Gypsum treatment in managing the internal phosphorus load from sapropelic sediments; experiments on Lake Laikkalammi, Finland

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The sediment of the hypertrophic Lake Laikkalammi, (area 8000 m^2 , max. depth 13 m) in southern Finland was treated with the industrial by-product Fe-gypsum (CaSO₄ × $2H_2O$) in summer 1998. A 90% reduction of the hypolimnic total phosphorus content was achieved and Secchi disk transparency increased from 50 cm to 270 cm during one year long observation period. Gypsum is hypothesized to limit the internal load through three interacting mechanisms: as a mechanical cover limiting the resuspension and gas ebullition, as a chemical offering binding sites for phosphorus (Ca, Fe) and finally by changing the microbiological composition of sediment by creating more favorable conditions for sulphur bacteria instead of methane bacteria. Fe-gypsum treatment can be seen as a complementary lake restoration method that is suitable for relatively deep, small lakes in which the water quality problems are a consequence of internal loading caused by oxygen deficiency in the hypolimnion.

Introduction

Eutrophication remains a major problem in Finnish lakes deteriorating their value as potable water and recreational resources. According to a recent study (Turunen and Äystö 2000), there are nearly 1000 named lakes suffering from eutrophication. Overloading of phosphorus is the main nutrient problem. The sources were earlier related to effluents from municipal and industrial wastewaters (Alhonen 1967), but nowadays the diffuse load from cultivated fields forms a key factor to

be taken into account when managing the problem (Ekholm 1998). In numerous lakes, the internal load from the sediments accelerates nutrient disturbances. This means that sediments recirculate nutrients back to the water column according to various principal mechanisms. Nutrient release from bottom sediments can be enhanced with resuspension driven by wind (Kettunen and Stenmark 1982), bioturbation caused by bottom animals or fish (Horppila 1994) or methane ebullition related to severe oxygen deficiency (E. Varjo et al. unpubl.).

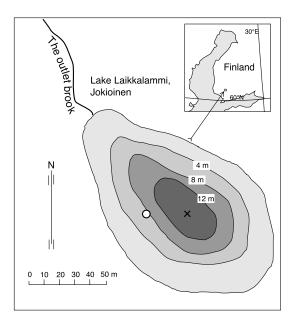


Fig. 1. Lake Laikkalammi; the water sample location (x) and the sediment sample location (o).

Public concern has initiated hundreds of lake- and watershed-scale restoration projects in Finland especially during the 1990s. The restoration of a lake is a multi-targeted long term operation aimed at reducing nutrient inflows from point and diffuse sources within the catchment. Biomanipulation is often included (Horppila 1994).

Only few restoration projects deal with the source of the internal load itself, the sediment. Experiments on the precipitation of phosphorus in a water mass using chemicals such as Al₂(SO₄)₃ and Fe₂(SO₄)₃ or their combinations have been conducted on about twenty lakes in Finland (Äystö 1997). The mechanical treatment of sediments has also been applied in certain cases abroad (Cooke 1980, 1986, Olem and Flock 1990, Baker *et al.* 1993, Sommarlund *et al.*

Table 1. Water quality variables 13 August 1997 (Kokemäenjoen Vesistön VSY 1997).

	5 m	12 m
36	160	4 600
970 8.7	1800 0.0	20 000 0.0
	970	970 1800

1998). Sediment inactivation based on chemical treatment of the sediment has been applied using Fe- and Al-compounds (Oravainen 1990), lime (Eiseltová 1994) or nitrate (Ripl 1976).

In our previous experiments (Salonen and Varjo 2000), gypsum addition to the sediment was shown to have potential in lake sediment-based restoration by reducing the internal loading of phosphorus from nutrient-rich sediments in anaerobic conditions. In Enäjärvi, the experimental addition of gypsum led to a decrease in oxygen demand by the sediment and to an increase in its nutrient binding capacity, as shown by the reduced values of tot-P and tot-N in the water mass. The chlorophyll *a* concentrations decreased remarkably as well.

