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Long Term Sediment Modification Effects after Applications of P Inactivation Method in Meromictic Lake (Starodworskie Lake, Olsztyn Lakeland, Poland)

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Abstract: Lake restoration is a part of geoengineering, which is a useful tool for landscape management. The phosphorus inactivation method is one of the most popular lake restoration methods. Using chemical compounds for P binding is leading to the creation of sediment "active layer", which should show higher P adsorption abilities, compared to non-modified sediment. However, it provides rather little information, how long the modified sediment remains active, and whether it is effective in continuous P binding. Lake meromixis is not commonly observed phenomenon, and sediment located in monimolimnion area is subjected long term anoxia. Therefore, observation of "active layer" in a meromictic lake can give very important data about durability of restoration effects. The object of our study was meromictic Starodworskie Lake (5.57 ha, max. depth 24.5 m), located in Olsztyn Lakeland, Poland. In the past the analyzed lake was subjected to various restoration methods, and phosphorus inactivation method by alum use (1994–1995) was the last used treatment type. The mixing regime of this lake had changed from bradimictic (before and during restoration time) into durable meromictic (post-restoration period). The research made two decades after implementing of P inactivation showed the presence of "active" sediment layer 10–15 cm below sediment surface. This sediment layer showed much higher content of P bound to aluminum, compared to surficial sediment layer. P binding molar ratio was assessed and amounted to 16.1 straightly after restoration and 6.1 after 21 years. This fraction amounts were higher that the values noted before restoration (ca. 358% higher than in 1994) and during restoration (ca. 86% higher than in 1995), which was probably the effect of continuous phosphorus adsorption by "active layer" in post-restoration period.

Keywords: geoengineering; lake; bottom sediment; alum; P inactivation; meromixis

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1. Introduction

Excessive eutrophication of surface water bodies is a common phenomenon worldwide [1–3]. The main reason for accelerated eutrophication is the multi-aspect human activity carried out in the catchment area—sewage production and discharge into water, intensive cultivation of plants requiring fertilization, industrial animal husbandry, reduction of forest area, etc. [1]. Moreover, negative effects of eutrophication are further enhanced by climate change, causing the increase of surface water temperature, and prolonging periods of stagnation of water bodies located in the temperate climate zone (middle latitudes) [4,5]. Water density is dependent on water temperature and salinity. Therefore, in the situation of rising water pollution, deep lakes with slow mixing regime (bradimictic) can form permanent meromixis, in which deep water layer (monimolimnion) has higher density, than water column above. This density difference is a factor blocking effectively the possibility of lake water full mixing. Low temperature, the absence of contact with atmosphere, and lack of photosynthesis possibility, as well as an oxygen depletion for sediment organic matter decomposition, lead to permanent anoxia in monimolimnion zone [5–8]. Due to this, negative symptoms of excessive eutrophication make water usage difficult, or even impossible, necessitating many types of geoengineering actions in the drainage basins as well as directly in the lake bowl, aiming to improve water quality [9,10]. Urban lakes are a very valuable part of landscape. Taking actions, which could help to improve and maintain good water quality of water bodies, is an important part of proper landscape management.

Phosphorus is the key factor controlling eutrophication. Hence many technical actions, realized in water bodies, are targeted to temporary or constant modification of P biogeochemical cycle [9,11–13]. One of the most popular methods is phosphorus inactivation via using preparations, which remove the P excess from water, and then block P in bottom deposits. The alum (hydrated aluminum sulfate $Al_2(SO_4)_3 \cdot 18\ H_2O$) was among the earliest used compounds for this purpose [9,11,13–17]. Starodworskie Lake was the first lake in Poland, in which the alum was used for P inactivation. The alum was dosed to the lake twice—in October 1994 and in October 1995 [4,18,19].

