Phosphorus retention at the redox interface of peatlands adjacent to surface waters in northeast Germany

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Abstract. It is demanded currently in public discussions to rewet peatlands and re-establish their function as nutrient sinks. But due to high phosphorus (P) concentrations in the pore water of rewetted peatlands (40–420 μM) it is hypothesized that they can act as a surplus P source for adjacent surface waters and consequently support the eutrophication of such waters. Our detailed investigations of processes at the redox interface in four fens with different geochemical character show the dependence of P retention from the chemistry of the pore water. The precipitation of Fe(III) oxyhydroxide led to high retention of phosphorus and other substances such as DOC and sulphate in the eutrophic fens. When molar Fe/P ratios were larger than about 3 the initially high P concentrations in the anaerobic pore water (20–210 μM) decreased to concentrations below 1 μM under aerobic conditions. Thus, after rewetting high pore water concentrations of P do not automatically result in an increased P load to adjacent surface waters compared to pre-rewetting conditions. An enhanced P export to adjacent surface waters from eutrophic fens can be expected when the Fe/P ratio is smaller than 3 in the anaerobic pore water. In our investigations of natural, oligotrophic to mesotrophic fens the precipitation of Fe(III) oxyhydroxide was inhibited by the formation of stable dissolved Fe $\sim$ humic complexes. P retention in these fens was only related to the DOC concentrations at the redox interface, so that lower DOC concentrations concurred with higher P retention. The P equilibrium concentrations in an aerobic environment can be higher than that of eutrophic fens with Fe/P ratios larger than about 3 in the anaerobic pore water.

Introduction

Fens that are recharged by groundwater (Mitch and Gosselink 1993) are the dominant type of peatlands in the glacial landscape of NE Germany as well as in large parts of Poland and of Baltic Counties. About 215,000 ha of fens (7.3% of the total area) cover originally the landscape of the state of Brandenburg (Zeitz 1997). Most of them are situated adjacent to surface waters. In the last few centuries and especially in recent decades more than 95% of the fens were drained for agriculture, leading to peat mineralization and transformation of organic bound phosphorus to phosphorus bounded by redox sensitive Fe(III) oxyhydroxides. Consequently fens lost their capacity for nutrient and water retention. A number of fens are currently rewetted to regenerate their lost functions. After rewetting, anaerobic conditions prevail leading to phosphorus release and pore water concentrations of up to about 420 μM (13 mg L^{-1}) (Gelbrecht and Lengsfeld 1998).

Thus, rewetted fens could potentially be an additional eutrophication source for adjacent lakes and rivers (Gelbrecht and Koppisch 2001). On the other hand, retention processes at the redox interface (anaerobic pore water/aerobic surface water) could lead to a strong decrease of phosphorus in the discharging water due to oxidation of Fe(II) and P sorption onto precipitating Fe(III) oxyhydroxides (Jensen et al. 1992; Roden and Edmonds 1997). Furthermore, it is well known that humic substances, which occur in high concentrations in fen pore water, can act as complexing agents thus influencing P retention at the anaerobic/aerobic interface (Steinmann and Shotyk 1997; Peiffer et al. 1999).

Therefore, this work investigated water chemistry changes at the redox interface and their contribution to phosphorus retention. We have chosen four different fens (two eutrophic and two oligotrophic to mesotrophic fens) to test the following hypothesis of P retention at the redox interface:

1. The main process for P retention is its sorption onto precipitated Fe(III) oxyhydroxides at the redox interface. Humic substances can inhibit precipitation due to stable dissolved complexes with Fe(II, III).
2. The extent of P retention does not depend on the P concentration in the anaerobic pore water but is controlled by the Fe(II)/P ratio.

Materials and methods

Study sites

The investigated fens are situated in the state of Brandenburg in the glacial landscape of NE Germany. The location of the fens and the sampling points for pore water are illustrated in Figure 1. Differences in geochemical conditions and human impact lead to different trophic states and thus to different vegetation communities in the fens.

The Fen at the River Schlaube (F1) is situated 70 km south east of Berlin (52°08′N, 14°28′E, 58 m above sea level) at the east site of the River Schlaube (Gelbrecht et al. 1999). In the past, the fen was drained for grassland use, but during recent decades without any agricultural use a regeneration to more natural conditions took place. The marginal area of the fen is eutrophic with a vegetation of *Lythrum salicaria*, *Urtica dioica* and *Phragmites australis*. The central area, which is permanently waterlogged, shows mesotrophic tendencies with for example, *Polygonum bistorta*, *Caltha palustris* and *Eriophorum* spec. The fen was sampled four times from October 1999 to September 2000 with altogether nine dialysis samplers.

