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Water pH and sediment resuspension regulating internal phosphorus loading in a shallow lake – field experiment on diurnal variation

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ABSTRACT

Resuspension-mediated aerobic release of P can be crucial to the phosphorus cycling of a shallow lake. The effect of resuspension on the aerobic release of phosphorus was studied in the field with experimental water columns. The study included four experiments that were conducted in May and August during the day and following night in the shallow Kirkkojärvi basin of Lake Hiidenvesi. In all the experiments, the concentrations of suspended solids (SS) and total phosphorus (TP) increased substantially, due to the 10-min resuspension treatment. However, the concentration of soluble reactive phosphorus (SRP) was strongly affected only at night in August, when high pH levels promoted SRP release by ligand-exchange reactions. During the day experiment in August, no SRP release was detected, due to high assimilation by phytoplankton. The results pointed out that coupling of resuspension and high algal blooms induced pH in the water column can liberate significant amounts of soluble P into the water column. The importance of this phenomenon for the lake studied was emphasized by the fact that the P pools susceptible to pH-dependent P release (Al-P and Fe-P) formed a large part of the total extractable P of the surface sediment. Additionally, the conditions of high pH are common from early July to late September and the water pH can stay at a high level also at night during strong phytoplankton blooms.

Key words: resuspension, internal loading, aerobic phosphorus release, experimental approach

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1. INTRODUCTION

In the majority of lakes, the phosphorus (P) burial or release is controlled by a complex coupling of sediment composition, external load, catchment hydrology, lake morphometry, and biogeochemical reactions (Hupfer & Lewandowski 2008). Periodically, some of these factors can take over others. In shallow lakes, the water body is well mixed and the surface sediment oxidized due to active surface wave action (Søndergaard *et al*. 2003). In addition to oxidized conditions, frequent surface wave action also leads to increased sediment resuspension, which can markedly contribute to internal P loading of a shallow lake (Kristensen *et al*. 1992; Niemistö & Horppila 2007). Although, beneath the aerobic sediment-water interface, anoxic conditions can govern P dynamics, resuspension-induced oxidized conditions play an important role in the P dynamics in the sediment-water interface and water column. The importance of sediment-water interactions in shallow lakes is further enhanced by the high sediment surface:water column ratio, which implies that the potential influence of resuspension on lake water concentrations is stronger than in deeper lakes (Søndergaard *et al*. 2003).

In a shallow lake, the aerobic release of P via ligand-exchange reactions can be substantial, because resuspension transports sediment P bound to Fe and Al oxides to the water layers where pH is elevated due to primary production (Hingston *et al*. 1967; Golterman 1976; Lijklema 1980; Koski-Vähälä & Hartikainen 2001; Søndergaard *et al*. 2003). In a previous study, the pH-dependent P release under oxic conditions was significant in the shallow Kirkkojärvi basin (Holmroos *et al*. 2009). The release was most pronounced in late summer due to coupling of sediment resuspension and high water pH induced by strong phytoplankton blooms. The seasonal variation in pH-dependent P release was also previously observed (Holmroos *et al*. 2009). However, the diurnal variation of aerobic P release was not studied and experiments conducted on this topic in the field are scarce. The present study was conducted to illuminate the possible diurnal alternation of aerobic P release during the different phases of the growing season. It was hypothesized that the P release would be most pronounced in daytime in late summer during high algal blooms when the water pH is high.

2. MATERIAL AND METHODS

2.1. Study area

The study was conducted in the shallow (mean depth 1.1 m, max. depth 3.5 m) Kirkkojärvi basin of Lake Hiidenvesi (area 30.3 km^2), which is situated in southern Finland (60°24'N, 24°18'E) (Fig. 1). The restricted Kirkkojärvi basin (area 1.6 km^2) is the most eutrophic basin of Lake Hiidenvesi due to agricultural actions in the catchment. The summertime total phosphorus (TP) concentration varies between 80 and 120 μg L^{-1} and total nitrogen (TN) concentration between 1000 and 1500 μg L^{-1}. Due to the shallowness of the lake, sediment resuspension is often high, constituting 85-95% of