Positive effects of restoration treatments are durable in the shorter or longer time period since the termination of treatment. It is dependent mainly on external nutrients loading in the post-restoration period, and, above all, on the type of used method. If a nutrient's loading (from the point and non-point sources) were effectively reduced or cut off, the positive effects of restoration should last longer. Then the very important part of research, which is carried out on the restored lakes is the observation of changes, induced by treatment, and taking place in the post-restoration period. Without that kind of data, the development of restoration techniques could not be possible.

Observation of restoration effects in specific water bodies, such as meromictic lakes, has additional value because of specific conditions in the near-bottom zone of these water bodies. Meromictic lakes have limited water exchange between monimolimnion and water column, as well as low redox potential in bottom zone because of permanent anoxia. This environment favors a specific microbial activity [8]. Then it is a good example for observing durability of restoration effects, particularly when restoration methods had an influence on bottom area. The aim of this study was the analysis of changes in the profundal water-sediment interface of meromictic Starodworskie Lake, which were induced by restoration treatment with alum use. The range of research included: The investigation of nutrients in the water-sediment interface of Starodworskie Lake, with a special emphasis on P compounds 21 years after the termination of restoration procedure.

2. Materials and Methods

Starodworskie Lake is a small (5.57 ha) and deep (24.5 m) kettle lake (Figure 1), located in Olsztyn city (Kortowo District). The topographic drainage basin area occupies 55 ha, but after modernization of storm the water sewerage system it had decreased to 14.34 ha only. 70% of the catchment area is occupied by barren lands, 13.4% by mixed forest, pastures' share amounts to 5.6%, and urban area covers 10.9% [4]. Starodworskie Lake in the past was classified as bradimictic, but anthropogenic activity in direct catchment (sewage discharge into lake, changes in the catchment use) and climate changes resulted in developing meromixis of this water body [4]. The basic morphometric characteristic of the investigated lake is given in Table 1.

In the past, the analyzed lake was subjected to various restoration methods—artificial mixing (1967–1968, 1972–1974, 1986–1987), hypolimnetic aeration (1988–1989), and, finally, phosphorus inactivation by alum using (1994–1995). The 667 kg Al was dosed into lake targeting bottom area below isobath 10 m [4,18–20].

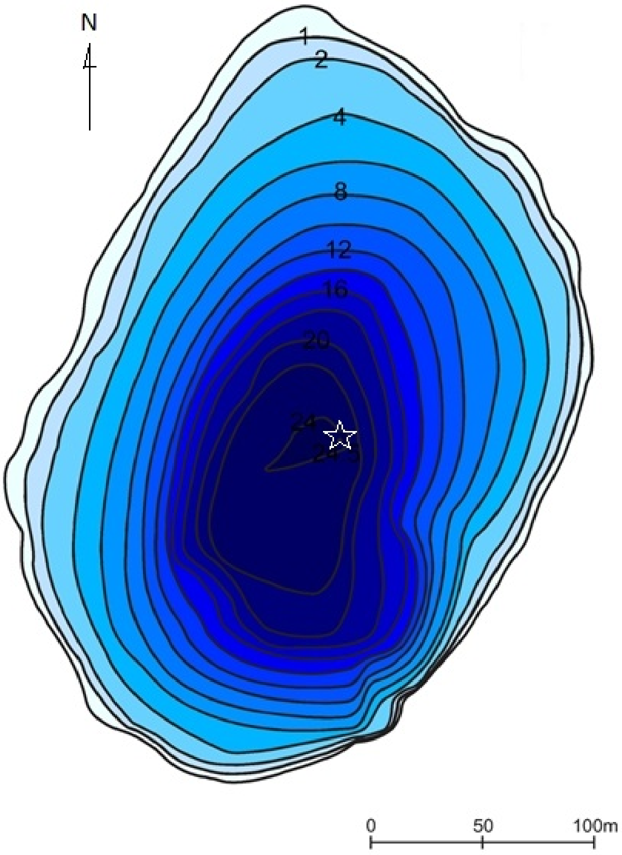
Bottom sediment was sampled in two periods—October 1994 (straightly before alum application), November 1995 (one month after second alum dose application), October 1996 (one year after second dose of alum), and September of 2016 year using Kajak sediment core sampler. In each case three sediment cores were taken from the deepest point of the lake (monimolimnion zone). After decantation of 10-cm thick layer of water above the core (which was treated as near-bottom water), and the 5-cm thick sediment layers were taken for further analysis (upper 0–5 cm layer and 5–10 cm layer below upper layer). In 1994–1996 years 10-cm thick sediment cores were subjected to analysis (0–5 cm, 5–10 cm); in 2016 20-cm thick sediment cores were analyzed (0–5 cm, 5–10 cm 10–15 cm and 15–20 cm). After transportation to laboratory the interstitial water was separated from sediment by centrifugation (MPW-321 centrifuge, MPW Med Instruments, Warsaw, Poland, 3000 r.p.m., time—20 min). In the near-bottom and interstitial water the phosphorus (SRP, TP and organic P) and nitrogen forms (N-NH_4, TKN, organic N) were determined according to Hermanowicz et al. [21,22] (using Spekol 11, Carl Zeiss, Jena, Germany, and MERCK SQ 118, KGaA, Darmstadt, Germany) spectrophotometers, and Kjeldahl method with using BÜCHI B-24 distillation unit (BÜCHI Labortechnik AG, Flawil, Switzerland). Sediment samples were dried, milled, and later the main chemical components (organic matter—OM, SiO_2, Al, Fe, Ca, TKN) were determined according to procedure described by [23]. Phosphorus fractions were analyzed using method described by [24].

