

THE INFLUENCE OF GYPSUM TREATMENT ON PHOSPHORUS RETENTION IN BOTTOM SEDIMENTS AND ON THE WATER OF MAN-MADE LAKE

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ABSTRACT

The paper presents a research covering the stability of phosphorus retention in the bottom sediments, resulting from application of gypsum in anoxic conditions and in slightly acidic environment (pH~5) of solutions. The present work also contains an analysis of possible effects of gypsum application for water from the reservoir, on the basis of selected parameters of overlying water. Undisturbed deposit cores extracted from two research stations on the Solina Reservoir have been a subject to a 10-weeks long exposure in determinate conditions, after application of gypsum. The conducted research indicated that retention capacities of deposits, which become increased after the use of gypsum, will decrease over time (e.g. after winter period). Application of gypsum led to a quite significant increase in concentrations of calcium in solutions. Intense release of iron from the sediments into the solutions and decrease in SO_4^{2-} content, during the said 10-week exposure in anoxic conditions, indicates the use of iron (III) and sulphates as electron acceptors in process of oxidization of an organic substance.

Keywords: reservoirs, bottom sediments, treatment of gypsum, stability of phosphorus retention

INTRODUCTION

Biogenic substances which reach the reservoirs, coming from external sources are used by the aquatic organisms and after the vegetation period, together with their dead remains, they deposit to the bottom sediments. Phosphorus compounds may also react with other substances present in water and precipitate directly into the deposits in the form of sparingly soluble inorganic or organic-inorganic compounds [Bajkiewicz – Grabowska 2002, Zbierska et al. 2016]. Various transformations take place in bottom sediments, including decomposition of organic matter, as a result of which soluble forms of nitrogen and phosphorus may be released into the water [Gruca-Rokosz et al. 2009, 2011]. Stability of phosphorus deposition in bottom sediments depends on compounds in which it occurs, oxygen conditions, temperature, pH, presence of ele-

ments which increase its retention in the deposits [Bartoszek and Koszelnik 2016].

Calcium is also one of the elements with which phosphorus creates sparingly soluble compounds in aquatic environment apart from iron, aluminium and manganese. Depending on concentration of calcium and phosphates in water and depending on conditions present in the water, phosphorus may precipitate directly in the form of calcium phosphates, be adsorbed on the surface of calcium carbonate particles, and may also co-precipitate with calcium carbonate [Berg et al. 2004, Lin and Singer 2006, Sørensen et al. 2011, Liu et al. 2012]. Effectiveness of calcium compounds in comparison to aluminium and iron compounds in retaining phosphorus in bottom sediments is weaker, but they are less harmful to the aquatic environment [Prepas et al. 2001a, 2001b, Zhang et al. 2001]. The research conducted in *ex situ* conditions on undisturbed deposit cores of the

Solina and Myczkowce Reservoirs indicated that addition of dihydrate calcium sulphate (gypsum) to bottom sediments significantly impacts on the increase in phosphorus retention. With the exception of sandy littoral deposits, gypsum application resulted in a complete removal of phosphorus from solutions by deposits subject to resuspension [Bartoszek and Tomaszek 2010].

The aim of the present paper was to verify the stability of growth of phosphorus retention in bottom sediments, as a result of gypsum application in anoxic conditions and decreased pH of solutions. The present work also contains an analysis of possible effects of gypsum application for water from the reservoir, on the basis of selected parameters of overlying water.

RESEARCH AREA AND METHODOLOGY

The Solina Reservoir is the deepest and the largest artificial lake in Poland in terms of capacity (Table 1). Together with the smaller Myczkowce Reservoir situated beneath, it is used for hydro-power purposes. Its mountainous, pasture-forest catchment area is scarcely populated and not much developed. Due to adverse landform features and unordered water and sewage management, especially within the direct catchment area, the object is considerably exposed to degradation [Koszelnik et al. 2002, Bartoszek and Czech 2014].

As part of the research, undisturbed deposit cores were extracted and placed in 6 perspex tubes (0–5 cm layer) in October 2007, with the use of a tubular sampler, at two research stations on the Solina Reservoir: 1. Centralny, 2. Skalki (average depth approx. 45, 15 m respectively) (Fig. 1).