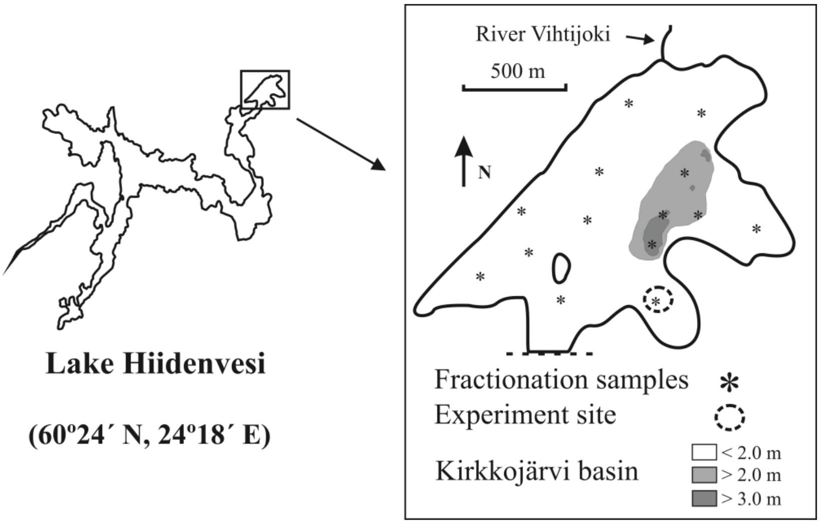

Fig. 1. Map of the study lake. Depth contours, experiment site, and phosphorus fractionation sample locations shown.

the gross sedimentation (Niemistö & Horppila 2007; Niemistö *et al*. 2009). In addition, the runoff from agricultural areas affects high water turbidity, which often varies from 20 to 70 nephelometric turbidity units (NTUs). Therefore, the Secchi depth usually remains below 0.5 m.

2.2. Experimental setup

The effects of resuspension events on P release were studied during the ice-free season of 2007. The study was conducted during two different periods, on 21-22 May and 2-3 August. In each period, the study included two identical experiments that were carried through during the day and following night in the Kirkkojärvi basin (Fig. 1). The day experiments were started at 11:00 hours. The night experiments were started 0.5-1 h after sunset and thus ended approximately 1 h before sunrise. The experiments were conducted in the experimental columns that were open to the lake bottom and placed at a 1 m depth. The height of each column was 1.3 m and the diameter 18.5 cm. Three columns were used as control units while in the other three columns resuspension was conducted with a piston by moving it in the uppermost 50 cm of water according to the maximum wave heights usually occurring in Kirkkojärvi (Horppila & Nurminen 2001). During the 10 min treatment, each column was mixed with 10-15-min bursts. The intensity of resuspension was evaluated with turbidity measurements. To be able to use turbidity as a measure of suspended solids (SS), a linear regression model between turbidity and concentration of SS was established, using different concentrations of sediment from the Kirkkojärvi basin (turbidity = 1.55x - 4.72, $R^2 = 0.9883$, $p < 0.0001$, $F = 1942.84$) (Holmroos *et al*. 2009). In the experiments, the aim was to create resuspension strong enough to transport all potentially resuspending material into the water column. The concentration of SS was elevated to 200-250 mg L^{-1} which corresponds to the mixing of the top 0.15-0.2 cm of the surface sediment material into the experimental column. Separate experimental column sets were set onto the lake bottom for the day and night experiments at the beginning of each experiment. In both study periods, all the columns were removed after the last experiment at night to avoid affecting the water quality at the study site between the day and night experiments.

