on the discharge of untreated urban sewage (Rocha, 1976); the high primary productivity may classify it as hypereutrophic at sites near tributary discharges (Caleffi et al., 1994). Total heavy metal and nutrient concentrations in the sediments show strong anthropogenic contamination (Mozeto et al., 1998; Patella, 1998). Heavy metal and nutrient concentrations progressively increase in the top 15 cm, which corresponds to the onset of the Brazilian industrial revolution (around 1950).

This study was carried out to provide information on nutrient concentrations in the sediment pore water of Guarapiranga reservoir, to determine the role of the sediment as a source or sink of nutrients, and to assess how sediment release of nutrients and contaminants may be affecting water quality.

## 2. Materials and methods

### 2.1. Sample collection for concentration profiles of nutrients in sediment pore water

Samples were collected in May 1998 from sites GU-101 (close to the dam) and GU-104 (central part), located along the long axes of the reservoir. The water depth at the sites was 18 and 12 m, respectively. Six sediment cores were collected using a transparent, hand operated Plexiglas pis-

ton corer (10 cm diameter; 50 cm height) (Ambuhl and Buher, 1975). The core liners were 50 cm long and recovered approximately 30–40 cm of undisturbed sediment. After retrieval, the cores were kept upright and tightly closed with plastic stoppers to minimize disturbance during transport by boat to the reservoir shore. Cores were immediately subsampled into 1-cm sections using a mechanical hand-operated extruder from the surface layers up to a 20-cm depth. The subsamples from different depths of each core were collected in plastic bags (high-density zip lock bags). The subsampling was conducted under a constant flux of pure gaseous nitrogen to minimize sediment oxidation. Subsampling was carried out in a Plexiglas glove box in which the core pipe was introduced through a bottom door. Zip lock bags were also flushed with nitrogen before being sealed and stored and kept refrigerated at 4°C in a cooler with ice cubes during transport to the laboratory.

### 2.2. Water sampling from the water column ('sediment–water column interface')

Samples of the overlying water were collected at three depths above the sediment–water interface (2, 6 and 10 cm) using a syringe and silicon tubing and taken from the top part of the Plexiglas corer before sediment extrusion (also while flushing with nitrogen as described above). Samples were immediately filtered under  $\text{N}_2$  through a cellulose membrane (0.45  $\mu\text{m}$ ). Different sub-fractions were saved and preserved for analysis of chemical species as follows:

1. dissolved organic carbon (DOC) analyses: 5 ml of 10% phosphoric acid added /100 ml of sample and preserved at 4°C;
2. total dissolved nitrogen (TDN) and soluble reactive phosphorus (SRP) analyses: no preservative added and stored at 4°C; and
3. acid volatile sulfide (AVS) analyses: 5 ml of 40% w/v of zinc acetate added and preserved at 4°C.

### 2.3. Sample collection for AVS and $E_H$ profiles on total sediment

The same protocol as above was adopted. The AVS was determined in the laboratory (see below) while  $E_H$  was measured in the field under  $N_2$  flushing (as described above) with a combined Ag/AgCl and Pt (s) electrode connected to a potentiometer using Zobell solution for calibration.

### 2.4. Pore water extraction and DOC, TDN, SRP and AVS analyses

Sediment pore water samples were extracted by centrifugation at 4°C while flushing with  $N_2$ , filtered immediately through a cellulose membrane (0.45  $\mu\text{m}$ ) and then preserved as described above. Membranes were dried initially for 2 h at 40°C and then pre-washed by filtering with 100 ml MilliQ® water (previously distilled and deionized). DOC was determined using a Shimadzu TOC-5000 as the difference between total carbon and dissolved inorganic carbon. Standard curves were prepared with analytical grade salts (potassium acid phthalate and sodium bicarbonate). The TDN speciation was carried out colorimetrically via a flow injection analysis (FIA) system according to the following techniques:  $\text{NO}_3^-$ , reduction of nitrate to nitrite via the Griess reaction where  $\text{NO}_2^-$  is determined by difference after the determination with and without a metallic Cd column;  $\text{NH}_4^+$ , Nessler reaction (Zagatto et al., 1981). The SRP was analyzed colorimetrically in a FIA system using the molybdenum blue method (Zagatto et al., 1981). The AVS was also colorimetrically determined in a FIA system using the methylene blue method modified by Mozeto et al. (1989) after acid extraction (HCl 6 M) in a closed line using  $N_2$  (g) as a carrier gas. This procedure presents a recovery rate for AVS of 83–90% in the concentration interval 0.1–1.5 ppm.