The pipes were being closed at the bottom with the use of a silicone cap. At the same time the water was taken from the reservoir, and after being thinned with distilled water it was used to prepare the solutions. In order to achieve anoxic conditions, the solutions were deoxidized after preparation with the use of anhydrous sodium sulphate (IV) [Wiśniewski 1995, 2006]. After application of gypsum in the amount of 300 and 500 mg, reactors with undisturbed sedi-

ment cores were filled with prepared solutions, and the sediments were subject to 10-minute resuspension through stirring of approx. 1 cm surface layer using a mechanical stirring device (RPM approx. 150 revolutions per minute). Approximately 12.6 cm³ of deposit in 314 cm³ of solution was subject to resuspension. All of the reactors were filled with solutions containing the same concentration of phosphate phosphorus 1.9 mgP dm⁻³ (Cp). Some of them were deoxidized, in case of some natural oxygen content was left within them, but with pH decreased down to 5. After the resuspension procedure was finished, the reactors were left in a cool, dark place (in the temperature of approx. 15° C) for the period of approx. 10 weeks. This time period was selected due to the fact that it corresponds to the approximate ice cover period on the lakes in the temperate transitional climate [Wiśniewski 1995]. The reactors with the sediments exposed in anoxic conditions were closed with plastic plugs and tightly wrapped in Parafilm. Solutions with decreased pH were carefully aerated using aquarium pumps for approx. 2 h daily and the reactors were closed with Parafilm. The pH value was monitored every 2 days and adjusted it accordingly. At the same time, the control samples with no gypsum within them were exposed in the same conditions and for the same period. After 10 weeks the supernatant solution was leveraged to measuring cylinders. Within the solutions, concentrations of mineral phosphorus, iron, calcium and sulphates were determined. Phosphates (reaction with ammonium molybdate), iron (reaction with phenanthroline) and sulphates (reaction with barium iodate and tannin) were determined spectrophotometrically (Aquamate Thermo Spectronic). Concentration of calcium was determined with the application of the titrimetric method with EDTA. Sediment cores recovered after exposure in anoxic conditions were exposed for the second time to the newly prepared deoxidized solution with P-PO₄³⁻ concentration of 1.9 mgP dm⁻³, and without repeated gypsum application underwent resuspension and resedimentation. After two hours exposure in anoxic conditions, within

Table 1. Morphometric parameters of the Solina Reservoir

Parameter	Area	Average (max.) depth	Maximum volume	Catchment area	Water retention time
Reservoir	[km ²]	[m]	[M m ³]	[km ²]	[d]
Solina	22	22 (60)	500	1174	214