2.3. Sampling and analysis

Sampling was conducted from the treated and control columns before the induced resuspension, immediately after the mixing was stopped, and then 20, 40, 60, 90, 120, 180, 240, 300, and 360 min after the resuspension treatment. The samples were taken from a depth of 45 cm with a small plastic bottle (V = 250 mL). Turbidity, pH, water temperature, chlorophyll-*a* (fluorescence), and the concentration of dissolved oxygen (DO) were measured with a YSI6600 sonde (YSI Corporation, Yellow Springs, OH, USA). The water samples were stored on ice until they were transported to and analyzed in the laboratory. The concentration of TP was analyzed from each sample according to the method of Koroleff (1979) and soluble reactive phosphorus (SRP) according to the method of Murphy and Riley (1962) (Lachat autoanalyzer, QuicChem Series8000, limit of detection 2 µg L^{-1}). For the analysis of SRP, 50 mL of water from each sample were filtered in the field immediately after sampling through a membrane filter (cellulose nitrate, pore size 0.45 µm; Sartorius AG, Goettingen, Germany).

The concentration of SRP in the sediment pore water in the experiment area was determined from two replicate surface sediment samples (0-1 cm) after centrifugation (15 min, 4000 rpm) and filtration (polycarbonate membrane filter, pore size 0.4 µm; Whatman (GE Healthcare), Fairfield, CT, USA) according to the method of Murphy and Riley (1962) (Lachat autoanalyzer).

To determine the concentration of chlorophyll-*a* in the experimental units, a linear regression model between fluorescence measurements and concentration of chlorophyll-*a* for Kirkkojärvi was used (May: chlorophyll-a = 2.19x - 3.88, $R^2 = 0.5167$, $p < 0.0001$, $F = 31.00$; August: chlorophyll-a = 6.48x - 22.75, $R^2 = 0.6269$, $p < 0.0001$, $F = 40.32$) (Holmroos *et al.* 2009). At the beginning and end of the experiments, the water temperature, pH, chlorophyll-*a*, and DO were also measured outside the columns to determine the possible effect of columns on these parameters.

2.4. Phosphorus fractionation

The chemical characteristics of the sediment P reserves affect their potential release (Williams & Mayer 1972). Despite the fractionation methods being approximate, they can be used for characterising sediment P, assessing the proportion of P which may, under certain environmental conditions, become available for production of new organic material in the aquatic system (Koski-Vähälä 2001; Lukkari 2008) The different pools of P in the surface sediment of Kirkkojärvi were determined in spring and midsummer 2007 to study the areal variation and seasonal development of these pools and thereby to aid in the interpretation of the experimental results. Surface sediments (0-1 cm) from 14 locations were collected (Fig. 1) with a Kajak corer (KC Denmark A/S, Silkeborg, Denmark) (Kajak *et al.* 1965) and the sediment P was fractioned according to the method of Chang & Jackson (1957) (modified by Hartikainen 1979). For the fractionation, two replicate samples were used and the sample volume was 8 mL. The fractionation procedure separates P into four fractions by sequential extractions with NH_4Cl (labile P), NH_4F (Al-related P, Al-P), NaOH (Fe-related P, Fe-P) and H_2SO_4 (calcium-related P, Ca-P).

2.5. Statistical analyses

The differences in the TP and SRP concentrations, temperature, chlorophyll-*a*, DO, and pH between the resuspension columns and control columns during the experiments were analyzed with analysis of variance for repeated measurements (ANOVAR) (SAS Institute Inc. 1990). Before the analyses, the normality of the datasets was verified with the Shapiro-Wilk test and the data were log-transformed if necessary. The differences in the turbidity, temperature, chlorophyll-*a*, DO, and pH between the samples taken from the control columns and outside the columns during the experiments were analysed with analysis of variance (ANOVA) (SAS Institute Inc. 1990).

3. RESULTS

*3.1. Concentration of SS, TP, SRP, and chlorophyll-*a

The resuspension treatment substantially increased the concentration of SS from the initial level in all the experiments (May, day: from 32 to 193 mg L^{-1}, night: from 29 to 193 mg L^{-1}; August, day: from 31 to 241 mg L^{-1}, night: from 30 to 233 mg L^{-1}) (Fig. 2). Thereafter, the SS concentration decreased constantly and at the end of the experiments it was approximately 50 mg L^{-1} in May and August (Fig. 2). In the control units, the concentration of SS remained at the initial level throughout the experiments and thus it differed significantly from the concentration in the treated units ($p < 0.001$) (Fig. 2).