The Fen at the Brook Pfefferfliess (F2) is situated about 20 km south west of Berlin (52°12′N, 13°5′E, 36 m above sea level). The fen was drained for intensive agriculture until 1990. Today the fen is permanently flooded in response to peat mineralization and shrinkage (Landgraf 1998). The vegetation of the polytrophic
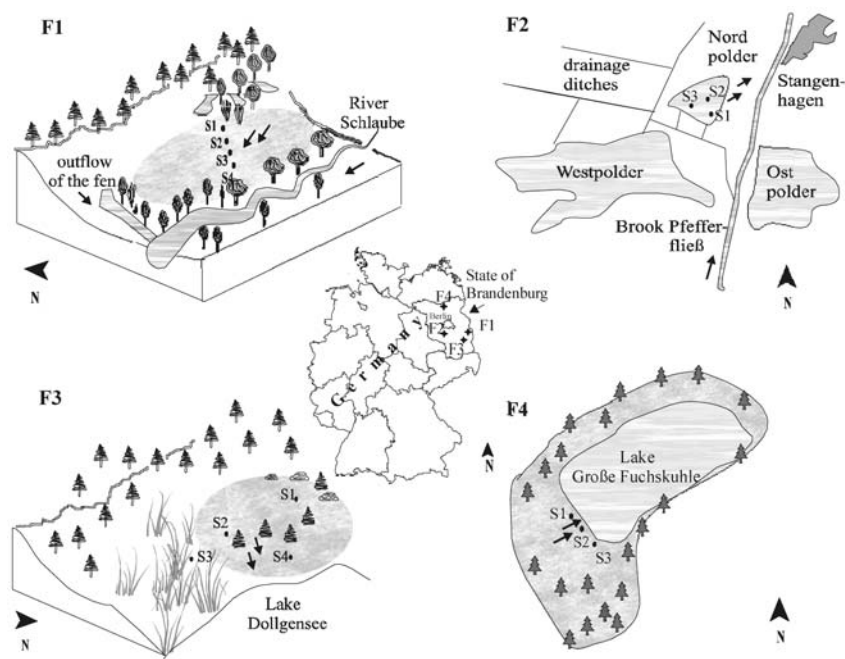
Figure 1. Location and sketches of the investigated wetlands F1–F4 in north east Germany. F1: fen at the River Schlaube; F2: fen at the Brook Pfefferfließ; F3: fen at the Lake Dollgensee; F4: fen at the Lake Große Fuchskuhle. S1, S2... indicate location of the dialyser samplers.

fen is dominated by *Typha latifolia* and *Ceratophyllum* spec. Pore water sampling was restricted to the flooded area of the so-called "Nordpolder" (Figure 1). Sampling was practiced once in November 1999 and once in August 2000 with altogether four dialysis samplers.

The Fen at the Lake Dollgensee (F3) is situated at the SW shore about 60 km south east of Berlin (52°01′N, 14°06′, 47 m above sea level). It is surrounded by a pine forest and the adjacent Lake Dollgensee (Figure 1). The fen is mostly waterlogged up to the surface and is not impacted by human activities. Since the catchment is nutrient poor an oligotrophic to mesotrophic fen was formed. The fen consists of two parts with different trophic state. One part is characterized by *Sphagnum recurvum*, *Oxycoccus palustre*, and *Eriophorum* spec. which are typical plant species for a weak acid and nutrient-poor fen. In the other part tendencies to a slight eutrophication are recognizable as *Phragmites australis* is the dominating species. The fen was sampled three times from October 1999 to August 2000 with altogether seven dialysis samplers.

The oligotrophic acid Fen at the shore of Lake Große Fuchskuhle (F4) is situated in a pine forest about 100 km north of the city Berlin (53°10′N, 13°02′E, 59 m above sea level). The fen discharges directly into the southwestern part of the

dystrophic Lake Große Fuchskuhle (Sachse et al. 2001). The vegetation of the drier main part of the fen is dominated by *Pinus sylvestris* and *Ledum palustre*. The fen is perennial waterlogged and habituated by *Sphagnum recurvum* close to the lake shore. Sampling was done in this area (Figure 1) three times from August 2000 to October 2000 with altogether nine dialysis samplers.