One of the proposed mechanisms causing the observed positive effects is that the gypsum addition limits gas ebullition from the benthic sediment, which according to Matinvesi(1996) enhances nutrient release. Gypsum treatment creates favorable conditions for sulphate-reducing bacteria to dominate the organic matter decomposition process and thus limits the microflotation effects of methane bacteria (E. Varjo et al. unpubl.). This means that gypsum treatment has a limiting effect on one form of the basic mechanisms of the internal load, i.e. microbiologically based nutrient release. Affecting the microbiological environment responsible for the internal load requires cutting down the methane generating bacterial activity. This can be done by oxidizing the hypolimnic environment or with chemical treatment of the sediment surface. Here we report for the first time on an experiment where the sediment of a small, relatively deep hypereutrophic lake was treated with gypsum in order to change the sediment environment to support sulphate reducing (SRB) instead of methane forming bacteria.

Description of the lake

Lake Laikkalammi (area 8000 m², max. depth 13 m), in the municipality of Jokioinen in southern Finland (60°50′N, 23°31′E) is a small lake occupying a kettle depression on the slope of an esker (Fig. 1). The average depth of the lake is 7 m, and the water volume is approximately

46 000 m³. In 1997, the water was anoxic below the 1.5 meters depth, and an intense internal load maintained a very high phosphorus level: a value of 4600 μ g l⁻¹ P at the depth of 12 m was measured (Table 1). The lake has a small catchment without any inflowing brooks, and it recharges directly from groundwater. People from the area remember that until the 1970s the basin was a pure clear water lake with high transparency of water. Intensive cattle, pig and horse farming are the probable reasons for the present nutrient problems in this tiny watershed.

The poor water quality of the lake has resulted in blue-green algae blooms and the Secchi depth transparency has been as low as 50 cm during the summertime. The only fish living in the lake is crucian carp (*Carassius carassius*), a cyprinid fish tolerating the anoxia of the water during the long winter period.

The surface sediment of the lake from a water depth of 2 m downward is black sapropel gyttja. It has an average water content of about 90% and loss-on-ignition of 40%. The total phosphorus content averages 3.5 mg g⁻¹ in dry matter as can be seen from the sample taken from a water depth of 10 meters (Table 3).

Experiment and monitoring

Ferrogypsum was used in the Laikkalammi sediment restoration experiment. Fe-gypsum is a byproduct from the production of titanium dioxide pigments. The composition of the gypsum used is listed in Table 2. Mineralogically gypsum is dihydrate (CaSO₄ × 2H₂O), which accounts for about 65%–70% of the material. The rest is composed of 10% calcium carbonate (CaCO₃) 10% titanium hydroxide (TiOOH) and 6%–7% iron hydroxide (FeOOH). The median grain size is 4 μ m, $d_{10} = 0.8$ μ m and $d_{10} = 45$ μ m. The gypsum has a water content of about 20%–27% per volume.

The Lake Laikkalammi sediment was treated with gypsum on 23 June 1998. Altogether 23 m³ of gypsum was distributed directly into the lake from a motorboat. Dry homogenized gypsum powder was moved from the tank vehicle with pressured air, and it was blown into the water by means of a special device constructed for liming

the acidified waters (Fig. 2). For the purposes of the experiment the sediment of the lake was covered with an average layer of 0.5 cm layer of gypsum. To compare this with the water volume, the dosage was about 500 ml m⁻³ equaling 500 g gypsum m⁻³ of lake water.

The water quality before and after the gypsum addition was monitored for tot-P, phosphate-P, tot-N, nitrate-N, ammonium-N, pH, O₂, Secchi depth, sulphate and Fe at about two weeks intervals. The water samples were taken from the deepest point of the lake from the depths of 12 m, 6 m and 1 m. The monitoring lasted one year, i.e. from 4 June 1998 to 25 October 1999. The lake was covered by ice from November 1998 to April 1999.