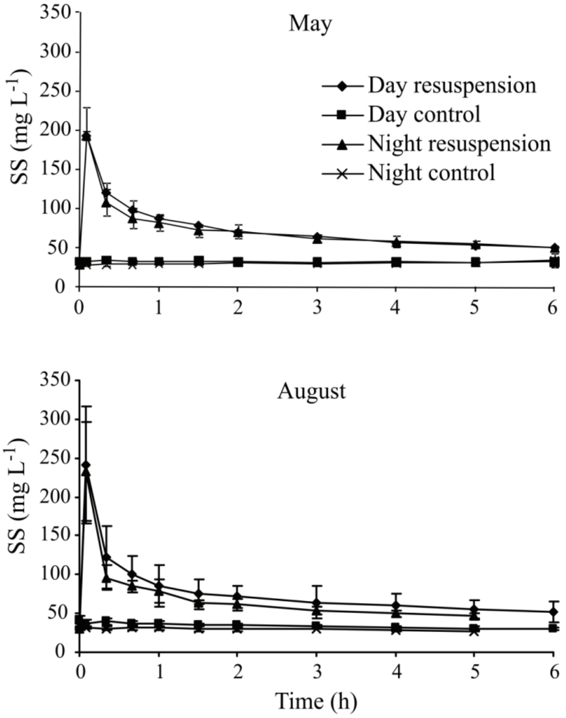

Fig. 2. Concentration of suspended solids (SS) in the day and night experiments in May and August (±95% confidence limits).

The concentration of TP closely followed the pattern of SS in the experiments of May and August during the day and night. A steep increase in the concentration after the resuspension treatment was followed by a rapid decrease and at the end of the experiment the concentration settled on close to the initial level (May, day: 77→248→84 µg L^{-1}, night: 65→231→86 µg L^{-1}; August, day: 144→372→185 µg L^{-1}, night: 151→349→179 µg L^{-1}) (Figs 3 and 4). In the control units, the concentration of TP did not change during the experiments and the difference between the treated units and the control units was significant in all the experiments ($p < 0.001$) (Figs 3 and 4).

In May during the day experiment, the resuspension treatment caused a significant increase in the SRP concentrations in the treated columns compared with the control columns ($p < 0.001$). The SRP concentration varied between 8 and 10 µg L^{-1} in the control units and between 9 and 11 µg L^{-1} in the treated units (Fig. 3). No

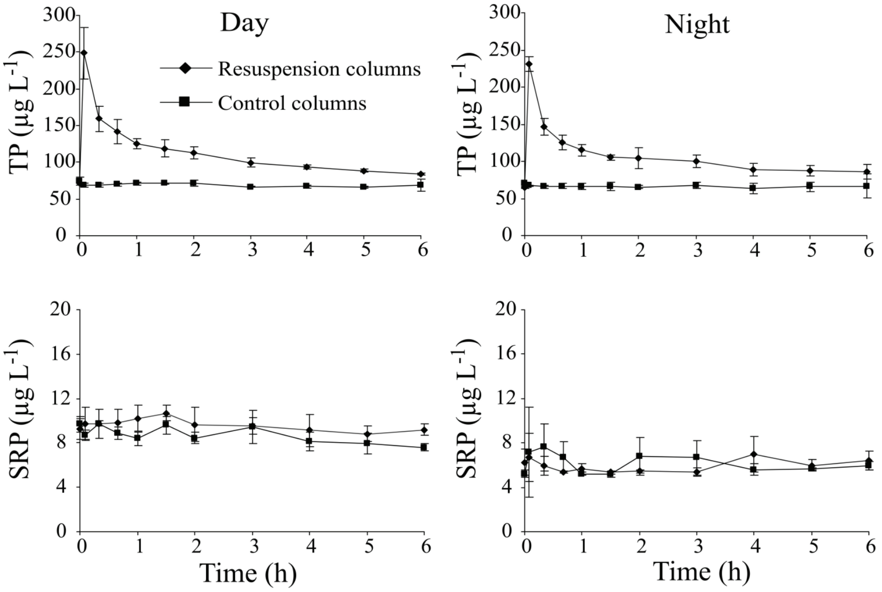

Fig. 3. Total phosphorus (TP) and soluble reactive phosphorus (SRP) concentrations in the day and night experiments in May (±95% confidence limits).