Sampling and analytical methods

Dialysis samplers (Hesslein 1976; Steinmann and Shotyk 1996) were inserted into waterlogged parts of the fens at fixed locations (Fig. 1) to obtain the pore water on a temporal and spatial scale. Samplers with sufficient chamber volume (0.7 L) were used to perform comprehensive analyses with the anaerobic and aerobic samples (see below). The sampling depth was restricted to about 0.7 m (length of the sampler). Prior to insertion, the chambers of the sampler, which were covered by a 0.2 μm pore membrane filter (Gelman$^{®}$ HT 200 tuffryn), were filled with deionised water. Oxygen from the chamber water and the sampler material (Perspex) was displaced by degassing with nitrogen for 24 h. The exposure time of the sampler in the peat was at least 7 days to guarantee an equilibrium between the concentrations of dissolved substances in the peat pore water and the chamber water.

After recovering the samplers, the water of all chambers was immediately combined in an oxygen free 1-L-polyethylene bottle by means of continuously adjustable pipettes. Quick sampling (few minutes), the storage of subsamples in argon-flushed vessels, and fixation with sulphuric acids prevented the oxidation of the redox sensitive parameter (Fe^{2+}, Mn^{2+}) and the precipitation of hydroxides prior to measurement. To deal with the strong supersaturation of the anaerobic pore water with respect to CO_2, subsamples for dissolved inorganic carbon (DIC) determination were collected in air tight, argon flushed vessels immediately after collection.

The rest of the samples was aerated to induce oxygen saturation and simulate the oxidation processes at the anaerobic/aerobic interface. The time to complete precipitation was adjusted according to results from prior investigations (Figure 2). A new chemical equilibrium was assumed to become approximately established in 5 h for samples from eutrophic fens (F1, F2) and in 24 h for those from the oligotrophic to mesotrophic fens (F3, F4).

Soluble reactive phosphorus (SRP) and total dissolved phosphorus after acid digestion (TDP) were measured by molybdenum blue method (Varian, Cary 1E). In the oligotrophic to mesotrophic fens (F3, F4) the SRP measurements were deemed unreliable because of interferences by humic substances and hydrogen sulphide with the molybdenum complex. $Fe^{2+,3+}$ was determined with the orthophenathroline method (UV-2101 PC, Shimadzu), whereas total dissolved iron (Fe_T) and Ca^{2+}, Mg^{2+}, Mn^{2+}, K^+, Na^+ were analyzed by flame atomic adsorption spectrometry (Perkin Elmer, 3300). If Fe_T concentrations were below the detection limit (4 μM), the results of the orthophenathrolin method were used (detection limit: 0.4 μM). The anions SO_4^{2-}, NO_3^-, Cl^- were measured by ionic chromatographic
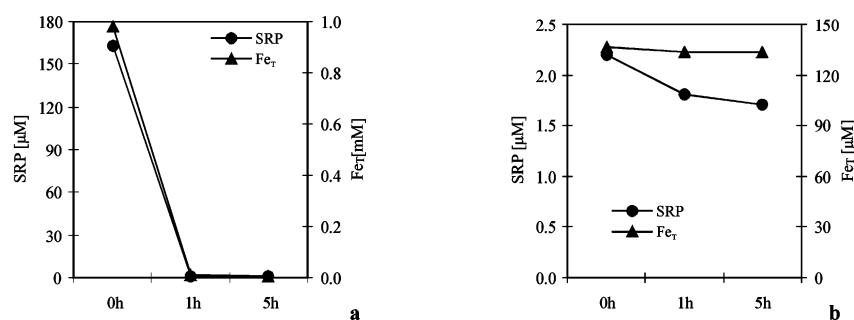
Figure 2. a–b: Preliminary examinations to detect the chemical equilibrium under oxygenated conditions related to decline of SRP and Fe_T after different reaction times (0 h, 1 h, 5 h). (a) fen at the River Schlaube, (b) fen at the Lake Dollgensee.

(Shimadzu), NH_4^+ photometrically (Flow Solution III, Perstop) and dissolved inorganic carbon (DIC), dissolved organic carbon (DOC) with the TC-DOC-Analyzer (TOC 5000, Shimadzu). Automated size-exclusion-chromatography with simultaneous UV- and organic carbon detection (IR) (Huber and Frimmel 1996; Sachse et al. 2001) was used for the measurements of DOC-fractions: polysaccharides (PS), humic substances (HUS), low molecular weight neutrals (LMWS), and low molecular weight acids (LMWA). Oxygen, pH, and conductivity were measured by means of probes (WTW®).

