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Preliminary Results from the Removal of Phosphorus Compounds with Selected Sorption Material

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Abstract. Due to the fact that resources of phosphorous are limited and are expected to get exhausted in the next 30 years the management of this resource has become extremely important. Most of the phosphorus compounds are lost forever, because they are discharged with sewage into surface water, causing eutrophication and in this way generating further issue and challenge. The aim of the study was to investigate the capacity to retain phosphorus compounds on sorption material. During the experiments, both synthetic and real wastewater were used. The synthetic wastewater simulated the composition of the reject water (RW) generated during the mechanical dewatering of the digested sewage sludge, and the real RW comes from WWTP in Gdansk. The investigation in steady conditions was carried out with Phoslock® which is chemically lanthanum clay. The results of the investigation are related to the determination of the sorption capacity with respect to the analyzed content of phosphorus compounds for stable conditions the determination of hydraulic load and way and time of mixing. For the synthetic wastewater the removal efficiency of phosphorous was 99.8% while for reject water (RW) generated during the mechanical dewatering of the digested sewage sludge was lower and equal to 85%.

Keywords: Adsorption, Phoslock®, phosphorus removal, wastewater.

Conference topic: Water engineering.

Introduction

The phosphorus compounds include the nutrient contamination, so they are the main cause of eutrophication. According to the provisions of Baltic Sea Action Plan (HELCOM Recommendation *28E/5* 2007; HELCOM Recommendation *28E/6* 2007), already for small wastewater treatment plants (with a load of 300 person equivalents) at least 70% reduction of phosphorus compounds is recommended. While for wastewater treatment plans with a load of 2000 person equivalents – at least 70% reduction of phosphorus compounds or concentration less than 2 mg/l in final effluent, when the wastewater is discharged directly or indirectly to marine areas is recomended. Along with the size of the wastewater treatment plants the limit concentrations of biogenic compounds increases (Council Directive 91/271/EEC (European Commission 1991; Polish Regulation (Year 2014, item 1800). The effect of these requirements are studies aimed finding the best method for removing phosphorus compounds.

The most common ways for phosphorus removal include chemical methods through precipitation of phosphate by salts of iron and aluminium (Nastawny *et al.* 2015; Jóźwiakowski *et al.* 2017). However, these methods are not suitable for use in many conventional wastewater treatment plants due to their high cost and difficulty in sewage sludge management. Many natural methods of wastewater treatment, with a very good effects of treatment, unfortunately, do not provide an appropriate degree of phosphorus removal (Paruch *et al.* 2011; Gajewska, Obarska-Pempkowiak 2009; Gajewska, Obarska-Pempkowiak 2011; Gajewska *et al.* 2011). More recently adsorbents of anthropogenic origin gained popularity, such as AAC (autoclaved aerated concrete) (Renman, G., Renman, A. 2012), Pollytag (Bus *et al.* 2014) or LECA (Karczmarczyk, Bus 2014) and natural, as Polonite (Karczmarczyk *et al.* 2016) or rock opoka (Renman 2008; Vohla *et al.* 2011). Adsorbents of anthropogenic origin are produced specifically for phosphorus removal, among other, from industrial by-products (ashes, blast furnace slag). Man-made materials include modified clays (Johansson Westholm 2006). Lanthanum-modified bentonite clay appeared on the market under the name Phoslock®. It is characterized by a significant reduction of Filterable Reactive Phosphorus (FRP), which is a growth limiting factor for algae. These material is used to reduce eutrophication, remediation of lakes and reduction of blue green algae (Phoslock water solutions… 2017).

According to the experiments carried out on natural waters, after the second treatment with application of Phoslock® achieved a reduction of FRP concentration varied from 50 µg/l to 5 µg/l (Robb *et al.* 2003). In addition to significant adsorption capacity of phosphorus compounds, Phoslock® also reveals the ability to reduce the growth of phytoplankton (Van Oosterhout, Lurling 2013) and does not show toxic activity, harmful to aquatic organisms (Van Oosterhout *et al.* 2014).

The aim of the study was to test the suitability of the Phoslock® material to remove phosphorus compounds in steady conditions. The experiment was carried out on synthetic wastewater and reject water generated during the mechanical dewatering of the digested sewage sludge, to determine the effectiveness of Phoslock® in phosphorus removal in adverse conditions such as high concentration of P, high turbidity, color and high concentrations of pollutants. The subject of research was also the time and manner of contact with wastewater.

Material and methods

Material

Phoslock® is an adsorbent based on lanthanum-modified bentonite clay, characterized by a high capacity of phosphate binding. This material is developed by the Australian company Phoslock Water Solutions Pty Ltd (PWS) (Haghseresht *et al.* 2009). The process of removing phosphorus depends on binding the molecules on the surface of the absorbent material and their sedimentation. At the bottom of the vessel is formed a layer of a bentonite clay that contains a significant amount of adsorbed phosphorus ions PO_4^{3-}.