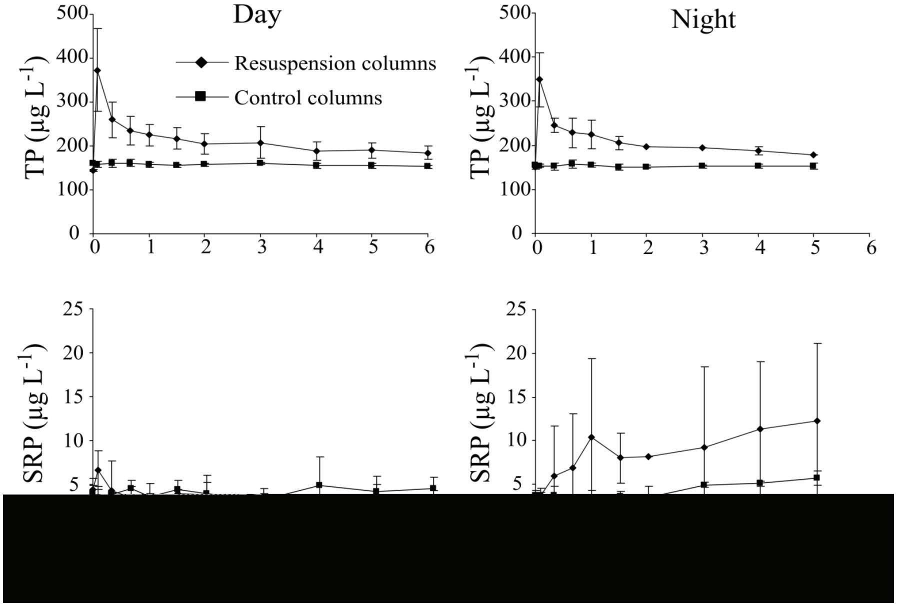

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Fig. 4. Total phosphorus (TP) and soluble reactive phosphorus (SRP) concentrations in the day and night experiments in August (±95% confidence limits).

difference was observed in the night experiment (p = 0.45). In August, the results were contrasting. In the day experiment, no difference was observed (p = 0.72), whereas in the night experiment the SRP concentration in the treated columns increased from 3 to 12 µg L^{-1} and thus was significantly higher in comparison to the control columns (p = 0.001), in which the concentration was 3-6 µg L^{-1} (Fig. 4).

In May, the concentration of chlorophyll-*a* increased slightly in the resuspension columns, due to mixing, but returned to the initial level at the end of the experiment (day: 25→32→24 µg L^{-1}, night: 22→31→25 µg L^{-1}) (p <0.001). In the control columns, the concentration of chlorophyll-*a* was 20-25 µg L^{-1}. In August, the concentrations of chlorophyll-*a* were higher than in May. During the day experiment, chlorophyll-*a* increased from 173 to 233 µg L^{-1}, due to mixing, and decreased to 211 µg L^{-1} by the end of the experiment. In the control units, the concentration of chlorophyll-*a* was 185-193 µg L^{-1}, being lower than in the treated units (p <0.001). In the night experiment, the concentration of chlorophyll-*a* was higher than during the day (p <0.001). The concentration varied from 210 to 249 µg L^{-1} in the treated units and from 197 to 223 µg L^{-1} in the control units.

3.2. Water temperature, pH, DO concentration, pore-water SRP

In May, the water temperature in the experimental columns was 15.5-17.0 °C during the day experiments and 16.0-16.8 °C during the night experiments. In August, the water temperature varied from 17.5 to 17.9 °C during the day and from 17.1 to 17.6 °C during the night. No statistical differences either between the resuspension columns and the control columns (p >0.01) or the day and night temperatures were found (p >0.01).