The water analyses for nutrients were performed at the laboratory of the Agricultural Research Centre of Finland, Jokioinen and the following standard methods were applied: SFS 3025, SFS 3026, SFS 3030 and SFS 3023. Tot-P, phosphate-P and tot-N were analysed with the Tectator FIAstar (autoanalysator) device and the others with the Skalar (autoanalysator) device. The samples were filtered with 0.45 μ m Sartorius cellulose nitrate filtrates (CAN for tot-N and PFN for the other nutrients) right before the analysis.

The sulphate and iron concentrations were analysed at the Geology department of the University of Turku with a spectrophotometer (HACH-DR/2000). Oxygen concentrations and pH were measured *in situ* with the YSI-600 XL field water quality monitor.

Table 2. The chemical composition of Fe-gypsum (analysed by ICP-MS and ICP-AES at the Geological Survey of Finland).

	mg kg ⁻¹		mg kg ⁻¹
Ca	145 000	Cr	273
S	59 000	V	270
Fe	40 000	Ni	76
Al	3 600	Р	60
Mg	2 100	Co	47
Ti	1 230	Cu	37
K	880	Pb	14
Mn	820	U	1.8
Na	730	As	0.68
Zn	384	Cd	0.47



Fig. 2. A motor boat was used to distribute the gypsum through the lake water to sediments of Lake Laikkalammi.

The sediment was sampled before and one year after the experiment to describe changes in sediment properties. Analyses were performed at the Geology department of the University of Turku. Total phosphorus was analysed with the Bengtsson and Enell method (1986) and the sequential extraction method (Hieltes and Lijklema 1980) was applied to determine phosphorus fractions.

Results

The addition of 23 m³ of gypsum made the water turbulent and the gypsum stayed in suspension in the topmost water column during the first two days. After that it started to flocculate and settle down, being all deposited on the bottom within a week. Gypsum was spread evenly within the water column of the lake, and no dead fish were observed. Results from water analyses are presented in Figs. 3–5.

Total phosphorus concentration at 12 m depth decreased rapidly after the treatment from 4640 μg l⁻¹ to about 500 μg l⁻¹ (Fig. 3). During the observation period a slightly increasing trend leading to a concentration of 960 μg l⁻¹ was noticed. Phosphate phosphorus fluctuated more (Fig. 3). The values varied from 2 to 57 μg l⁻¹ without any visible trend in time.

The concentration curve for nitrogen greatly

resembled that of the total phosphorus. A sudden decrease in concentration followed the gypsum treatment. The initial concentration was about 26 mg l⁻¹, and it decreased to 5 mg l⁻¹ following the gypsum addition. The total nitrogen level rose steadily approaching a concentration of 16 mg l⁻¹ at the end of the experiment (Fig. 3). The ammonium trend followed closely that of the total nitrogen (Fig. 3), whereas nitrate (Fig. 3) levels also decreased remarkably due to the gypsum treatment, from 580 μ g l⁻¹ to about 50 μ g l⁻¹, but remained at that lower level until the end of the experiment.

The sulphate concentrations showed the opposite trend. The background levels were at the detection limit, but they increased to 130 mg l^{-1} , and later to 190 mg l^{-1} during the experiment. However, during the last half of the experiment, sulphate levels decreased to about 100 mg l^{-1} .

The iron concentrations fluctuated throughout the experiment, with seemingly no effects from the gypsum addition. The same holds true with the pH; no clear trend. Oxygen levels stayed low, normally around 0.5 mg l⁻¹. An oxygen deficit occurred for the first time in winter 1998, but summer values were the highest measured during the observed period.

