

The distribution of phosphate in sediments and its relation with eutrophication of a Mediterranean coastal lagoon

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Abstract

A major problem of the Mediterranean coastal lagoons is an excessive input of nutrients (*i.e.* N and P), causing eutrophic conditions in summer. The sediments of these lagoons can serve as a reservoir by fixing phosphate, or as a source when this phosphate is released under certain conditions. Knowledge of nutrient sources and fluxes is needed if coastal lagoons are to be protected against eutrophication. Therefore, we have evaluated the total pool of phosphate in the lagoon sediments, and the quantity of phosphate which may be released.

Sediment profiles have been analysed at two stations of the 'Lagune de Thau' both in and outside the oyster-bank zone. A sequential fractionation, using chelating agents was performed to extract the inorganic (iron and calcium bound phosphate) and the organic phosphate fractions. A statistical analysis of the data set has revealed several significant factors which explain the fluctuations of the concentrations of each phosphate fraction. These factors are: the time of year (seasons), the depth (5 cm layers of sediment), and the site (station).

A spatial and a temporal variation of the concentration of Tot-P was found. The largest variation between the two different zones appeared only in the first five cm of sediment. There is only a slight seasonal variation in the amount of phosphate at other depths at the two different zones. Season and station are the factors which control the variations in distribution of phosphate fractions. The spatial and temporal variations of the iron and calcium bound phosphate are explained by the redox potential and pH in the top layer of the sediment.

Abbreviations In principle the general abbreviations are used (see page vi). Furthermore:

Fe(OOH)-P = Ferric hydroxide bound phosphate

CaCO₃-P = Calcium carbonate bound phosphate

ASOP = Acid soluble organic phosphate

ROP = Residual organic phosphate

Introduction

Near the coast of the Mediterranean sea are several shallow lagoons, which form a transition zone between continent and sea. The rate of water renewal of these lagoons is very low. Water exchange with the Mediterranean sea is limited, via narrow canals; because of this low water exchange the ecosystem is susceptible to urban and agricultural pollution.

In summer the lagoons have a problem of acute eutrophication: an excess input of nutrients creates algal blooms; the subsequent degradation of organic matter produced is enhanced by the season's high temperature (25 °–30 °C water temperature). O₂ is then rapidly depleted and the lagoon water may become turbid and smell of H₂S. The growth of sulfur bacteria may even result in the water being red.

Besides the exogenous input, there is also a supply by recycling of endogenous phosphate fixed previous-

ly in the sediment. Particulate phosphate in sediment is organic or forms inorganic complexes with iron or calcium (Williams, 1976). It is a source as it releases phosphate under certain well-defined conditions (Boström, 1982). Depending on their chemical composition some phosphate fractions can be released from the sediment and may become available to algae (Golterman, 1977). Methods for fractionation described in previous studies are often based on sequential extractions, which are supposed to release the phosphate fractions bound to different cations successively (Williams, 1971; Hieltjes & Lijklema, 1980; Psenner, 1984, 1988; Golterman & Booman, 1988). After previous work (Messagne & Picot, 1993) comparing two chemical methods of phosphate extraction (Bonzongo, 1989; Golterman & Booman, 1988) we have chosen the method using chelators (Golterman & Booman, 1988 as modified by De Groot & Golterman, 1990).

In order to know the influence of the oysters on the phosphate distribution we measured the Tot-P in the sediment in two different areas of the lagoons. One station was chosen inside and the other outside an oyster bank zone. Phosphate in the sediments from the two stations was fractionated at each season, in order to estimate the percentage of phosphate which may be released from the sediment. We analysed iron (Fe(OOH)-P) and calcium (CaCO₃-P) bound phosphate and two organic fractions, an Acid Soluble Organic Phosphate (ASOP) and a residual one (Residual Organic Phosphate - ROP). Recent work has shown that a large part of the ROP fraction is mainly composed of FeOOH-phytate complex, a very insoluble compound, (De Groot & Golterman, 1993), which explains the fact that this fraction is not bioavailable.

Lijklema (1977, 1980) has shown that phosphate and iron are strongly associated in many soils and sediments, with iron playing a major role in controlling the phosphate exchange. The phosphate is bound onto Fe(OOH), the solubility of this complex is a function of the pH in the sediment. Reducing conditions in sediments may increase the solubility of both iron and phosphate; the determination of this fraction is therefore useful in estimating the quantity of phosphate which may be released from the sediment.

The formed complex of calcium carbonate and hydroxyapatite is unstable and can be solubilised by a decrease in pH; whereas the mineral form, apatite, is more insoluble.

As the aim of the study was to evaluate the phosphate mobility of the sediments, we have applied a statistical analysis to the data sets of Tot-P and of the

five phosphate fractions to answer the following questions:

Do phosphate concentrations differ significantly with depth, taking seasons and stations separately?

Are there any significant seasonal variations of the phosphate concentration at the different depths in the sediments?

Material and methods

Site

The study was carried out with sediments of the 'Thau' lagoon, a Mediterranean coastal lagoon, in the South of France (3 °32' to 3 °42'E and 43 °20' to 43 °28'N). This lagoon has a total surface of 70 km², a length of 19.5 km and a width of 4.5 km. The depth varies from 4 m to 10 m. One of the characteristics of the lagoon is the presence of an oyster bank zone with a surface of 13.5 km², which represents 1/5 of the total surface of the lagoon (Fig. 1). Two different sampling stations were chosen, inside (station n ° 1) and outside (station n ° 2) the oyster bank zone B.

Sampling techniques

Undisturbed sediment samples were taken with a plexiglas corer (diameter: 6 cm), by diving, at both stations in July 1991, October 1991, January 1992 and April 1992 reflecting the four seasons.

The cores were cut into slices of 5 cm (0–5 cm/5–10 cm/10–15 cm/15–20 cm/20–25 cm), stored in a plastic flask and treated the same day. In order to have a homogeneous suspension for the chemical extractions of phosphate, the sediment was first sieved over 2 mm and then over 200 µm.