The water pH varied between 7.1 and 7.8 in May and between 9.3 and 9.9 in August. In the night experiments in May, the pH was slightly lower in the control units (7.2) than in the treated units (7.3) and *vice versa* in August (control: 9.9, treated: 9.8) (p <0.01). In the day experiments, no difference was observed in May and August (p >0.01). Comparison between the day and night experiments showed that the pH was higher during the day in May (7.4 *vs* 7.3) and during the night in August (9.6 *vs* 9.8) (p <0.01).

Apart from the day experiment in May, the resuspension treatment decreased the concentration of DO significantly in comparison to the control columns in all the experiments in May and August. In May, the DO concentration was higher during the day experiment (8.7-11.1 mg L^{-1}) in comparison to the night experiment (8.1-8.7 mg L^{-1}) (p <0.01). In August, the results were contrasting (p <0.01). During the day experiment, the DO concentration varied between 10.1 and 10.8 mg L^{-1} and during the night experiment between 9.8 and 11.6 mg L^{-1}.

Some of the water quality variables differed only slightly between the control columns and the lake water outside the columns in May. During the day experiment, the temperature and concentration of DO were higher in the lake water than in the control columns (p <0.01) (temperature: 17.0 *vs* 16.5 ºC, DO concentration: 10.1 *vs* 9.2 mg L^{-1}). During the night experiment in the lake water, the pH was higher and the DO concentration lower than in the control columns (p <0.01) (pH: 7.4 *vs* 7.2, DO concentration: 8.3 *vs* 8.6 mg L^{-1}). No differences were detected in August (p <0.01).

The SRP concentration in the sediment pore water in all the experiments was low, varying between 6 and 8 µg L^{-1} in May and August.

3.3. Phosphorus fractionation

The total extractable P concentrations (sum of labile P, Al-P, Fe-P, and Ca-P) measured from 14 different sites varied from 494 to 755 mg kg^{-1} in May and from 478 to 847 mg kg^{-1} in August. The concentrations were greatest in the deepest areas (depth >2 m, 731 mg kg^{-1} on average in May and 771 mg kg^{-1} in August), which cover 10% of the entire lake area (Fig. 1). In the area where the water depth was less than 2 m, the average concentration of total extractable P was 596 mg kg^{-1} in May and 651 mg kg^{-1} in August. In May, the P pools from the greatest to the smallest were Ca-P, Fe-P, Al-P, and labile P, consisting of 54.8%, 32.9%, 11.6%, and 0.6% of the total extractable P, respectively (Fig. 5). In August, the proportions of different pools were similar (Ca-P 54.0%, Fe-P 36.8%, Al-P 8.6%, and labile P 0.6%) (Fig. 5). The P pools potentially affected by pH-dependent release (Fe-P and Al-P) (Boström 1984) were, in contrast to the total extractable P, greatest in the shallow areas (depth <2 m). In May and August, the sum of these pools comprised 54% of the total extractable P.

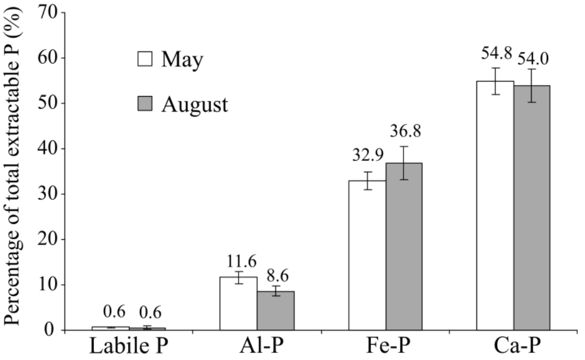

Fig. 5. Average content (samples from 14 different sites) of different sediment P pools of total extractable P in Lake Kirkkojärvi in spring and midsummer (±95% confidence limits).

4. DISCUSSION

4.1. Effect of resuspension treatment on the concentrations of SS, TP, and SRP: role of ligand-exchange reactions

The resuspension treatment increased the concentration of TP in the treated columns along with the SS concentration in all the experiments, since plenty of P was bound to particles (Holmroos *et al*. 2009). At the end of the experiments, the TP concentrations in the treated columns remained higher compared to initial concentrations due to slowly settling fine grained particles (Horppila & Nurminen 2001, 2003).