Table 1. Basic morphometrical parameters of Starodworskie Lake [4].

Parameter	Unit	Value
Location	m a.s.l.	110.85
Surface area	ha	5.57
Maximum depth	m	24.5
Average depth	m	9.4
Relative depth index	-	0.1038
Depth index	-	0.382
Volume	m^3	522,014
Maximum lenght	m	341
Maximum width	m	226
Shoreline lenght	m	892.0
Shoreline development index	-	1.07

(**a**)

Figure 1. *Cont.*



(**b**)

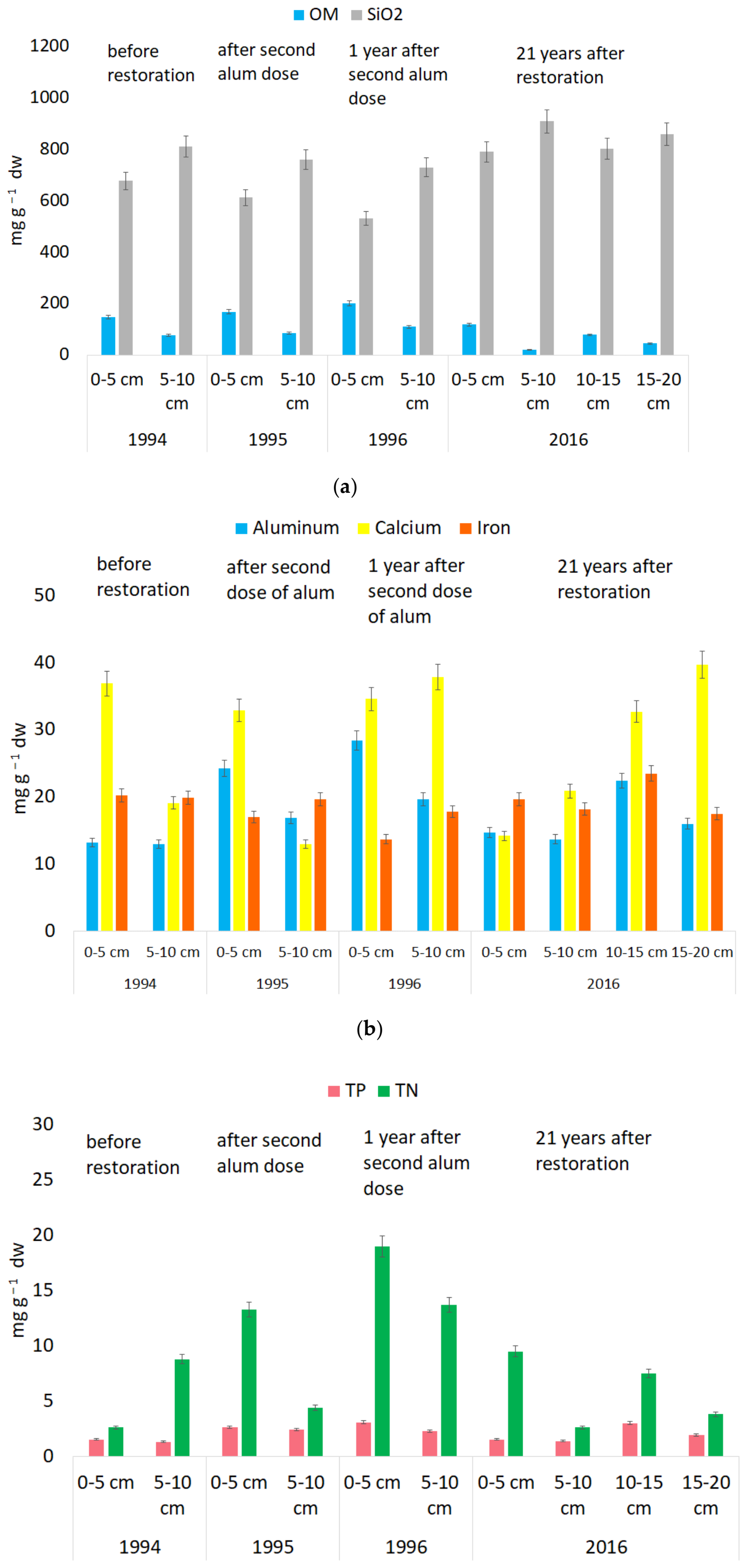
Figure 1. Starodworskie Lake and its surroundings (watershed line is marked as a yellow line). (**a**) Location of sampling station (source: [25], modified); (**b**) bathymetric map of Starodworskie Lake (after [4], modified). * sampling station coordinates: 53.7475905 N 20.45347389 E (WGS84).

The relationships between particular sediment compounds were analyzed using correlation coefficient. An assessment was made using Statistica 13.3 software package (Tibco Inc., Palo Alto, CA, USA) [26].

3. Results and Discussion

3.1. Sediment Chemical Composition

Profundal bottom sediment of Starodworskie Lake can be classified as silica type (according to classification of Stangenberg) [27] with the highest share of silica (>50% dw) and rather low organic matter contents and total nitrogen (within the range from 17 to 208 mg OM g^{-1} dw and from 2.5 to 20.2 mg N g^{-1} dw), which is rather unusual for that type of sediment in deep, stratified and eutrophic lake (Figure 2). The amounts of other sediment components were rather low and did not exceed few percent of dw (Figure 2). This situation resulted in highly significant negative relationship between OM and SiO_2 in analyzed sediment ($r = –0.860$, $n = 30$, $p < 0.05$) (Table 2). Correlation between these two components can be weaker for sediment with higher share of other mineral compounds e.g., calcareous minerals [28].



(a)

(b)

(c)

Figure 2. The content (mean values ± SD) of selected components of the bottom sediment of Starodworskie Lake in 1994, 1995, 1996 and 2016: (**a**) Organic matter and silica content; (**b**) aluminum, iron and calcium content; (**c**) nitrogen and phosphorus content.

Table 2. Correlation matrix between bottom sediment components of Starodworskie Lake ($n = 30$, $p < 0.05$).