Granulometric characteristics of sediment

A mineralogic study of the Thau lagoon sediments has given the following composition: CaO = 18.4%, MgO = 1.97%, K₂O = 1.71%, Na₂O = 3.72%, Fe₂O₃ = 2.02%, Al₂O₃ = 8.54% (Péna & Picot, 1991). Granulometric distribution (laser granulometer) of the sediment sampled at the two stations has been performed at the four seasons and at five depths from 0 to 25 cm (each 5 cm).

For the description of the granulometric distribution of the sediment we followed the pedologic nomenclature of Duchaufour (1988).

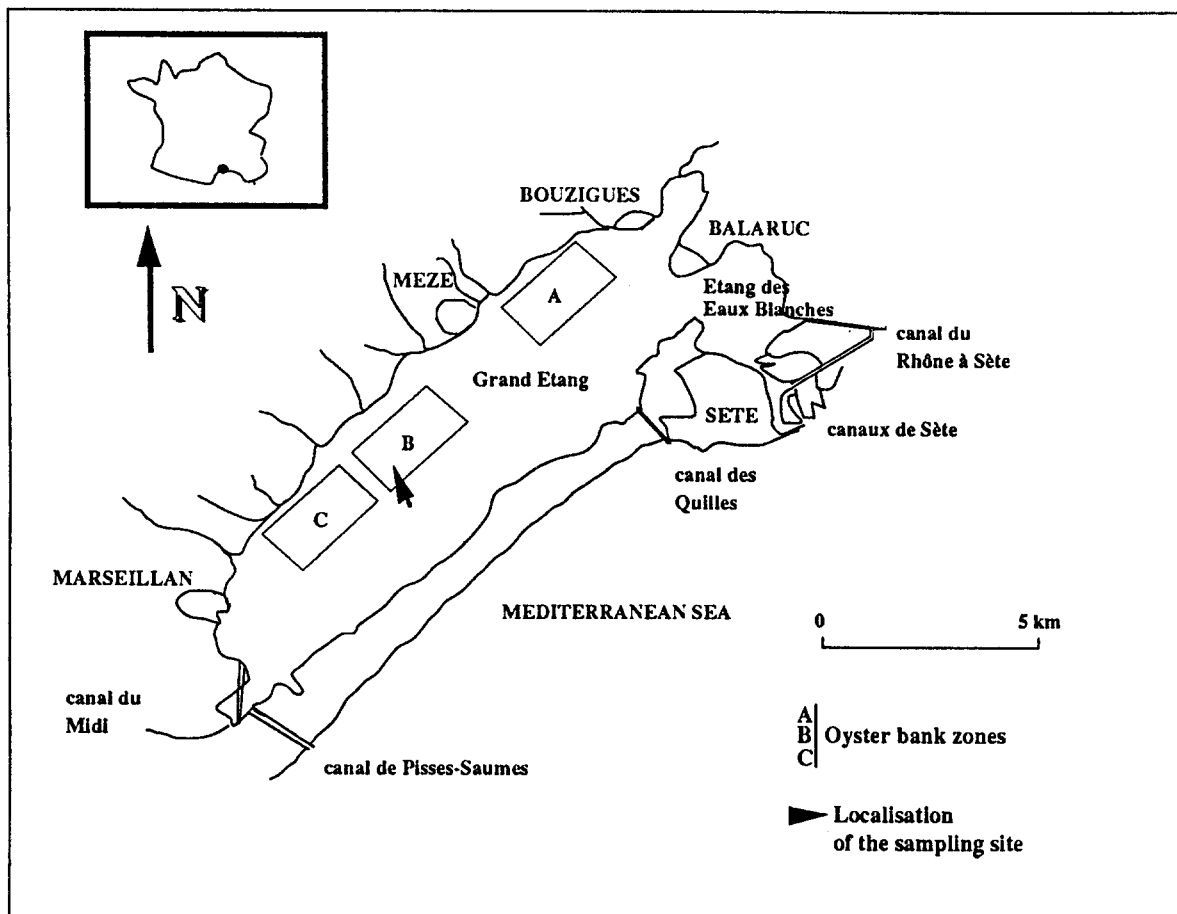


Fig. 1. Location of the study site: the 'Thau' lagoon (France)

Chemical extraction

Chemical analyses of o-P were carried out according to the French norms (AFNOR, 1982), using molybdate-antimony.

Phosphate fractionation was carried out, using chelators such as NTA and EDTA to extract the inorganic phosphates, according to Golterman & Booman (1988) and De Groot & Golterman (1990) (Fig. 2). The soluble phosphate fraction was extracted with H_2O . The inorganic fraction of iron bound phosphate, $Fe(OOH)-P$, was extracted with a chelating agent Ca-NTA (0.02 M + dithionite). The iron hydroxide ($FeOOH$) is also removed by this extraction. The $FeOOH$ concentration was measured with the o-Phenantroline method (Golterman, 1978). The inorganic fraction of calcium bound phosphate ($CaCO_3-P$) was extracted with Na-EDTA (0.05 M). The extractions of these 2 fractions were repeated until depletion. The extraction of the acid soluble organic fraction (ASOP)

requires a single extraction with H_2SO_4 0.5 M. Secondly, the residual organic fraction (ROP) was estimated by destruction of the remaining pellet.

pH and redox potential

Profiles of pH and redox potential have been measured using a combined rH-electrode against a $Ag/AgCl$ reference (Ingold). The results have been corrected to standard- H_2 electrode (Bühler & Galster, 1980).

Statistical analysis

A statistical analysis (Anova, Statview 4) was carried out to evaluate the spatial (influence of the factors 'station' and 'depth'), and the temporal (factor 'season') variations of phosphate concentrations in the sediments. We had 3 replicates of the five depths of sediments sampled.