In the intermediate water mass (Fig. 4) the phosphorus concentration of the water stayed at about the same level (370 µg l⁻¹1) or even rose after the gypsum treatment. The phosphorus

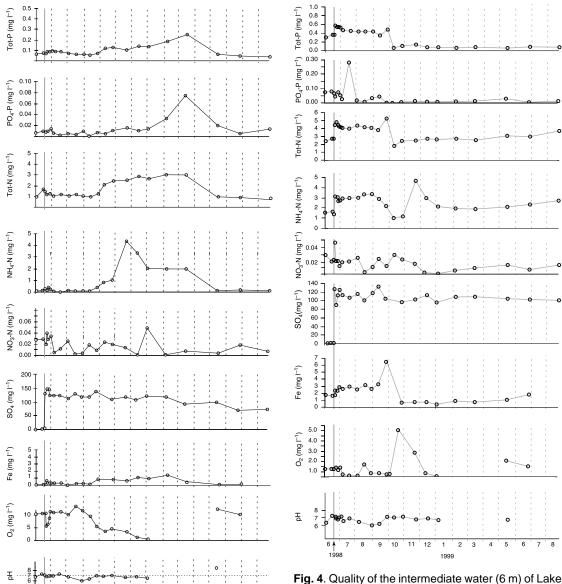


Fig. 3. Quality of the hypolimnic water of Lake Laikkalammi (12 m) from 4 June 1998 to 26 October 1999.

content decreased later. The decrease from 486 to 93 μg l⁻¹ recorded first time in the sample from 13 October. Phosphate phosphorus fluctuated at the beginning (Fig. 4), but stayed at a low level (ca. 10 μg l⁻¹) towards the end of the experiment.

Nitrogen behaved similarly to total phospho-

Laikkalammi from 4 June 1998 to 26 October 1999.

rus. At first there was a rise in concentrations, which then decreased in October. Sulphate levels increased from the background to about 120 mg l⁻¹, and the concentration only decreased slowly to about 100 mg l⁻¹ at the end of the experiment. The iron concentration increased in the intermediate water mass from 2 to nearly 7 mg l⁻¹, but went down in October 1998, and remained there until the end of the experiment.

Oxygen concentration was low during the first summer, but in October O₂ levels rose

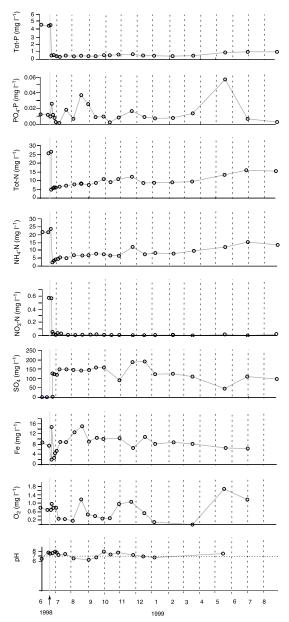


Fig. 5. Quality of the epilimnic water of the Lake Laikkalammi (1 m) from 4 June 1998 to 26 October 1999.

temporarily to about 5 mg l⁻¹. Two measurements from summer 1999 indicated some higher oxygen values than earlier (Table 1). The pH was rather steady throughout the experiment.

In the epilimnic water (Fig. 5) the total phosphorus levels did not seem to change much following the gypsum addition. The concentration stayed at about 70 µg l⁻¹ increasing during

late winter to its maximum, 250 μ g l⁻¹. In summer 1999 tot-P stayed at about 40 μ g l⁻¹. Phosphate followed the trend of tot-P (Fig. 5). Nitrogen remained the same, its concentration not being directly affected by the gypsum treatment. Nitrogen levels became higher during the wintertime, and fell back to about 1 mg l⁻¹ in summer 1999. Ammonium (Fig. 5) had the highest values during the ice cover period.

Sulphate (Fig. 5) also rose in epilimnion as it did in the whole water column. The highest value (148 mg l⁻¹) was measured soon after the treatment, and the concentration decreased steadily to about 70 mg l⁻¹ by the end of the experiment. The iron concentration fluctuated showing the highest value in the winter. The pH was circumneutral and oxygen was high in the summer, but even in the epilimnic water it decreased to about 0.5 mg l⁻¹ during the wintertime. There was a clear trend towards higher transparency of water during the experiment (Fig. 6).