Parameter	Al	Ca	Fe	OM	SiO_2	TP
Al	-	-	-	-	-	-
Ca	0.389	-	-	-	-	-
Fe	−0.454	n.s	-	-	-	-
OM	0.629	n.s	−0.427	-	-	-
SiO_2	−0.678	n.s.	0.503	−0.860	-	-
TP	0.921	0.369	n.s.	0.460	−0.548	-
TN	0.782	n.s.	−0.553	0.714	−0.654	0.578

n.s.—non significant.

The high share of Si in sediment is a factor, which can have limiting influence on P binding [14,29], and it seems to be responsible for rather low P level in sediment noted before P inactivation (which did not exceed 2 mg P g^{-1} dw). Highly mineral character of profundal Starodworskie Lake sediment was also noticed by Gawrońska [20] during artificial mixing (1986–1987). It is possible, that relatively long period of previous artificial aeration influenced on sediment organic matter content, resulting in lower amount of this component.

Research made by Gawrońska [20] showed, that sediment P was mainly stored in mineral forms, and that situation was also observed in present research (before P inactivation) (Figure 3).

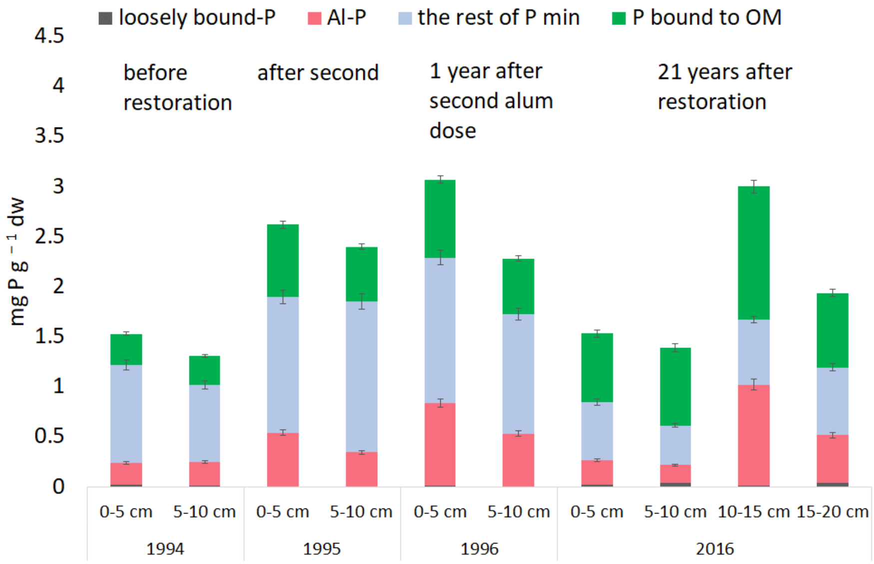


Figure 3. The amounts of selected P fractions (mean values ± SD) in the bottom sediment of Starodworskie Lake in 1994, 1995, 1996 and 2016. Bars' heights represent total amount of element.

3.2. *Changes in Sediment Chemical Composition and Near-Bottom and Interstitial Water Induced by P Inactivation*

The main purpose of the phosphorus inactivation method is to artificially modify the circulation of this element in a water body, and, thus, to limit its availability to primary producers. This modification involves the removal of excess mineral phosphorus from the lake water by the added active compound. The phosphorus removed from the water is directed to the bottom sediment, and also is temporarily or permanently excluded from circulation. It depends mainly on the type and used dose of a preparation. In addition, the active substance forms so-called "active layer", which theoretically should continue to adsorb phosphorus on the available adsorption surface. This limits an internal loading phenomenon [9,13,14,16,17,23].

The studied lake in the period before and during the reclamation still was a bradimictic lake, but as a result of climate change and changes in the use of the catchment (mainly expansion of built-up area by high buildings), it turned into a meromictic lake. Permanent meromixis has been recorded in this lake since 2001.Undoubtedly, the phosphorus inactivation procedure also had its share in this transformation, because of increasing in water salinity by sulfates from coagulant [4]. Monimolimnion in lake is stable and includes water masses below a depth of ca. 13–15 m [4].