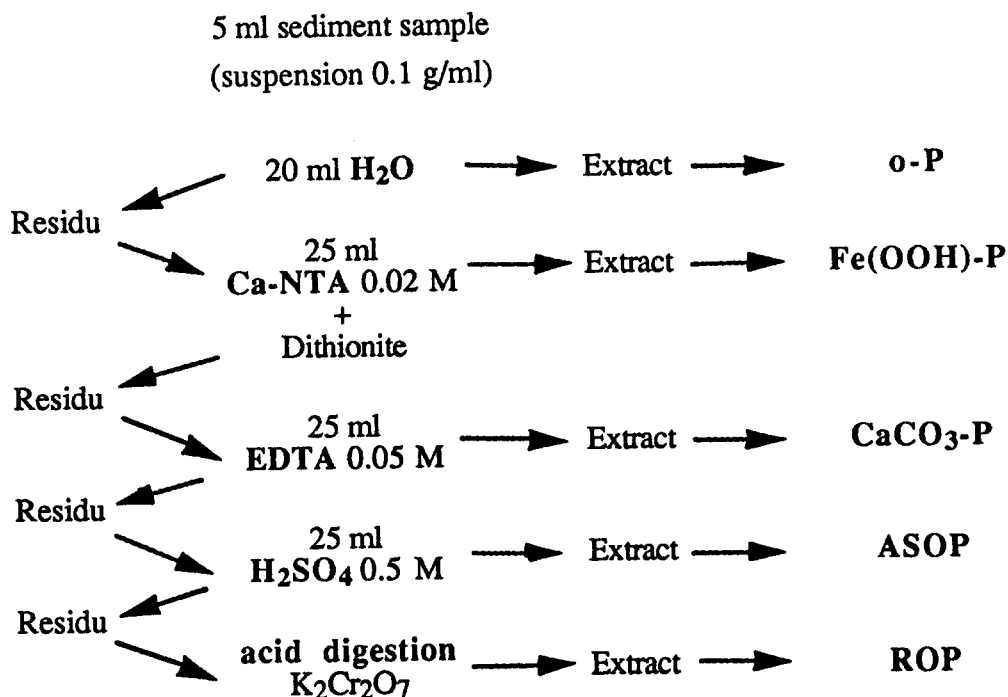


Fig. 2. Sequential extraction of sediment phosphate

Table 1. Granulometric composition of the 'Thau' sediment at station n° 1 and 2

Depth under the sediment surface (cm)	Station	Fine sand (50–200 μm)	Coarse silt (20–50 μm)	Fine silt (2–20 μm)	Clay (<2 μm)
	Station n° 1				
0–10		23%	27%	38%	2%
10–25		23%	23%	43%	2%
	Station n° 2				
0–10		18%	23%	36%	3%
10–25		18%	19%	40%	3%

Results

The granulometric distribution (Duchaufour, 1988) of the sediment of the two stations in the 'Thau' lagoon is summarized in Table 1.

The profiles of pH and redox potential measured at the two stations and at the four seasons are presented in Figs 3 and 4. Whatever the station and the season the pH is lower in the sediment than in the overlying water. pH values in the sediment in July are always

lower than those of the other seasons. At station n° 1 the pH fluctuation with depth is significant with the season. Whatever the station the pH in July is always lower than the pH of the other seasons (Fig. 3).

The redox potential in the overlying water is about 200 mV whatever the station and the season whereas the redox measured in the first 10 cm of sediment showed a significant seasonal variation (Fig. 4). The redox values decrease during the year: Eh = 125 mV in

