The Efficiency of P-Removal from Natural Waters with Sorbents Placed in Water Permeable Nonwovens

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Abstract. An important aspect of lake restoration is the reduction of phosphorus (P) concentrations in water. To achieve this effect, various methods are used, including chemical inactivation by means of the addition of different substances into the water column or sediment. Although the efficiency of chemical methods is high, there are numerous concerns related to the introduction of large amounts of atypical compounds to aquatic ecosystems, eg. the possibility of the appearance of their toxic forms, such as aluminium as a result of pH changes. The aim of the research was to evaluate the possibility of using water-permeable nonwovens as P sorbent carriers. A bag made of nonwoven is filled with the sorbing material. When placed in the water column, sorbent present in the bag reduces orthophosphate (PO$_4^{3-}$) concentration in the water due to the water inflow into the bag. The bag is then removed from the water together with the sorbent and sorbed PO$_4^{3-}$, which enables permanent removal of P and sorbent from the water reservoirs. Three synthetic sorbents were used in the study: SINOBENT (form: paste, composition: calcium, iron and magnesium embedded in a bentonite carrier), HYDROPHOSBIND (hydrogel capsules, biopolymer with embedded FeCl$_3$), precipitated calcite (PCC, powder, CaCO$_3$) and one natural sorbent: ground calcite (GCC, powder, CaCO$_3$). Water samples for the study were taken from the eutrophic lake from the epi- and hypolimnion zone. Sorbents efficiency was tested in low (0.043 and 0.067 mg PO$_4^{3-}$/L) and high (0.230 and 0.540 mg PO$_4^{3-}$/L) concentrations at two temperatures (6°C and 20°C). Sorption of PO$_4^{3-}$ was tested for the sorbents placed in a bag made of permeable nonwovens. The dose of materials was 1g/L, except for HYDROPHOSBIND (5 g/L). Additional experiments were carried out with the addition of effective microorganisms to check their effect of the sorption behaviour of the tested sorbents. An unambiguous PO$_4^{3-}$ loss from the solution was observed for three materials: PCC, GCC and HYDROPHOSBIND. Given for 1g of the sorbent, the highest losses (19-79%) were found in PCC and the lowest (5-13%) in HYDROPHOSBIND treatments. For these materials, the effectiveness of PO$_4^{3-}$ removal raised with rising temperature and the initial PO$_4^{3-}$ concentration. The addition of probiotic organisms favoured the reduction of PO$_4^{3-}$ concentration, especially in the presence of PCC. The addition of SINOBENT usually resulted in an increase in PO$_4^{3-}$ concentration and pH decrease even to pH=6.45.

1. Introduction
The long-term monitoring and experimental studies on the causes and rate of eutrophication of water ecosystems have shown that in most cases the main factor controlling the development of planktonic organisms in water is the content of P. The deficiency of this element in the aquatic environment leads to a significant reduction of the biomass growth, while the excess usually accelerates this process. Bioavailability of P in the water column can be limited by immobilization of dissolved P and its deposition in the bottom sediment [1, 2]. In the lakes’ restoration practice, coagulants (iron chloride and sulphate (VI)) or aluminium (sulphate (VI)), added directly into the water column, are most often used for P inactivation [3-6]. The most modern P adsorbent used for this purposes is Phoslock - a mixture of sodium bentonite and lanthanum operating in a wide pH range and under anaerobic conditions [7, 8].
Although the applied chemical methods show high efficiency, they raise numerous concerns related to the introduction of large amounts of atypical compounds to aquatic ecosystems and their effect on biota [9-11]. The optimal solution would be to bind phosphates on natural sorption material and to remove them from the lake's ecosystem.

The aim of the research was to assess the possibility of using water-permeable nonwovens as carriers of phosphate sorption materials. When placed in the water column, sorbent present in the package (bag) reduces phosphate (PO\(_4^{3-}\)) concentration in the water due to water inflow into the bag. The bag is then removed from the water together with the sorbent and sorbed PO\(_4^{3-}\), which enables permanent removal of P and sorbent from the reservoir.

### 2. Materials and methods

#### 2.1 Sorbing materials

As sorption materials for phosphates dissolved in lake water, three synthetic and one natural material were used.