Charge balance calculations

Accurate measurements of all important cations and anions allowed the assessment of the total organic anionic charge $[X^-]$ from humic acids and low molecular acids (Steinmann and Shotyk 1997). Their amount can be calculated as the difference between the electrical charges (μeq/L):

$$[X^-] \quad = \quad \sum (\text{inorganic cationic charge}) - \sum (\text{inorganic anionic charge})$$

Chemical equilibrium calculations were used to quantify the anionic composition of DIC and P (SRP, TDP) in dependence of the measured pH. Results indicate the potential metal binding capacity of the functional groups of the organic ligands.

Results

The temporal and spatial variation of the dissolved substances in the pore water of each fen and variation between them was high (Table 1). Typically the pore water was anaerobic and therefore lacked nitrate; it was extremely supersaturated with respect to CO_2. Large amounts of CO_2 degassed during aeration and CO_2 reached

Table 1. The range of conductivities (Cond.), pH and molar Fe/P ratios and concentrations of dissolved substances in the pore water of the investigated fens.

	Fen			
	River Schlaube (F1)	Brook Pfefferfließ (F2)	Lake Dollgensee (F3)	Lake Große Fuchskuhle (F4)
	$n = 9$	$n = 4$	$n = 7$	$n = 9$
Cond. [µS/cm]	279–656	1604–2210	150–252	54–193
pH	6.2–7	6.4–6.8	5.8–6	3.3–3.9
Fe/P ratio	4–51	1–2	21–111	1–8
Fe^{2+} [µM]	320–1760	410–510	100–140	2–12
P [µM]	30–210	240–440	2–5	0.6–14
SO_4^{2-} [µM]	20–100	1470–3800	81–356	7–30
Cl^- [µM]	30–120	2800–3430	170–547	138–313
DIC [µM]	4190–12870	16320–35760	3430–5620	841–1532
DOC [µM]	1600–2690	11820–21960	2190–3705	2348–9159
Ca^{2+} [µM]	970–2060	1220–7740	462–866	12–40
Mg^{2+} [µM]	90–220	630–910	103–169	5–17
Na^+ [µM]	330–640	2870–3480	222–470	97–231
K^+ [µM]	20–80	250–440	19–87	12–56
Mn^{2+} [µM]	10–70	70–140	3–6	<1*
	$n = 3$	$n = 3$	$n = 3$	$n = 9$
PS [µM C]	70–210	470–700	30–80	0–40
HUS [µM C]	560–820	10100–15800	2030–2540	1630–7800
LMWA [µM C]	340–720	17–640	180–290	0–220
LMWS [µM C]	280–530	800–1600	1030–1320	40–1250

PS – polysaccharides; HUS – humic substances; LMWS – low molecular weight neutrals; LMWA – low molecular weight acids.
*Detection limit.

equilibrium concentrations. This process resulted in a strong decrease of DIC and a pH increase in all fens except in F4 (Table 2). Furthermore the pore water was characterized by the presence of hydrogen sulphide, and the yellowish color from humic substances. Humic substances were the dominant fraction of DOC in all fens (Figure 3). Beside them above all LMWS were of importance in the fens, although the total amount and composition of DOC differed strongly between the investigated fens (Table 1). The proportion of dissolved organic substances in the total charge balance is presented in Figure 4.

Already after a few minutes of aeration and oxygen saturation the precipitation of iron compounds was visible through brownish coloring and clouding in the samples of the eutrophic fens (F1, F2). The initially high concentrations of iron (Table 1) diminished to and below the detection limit of the AAS method (Table 2). Whereas precipitation of Fe(III) oxyhydroxides did not occur in the oligotrophic to mesotrophic fens but in F3 the starting Fe(II) concentration was up to 140 µM (Table 1) and about 50% of ferrous iron was oxidized under aerobic conditions

Table 2. The pH change, the concentrations of $Fe^{2+,3+}$ and P in the aerated pore water samples and the decreases of single dissolved substances in μM and % after aeration (medians).