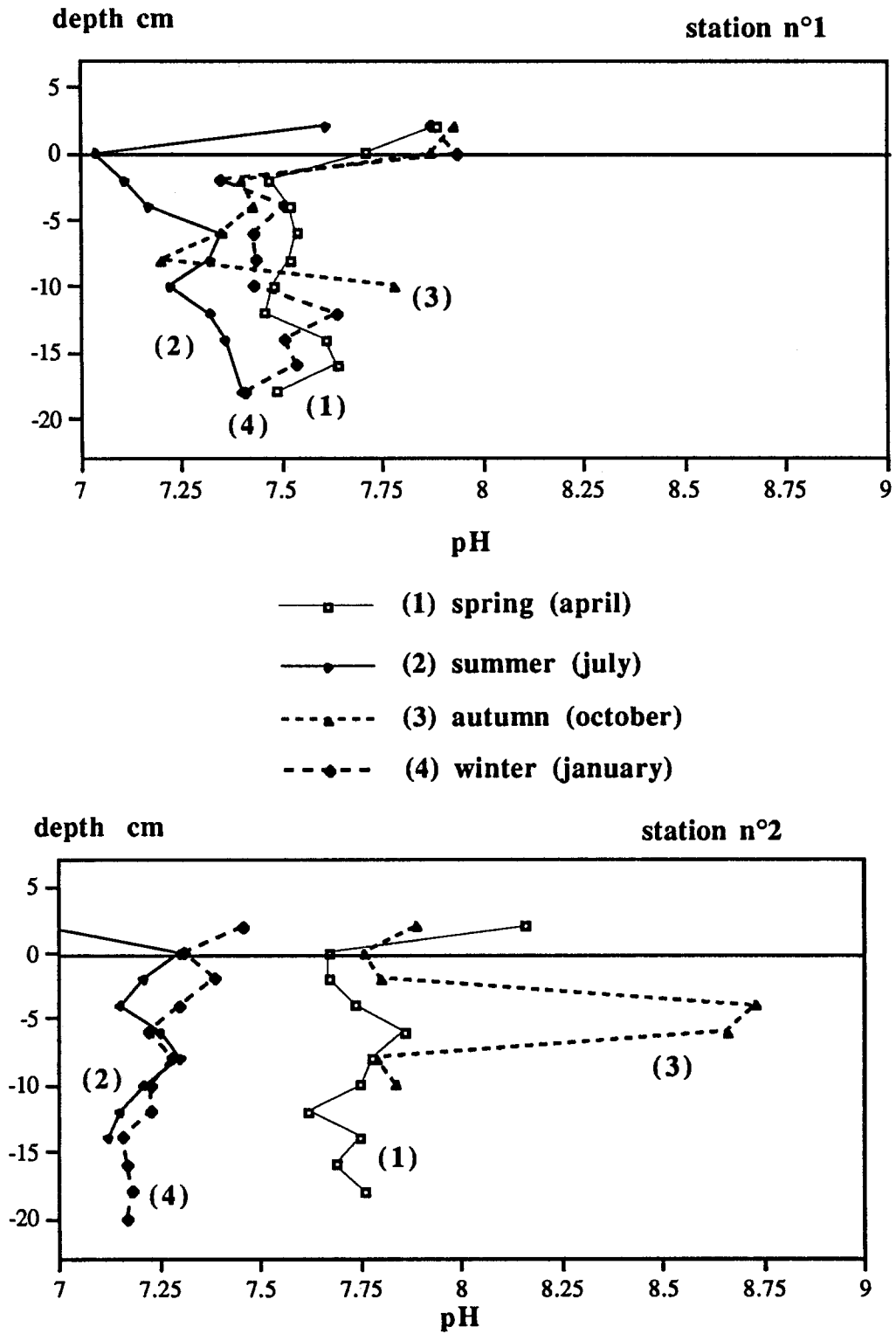


Fig. 3. Seasonal variation in pH profiles in sediment and overlying water

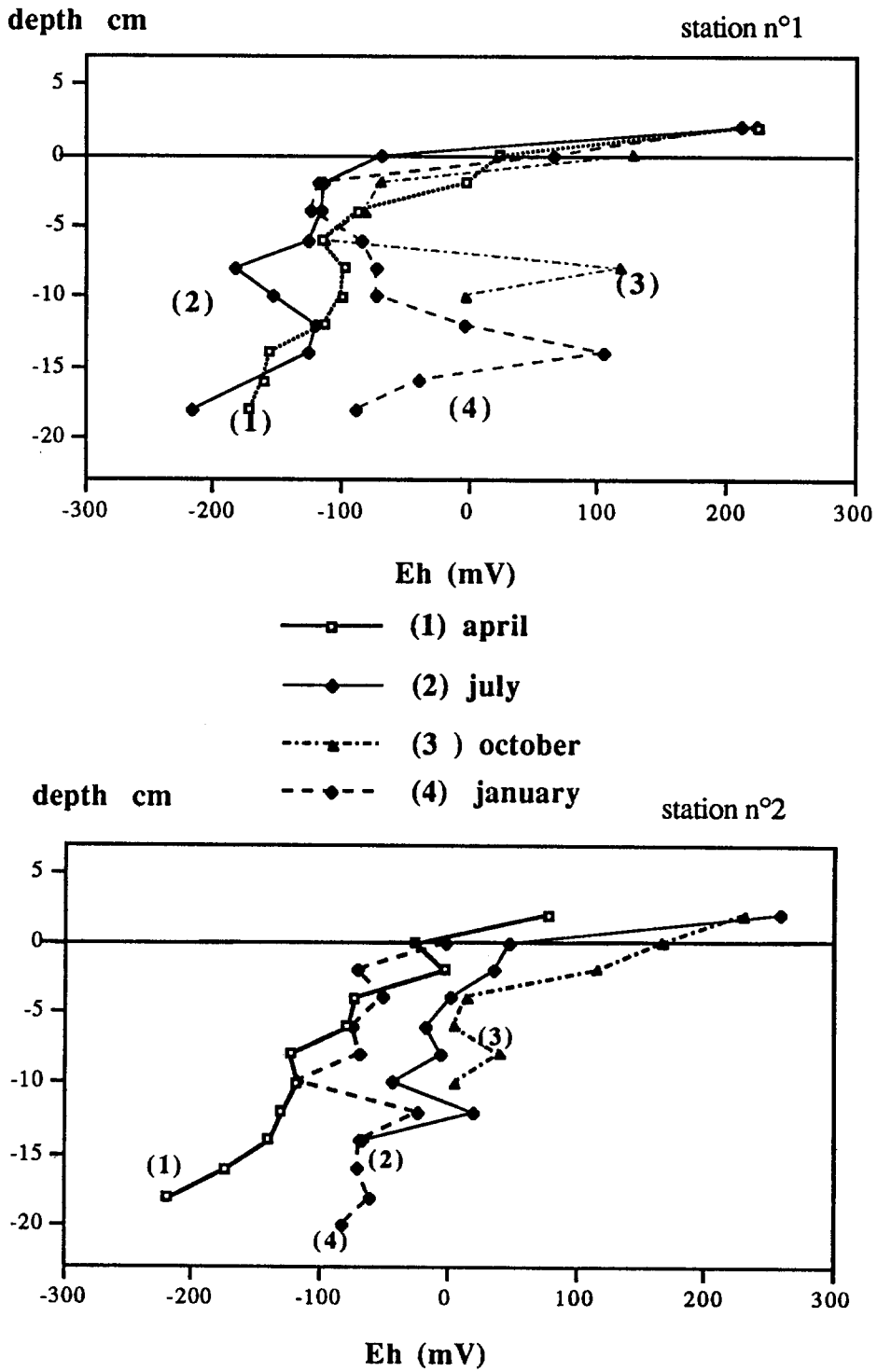


Fig. 4. Seasonal variation in redox potential profiles

Table 2. Tot-C_{org} and FeOOH extractable concentrations

Season	Station	Depth under the sediment surface (cm)	Organic carbon mg g ⁻¹	FeOOH extractable by Ca-NTA mg g ⁻¹	Fe(OOH)-P μg g ⁻¹
Summer (July 91)	n° 1	0-5	48.4	2.4	216
		20-25	22	0.04	50
Winter (January 92)	n° 1	0-5	44.7	1.5	114
		20-25	30.2	0.1	37
Summer	n° 2	0-5	6.7	0.5	115
		20-25	8.7	0.08	46
Winter	n° 2	0-5	20.2	0.5	98
		20-25	22.8	0.1	39