**SINOBENT** - a synthetic preparation in the form of a paste, containing calcium, iron and magnesium embedded in a mineral carrier (bentonite). Its action consists mainly in the liberation of iron, which then forms iron III hydroxide, adsorbing phosphates on its surface. The presence of nitrates in the material provides the redox potential suitable for this process. Calcium and magnesium additionally affect sorption. The product was developed for the purpose of surface water reclamation and was applied in Polish lakes by application in a loose form to the water column to bind P in water, and after sedimentation – to inhibit of P release from bottom sediments [12, 13].

**HYDROPHOSBIND** - a synthetic preparation developed for the removal of dissolved P from surface water, in the form of hydrogel capsules made of biopolymer with embedded iron (III) in the form of FeCl\(_3\) coagulant. Sorption of P by capsules can take place in two ways: by adsorption on the capsule activated by the presence of metal and by P precipitation P with Fe. The maximum phosphate adsorption determined in deionized water and waters of two lakes was 30.3-50.0 mg P/g [14].

**Precipitated calcium carbonate (PCC)** - synthetic pure calcite (one of calcium carbonate polymorphs) with a grain diameter \(d_n\) equal to 19 \(\mu\)m and BET surface area of 13.2 m\(^2\)/g. The preparation is used, among others, in the pharmaceutical and chemical industry. This material was previously tested as a P binding agent in bottom sediments undergoing artificial resuspension on a laboratory scale when applied directly to bottom sediments. The maximum PCC adsorption, determined in the synthetic solution and porewater of the eutrophic lake, was 0.76-1.72 mg P/g, depending on the concentration of phosphates. The preparation was previously tested as a P binding tool in bottom sediments undergoing artificial resuspension on a laboratory scale when applied directly to bottom sediments to bind P in sediments [15].

**Ground calcium carbonate (GCC)** - limestone grinded to a limestone powder, containing >96% calcium carbonate as calcite, produced for the removal of sulphates from the exhaust. A<63 \(\mu\)m fraction was used in the studies. The maximum adsorption on this material, determined in the water of the eutrophic lake, is 0.65 mg P/g [16]. BET surface area is 6 m\(^2\)/g.

#### 2.2 Experimental design

Sorbents were placed in bags made of a water-permeable nonwoven, which were immersed in containers with lake water characterized by different concentrations of phosphates (table 1). The bags containing sorbing materials were then incubated in darkness for 48h. In the case of SINOBENT, PCC and GCC, a dose of 1g/L was used for testing, whereas for HYDROPHOSBIND it was 5g/L.

In the experiments, the natural lake water was used, taken in autumn from the Stężyckie Lake (Poland). It is a gutter lake with an area of 62 ha and the maximum depth of 12 m, located in the
Kashubian Lake District. Surface and bottom water samples were taken from two stations: "littoral" and "profundal". Lake water was filtered through paper filters. Incubations were carried out in the dark at two temperatures: 6°C - "profundal" and 20°C - "littoral". An additional aspect of the research was to analyse the effect of the presence of beneficial microorganisms on the removal of P from lake water using sorbents placed in nonwovens. For this purpose, a liquid biopreparation (commercially named, EmFarma PlusTM) was used at a dilution of R = 100,000, taking into account the typical dose recommended for introduction into the water reservoirs. In all experiments, the same scheme of the sample set was used: 1) control (lake water and lake water with the addition of probiotics) and 2) sorbent treatments (lake water incubated in the presence of bags with sorbents, with and without the addition of probiotics). Phosphate concentration and pH were measured before the experiment and during incubation after 2, 4, 6, 12, 24, 48 hours. Total P (P\textsubscript{tot}) was determined in the samples before and after the experiment to check, if the decrease in phosphate concentration is caused by sorption onto sorbing materials (allowing for long term phosphate removal after the bag is taken out of water) and not by transformation processes, such as precipitation from the solution, e.g. with the iron or calcium ions released by sorbing materials.

Table 1. The characteristics of lake water used in the sorption experiment

| Station (depth) | Temperature [°C] | Layer | Phosphates (PO\textsubscript{4}\textsuperscript{3-}) [mg/L] | Total phosphorus (P\textsubscript{tot}) [mg/L] | P-PO\textsubscript{4}/P\textsubscript{tot} [%] |
|----------------|------------------|-------|----------------------|---------------------|--------------------------|
| "profundal" (12 m) | 6 | surface | 0.043 | 0.035 | 40 |
| □ bottom | | | 0.540 | 0.240 | 73 |
| "littoral" (2 m) | 20 | surface | 0.067 | 0.043 | 52 |
| □ bottom | | | 0.230 | 0.164 | 46 |

2.3 Physio-chemical and chemical measurements
Phosphate concentration was measured spectrophotometrically using blue molybdenum method after filtration through glass fibre filters (GF/F), P\textsubscript{tot} was measured the same way after sample mineralization (at elevated temperature and pressure in acidic medium with potassium persulfate as an oxidizing substance) and the pH was measured potentiometrically (Elmetron pH meter).