Table 3 shows the sediment properties measured at the 10 m deep sampling point before the experiment and from the samples taken after the one year long experiment period. The effects of gypsum can be observed by comparing the topmost 2-cm layers of samples. It can be noted that the gypsum treatment slightly increased the concentration of total phosphorus in the sediment, but significantly the amount of NaOH-extractable fraction. The residual fraction (organic phosphorus) decreased, as did the proportion of labile phosphorus (extracted by ammoniumchloride).

Discussion

Water quality parameters

The rapid and remarkable decrease of tot-P at 6 and 12-m depths was most probably a consequence of the chemical binding reaction caused by fine-grained gypsum as it sank through the water mass into the sediment. The reduction for the whole water mass can be calculated to 62% after one year of the experiment.

When the gypsum settled in the sediment, it also enhanced the binding of phosphorus by changing the properties of the sediment. Phos-

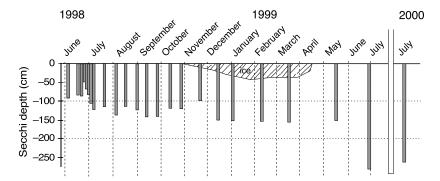


Fig. 6. Transparency (Secchi depth) in Lake Laikkalammi from 4 June 1998 to 31 July 2000.

phorus forms precipitates and complexes with iron and calcium (Cooke *et al.* 1993). Both Fe and Ca were included in the gypsum used in the experiment (Table 2). Fe and Ca can result in significant phosphorus removal by forming Ca-and Fe-phosphates in both the water mass and sediment (Cooke *et al.* 1993). This explains why the concentrations of the hypolimnic phosphorus remained low. Gypsum prohibited the internal load (Fig. 7).

The temporary increase in phosphorus concentrations at the 6-m water depth can be interpreted as a consequence of the mixing of water mass during the injection of the gypsum into the lake following phosphorus transportation from the hypolimnion. However, the autumn overturn decreased the metalimnic phosphorus concentration to a much lower level than that before the treatment (Fig. 4). The low concentration remained until the latest observation. The autumn circulation period increased slightly the concentration of the epilimnic phosphorus (Fig. 5) as well. After the winter ice period and the beginning of the growing season in spring, the epilimnic concentration also reached a level lower than before the treatment.

The effects of the treatment on the phosphorus concentrations in the water mass can be first and most strongly seen in the hypolimnion due to the strong effects of the gypsum addition on the sedimental phosphorus binding capacity. As a consequence of the reduction of the internal load from the sediments, the phosphorus concentrations in the total water mass started to decrease in time. However, the PO₄-P concentrations fluctuated throughout the observation period without any clear or significant response to the treatment.

The changes in nitrogen concentrations are as pronounced as those observed in phosphorus. The rapid decrease of the concentrations right after the treatment was probably a result of the transportation of nitrogen into the sediment together with the organic material bound to the gypsum and the mixing of the water mass. The turbulation may also have accelerated bacterially mediated changes in nitrogen compounds (NH⁴⁺, NO²⁻, NO³⁻, N₂O, N₂) and removed them from the water mass.

In the epilimnion, the maximum concentration of nitrogen can be found in the winter ice cover period due to increased solubility of nitrogen and the colder temperature of the water (Wetzel 1983). Under warmer conditions in spring the solubility and the concentration of nitrogen decrease again. The short-term decrease of the NH₄-N at 6-m depth is a consequence of circulation of the water mass after which the oxygen content decreases and the NH₄-N content increases again. The winter ice cover and

Table 3. Sediment properties of Lake Laikkalammi (DM = dry matter).