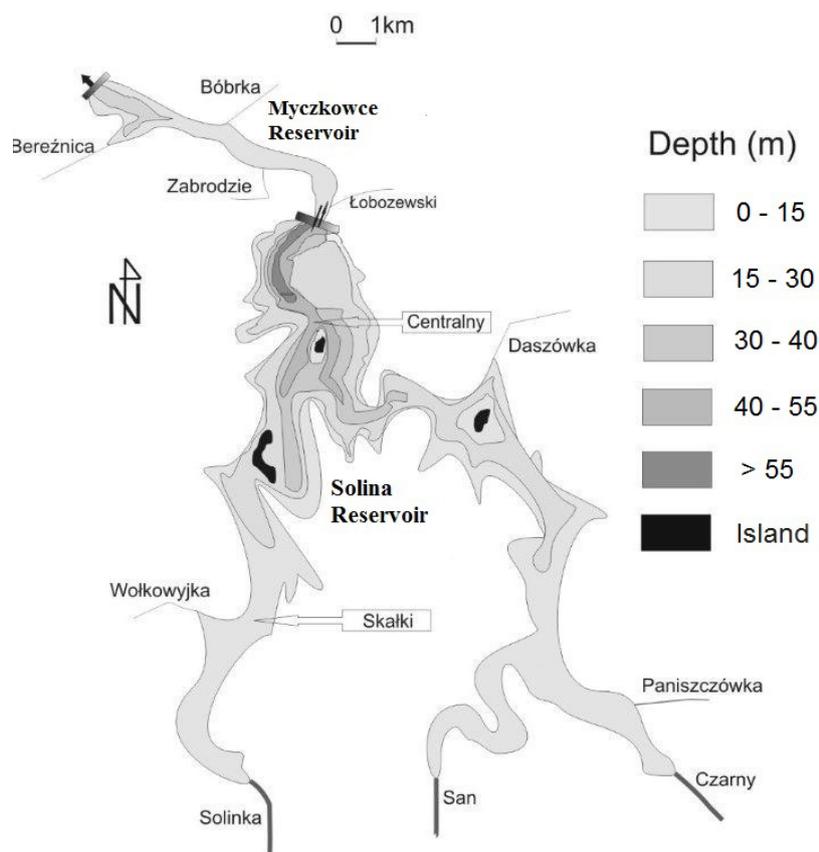


Figure 1. Localisation of the sediment sampling points in the Solina Reservoir

the leveraged solutions, phosphate phosphorus content was being determined. Further analysis used 1 cm top layer of the sediment cores. The sediments, after drying and thorough grinding, were mineralized and phosphorus was fractionated. As a result of phosphorus fractionation with the use of the SMT method, the fractions of NAIP (non-apatite, inorganic phosphorus i.e. compounds with Fe, Al, Mn), AP (apatite phosphorus i.e. compounds with Ca) and OP (organic phosphorus) were obtained [Bartoszek 2008]. After microwave mineralization of the deposits in concentrated HNO_3 (2–4.5 MPa – UniClever II Plazmatronika), the phosphorus content in achieved mineralisates was determined.

The content of dissolved oxygen, pH and selectively the redox potential in solutions before and after the exposures in given experimental conditions, using the MultiLine P4 multiparameter gauge (WTW Germany) with CelloX 325 oxygen probe (oxygen – O_2), Sen Tix 61 combined electrode (pH) and Sen Tix ORP combined electrode (redox potential – Eh) were also measured.

RESULTS AND DISCUSSION

Phosphorus retention after 10-week ex situ exposure in anoxic conditions and in slightly acidic environment of solutions

After 10-week exposure of deposits in a slightly acidic environment (pH~5) in control samples, phosphorus retention at the level of 99.3% and 99.8% (Centralny; Skalki respectively) was observed (Table 2.). Samples with 300 and 500 mg doses of gypsum were characterized by similar retention from 99.7% to 100.0%. Thus, no significant differences were found regarding the phosphorus retention phenomenon within the samples with and without gypsum. Research conducted in the years 2005–2006 indicated that sediments of the Solina Reservoir are characterized by high content of iron, aluminium, manganese and relatively low content of organic matter, phosphorus and calcium [Bartoszek et al. 2015]. In deposits in which the retention capacity of this element results mainly from creation of compounds with

Table 2. Phosphorus retention [%] in the bottom sediments after 10 weeks of *ex situ* exposure in the conditions of decreased pH, and 10 weeks (I) and 2h (II) of exposures in anoxic conditions

Dosage gypsum [mg]	pH~5	Anoxic	
	Retention P-PO ₄ ³⁻ [%]	Retention I P-PO ₄ ³⁻ [%]	Retention II P-PO ₄ ³⁻ [%]
<i>Centralny</i>			
-	99.3	96.0	61.4
300	99.7	99.6	62.8
500	100.0	99.9	72.3
<i>Skalki</i>			
-	99.8	90.6	35.1
300	99.8	92.2	38.5
500	99.9	96.3	52.8

iron (III), decrease of the pH value may cause an intense flow of phosphorus towards the deposits, while in deposits with high calcium content the decrease of pH may initiate its release into the water [Bajkiewicz-Grabowska 2002]. According to Lossow and Gawrońska [2000], optimal pH level for removal of phosphorus from water using iron coagulant containing iron III is 4.5–5.