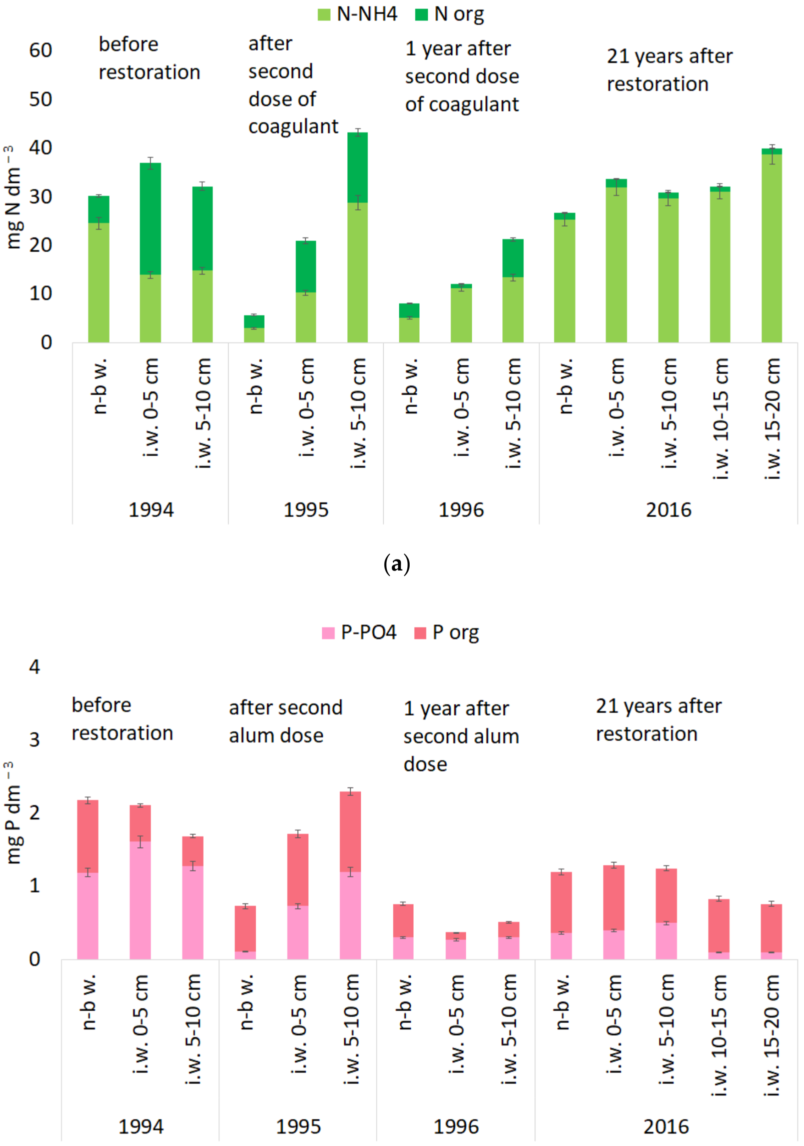
During P inactivation treatment 667 kg Al (as alum, in two doses) was added into Starodworskie Lake (which corresponds to 18.6 g Al/m^2 of targeted bottom area) [19]. The amount of added Al was equal ca. 4.8 mg Al g^{-1} dw of sediment. The treatment resulted in increase of Al level in surficial sediment layer (Figure 2) and the Al-bound P had risen as well (Figure 3), despite of silica type of sediment. P binding molar ratio [14], which means the molar ratio of added Al to P bound to this Al pool (P_{Al}) ($Al{:}P_{Al}$) was assessed and amounted to 16.1 in 1995, (one month after ending of alum application). After one year this ratio decreased to 9.0, highlighting the effectiveness of the active layer. The research, which was made in 2016 year showed, that higher level of Al, organic matter, as well as Al-bound P pool was observed in the sediment layer at the depth of 10–15 cm (Figures 2 and 3). The assessed $Al{:}P_{Al}$ ratio in that sediment layer amounted to 6.5, which is more than twice lower value than in 1995, and one-third lower than that in 1996. Then it seems to confirm the supposition, that "active layer" created during P inactivation has acted as net trapping P, and the rate of P trapping decreased with elapsed time. However, it is worth of noting, that assessed changes in $Al{:}P_{Al}$ ratio could confirm the continuous ability of modified sediment to retain P, even during constant monimolimnion anoxia. The increase of sediment TP, and higher Al-P fraction amount, observed in 2016 in the sediment layer 10–15 cm also could confirm this hypothesis. Lowering $Al{:}P_{Al}$ ratio with time was also reported by Huser [10], and Welch et al. [12,13]. The role of aluminum in P binding in bottom sediment of Starodworskie Lake monimolimnion zone was also confirmed by highly significant correlation between sedimentary Al and TP (r = 0.921, $n = 30$, $p < 0.05$) (Table 2). Significant correlations were also noted between Ca and TP (r = 0.368, $n = 30$, $p < 0.05$) and OM and TP (r = 0.460, $n = 30$, $p < 0.05$) (Table 2), which confirms the significant role of this sediment components in the P binding in analyzed sediments. P bound with organic matter is easily mobilized in high pH range and during intensive mineralization processes [23,24] (both factors are of minimum meaning in the monimolimnion environmental conditions). P bound with Ca compounds also is perceived as hardly mobile fraction, resistant to redox potential changes, and its mobilization is possible mainly in low pH [24].