The effect of resuspension treatment to the SRP concentrations was more complicated. The SRP release was observed in May during the day and in August during the night. We concluded this release to be due to aerobic pH-dependent ligand-exchange reactions. This conclusion was supported by the observations that in August (during the night), when the release was strongest, the water pH was much higher than in May (9.3-9.9 *vs* 7.1-7.8) and the P pools susceptible to pH-dependent release were strongly represented in the surface sediment of the Kirkkojärvi basin. The Al-P and Fe-P pools, which are known to give the highest pH effect (Andersen 1975; Boström 1984), formed ~45% of the total extractable P in the entire lake area and the water pH was in the range at which the highest net release of P occurs. The minimum concentration of DO measured in the treated columns was 8.1 mg L^{-1}, representing well-oxidized conditions in all the experiments. High levels of pH in the water trigger phosphate release from aerobic sediments by OH^- ion exchange with PO_4^{3-} on the surfaces of metal oxides-hydroxides (Andersen 1975; Jensen & Andersen 1992). Our resuspension treatment brought sedimentary P into the water column, where the buffering capacity of sediment against pH changes is decreased (Drake & Heaney 1987). Due to lower pH, the P release was lower in May than in August. However, the slight increase in the SRP concentration in May was probably also due to ligand-exchange reactions, which can be accelerated by degradation of biogenic silicon after the spring diatom bloom. Large additions of Si may affect the release of P even without an increase in pH (Koski-Vähälä *et al.* 2001). This has been observed in experiments conducted with the sediment of the lake studied (Tallberg & Koski-Vähälä 2001; Holmroos *et al.* 2009). The P desorption from the particle surfaces is also affected by the initial SRP concentration of the solution (Søndergaard *et al.* 1992). This could have been one factor that led to lower P release in May, since the SRP concentration at the beginning of the experiment during the day was almost three-fold higher than in the August experiment during the night.

The conclusion that the ligand-exchange reactions were the main factor inducing SRP release was also supported by the low SRP concentrations in the sediment pore water (6-8 µg L^{-1}). Centrifugation used in separating the pore water from the sediment before the SRP concentration determination is considered to be a rough method that may result in overestimates of the real concentrations in the surface sediments (Angelidis 1997). However, even if overestimates, the measured concentrations were too low to have been the sole cause of the SRP increase observed in the water column since the amount of SRP in the sediment layer approximately entrained (~2 mm) was small (Holmroos *et al.* 2009). Centrifugation also affects the redox conditions of sediment (Angelidis 1997). The oxidizing of anoxic sediments may decrease the SRP concentration of pore water. However, the surface sediment was most likely well oxidized at the study site, due to the shallowness of the water and frequent sediment resuspension (Niemistö et al. 2009; Holmroos *et al.* 2009) as well as due to the high DO concentration measured in the water column. Increased temperatures stimulate overall mineralization and thereby liberate organic-bound P into the sediment pore water (Jensen & Andersen 1992). On the other hand, higher temperature is known to promote P retention of particles (Barrow 1979; Yli-Halla & Hartikainen 1996). However, small differences in the temperature between May and August (averaging from 1 to 1.5°C higher in August) had no effect on the SRP concentration of the pore water and were unlikely the cause for the seasonal difference in P release, since the temperature as well as the SRP release were higher in August. In part, the dissolving of the labile P pool probably affected the SRP concentrations in May and August; however, being rather low it could not explain all of the increase.

4.2. Diurnal variation in SRP release

The difference between May and August experiments in SRP release was due to differences in water pH, but the diurnal variation was more complicated to explain. As pointed out earlier, no SRP release was observed due to the resuspension treatment during the night experiment in May and during the day experiment in August. The reasons for this can be varied.