After 10-week exposure of sediments in anoxic conditions, retention in control samples at the level of 96.0% and 90.6% (Centralny; Skalki respectively) was observed (Table 2). In samples with gypsum, retention occurred at the level of 99.6% and 99.9% (300; 500 mg doses respectively) within the sediments from the Centralny station and 92.2% and 96.3% (300; 500 mg doses respectively) within the sediments from the Skalki station. Two hour exposure of the same sediment cores conducted for the second time in anoxic conditions, using newly prepared solutions with phosphate phosphorus concentration of 1.9 mgP dm⁻³, but without second gypsum application, did not confirm preservation of retention capacities of the sediments at the same level. In the case of the sediments from the Centralny station (in spite of previously retained phosphorus portions) 61.4% retention in the control sample was only slightly lower than 68.3% observed in previous research during 2 h exposure (Table 3) [Bartoszek and Tomaszek 2010]. However, the natural retention capacity of sediments from the Skalki station weakened significantly. The deposits from this station retained only 35.1% of subsequent phosphorus portion, whereas directly after 2 h exposure in anoxic conditions retention at the level of 61.7%

was achieved. The sediments to which dihydrate calcium sulphate (VI) was applied 10 weeks earlier indicated retention at the level of 62.8% and 72.2% (Centralny) as well as 38.5% and 52.8% (Skalki). The impact of gypsum on phosphorus retention in deposit after 10-week exposure in anoxic conditions was insignificant (Table 2). In relation to the control sample, only up to 10% more phosphorus became retained in deposits from the Centralny station. Deposits from the Skalki station retained slightly more phosphorus, approx. 18%.

Analysis of phosphorus fractionation in the sediments left after the experiment indicated a slight increase in the AP fraction content in deposits with added dihydrate calcium sulphate (VI) (3.2–11.8%), in relation to deposits of control samples in slightly acidic environment (Table 4). In deposits exposed in anoxic conditions, the observed changes in AP fraction content were similar (at the level of 4.5–12.6%). The NAIP fraction content would vary very irregularly, often alternately indicating a slight upward and downward trend. In the sediments from the Skalki station, after exposure in both experimental conditions, slight decrease in content of OP was noticed.

Due to long time of value stabilization, measurement of redox potential (Eh) was conducted for deoxidized initial solutions as well as for selected solutions after 10-week exposure (Table 5). The concentration of O₂, which, in deoxidized initial solutions, fluctuated between 0.01 and 0.03 mgO₂ dm⁻³, after 10-weeks the respective values amounted between 0.08 and 0.15 mgO₂ dm⁻³ was monitored (Table 5). The observed decrease of pH in the solutions after 10-week exposure in anoxic condition was probably caused by creation of weak organic acids and CO₂ during decomposition of organic matter.

Table 3. Phosphorus retention [%] in the bottom sediments under the influence of gypsum after 2h of *ex situ* exposure in anoxic conditions [Bartoszek and Tomaszek 2010]

Initial concentration P-PO ₄ ³⁻ in solution Cp = 1.9 mgP dm ⁻³	Anoxic	
	Retention P-PO ₄ ³⁻ [%]	
Dosage gypsum [mg]	<i>Centralny</i>	<i>Skalki</i>
-	68.3	61.7
200	99.8	98.6
300	99.9	99.9
400	99.5	99.8

Table 4. The contents of the total phosphorus ($P_{tot.}$) and its fractions in sediments after 10 weeks of exposure in anoxic and decreased pH conditions