	Before the treatment	After the treatment		
	0–2 cm	0–2 cm	2-4 cm	4–6 cm
Water content (%)	96.0	97.5	95.1	95.5
Loss-on-ignition (%)	60.5	43.5	47.1	53.8
Tot-P (mg g ⁻¹ DM)	3.56	3.74	3.25	3.31
NH ₄ CI-P (mg g ⁻¹ DM)	0.19	0.00	0.00	0.01
NaOH-P (mg g-1 DM)	1.47	3.16	1.79	1.12
HCI-P (mg g ⁻¹ DM)	0.46	0.47	0.19	0.29
Res-P (mg g ⁻¹ DM)	1.44	0.11	1.26	1.88

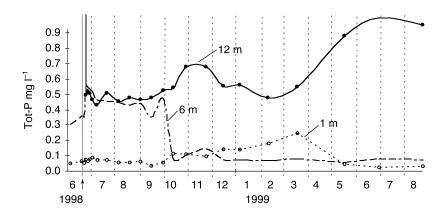


Fig. 7. The effect of gypsum addition (23 June 1998) on the tot-P concentration in water mass of Lake Laikkalammi.

lack of oxygen keep the NH₄-N content high. In the lower hypolimnion, the decrease in the nitrogen contents above the sediment is presumably related to bacterial fixation.

The increase in sulphate concentrations in the whole water mass after the gypsum treatment was expected because of the relative high solubility of gypsum. During the first two days of settling down into the sediment, the gypsum dissolved in some extent into the water column. Thereafter, the sulphate concentration trend decreased indicating that the dissolution from the sediment was not increasing and that the metabolic activity of sulphur bacteria was reducing sulphate from the water mass. The discharge of the lake water is almost nonexistent and thus the decreasing trend of sulphate can be interpreted to indicate an improved environment for SRB in the sediment. When sulphate from the gypsum in the sediment has been used up, sulphur bacteria tend to use the sulphate from the overlying water as electron acceptors in organic matter decomposition.

Changes of iron concentrations in the water mass are related to short-term mixing right after the treatment and the autumnal water circulation, but no permanent effect of the treatment can be observed.

The turbulation of the water mass right after the gypsum addition resulted in short-term changes as well in oxygen concentrations. This was observed as increasing concentrations in the hypolimnion and decreasing concentrations in the epilimnion. The transportation of epilimnic water closer to the benthos accelerated the bacterial decomposition of organic material. The effects of gypsum treatment into oxygen concentrations was indicated by a slight increase the following summer, most probably due to the decrease of decomposable organic material as a consequence of the lowering of nutrient concentrations.

A slight decrease in pH is usual with the dissolution of gypsum. In our experiment, the addition of gypsum did not affect the pH values of the water mass due to the relative low amount of gypsum in proportion to the lake volume.

Transparency reflects the trophic state of the lake. As the gypsum sank through the water mass it absorbed particular matter which then settled on the bottom. This was observed in the form of increased Secchi depth right after the final deposition of the gypsum. The Secchi depth, unusually high in relation to the recent history of the lake, remained high throughout the whole observation period. Two years after the treatment, the Secchi depth was still fivefold compared to the pre-experimental conditions indicating decreased productivity of the lake (Fig. 6).

Sediment properties

The changes in water quality can be explained not only by the binding effect of gypsum when sinking through the water mass but also by the improvement in sediment quality. The gypsum addition into the sediment created more sites for phosphorus to bind (Fe, Ca) and thus led to more stabile phosphorus fractions formation. The tot-P concentration in the sediment somehow increased despite the clearly lower P-con-

centration of the gypsum (Table 2) compared with the original sediment (Table 3). This indicated that more phosphorus from the water had been captured by the sediment and/or that the phosphorus did not escape from its sedimentary reserves back to the water column. The easily leachable fraction (NH₄Cl) was bound more strongly and increased the proportion of NaOH-extractable fraction including Fe- and Al-bound and weak Ca-P compounds. Due to the fact that the chrystallization of Ca-P-compounds is a slow process, the changes in HCl-extractable fraction could not be detected (Table 3).