|	Fen							
	River Schlaube (F1)		Brook Pfefferfließ (F2)		Lake Dollgensee (F3)		Lake Große Fuchskuhle (F4)	
	$n=9$		$n=4$		$n=7$		$n=9$	
pH-change (median)	0.5		1.9		1.3		−0.1	
Concentrations in the aerobic samples (median)								
$Fe^{2+,3+}$ [μM]	5		8		126		7	
P [μM]	1		26		2		4	
Decreases of dissolved substances (median)								
	μM	%	μM	%	μM	%	μM	%
Fe	978	100	448	98	0	0	0	0
P	92	99	336	93	2	54	2	32
SO_4^{2-}	80	85	137	6	0	0	3	18
DIC	3301	49	8301	40	3805	83	1153	98
DOC	508	28	4113	25	583	18	458	10
Ca^{2+}	0	0	2481	62	0	0	0	0
Mn^{2+}	10	52	29	27	0	0	–	–
	$n=3$		$n=3$		$n=3$		$n=9$	
PS	90	29	620	100	−250	−375	0	0
HUS	370	55	2400	16	180	9	−60	−2
LMWA	−80	−16	270	100	220	100	0	0
LMWS	380	74	420	30	520	46	170	26
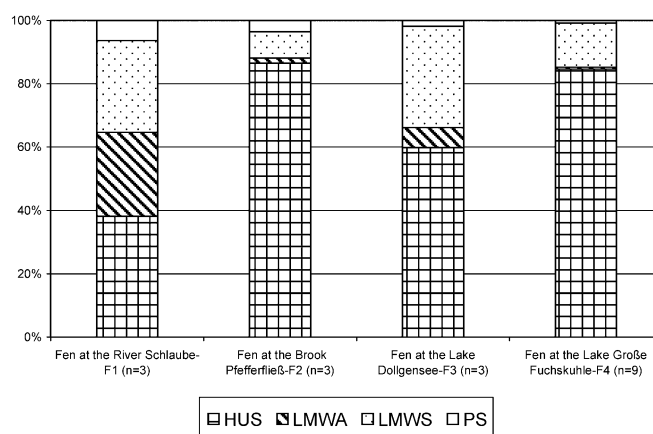
Figure 3. Mean composition of the DOC in the anaerobic pore water of the investigated fens. (HUS: humic substances, LMWS: low molecular weight neutrals, LMWA: low molecular weight acids, PS: polysaccharides).
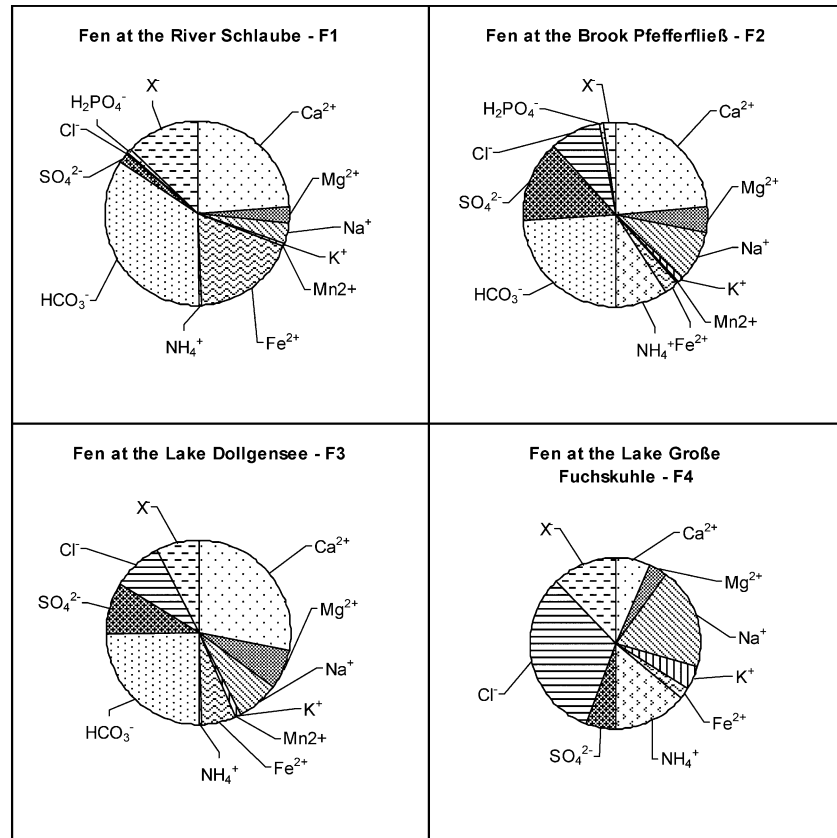
Figure 4. Mean proportion of the dissolved substances at the charge balance in the anaerobic pore water of the investigated fens (X^- = organic anionic charge).