Dosage gypsum [mg]	Conditions	$P_{tot.}$	NAIP		AP		OP	
		[mg g ⁻¹ of d.w.]	[mg g ⁻¹ of d.w.]	[%]	[mg g ⁻¹ of d.w.]	[%]	[mg g ⁻¹ of d.w.]	[%]
		<i>Centralny</i>						
-	pH ~ 5	0.943	0.255	27.0	0.373	39.5	0.305	32.3
300		0.983	0.265	27.0	0.385	39.2	0.315	32.1
500		0.965	0.251	26.0	0.417	43.2	0.302	31.3
-	anoxic.	0.920	0.251	27.3	0.347	37.6	0.279	30.3
300		0.978	0.295	30.2	0.374	38.2	0.270	27.6
500		0.962	0.270	28.0	0.377	39.2	0.275	28.6
		<i>Skalki</i>						
-	pH ~ 5	0.725	0.140	19.3	0.322	44.5	0.241	33.2
300		0.738	0.148	20.1	0.343	46.5	0.236	32.0
500		0.730	0.142	19.4	0.339	46.4	0.231	31.6
-	anoxic	0.718	0.172	24.0	0.333	46.4	0.182	25.3
300		0.720	0.170	23.7	0.348	48.2	0.170	23.6
500		0.706	0.159	22.6	0.375	53.1	0.147	20.8

Table 5. Monitored parameters in the solutions before and after 10 weeks of exposure in anoxic and decreased pH conditions

Dosage gypsum [mg]	Station	Conditions	pH		Oxygen O ₂ [mgO ₂ dm ⁻³]		Potential Eh [mV]	
			before	after	before	after	before	after
			10 weeks		10 weeks		10 weeks	
-	Centralny	anoxic		7.26		0.11		160.5
300			7.82	6.66	0.02	0.09	127.6	-
500				6.75		0.12		177.9
-	Skalki			7.41		0.09		159.6
300			7.95	7.22	0.03	0.08	130.1	-
500				7.10		0.15		182.1
-	Centralny	pH ~ 5		5.22		6.09	-	-
300			5.02	5.26	7.11	5.94	521.3	-
500				5.16		6.49	-	-
-	Skalki			5.29		5.42	-	-
300			4.99	5.14	7.16	6.06	-	-
500				5.20		6.28	-	-

Impact of gypsum application on selected parameters of water

Introduction of dihydrate calcium sulphate (VI) into the reservoir may cause significant increase in concentration of calcium and sulphates in the water. Not only does this bear a significance in case of using the water from the reservoir for drinking, as the aforesaid condition is also important for the industrial and farming purposes [Bartoszek 2008]. In samples with applied gypsum, calcium concentrations in solutions after 10-week exposure were approx. 5–10 times higher than in test samples depending on the dose used

and on place from which the deposits were drawn (Table 6). However, no significant impact of exposure conditions on concentration of calcium in water was noticed. In case of the calcium concentration value so high, it is required that water undergoes purification before becoming suitable for consumption [Dz.U. z 2015 r. poz. 1989]. Concentrations of sulphates in water in samples with gypsum were also higher than those observed in the control samples, but not as high as to make the water unsuitable for consumption. After 10-week exposure in anoxic conditions, concentrations of sulphates in solutions were often lower than after

Table 6. Influence of gypsum application on concentrations of calcium, sulphate and iron in the solutions after 10 weeks of exposures in decreased pH and anoxic conditions

Dosage gypsum [mg]	pH~5			Anoxic		
	Ca	SO ₄ ²⁻	Fe	Ca	SO ₄ ²⁻	Fe
	[mg dm ⁻³]					
<i>Centralny</i>						
-	22.0	47.4	0.06	24.0	81.6	1.09
300	125.3	107.1	0.00	160.3	99.3	3.16
500	236.5	124.1	0.00	244.5	106.5	9.00
<i>Skałki</i>						
-	34.5	45.0	0.09	27.0	94.3	0.93
300	170.3	107.7	0.09	168.3	115.1	2.65
500	295.6	133.0	0.06	263.5	118.7	6.51

exposure in conditions with decreased pH, despite the fact that sodium sulphate (IV) was applied into this system in order to deoxidize the solutions.