Table 3. Distribution of phosphate fractions in the sediment (μg g⁻¹) - standard error values in brackets, n = 3

Sediment layer: 0-5 cm								
	April		July		October		January	
	Station n° 1	Station n° 2	Station n° 1	Station n° 2	Station n° 1	Station n° 2	Station n° 1	Station n° 2
Tot-P	751(152)	775	914(28)	561(60)	815(125)	667(6)	632(48)	473
o-P	7.6(2.4)	11(8)	6.6(1.2)	4.7(0.5)	1.6(1.5)	5.9(0.4)	2.2(0.3)	4.6(1.5)
Fe(OOH)-P	168(27)	169(8)	216(2)	115(28)	264(69)	207(15)	114(7)	98(6)
CaCO ₃ -P	293(44)	287(13)	255(25)	159(26)	276(33)	233(14)	296(29)	169(17)
ASOP	18(5)	30	25(10)	30(12)	31(11)	26(3)	26(5)	30(1)
ROP	283(32)	260(47)	411(38)	254(48)	242(38)	196(25)	194(14)	164
Sediment layer: 20-25 cm								
	April		July		October		January	
	Station n° 1	Station n° 2	Station n° 1	Station n° 2	Station n° 1	Station n° 2	Station n° 1	Station n° 2
Tot-P	481(25)	—	396(16)	390(10)	367(4)	310(65)	369(11)	290
o-P	4.5(1)	5.2	3.5(1)	3(1)	1.1(1.1)	2(0.6)	3.1(0.6)	2.1(0.1)
Fe(OOH)-P	39(14)	72	50(13)	46(8)	55(2)	52(10)	37(12)	39(1)
CaCO ₃ -P	168(4)	319	144(21)	137(14)	124(5)	135(22)	172(16)	132(4)
ASOP	20(2)	—	21(1)	21(1)	65(1)	50(3)	43(8)	28(2)
ROP	249(48)	126	178(20)	184(13)	123(6)	72(36)	122(36)	84

autumn-winter (October, January) and Eh = -50 mV in summer.

The mean composition of the sediments was measured in the middle part of the lagoon. The mean concentration of Tot-C_{org}, Tot-N, Tot-P was measured in 1991 by Péna and Picot: Tot-C_{org} = 24 mg g⁻¹, Tot-N = 3 mg g⁻¹ Tot-P = 0.6 mg g⁻¹.

Recent data of Tot-C_{org} measured by Gadel (personal communication) at the two different stations with depth and season are summarised in Table 2.

Total phosphate

The Tot-P concentration changed with depth and season. These variations were greater at the station n° 1 than at station n° 2. In July, for example at station

n ° 1, the concentration of Tot-P was $914 \mu\text{g g}^{-1}$ (standard error = 28) at 0–5 cm and $396 \mu\text{g g}^{-1}$ (standard error = 16) at 20–25 cm (Table 3). This difference over depth is significant (Fig. 5).

There is also a seasonal variation inside the oyster bank zone but only significant for the surface sediment. The concentration of Tot-P at this station is $914 \mu\text{g g}^{-1}$ (standard error = 28) in July, $815 \mu\text{g g}^{-1}$ (standard error = 125) in October and $632 \mu\text{g g}^{-1}$ (standard error = 48) in January. On the other hand, for the station outside the oyster bank zone, the mean seasonal concentration (0–5 cm) is $600 \mu\text{g g}^{-1}$ (Table 3).

Phosphate fractions

The statistical test has shown that there is neither a significant spatial nor a temporal variation of the soluble phosphate fraction. The concentration was always lower than 5% of the Tot-P.

The mean concentration of Fe(OOH)-P in the top 5 cm of sediment was about $170 \mu\text{g g}^{-1}$ (all stations and seasons), decreased sharply at the layer below (5–10 cm) and reached $36 \mu\text{g g}^{-1}$ for the 20–25 cm sediment layer. This fraction represents 25% of Tot-P in the surface sediments (mean value for the 2 stations) and 12% in the sediments at 20–25 cm (Table 3).

The main factor controlling the variation of this fraction is sediment depth. Concentrations are maximal in July and October, and minimal in January and April (Fig. 6). Even with an anoxic sediment surface (Fig. 4) in summer (July and October) the concentration of Fe(OOH)-P is maximal, this fraction is therefore not released from the sediment.

The difference between the two stations was only significant for the 0–5 cm sediment layer.

In April and October the mean value of $\text{CaCO}_3\text{-P}$ in the surface sediment was $200 \mu\text{g g}^{-1}$ while the concentration reached a mean value of $300 \mu\text{g g}^{-1}$ in January and July (Fig. 6). These concentrations represent 30% of Tot-P (surface layer) and 40% (the 20–25 cm sediment layer). Concentrations of $\text{CaCO}_3\text{-P}$ in the 20–25 cm sediment layer were always lower than in surface sediments.

At station n ° 1, this fraction decreased with depth, that of the first layer being always higher than those in the lower ones. In all seasons the concentration of $\text{CaCO}_3\text{-P}$ decreased from $250\text{--}300 \mu\text{g g}^{-1}$ (0–5 cm) to $150 \mu\text{g g}^{-1}$ (20–25 cm).

The concentration of the ASOP is not very different in the two types of sediment ($23 \mu\text{g g}^{-1}$ surface sediment, $35 \mu\text{g g}^{-1}$ in the 20–25 cm sediment layer).

This organic fraction represents a relatively small part of the tot-P: 5% in the surface sediment and 10% in the other sediment layers (Table 3).

The concentration of this fraction remained constant with depth, and unlike the other fractions (Fe(OOH)-P, $\text{CaCO}_3\text{-P}$), there is no difference in concentration between the top 5 cm and lower layers, nor is there any significant difference between the stations (Fig. 7).