The LOI (loss on ignition) of the sediment along with the residual fraction of phosphorus decreased indicating accelerated decomposition of organic matter and also dilution of gypsum. The sulphate addition into the sediment from the gypsum presumably created favorable conditions for sulphur bacteria to grow and decompose organic material. These conditions are enhanced due to the elevated sulphate concentration of the water mass. The dark-colored sulphide-containing surface layer on gypsum-containing sediment found in this experiment is an indication of the SRB activity. As long as sufficient sulphate is present, the sulphate-reducing bacteria will dominate methane-producing bacteria (Steenberg et al. 1993). In our previous experiments (E. Varjo et al., unpubl.) the inverse relation between methane bacteria activity and gypsum treatment has been pointed out. The treatment limits the methane convection currents and transportation of particles and dissolved substances from sediment to water mass thereby decreasing the internal loading.

Applicability

Gypsum treatment can be applied to phosphorus precipitation from the water column and to sediment inactivation. Earlier, aluminium and iron compounds were used for the most part for similar purposes (Eiseltová 1994). The use of lime has also been recommended. As compared with iron, the use of aluminium is more costeffective, but aluminium may have harmful effects on organisms when used in high concentrations (Eiseltová 1994). The problem in precipi-

tations in soft water lakes is usually that the dosage of chemical should be as high as possible to maintain the phosphorus precipitates in sediment. However, the low buffering capacity of waters and the phytotoxity of aluminium prohibit high doses and the reaction between aluminium and H₂S liberates phosphorus from the sediment back into the water column (Oravainen 1990). Gypsum treatment does not have these drawbacks. It can be applied in much larger amounts (500 g m⁻³ in this experiment) compared to other chemicals used in phosphorus precipitation and sediment treatment. In Finnish softwater lakes doses of 20-90 g m⁻³ of iron and aluminium compounds have been safe to use (Äystö 1997). Evidently this is much too little to maintain a permanent or prolonged decrease in phosphorus concentration.

Conclusions

Ferro-gypsum treatment appears to be a versatile method in lake restoration as compared with other sediment inactivation methods. It seems to act through three interacting mechanisms: as a mechanical cover, a chemical offering binding sites for phosphorus and finally it changes the microbiological composition of sediment by creating more favorable conditions to sulphur bacteria instead of methane bacteria (E. Varjo *et al.* unpubl.).

In the Lake Laikkalammi experiment, we found that a 90% reduction of total phosphorus content was reached in the hypolimnic water. After one year the reduction in P concentration for the whole water mass was calculated to be 62%. The gypsum treatment increased the sulphate content of the water body from close to zero to about 150 mg l⁻¹. Due to precipitation of monosulphides and use of sulphate as an electron acceptor, the sulphate concentration decreased to about 100 mg l⁻¹. There was no marked change in the Fe-concentration or pH-value. Transparency increased from 50 cm to 270 cm during the observation period as a result of the experiment.

These results support the hypothesis of gypsum effects on changing sediment properties: The Fe-gypsum addition improves the living conditions for heterotrophic sulphate reducing bacteria and has a moderating effect on methanogenesis. Furthermore, the gypsum treatment adds new binding sites (Fe, Ca) for phosphate to precipitate, which can be observed in the increase of stabile phosphorus fractions in the sediment. This also prohibits the internal loading from the sediment.

Fe-gypsum is interesting as a lake sediment restoration chemical for the following reasons: Fe-gypsum is an industrial by-product for which no large-scale use has been found to date. The use of gypsum offers a comprehensive control of materials lifecycle. The reactions of gypsum in sediment are natural and resistant. Gypsum can be portioned out in such large quantities that water quality problems caused by bacteria in the sapropelic sediment can be solved.

Fe-gypsum treatment can be seen as a complementary lake restoration method that is suitable for relatively deep, small-scale lakes where water quality problems are a consequence of internal loading caused by oxygen deficiency in the hypolimnion. However, this does not imply that other gypsum by-products, e.g. phosphogypsum could be applied to lake sediment restoration as well.

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