### 2.5. Water content of bulk sediment

The water content in sediment samples was gravimetrically determined by weight difference

on wet samples dried in an oven at 105°C for 6 h (Hakanson and Hansson, 1983).

## 3. Results and discussion

The water content of the sediment decreased from 87.2% (w/w) at the surface to 67.1% (w/w) at a 20 cm depth, and from 90.7% (w/w) at the surface to 71.7% (w/w) at a 20 cm depth for sampling points GU-101 and GU-104, respectively. The average content of total organic carbon, total nitrogen and total phosphorus ranged from 4.5–2.6, 0.76–0.31 and 0.18–0.08% for GU-101, and from 5.0–2.4, 0.90–0.33 and 0.18–0.07 for GU-104, respectively (Patella, 1998).

### 3.1. Concentration profiles of nutrients, AVS and $E_H$

The concentration profiles of all nutrients in the sediment pore water, as well as AVS and  $E_H$  in the bulk sediment, are shown in Figs. 1 and 2.

The redox status of natural sediment–water systems is one of the most difficult variables to determine, as these systems are seldom at equilibrium. The  $E_H$  is thermodynamically defined, and when measured in the field corresponds to the dominant redox couple present in the system (Grundl, 1995). Frequently the  $E_H$  values measured with a Pt electrode differ from computed values, suggesting that the differences are a result of various factors (misbehavior of the electrodes; irreversibility or slow kinetics of reactions; existence of mixed potentials) (Langmuir, 1997).

Although the natural complexity of reservoir sediments applies here, some conclusions/observations can be made. First, the  $E_H$  and AVS data collected on bulk sediment (Fig. 1a, b) confirm that the reservoir is anaerobic. Second, the sediment–water interface of the Guarapiranga reservoir can be classified (according to Berner, 1981) as an anoxic sulfidic environment. The AVS profiles display basal concentrations that are at, or somewhat above, the value of  $10^{-6}$  M  $\text{H}_2\text{S}$ , which has been suggested as the limiting concentration separating non-sulfidic and sulfidic systems (Berner, 1981). The average concentration

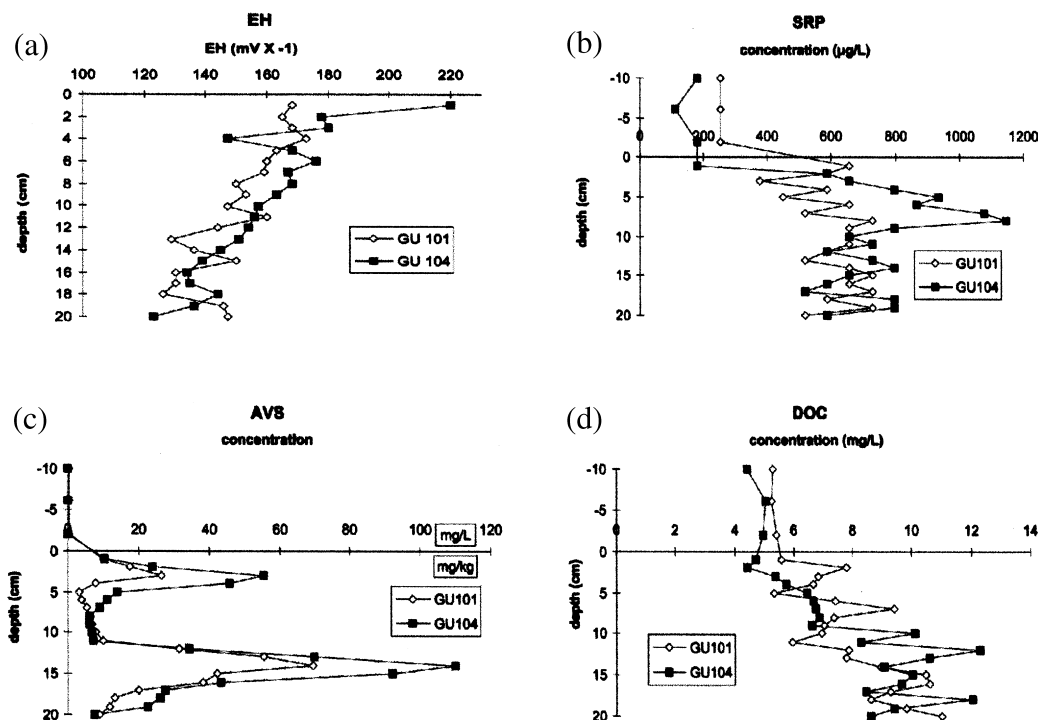


Fig. 1. Redox potential ( $E_H$ ) (a) and acid volatile sulfide (AVS) (b) determined on bulk sediment and concentrations of soluble reactive phosphorus (SRP) (c) and dissolved organic carbon (DOC) (d) from sediment pore water of Guarapiranga reservoir.