In the samples with applied dihydrate calcium sulphate (VI), after 10-week exposure in anoxic conditions much greater release of iron from deposits to solutions than in control samples was observed (Table 6.). The higher concentration of sulphates in solutions favoured the creation of soluble compounds of Fe and SO₄²⁻. Soluble iron (III) salts include, among other substances: Fe₂(SO₄)₃ as well as alum, with formula as follows: FeMe(SO₄)₂·12H₂O (Me=NH₄⁺,K⁺,Na⁺). Iron (II) salts include the following compounds: FeSO₄ and Mohr's salt Fe(NH₄)₂(SO₄)₂·6H₂O [Minczewski and Marczenko 2004]. In anoxic conditions, the reduction of Fe (III) which accompanies oxidization of the organic substances may lead to emergence of higher concentrations of Fe (II) in the water. Hydrogen sulphide, which is formed as a result of microbiological reduction of sulphates, should bond the iron (II), creating a sparingly soluble iron (II) sulphide. In conditions of natural aquatic environment, the reactions of Fe (III) and sulphate reduction may take place simultaneously. Competitiveness of these reactions depends on permanence of iron (III) forms as well as on the pH value. The sulphate content has a lesser impact on the above phenomena [Postma and Jakobsen 1996]. Hydrogen sulphide could also cause reduction of iron (III) and increase the rate its release into the solution. For iron (III) reduction to take place via H₂S an extremely high excess of hydrogen sulphide is necessary [Golterman 2001]. Black colour of the surface layer of deposits as well as intense black coating on the reactor walls above the deposit clearly indicated occurrence of strong reduction conditions inside the reactors during the 10-week exposure, par-

ticularly in samples with higher sulphate (VI) calcium dose. Creation of iron (II) sulphide has been observed.

Lesser degree of iron release was observed in the coarse-grained, partly sandy deposits from the Skałki station. The largest quantity and diversity of bacteria occurs in sandy deposits, lower – in argillaceous deposits. Rate at which mineralization occurs is much slower in argillaceous deposits than in sandy deposits [Olańczuk – Neyman 2001]. However, the research conducted in the years 2005–2006 indicated that deposits from both these sites differed significantly in terms of content of selected substances. Sediments from the Skałki station were characterized, among other features, by lower content of iron and organic matter in comparison to sediments from the Centralny station [Bartoszek et al. 2015].

In hypertrophic Laikkalammi lake, after introduction of gypsum, concentrations of sulphates in the water increased from almost 0 to approx. 150 mg dm⁻³. As a result of sulphate depletion as an electron acceptor and of sulphide creation, concentration of SO₄²⁻ ion decreased to approx. 100 mg dm⁻³ [Salonen et al. 2001]. However, neither increased iron concentration nor changes in pH values of the water were found.

CONCLUSIONS

In the case of high content of iron, aluminium and manganese as well as of resulting high natural phosphorus retention capacities in the sediments of the Solina Reservoir, gypsum had no impact on the process of phosphorus retention in deposits in slightly acidic environment in aerobic conditions. Apart from that, in sediments exposed in anoxic conditions, it was found no significant impact

of gypsum on increase in phosphorus retention, which was already at a very high level in control samples. From subsequent phosphorus portion in anoxic conditions, sediment cores without second application of gypsum retained only up to 18% more of this element from the solution. The conducted research indicated that retention capacities of deposits, which become increased after the use of gypsum, will decrease over time (e.g. after winter period). Therefore, it will be necessary to repeat the application within several subsequent years, as it occurs in case of iron and aluminium compounds most commonly used to increase sorption capacities of the sediments with regards to phosphates.

The analysis of phosphorus fractionation in the sediments showed a slight increase in apatite fraction content and a slight decrease of organic phosphorus content in case of the deposits with added gypsum. However, those differences were small and irregular, they could also be caused by heterogeneity of undisturbed sediment cores in terms of phosphorus distribution.

Application of gypsum significantly increased calcium concentration in solutions, which exceeded the standard values acceptable for drinkable water. Intense release of a water-soluble form of iron from deposits to the solutions and decreased SO_4^{2-} content during the 10-week exposure in anoxic conditions indicates the use of iron (III) and sulphates as electron acceptors in the process of oxidization of organic substances. More intense release of Fe in the presence of gypsum indicates occurrence of iron (III) reduction also with participation of H_2S .

Lack of more evident effects of gypsum application is caused mainly by high natural retention capacities of deposits in the Solina Reservoir, which result from their significant content of iron, aluminium and manganese, i.e. elements that have a great influence on the increase in phosphorus retention.

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