(data not shown). Note that SRP and TDP was not distinguished in the following presentation, because in F1 and F2 both fractions were similar and in F3 and F4 only TDP could determined (see above). P decreased strongly from more than 90% in the eutrophic fens to about 30–50% in the oligotrophic fens (Table 2). P equilibrium concentrations under aerobic conditions were approximately 1 μM (range: 0.4–2.6 μM) in fen F1 independent of the strongly varying original P concentrations (Table 1). In contrast to F1 the aerobic P equilibrium concentrations were higher and more variable in F2 (6–48 μM). The P concentrations (median) in the aerobic samples of the oligotrophic to mesotrophic fens were 4 μM in F3 and 6 μM in F4.

A decline in DOC upon aeration of 10–30% was measured in all fens with highest responses in the eutrophic fens F1 and F2. Detailed investigations on the decline of different DOC fractions show that changes in PS, HUS and LMWS were
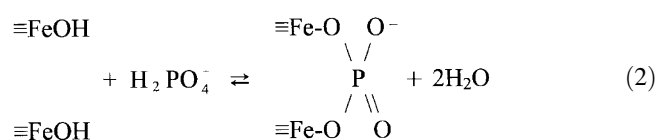
mainly responsible for the decline of DOC in the eutrophic fens while in the oligotrophic fens mostly LMWA (F3) and LMWS (F3, F4) (Table 2) decreased. The changes of humic substances were within measurement error of $\leq 10\%$ of the LC-OCD method (E. Zwirnmann, personal communication).

Discussion

Due to the changed conditions after aeration of the anaerobic pore water (pH, redox state etc.) complex precipitation and sorption reactions occurred. The lack of pH increase in F4 despite high loss of DIC (Table 2) is most likely related to the buffering by humic acids.

The decrease of P at the redox interface together with other ions such as sulphate and the fractions of DOC (see Table 2) can be summarized with the following simultaneously occurring processes:
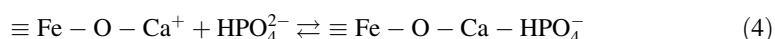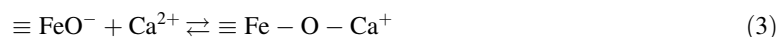
(a) After aeration of the anaerobic pore water ferrous iron was oxidized, hydrolyzed and precipitated completely as Fe(III) oxyhydroxide in the eutrophic fens (F1, F2). It explains the removal of phosphorus exceeding 90% as precipitation of $\{Fe(PO_4)_x(OH)_{3-x}\}$ and/or sorption on precipitated Fe(III) oxyhydroxides (Eqs. 1 and 2).

$$\equiv FeOH + H_2PO_4^- \rightleftarrows \, \equiv FeHPO_4 + H_2O \tag{1}$$

$$
\begin{array}{l}
\equiv FeOH \\
\qquad\qquad + H_2PO_4^- \rightleftarrows \\
\equiv FeOH
\end{array}
\quad
\begin{array}{l}
\equiv Fe\text{-}O \quad O^- \\
\qquad\quad \backslash \, / \\
\qquad\qquad P \qquad + 2H_2O \\
\qquad\quad / \, \backslash\!\backslash \\
\equiv Fe\text{-}O \quad O
\end{array}
\tag{2}
$$

Our study shows that SRP can decrease to concentrations of 1 μM or less if the molar Fe/P ratio is larger than about 3 (F1). These findings agree well with several authors (Lijklema 1980; Buffle et al. 1989). Consequently, lower Fe/P ratios result in higher aerobic equilibrium P concentrations and indicate an increasing risk of P export to adjacent surface waters, as was found in F2. Here, SRP aerobic equilibrium concentrations ranged from 6–48 μM (Fe/P ratio = 1.3, median). The role of Mn(IV) oxides as important absorbents for phosphorus can be neglected due to minor concentrations compared to iron (Yao and Millero 1996).