3. Results and discussions
Surface waters in the coastal zone (littoral) were characterized by higher concentrations of P (PO\textsubscript{4}\textsuperscript{3-}=0.067 mg/L i P\textsubscript{tot}=0.043 mg/L) than water in the open part of the lake (profundal; PO\textsubscript{4}\textsuperscript{3-}=0.043 mg/L i P\textsubscript{tot}=0.035 mg/L). The proportion of phosphate P in the total P pool was 40% and 52%, respectively (table 1). The surplus of phosphates in the littoral zone has likely resulted, among others, from their inflow from the catchment (a small agro-industrial farm close to the shore). The highest concentrations of P characterized the profundal bottom waters (0.540 PO\textsubscript{4}\textsuperscript{3-}/L i 0.240 mg P\textsubscript{tot}/L). The pH of the tested waters ranged from 8.09 to 8.22.

3.1 Macroscopic observations
Macroscopic observations were made to check for a visible transition (washing or falling out) of the sorbing material from the bags into the solution (figure 1). This process was most marked in the case of SINOBENT, where yellowish-brown suspension in the lake water appeared and quickly settled to the bottom. This caused the problem to take a homogeneous sample for further analysis, as even during strong mixing in the container, a relatively inhomogeneous solution with distinct clusters and clear water were observed. Most likely, this suspension resulted from the dissolution of SINOBENT and the release of iron, which precipitated as iron hydroxide, which is confirmed by distinct pH decrease, as further discussed. In the case of PCC and GCC, the loss of the materials from the bags was smaller and more pronounced in the first one. As this material is very fine grained, it poorly settled and caused slight water turbidity. In the presence of HYDROPHOSBIND, the water was clear, indicating that FeCl\textsubscript{3} did not get out of the capsules and bags.
Figure 1. SINOBENT (S1), PCC (S2), GCC (S3) and HYDROPHOSBIND (S4) sorbents in lake water

3.2 Efficiency of phosphate removal
The effectiveness of sorbing materials placed in bags was estimated after 48 hours of the experiment, although the maximum phosphate loss occurred depending on the type of sorbent and temperature also in the earlier stage of the incubation (after 12 or 24 hours) (figure 2). Each time the largest loss of phosphate was observed in PCC treatments, next GCC and then HYDROPHOSBIND. Most often, the highest decrease in phosphate concentration was observed between 4th and 6th hour of the experiment. This was particularly evident in the case of lower initial phosphate concentrations. At higher phosphate concentrations, the decrease was more even. The lowest concentrations were observed after 12 hours at 6°C and 24-48 hours at 20°C.
Figure 2. The concentration of phosphates after 2, 4, 6, 12, 24 and 48 hours in natural waters in the presence of sorbing materials placed in bags at a temperature of 6°C (PO$_4^{3-}$ initial concentration - 0.04 mg/L and 0.54 mg/L) – a) and 20°C (PO$_4^{3-}$ initial concentration -0.07 mg/L and 0.23 mg/L) – b) and with the addition of liquid probiotic (graphs in a blue frame).

In the presence of hydrogel capsules, there was no change in pH and turbidity of water (figure 3), which indicates that P binding actually occurred in the capsules in bags, without a release of iron into the water. The effectiveness of HYDROPHOSBIND in the PO$_4^{3-}$ binding is not high (<0.20 mg PO$_4^{3-}$/
g), but when we compare this result to the amount of FeCl₃ used, the value increases to 35.7 mg PO₄³⁻/g Fe.

The influence of probiotic microorganisms on the effectiveness of sorption is not unambiguous. In their presence a much higher phosphate loss from water in the presence of PCC, GCC and HYDROPHOSBIND was observed in two experimental series: at 20°C when the initial concentration of phosphate was high (0.23 mg/L) and at 6°C when the initial concentration of phosphate was lower (0.04 mg/L). The noticed greater decrease in phosphate concentration in the presence of probiotics could be associated with adsorption and/or incorporation into the cell structure of probiotic microorganisms.