In August, the concentration of chlorophyll-*a* was high and thus the assimilation of P by phytoplankton probably concealed the P release in the experiment. Additionally, the orthophosphate uptake of the entire planktonic community, bacteria and phytoplankton, can be higher during the day than at night (Richey *et al.* 1975; Harrison *et al.* 1977; Currie & Kalff 1984). Bacterial activity in the water column of the Kirkkojärvi basin is known to be high, since even during strong phytoplankton blooms, bacteria can potentially assimilate 20% of the phytoplankton standing stock of carbon per day (Tallberg *et al.* 2005). Consequently, due to high levels of bacterial activity, the mineralization and bacterial excretion can also be expected to be high. However, since the SRP did not accumulate in the water, the excretion rates were most likely relatively slow (Currie & Kalff 1984). Moreover, under depleted conditions the turnover and thus the uptake of phosphate can be rapid (Richey 1979). During the night experiment in August, the concentration of chlorophyll-*a* as well as the DO concentration and pH were even higher than during the day. In midsummer during intensive cyanobacterial blooms, high nocturnal pH values have been previously measured in Lake Kirkkojärvi (Nurminen & Horppila 2002). Consequently, since the pH remained high but the assimilation of P by phytoplankton was most likely lower (Litchman *et al.* 2004), the ligand-exchange reactions increased the SRP concentrations in the treated columns. It could be argued that nocturnal increment in the SRP concentration was due to slowly settling fine grained particles (smaller than 0.45 µm) which remained in the water in the end of the experiment, but then increment should have been observed in all the experiments in May and August. Additionally, this would not explain the fact that the highest SRP concentration was measured in the end of the night experiment in August.

Sediment resuspension can bring the sedimented phytoplankton cells back into the water column (Carrick *et al.* 1993; Schelske *et al.* 1995). Thus, the rapidly increased chlorophyll-*a* concentration in the treated columns after the mixing was due to resuspended phytoplankton in the day and night experiments in August. Since resuspended phytoplankton may photosynthesize at rates similar to those of surface populations (Carrick *et al.* 1993), they probably attenuated (at night) or prevented (during the day) the SRP increment in the treated columns.

In May, the differences between the day and night experiments could have resulted from slightly higher water pH during the day (7.4 *vs* 7.3). On the other hand, the SRP release in the day experiment was close to the resolution limit of the determination method. Consequently, sediment resuspension under prevailing conditions caused only very small or negligible SRP release.

4.3. Importance of aerobic phosphorus release for the Kirkkojärvi basin

The differences in the water quality parameters between the control columns and in the water outside the columns were only minor (in May) or negligible (in August), and therefore the SRP release observed can also be expected to occur during a resuspension event in the lake. The importance of resuspension-mediated pH-dependent aerobic SRP release for the Kirkkojärvi basin was emphasized by the fact that the lake is very shallow and its bottom sediment is often prone to resuspension. There were no marked seasonal differences in the different P pools of the surface sediment and when the quite restricted deepest areas of the lake (depth >2 m) are excluded, the P pools susceptible to the pH-dependent P release (Al-P and Fe-P) formed 54% of the total extractable P. The lake area shallower than 2 m covers 90% of the entire lake area and the model of Scheffer (1998) suggests that 50-85% of this area is potentially resuspended when the wind velocity is 3-4 m s^{-1}. Such conditions occur very often in the Kirkkojärvi basin (Horppila & Nurminen 2005). The pools of Al-P and Fe-P become susceptible to the aerobic SRP release as the phytoplankton assemblage attains high levels of abundance and creates conditions of high pH and as the wind velocities mentioned above are exceeded. The aerobic release of P can occur over a long period during the growing season, since sediment is frequently resuspended and strong phytoplankton blooms creating conditions of high pH are common from early July to late September (Horppila 2005). Additionally, the water pH can stay at a high level also at night during strong phytoplankton blooms.

5. CONCLUSIONS

The effect of resuspension treatment led to significant aerobic SRP release during the night experiment in August, when the water pH was high due to intensive phytoplankton blooms. Conditions of high water pH enabled SRP release by ligand-exhange reactions. The SRP release was not observed during the day experiment, probably due to strong P assimilation of the planktonic community (bacteria and phytoplankton). The aerobic release of P can occur over a long period during the growing season in the Kirkkojärvi basin, since sediment is frequently resuspended and strong phytoplankton blooms creating conditions of high pH are common from early July to late September. During strong phytoplankton blooms, the water pH can stay at a high level also at night.

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