(b) Unspecific and specific binding of SRP on Fe(III) oxyhydroxides (Eqs. 1 and 2) depends on pH since P-sorption will occur primarily at the positive sites (Goldberg and Sposito 1984; Buffle et al. 1989). Considering that the zero point charge (ZPC) of amorphous FeOOH happens at pH 7.9–8.1 (Buffle, 1988) a positive charged surface can be assumed in F1 whereas a negative surface is expected in F2 due to pH shifts above 8 (Table 2). Furthermore, sulfate sorption onto Fe(III) oxyhydroxides cannot be excluded as a competitive reaction to P, especially in F2.

(c) The high decline of P in F2 (93%) despite above mentioned reactions can be attributed to high calcium concentrations. Calcium is known to support sorptive binding of phosphate as coupling ion (Kawashima et al. 1986) at the surface of Fe(III) oxyhydroxide according to equations (3) and (4) (Driescher and Gelbrecht 1999):

$$\equiv FeO^- + Ca^{2+} \rightleftarrows \equiv Fe - O - Ca^+ \qquad (3)$$

$$\equiv Fe - O - Ca^+ + HPO_4^{2-} \rightleftarrows \equiv Fe - O - Ca - HPO_4^- \qquad (4)$$

But the strong decrease of calcium concentrations and the pH increase above 8 in F2 (Table 2) suggest that calcite precipitation also occurs which was not investigated in detail.

(d) It is also possible that humic substances (HUS) act as coupling ion (Laxen 1985; Dolfing et al. 1999) in F1 and F2 and bind phosphates via metal (M) bridges according to equation (5):

$$\equiv Fe - O - HUS - M^+ + HPO_4^{2-} \rightleftarrows \equiv Fe - O - HUS - M - HPO_4^- \quad (5)$$
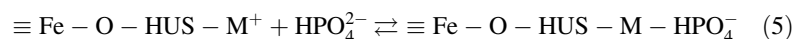
Humic substances, which were markedly retained in the eutrophic fens F1 and F2 (Table 2) by precipitation of Fe(III) oxyhydroxides, which is confirmed by several authors (Tipping 1980; Buffle 1988) are also known to form stable dissolved Fe(II/III)-humic complexes (Ziechmann 1980; Steinmann and Shotyk 1997; Peiffer et al. 1999). According to the charge balance (Figure 4) a binding of iron at acid groups of organic ligands can be expected in anaerobic pore waters of all investigated fens. But only in the oligotrophic to mesotrophic fens F3 and F4 dissolved complexes appear to be stable and hinder the precipitation of iron. We assume that a lower pH and special complexing properties of HS originated from *Sphagnum* peat are responsible for the lack of Fe precipitation in these fens. The decline of P in oligotrophic to mesotrophic fens (F3, F4) without a simultaneous decrease of Fe, can only be explained by the decrease of DOC (Table 2).

In conclusion, the hypothesis of enhanced P export from rewetted fens into adjacent surface waters needs detailed consideration. Our investigation shows that chemical reactions at the redox interface of fens are the controlling process for the retention of phosphorus and other dissolved substances and their emissions into adjacent surface waters. Different mechanisms can be assumed for rewetted eutrophic and natural oligotrophic to mesotrophic fens:

Eutrophic fens

The precipitation of Fe(III) oxyhydroxides is the main process for P retention. Phosphorus equilibrium concentration $\leq 1\,\mu M$ ($\leq 0.031\,mg\,L^{-1}$ P) in an aerobic environment can be found only when Fe/P ratios are larger than about 3. Thus, high

pore water concentrations of P between 100–450 μM (3–14 mg L^{-1}), as often found in rewetted eutrophic wetlands, do not automatically result in an increased P load to adjacent surface waters compared to pre-rewetting conditions. Enhanced P contamination of adjacent surface waters can be expected if the Fe/P ratio is smaller than 3 in the anaerobic pore water or if the receiving water is anaerobic. High Ca concentrations promote P sorption on Fe(III) oxyhydroxides and thus P retention at the redox interface.

Depending on the pore water chemistry before aeration also other dissolved substances (e.g., HUS, LMWA, LMWS, sulphate) can be retained.

Oligotrophic to mesotrophic fens

In general, pore water of these fens is characterized by a low ion content. The precipitation of Fe(III) oxyhydroxides can be inhibited by the formation of stable dissolved Fe ∼ humic complexes. In such cases P retention does not depend on the Fe/P ratio of the anaerobic pore water but is related to a decline of DOC at the redox interface. The P equilibrium concentrations in an aerobic environment can be higher than that of eutrophic fens with Fe/P ratios larger than about 3 in the anaerobic pore water.

Acknowledgements

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