of  $3.7 \cdot 10^{-6}$  M  $H_2S$  in the water column at the sediment–water interface (see Fig. 1b) confirms this. The AVS concentration peaks (ranging from 27–110  $mg\ kg^{-1}$  or  $0.78$ – $3.2 \cdot 10^{-3}$  M) shown in Fig. 1b are two to three orders of magnitude larger than the basal value defined by Berner's classification as the 'measurable concentration of

total dissolved sulfide' using classical techniques, corresponding to 0.1–1.0% of the total dissolved sulfide found in a typical nearshore sulfidic sediment (Berner, 1981). As the redox status of sediment–water systems is one of the major factors influencing speciation and mobility of heavy metals, it follows that the concentration of AVS and

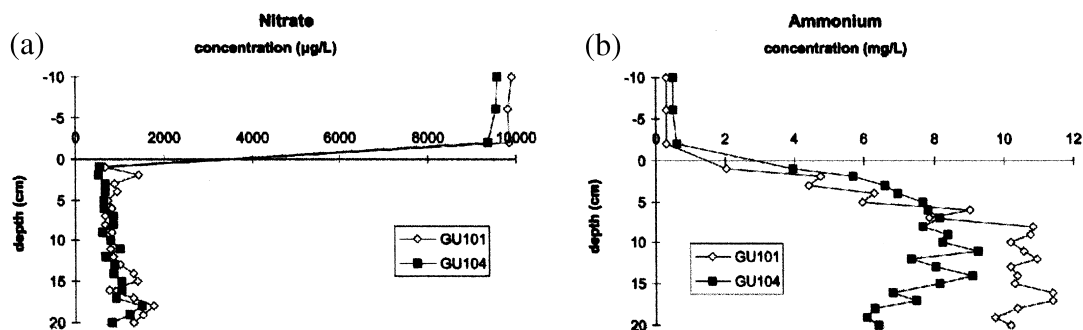


Fig. 2. Concentrations of nitrate (a) and ammonium ions (b) from sediment pore water from Guarapiranga reservoir.

value of the redox potential in the reservoir will play a major role in controlling the distribution and, consequently metal toxicity, and is currently being investigated.

Profiles of DOC in the sediment pore waters (Fig. 1d) are very similar at the two sampling sites. Although there is no DOC enrichment at the surface sediment (enrichment factor is approximately three times that of the overlying water), both sites show an increase in concentration with depth, reaching values as high as  $12 \text{ mg l}^{-1}$  at approximately 20 cm. As expected, based on the relatively elevated concentration of total organic carbon (TOC) existing in the sediment (average concentration of 3.6%, although there is a decrease in TOC with increasing depth) (Patella, 1998), pore waters are, in general, richer in DOC than the overlying water, which, on average, has a concentration of  $5.1 \text{ mg l}^{-1}$ .

A pattern similar to the DOC profiles was observed for SRP (Fig. 1c). Surface pore waters (depth 0–1 cm) are, on average, approximately three, seven and 75 times richer in SRP, ammonium ions (Fig. 2b) and AVS, respectively than sediment–water column interface waters (depth interval of 0–2 cm). Nitrate concentrations (Fig. 2a) show a reverse trend, with the overlying water concentrations being 16 times greater than the pore waters (nitrite, not shown in this figure, is 190 times higher).

Except for the AVS differences between sampling locations, practically no enrichment or depletion was detected in the other measured species, although the distance between sites is only 2 km. Sampling point GU-104 showed an enrichment factor for AVS 6 times above that of GU-101. However, potential fluxes of sulfide are equivalent between these two sites.