![Figure 3](image.png)

**Figure 3.** Effectiveness of phosphate sorption (phosphate loss from water) after 48 hours of the experiment. The results take into account the natural loss of phosphates from lake water. PR indicates the presence of probiotic microorganisms; HYD - HYDROPHOSBIND

Concentration of the Ptot decreased as a result of the application of PCC, GCC and HYDROPHOSBIND with promoting an effect of probiotics in case of last 2 materials (figure 4). This confirms that phosphates were removed from lake water, although this result may be partly related to the sorption of other forms of P than phosphates: e.g. [21] reported polyphosphates fixation onto calcite. The observed greater loss of Ptot in samples enriched with probiotics as compared to the treatments without these organisms suggests that mineralization of organic matter took place, leading to phosphate release, which has been adsorbed by the sorption materials. Another explanation may be the adsorption by microorganisms on sorbents. In both cases, phosphates would be removed from the lake water along with the packaged sorbent, which provides a possibility of its long term elimination from the water. However, for SINOBENT, an increase of the concentration of Ptot was observed, showing that the preparation may be a source of P for the aquatic environment, which further eliminates this material for use in the tested method.
The concentration of P_{tot} in the experiment carried out at 20°C when the initial concentration of phosphate was 0.164 mg/L (exemplary diagram) should be mentioned that during the experiment a loss of phosphate was also observed in the lake water itself (the control). The concentration of phosphate in the control dropped from 0.043 to 0.005 mg/L. The reason for this may be the natural sorption of phosphates on the particles of organic and inorganic matter remaining after initial filtration in lake water. This process takes place particularly well under aerobic conditions and is one of the natural processes that remove phosphates from the water column. Another reason may be the consumption of phosphates by microorganisms. The phosphate loss was the higher, the higher the initial concentration of phosphates was. The highest decrease in phosphate concentration in the control was found at low temperature when the initial phosphate concentration was 0.54 mg/L and when probiotic microorganisms (figure 4) were added to the experiment.

The obtained phosphate binding efficiency of sorption materials placed in bags is smaller than in case of loose material, which was previously tested by EKOLAB Ltd. For example, the highest sorption efficiency of PCC achieved in the experiment was 0.087 mg PO_4^{3-}/g, whereas this material in a loose form reached the sorption of 0.5 mg PO_4^{3-}/g. It seems that the reason for the clearly lower sorption of phosphates is the packaging used - the "bag" - can reduce the surface of the material participating in the binding. This is probably related to the agglomeration of the sorbent particles. Confirmation for this is the fact that in the case of GCC, characterized with larger grains, the differences between packed and loose form are much smaller (0.046 and 0.170 mg PO_4^{3-}/g respectively).

Calcite precipitates naturally in surface waters [22, 23]. As phosphate is co-precipitated with newly forming calcite and removed from the water column during crystals settling, this process is an important self-purification mechanism [24-26]. Calcite, in various forms (synthetic precipitates and ground limestone), was already tested under laboratory conditions as a tool for P binding in surface waters, in a loose form applied to water [17, 27-32] and as active barriers for inhibiting P release from lakes’ sediments [18, 33-36]. It was also used in the restoration of several lakes in Canada and Germany [18, 37, 38].

4. Conclusions
The HYDROPHOSBIND sorption per gram of preparation is not high. However, the comparison of results obtained in sachets (0.20 mg PO_4^{3-}/g) with the results of a "loose" (0.21 mg PO_4^{3-}/g, unpublished data) preparation is similar. This confirms the usefulness of the selected packaging. The sachet does not constitute a barrier in the sorption process, and the reason for the reduced efficiency of this process is the "caking" of very fine particles of carbonate sorbent. The sorption efficiency is the worse the finer
the sorbent is used. Sorption efficiency is lower 3.7 for GCC while for PCC almost 6 times lower in comparison to the powdery sorbent.

Another possible reason for the lower sorption efficiency in the conducted experiment is closing the "bags" holes with the dissolved organic compounds, which are present in natural lake water in high concentrations. This is particularly noticeable in the case of the bottom waters.

The strong effect on the pH and the fact of washing out of the SINOBENT from the bag eliminates this sorbent for use in the tested method.

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