At station n ° 1, there was a significant decrease of ROP with depth, the mean value being $250 \mu\text{g g}^{-1}$ in the first layer (0–5 cm) and $100\text{--}150 \mu\text{g g}^{-1}$ in the lower layers of sediment (20–25 cm).

The quantity decreased with depth, and was higher inside than outside the oyster bank zone. This can be explained by the organic load being higher inside the oyster bank zone (Fig. 7).

Discussion

The fact that the concentration of Tot-P is higher in the sediment just under the oysters cultures confirms the direct influence of the oysters on the total concentration of phosphate in the sediment. The total stock of phosphate is maximal in summer because of the metabolism of the oysters.

Because of the concentration of the o-P represented 1% to 5% of Tot-P (Table 3), the concentrations are too low to detect seasonal or spatial variations.

The significant change with depth and season of the Fe(OOH)-P, $\text{CaCO}_3\text{-P}$ and ROP phosphate fractions can be correlated with variations of environmental parameters such as pH, redox potential or organic matter discharge in the sediment.

The maximal concentration of Fe(OOH)-P in the sediment is related with the maximal concentration of Tot- C_{org} (Table 2) in the sediment. In a case of a sediment rich in organic matter a complexation of Fe(OOH)-P with organic matter may happen, as was already shown in river sediments (Suzuki, 1992). In summer, because of the high oysters metabolism, there is a maximal input of organic matter at the surface of the sediment. In this case the Fe(OOH)-P can form organic complexes. Even from a reduced surface sediment (Fig. 4) the Fe(OOH)-P is not released. The release of this fraction will probably appear between October and January when the organic-matter content of the sediment decreases.

The decrease in Fe(OOH)-P concentration with depth in all seasons is correlated with the iron hydrox-

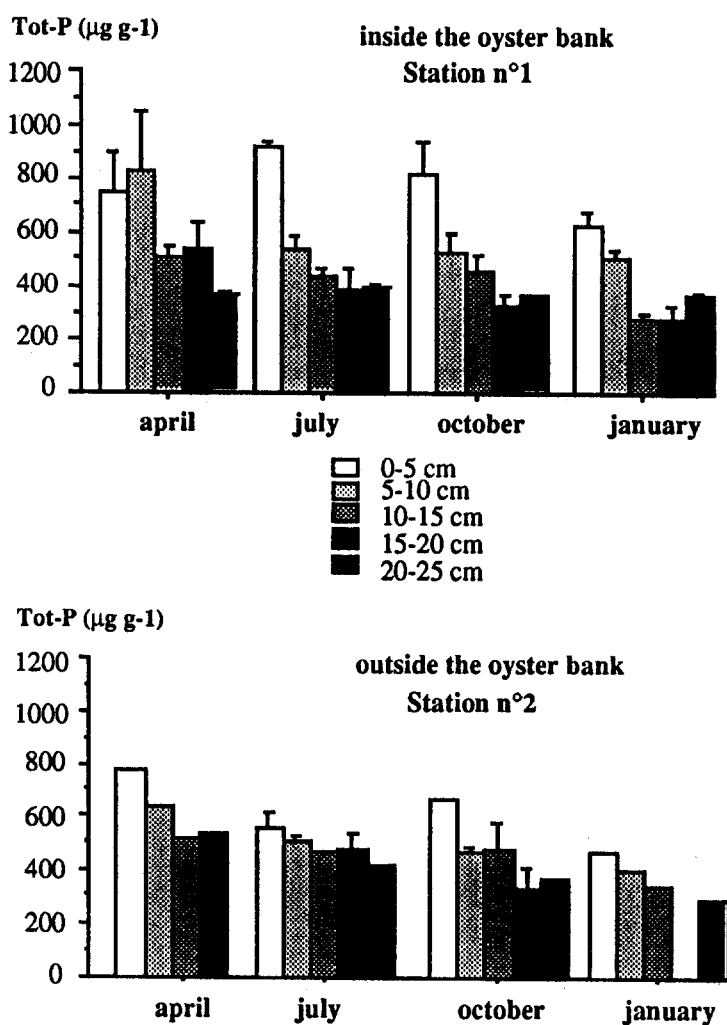


Fig. 5. Seasonal variation in Tot-P as function of depth

ide concentration (FeOOH), which decreased with depth between 0–5 cm and 20–25 cm (Table 2). FeOOH which has a high phosphate adsorption capacity may control the phosphate fixation in the sediment. The decrease of FeOOH explains the low concentration of Fe(OOH)-P at 20–25 cm depth. The maximal concentration of FeOOH was found in April (Table 2) which explains the high concentration of FeOOH-P at this season.

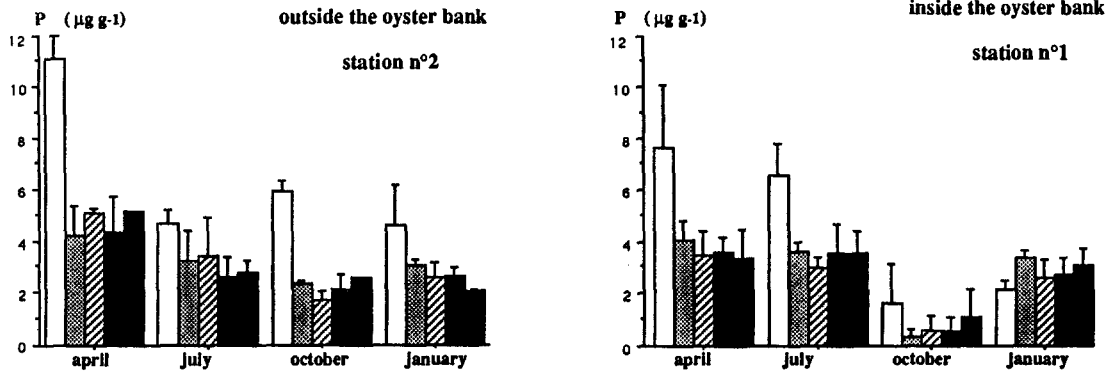
In other Mediterranean coastal lagoons, the maximal concentration of Fe(OOH)-P in the top 5 cm was found in summer (September)–(Moutin, 1993). These authors have found that the maximal concentration of Fe(OOH)-P appeared during spring and summer, and the minimal in autumn and winter. An explanation of

this inverse development may be the presence of a high concentration of organic matter, part of which may form an iron-organic-phosphate complex, which may be extracted in the Ca-NTA fraction.