No significant correlations were observed between Fe and TP (Table 2), which could be explained by two potential phenomena: Blocking of part of sediment iron in the iron sulfide form, as well as a partial transition of mobile pool of P, bound to Fe and Mn compounds into Al-P form. Rydin [30] as well as Welch et al. [17] suggest that mobile P fractions with elapsed time are replaced by Al-P fraction. In the Starodworskie Lake a share of Al-P fraction in the mineral P in 2016 year was higher comparing to 1994, 1995, and 1996 years. As well, the share of P bound to organic matter was higher (Figure 3), which could be explained by lower organic matter mineralization possibilities in long-term anoxic conditions.

The total amount of P in sediment layers created after the termination of restoration treatments (0–5 cm and 5–10 cm) was similar to the level observed before P inactivation (Figure 3). Low sedimentation rate (<0.5 cm per year) probably is connected to monimolimnion zone creation, which substantially slows the sedimentation process.

In general, the phosphorus inactivation method has no direct effect on the content of total nitrogen in lake water and near-bottom and pore water [23,31]. In Starodworskie Lake total nitrogen concentration recorded in 2016 was at a similar level to the period before and during the reclamation (1994–1996). The domination of the mineral N form over organic form was noticeable (Figure 4). In 2016, ammonium nitrogen comprised from 94% to 97% of TKN, while before and during the reclamation its share in total nitrogen was clearly lower (between 38% and 46% of TKN before restoration and between 49% and

66% of TKN during reclamation). This demonstrates the processes of ammonification of organic matter in monimolimnion. However, the ammonia produced in this process under permanent anaerobic conditions does not undergo further nitrification [6,32]. The anammox process may also occur in lake monimolimnion zone, in the area of redoxcline [7,8], and sediments [7,32], however, it requires the presence of nitrites, and in addition, H_2S is a factor inhibiting the activity of microorganisms carrying out this process [7,8,32]. Tandyrak [4,19] reported the increase in H_2S concentration in deeper parts of lake during stagnation periods, especially after restoration, and this compound is still present in monimolimnion zone in concentrations rarely exceeding 2 mg dm^{-3} [4].



(**a**)

(**b**)

Figure 4. Nutrients concentration (mean ± SD) in the near-bottom water (n-b w.) and interstitial water (i.w.) layers of the water-sediment interface of Starodworskie Lake in 1994, 1995, 1996, and 2016: (**a**) Nitrogen compounds; (**b**) phosphorus compounds. Bars' heights represent total amount of element.