### 3.2. Estimates of nutrient fluxes

The vertical diffusional flux of dissolved nutrients from sediments to the overlying water may be calculated from the pore water dissolved gradients  $(dC/dZ)_{Z=0}$  across the sediment–water in-

terface, according to Fick's law in one dimension (Azcue et al., 1996) represented by Eq. (1) below:

$$F = -\Phi D_s(\Delta C/\Delta Z) \quad (1)$$

where  $F$  is the diffusive flux of the nutrient species ( $\text{mg cm}^{-2} \text{ year}^{-1}$ ),  $\Phi$  is the porosity of that species, and  $D_s$  is the diffusion coefficient in pore water ( $\text{cm}^{-2} \text{ year}^{-1}$ ). We have used the assumptions of Azcue et al. (1996) regarding the position of the sediment–water interface, viscosity and the effects of charge coupling, precipitation and biogenic uptake of dissolved species and concentration. The porosity was assumed to average 0.91 (91%) for the entire sediment profile (0–20 cm), which is a typical value for lakes with a relatively high primary productivity (Smith and Fisher, 1986). The diffusion coefficient ( $D_s$ ) was estimated from the empirical relationship  $D_s = \Phi^n D$  (Lerman, 1979), where  $n$  is a constant ( $n = 2$ ) and  $D$  is the diffusion coefficient at infinite dilution corrected by the Stokes–Einstein relation (Li and Gregory, 1974) at the in situ temperature of  $18^\circ\text{C}$  measured at the sediment–water column interface.

The estimated upward fluxes of nutrients across the sediment–water interface are shown in Tables 1 and 2 and are typically bi-directional for some of the species such as SRP (assumed to be  $\text{H}_2\text{PO}_4^-$  due to the low pH found in the pore water) and ammonium (see Fig. 1c Fig. 2b, respectively) which are transported from layers of high concentrations downwards to deeper layers (i.e., from approx. 5 cm to between 10 and 15 cm deep, to layers underneath); they also migrate across the sediment–water interface into the overlying water column. As anticipated, given the anaerobic status at the sediment–water interface and in the deeper layers (see Fig. 1a,b for  $E_H$  and AVS profiles), the sediments are a sink for nitrate (see Fig. 2a), as well as for nitrite (not shown in this figure).

The sediments are also a source of DOC because of the strong gradients between sediment pore water and the water column (see Fig. 1d). The DOC concentration increases with depth to a maximum value of  $12 \text{ mg l}^{-1}$ , which is much

Table 1

Positive (water column → sediment) and negative (sediment → water column) fluxes of dissolved species of nutrients across the sediment–water interface at the Guarapiranga reservoir (GU-101)

Species	Depth $C_{\max}$ (cm)	$\Delta C/\Delta Z$	$Ds$ ( $\text{cm}^{-2} \text{ year}^{-1}$ )	Flux ( $\text{mg cm}^{-2} \text{ year}^{-1}$ )
$\text{NH}_4^+$	11.5	0.848	438.7	– 338.5
$\text{NO}_3^-$	0.5	367.8	420.0	+ 140.6
$\text{NO}_2^-$	0.5	14.7	339.1	+ 4.5
SRP ( $\text{H}_2\text{PO}_4^-$ )	8.5	45.3	186.5	– 7.7
AVS ( $\text{HS}^-$ )	3.5	4.8	386.1	– 1.6

higher than the range of 4–5  $\text{mg l}^{-1}$  measured for the interface waters. It is not possible to calculate a flux for DOC since the speciation of charged species of the DOC is unknown.

Because of the nature of the AVS (i.e. sulfide forms will be released from sediments by dissolution in a very acidic medium only), the calculated flux must be seen as a latent or potential pool of sulfides existing in the sediments. This pool represents the pool of sulfides that are volatile or soluble after the addition of a strong acid (HCl 0.1 M) (e.g. Allen et al., 1993). These mineral phases (actually heavy metal sulfides) are recognized as the fraction that controls the bioavailability of heavy metals in the aquatic system. Recently this procedure has been questioned, as some of these mineral phases are insoluble in an acidic medium (Cooper and Morse, 1998). The estimated flux of AVS should be regarded as indicative of the potential of the sediment to maintain reducing conditions at the sediment–water interface. AVS can therefore play a significant role in the storage and bioavailability of

heavy metals and particularly in the cycling of nutrients across the sediment–water interface.