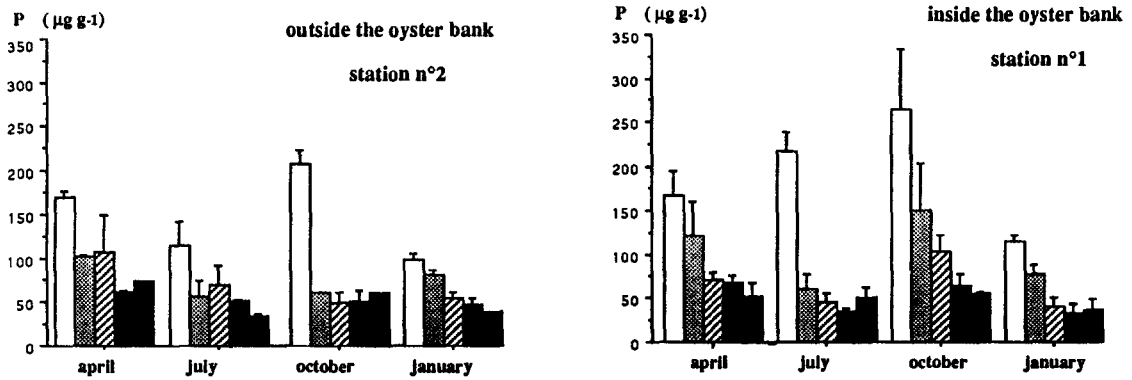
When the O_2 demand is high because of the mineralisation of the high concentration of Tot-C_{org} in the sediment, iron is used as electron acceptor (Fe^{2+}). When all FeOOH is reduced, the FeOOH-P complex can be reduced and o-P may be released from the sediment. This mechanism of iron-bound phosphate reduction at the anoxic interface described by Mortimer (1941, 1942) happens in the Thau lagoon between October and January.

The seasonal variation in the concentration of $CaCO_3$ -P can be explained by pH fluctuations. At both

SOLUBLE PHOSPHATES



IRON-BOUND PHOSPHATES



CALCIUM-BOUND PHOSPHATES

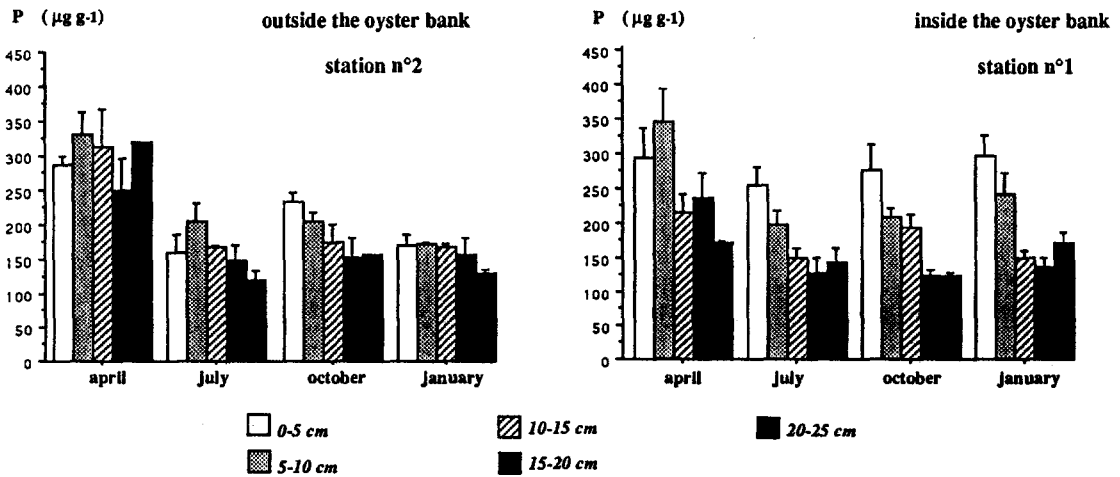
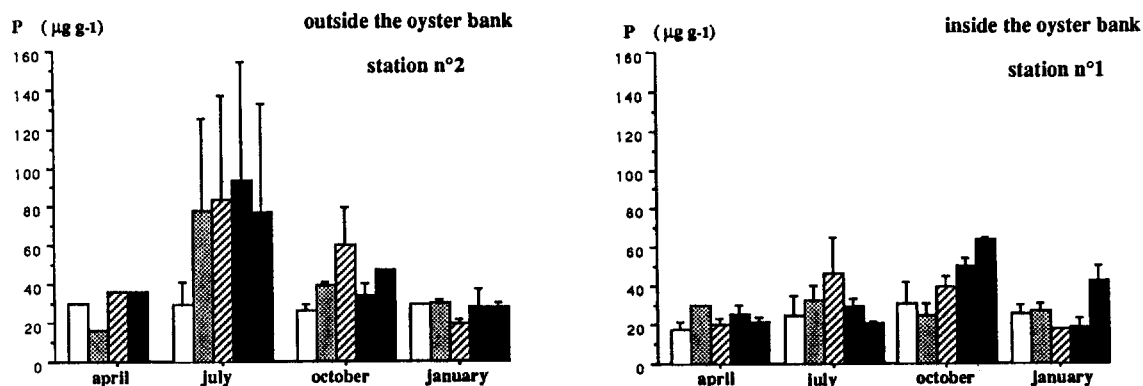