The P inactivation is targeting into a removal of bioavailable P forms from the lake water and the binding of removed P compounds in sediment, as well as blocking P internal loading phenomenon [9,10,23]. That effect was observed in the water-sediment interface of

Starodworskie Lake. Before restoration (in 1994) the average total P concentration in analyzed water strata (near-bottom and interstitial water) were in range from 1.7 mg P dm^{-3} (sediment layer 5–10 cm) to 2.2 mg P dm^{-3} (in near bottom water), with domination of mineral P form [Figure 4]. P inactivation caused the decrease in TP, particularly in the near-bottom water (to the level of 0.7 mg P dm^{-3} on average in 1995). The share of mineral P in TP decreased from ca 54%TP before restoration to 15%TP straightly after second dose of alum (1995) in near-bottom water. In 1996, after one year since alum dosing the lowest TP level was observed, with minimum for pore water of surficial sediment layer (0–5 cm)—0.37 mg P dm^{-3} on average (Figure 4). The research conducted two decades later (2016) revealed that TP level in near-bottom and interstitial water strata was higher than in 1996. However, it is worth of noting that mineral P share in TP was lower (amounting to ca. 30–40% TP in the near-bottom water and pore water of 0–10 cm sediment layer, created after restoration). Mineral P share in TP for deeper sediment layer 10–15 cm (the modified "active"sediment) was the lowest and amounted to 12% TP only. It seems to confirm the supposition that "active" layer created during restoration is still effective in mineral P binding, despite of permanent anoxia in water- sediment interface of monimolimnion. It is in concordance with opinion of Cooke et al. [11] or Rydin [30], that P bound to Al is non-sensitive to changes in redox potential.

Meromixis of Starodworskie Lake can be seen as a positive factor, limiting internal P loading. The process could influence the nutrients' internal loading from monimolimnion into mixolimnion is methane convection [33]. However, calculated values of diel nutrient loading by Tandyrak [4] from sediment in monimolimnion zone in Starodworskie Lake were low and ranged for phosphorus between −0.035 and 0.065 mg P m^2 d, depending on the season. Caraco et al. [34] and Kowalczewska-Madura et al. [2,35] reported much higher P internal loading values, which seems to confirm the limited possibility of P loading from monimolimnion zone sediment in Starodworskie Lake.

4. Conclusions

Phosphorus inactivation method using alum, which was implemented on Starodworskie Lake in Olsztyn in the 1994 and 1995 years, created an "active layer" in sediment of profundal zone, which was able to bind phosphorus in the post-restoration period, despite of long-term permanent anoxia in monimolimnion zone (since 2001). It resulted in an increase of Al-P bound fraction, and the P binding molar ratio value decreasing from the level of 16.1 in 1995 and 9.0 in 1996 to 6.5 in 2016. Al-P amounts were higher than the values noted before restoration (ca 358% higher than in 1994) and after second alum dose (ca 86% higher than in 1995), which was probably the effect of continuous phosphorus adsorption by "active layer" in post-restoration period and transition of mobile P fractions into Al-P fraction. The presence of "active layer" was also the possible reason of observed quantitative domination of organic phosphorus form in the analyzed water strata. Then long-term anoxia in monimolimnion of Starodworskie Lake seems to have minor influence on functioning of "active" sediment layer.

The beginning of permanent meromixis in lake in 2001 limited the sedimentation rate and also influenced nitrogen forms and mutual relations in the water-sediment interface (quantitative domination of ammonia, very low level of organic N). However, a meromixis can also be seen as a slight advantage, limiting internal nutrients loading to trophogenic zone.

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Data Availability Statement: Data presented in this study are available in the archive of Department of Water Protection Engineering and Environmental Microbiology, University of Warmia and Mazury in Olsztyn, Poland.

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