### 3.3. Upward diffusive fluxes versus depositional rates of nutrients

From Table 3, the average (sampling points GU-101 and -104) calculated diffusive fluxes from the sediment to the overlying water column for N (as  $\text{NH}_4^+$ ) and P (as  $\text{H}_2\text{PO}_4^-$ ) were 308.2 and 12.5  $\text{mg cm}^{-2} \text{ year}^{-1}$ , respectively, while the depositional fluxes (in terms of total N and P) were 565.0 and 73.0  $\text{mg cm}^{-2} \text{ year}^{-1}$ , respectively. On average, the amount of N and P deposited and buried in the sediment corresponds to approximately 56.0 and 17.2%, and 42.1 and 82.9%, respectively. The release of dissolved nitrogen and ammonium from the sediments to the water column varied from 46–70% (sampling points GU-101 and -104, respectively) of the total amount of nitrogen deposited to the sediment

Table 2

Positive (water column → sediment) and negative (sediment → water column) fluxes of dissolved species of nutrients across the sediment–water interface at the Guarapiranga reservoir (GU-104)

Species	Depth $C_{\max}$ (cm)	$\Delta C/\Delta Z$	$Ds$ ( $\text{cm}^{-2} \text{ year}^{-1}$ )	Flux ( $\text{mg cm}^{-2} \text{ year}^{-1}$ )
$\text{NH}_4^+$	11.5	0.696	438.7	– 277.9
$\text{NO}_3^-$	0.5	351.8	420.0	134.5
$\text{NO}_2^-$	0.5	15.1	339.1	4.7
SRP ( $\text{H}_2\text{PO}_4^-$ )	8.5	91.9	186.5	– 17.3
AVS ( $\text{HS}^-$ )	3.5	10.1	386.1	3.5

Table 3

Upward dissolved fluxes of  $\text{NH}_4^+$  and  $\text{H}_2\text{PO}_4^-$  and depositional fluxes of total N and P at the Guarapiranga reservoir (GU-101 and -104) (Patella, 1998)

Sampling points	Diffusive flux ( $\text{mg cm}^{-2} \text{ year}^{-1}$ )		Depositional flux ( $\text{mg cm}^{-2} \text{ year}^{-1}$ )		Deposition (%)		Burial (%)	
	N	P	N	P	N	P	N	P
GU-101	338.5	7.7	730	73	46.4	10.6	53.6	89.4
GU-104	277.9	17.3	400	73	69.5	23.7	30.5	76.3

surface. Therefore, an amount varying from 30–54% is buried.

As suggested by Azcue et al. (1996), it is difficult to calculate the contemporary fluxes of elements incorporated into the sediment because of mixing and diagenetic processes at the sediment–water interface. In Table 3, there appears to be a balance between N (in form of  $\text{NH}_4^+$ ) and P (in the form of  $\text{H}_2\text{PO}_4^-$ ) which diffuse back from sediments to the water column and the depositional fluxes of total N and P obtained from suspended particulate traps determined by Patella (1998). Acceptance of these comparisons requires there to be an essential difference between the deposition and incorporation processes. For heavy metals for instance, incorporation results from the scavenging of elements by suspended particulates, hydrolysis to sedimentable forms, or biological assimilation (Azcue et al., 1996), which may in part be also true for the incorporation of nutrients to sediments.

With respect to phosphorus, there is a larger burial rate than for nitrogen: 76–89% is buried as the flux to the water column varied from 24 and 11% of the total amount deposited on surface sediments. Fluxes of SRP calculated in this study are similar to those of Azcue et al. (1996) for Lake Erie, particularly at GU-104 ( $17.3 \text{ mg PSR cm}^{-2} \text{ year}^{-1}$  vs.  $16.8 \text{ mg PSR cm}^{-2} \text{ year}^{-1}$ ).

It is important to keep in mind that these fluxes are conservative estimates, as they were calculated considering molecular diffusion only. It should also be mentioned that the estimates of nutrient fluxes presented here should be considered as potential nutrient fluxes, because they were calculated based on the maximum concentrations found in the sediments (concentration

peaks). Perhaps more realistic fluxes should be those calculated based on the concentration gradient existing at the sediment–water interface. However, one has to bear in mind that the concentration peaks fall at depths lower than 10 cm, which is very close to that interface. Total benthic fluxes would, however, be higher, as many studies have demonstrated. Usually these are measured in situ using bottom chambers. Since our data represent only two sampling points on one collection date, significant spatial and temporal variations may occur, and extrapolation to the whole Guarapiranga reservoir over the year should be made with caution.

An evaluation of the incremental phytoplankton productivity resulting from the release of SRP to the water column is presently being carried out for the Guarapiranga reservoir. Although such a calculation would require several assumptions, it could be anticipated that this release would amount to only a minor part of the total SRP uptake by algae, considering the extremely high external load of total phosphorus to the reservoir.

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