Fig. 6. Seasonal variation in inorganic phosphate fractions, with depth

ACID SOLUBLE ORGANIC PHOSPHATES



RESIDUAL ORGANIC PHOSPHATES

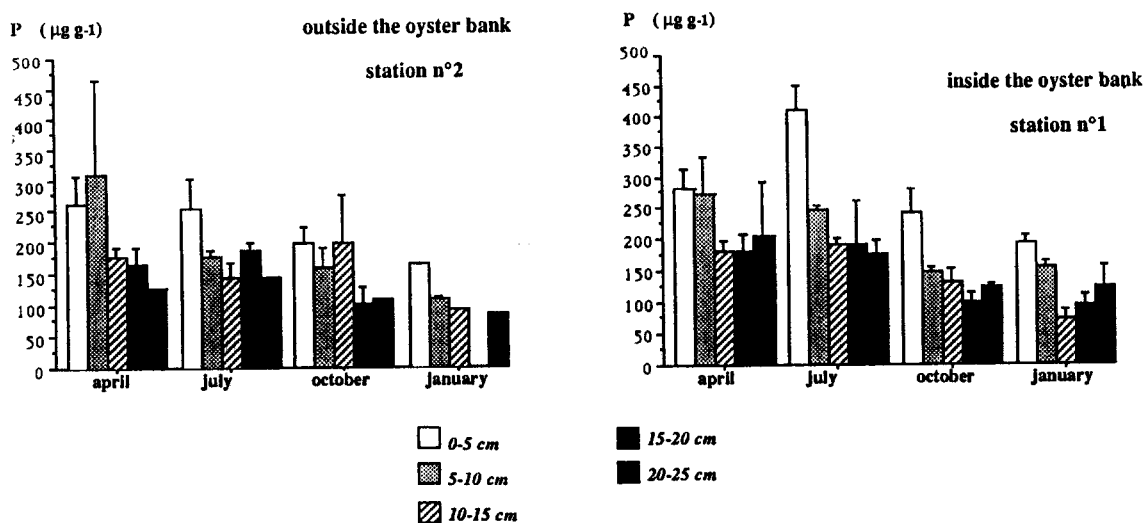


Fig. 7. Seasonal changes in organic phosphate fractions as function of depth

stations pH showed a seasonal variation. These variation can be explained by the bacterial degradation of organic-matter in the sediment. The input of CO_2 into the porewater is maximal in the first 10 cm of sediment where the bacteria metabolism is high compared with the sediment at 25 cm under the sediment surface.

The photosynthetic activity of algae growing at station n° 1 increased the pH of the overlying water. These high pH values in summer increased the precipitation of $\text{CaCO}_3\text{-P}$ at the sediment surface. The maximal concentration of $\text{CaCO}_3\text{-P}$ in spring can also be explained

by the pH, whereas the pH at the sediment-water interface decreased in summer explaining the minimal concentration of $\text{CaCO}_3\text{-P}$ at this season.

The ROP fraction decreased with depth. The higher concentration of this fraction at station n° 1, can be explained by the organic load being higher inside the oyster bank zone (station n° 1) than outside. De Groot & Golterman (1993) have shown that this fraction is mainly composed by phytate. Its extraction requires an acid digestion with sulfuric acid, in the presence of potassium persulfate. I have shown that this frac-

tion which is considered as immobile in the sediment can be mineralised when the bacterial activity is high (Mesnage, 1994). The ROP fraction can also become available for the algae growth.

Conclusion

The statistical analysis of the spatial and temporal variations of Tot-P, has first shown a high dispersion of the mean values (standard error) of the five phosphate fractions, especially in July in the surface sediment (Table 3). The top sediment (0–5 cm) is highly heterogeneous during this period, because of the deposition of oyster faeces.

The spatial variation (factor 'depth') is more pronounced under the oyster bank, because the top 10 cm of sediment are more recently loaded with phosphate than the layers below. The concentration of phosphate remained almost constant below a depth of 15 cm for the two zones. The decrease of Tot-P with depth shows that the variation of phosphate is caused by an increasing load of phosphate from the watershed. This load, which has considerably increased in the past fifty years with the growth of the population around the lagoon, accumulates in the sediment. Therefore the sediments are a good indicator of the increase in phosphate load in this lagoon.

At the anoxic interface of the sediment the phosphate release is not limited to the reduction of Fe(OOH)-P as it was usually found in the litterature (Mortimer, 1941; 1942). Other fractions (CaCO₃-P, ROP) susceptible to certain parameters (pH, redox potential) can be released as well. The significant seasonal variation of phosphate fractions confirm the mobility of these fractions.

Under the oyster bank, the Tot-P in the first 5 cm of sediments is maximal in summer and minimal in winter, showing the influence of the oysters on the phosphate distribution in the sediment. Outside, we found no seasonal variation in agreement with the oyster excretion which is maximal in summer and minimal in winter.

As each fraction has its own capacity of release from the sediment, depending on the pH conditions in the sediments, the knowledge of the different phosphate fractions is necessary to evaluate the percentage of bioavailable phosphate.

The amount of phosphate available to algae from sediments has been assessed by means of bioassays, which are cultures of algae with sediment as the only

source of phosphate (Golterman, 1969; Hegemann *et al.*, 1983; Klapwijk, 1982).

These bioassays suggested that soluble phosphate in interstitial water, Fe(OOH)P and CaCO₃-P are directly available for algal growth. The acid soluble organic fraction (ASOP) may become available after remineralisation (De Groot & Van Wijck, 1993).

In the sediment of the 'Thau' lagoon the percentage of bioavailability is always different in the surface sediment from that at 20–25 cm below the interface. The percentage of available phosphate in the top 5 cm varies according to season. The proportion of phosphate available is maximal in summer (30% of Tot-P), and minimal in winter (20%). Phosphate release is also higher in summer than in winter, explaining high concentration of phosphate at the sediment interface, which is the first step of eutrophication in this ecosystem. Contrarily, in winter (January) we noticed the same proportion of bioavailable phosphate in the surface sediments as in the sediments below (22% of Tot-P).

Acknowledgments

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