Lanthanum is a chemical element that binds phosphorus in the ratio 1: 1 according to equation (1):

$$La^{3+} + PO_4^{3-} \rightarrow LaPO_4. \quad (1)$$

The product of the above reaction is a stable mineral of low solubility (lanthanum phosphate) known as rhabdophane, which occurs in nature (Zamparas *et al.* 2015).

Phoslock® is increasingly used as a material for phosphorus removal due to a number of advantages. Rhabdophane is the only product created in above reaction (1), and the lack of by-products is extremely important during the process of wastewater treatment.

The particles of the Phoslock® are relatively small, however, with a large specific surface area and total pore volume (Table 1), the adsorption area of phosphate ions is maximum.

Table 1. Physical characteristic of Phoslock® (Haghseresht *et al.* 2009)

Physical properties	Value
Specific surface area (m^2/g)	39.3
Total pore volume (cm^3/g)	0.171
Average particle size (µm)	22
Moisture content (%)	7–9
pH	7–7.5
Bulk Density (kg/m^3)	910–960

The chemical composition of lanthanum-modified bentonite clay (Table 2) shows a significant content of silicon Si and aluminium Al. The product contains approximately 49 mg of lanthanum per 1 gram of Phoslock®, so that the maximum adsorption capacity of phosphorus is 10.6 mg per 1 gram of the adsorbent.

Table 2. Chemical composition of Phoslock® (Haghseresht *et al.* 2009)

Chemical composition	Concentration [%]
SiO_2	63.36
Al_2O_3	14.73
MgO	2.76
Fe_2O_3	3.64
CaO	1.79
La_2O_3	0.058

The adsorption efficiency of phosphate ions is optimal at pH in the range 5.0–9.0, but most higher efficiency is achieved at pH in the range 5.0–7.0. According to Ross, 2008, the degree of phosphorus removal is significantly decreases when the pH increased to 7.0–9.0. This may be result of a reduction of phosphorus binding sites on the surface of the Phoslock®.

Experiment design for synthetic wastewater

To determine the effectiveness of application Phoslock®, the model solution was prepared in 4 beakers with 2 dm^3 capacity. The chemical compounds (di-potassium hydrogen phosphate K_2HPO_4) was used to prepare model solution (1 cm^3 – 10 $mgPO_4$/dm3) based on distilled water. Synthetic wastewater was prepared in such a way that in each beaker were 1.5 dm^3 of model solution with a concentration of 10 $mgPO_4$-P/dm^3.

Before adding the Phoslock®, the following solution parameters were specified: phosphate phosphorus concentration, pH, temperature, conductivity and total suspended solids (TSS). To each beaker 100 g of adsorbent material was added. The first of them was not subjected to mixing, in next two beakers mixing was done with glass rod successively for 5 and 10 seconds after the addition of Phoslock®, and then in the cycle: sampling- mixing – sedimentation – sampling and so on. In the last one mixing was done by using a magnetic stirrer (1000 rpm) and sampling was preceded by a 15 minutes sedimentation over time. Sampling of each beaker held 5 times, after certain times of contact of the solution with Phoslock®, respectively after 30, 150, 1110, 1170 and 1410 minutes. All the tests were conducted at room temperature approx. 23°C.

Experiment design for reject water generated during the mechanical dewatering of the digested sewage sludge

In the second stage of the research, to determine the effectiveness of Phoslock® in phosphorus removal, the reject water generated during the mechanical dewatering of the digested sewage sludge from the wastewater treatment plant in Gdansk was used (Table 3). The current receiver of treated wastewater is the Gulf of Gdansk.

Table 3. Quality of RW from wastewater treatment plant in Gdansk (Gajewska 2013; Gajewska *et al.* 2015)

Parameter	Average value
pH	6.6
TSS (mg/dm^3)	517.05
Organic SS (mg/dm^3)	358.87
TN (mg/dm^3)	785.65
N-NH_4^+ (mg/dm^3)	706.37
Organic N (mg/dm^3)	78.67
TP (mg/dm^3)	421.0
COD (mg/dm^3)	1215.42
BOD_5 (mg/dm^3)	422.43
Akalinity (mval/dm^3)	13.4
T (^{0}C)	22
O_2 (mg/dm^3)	0,1

Basic parameters of wastewater treatment plant in Gdansk:

- amount of flowing wastewater: approx. 92 200 m^3/day;
- 6 bioreactors: 158 100 m^3;
- 4 digesters: 28 000 m^3;
- biogas production: 16 500 m^3/day, composition: 55–65% methane, 31.5% carbon dioxide;
- sludge fermentation time: 21–28 days;
- the amount of sewage sludge: 140 tons/day (Otwarta oczyszczalnia 2016, Klimowicz *et al.* 2016).

As for the synthetic wastewater, there were 4 beakers with capacity 2 dm^3 with volume of 1.5 dm^3 reject water generated during the mechanical dewatering of the digested sewage sludge.

Before adding the Phoslock®, the following solution parameters was specified: phosphate phosphorus concentration, pH, temperature, conductivity and total solids suspension To each beaker 10 g, 50 g, 100 g, 150 g of Phoslock® was added respectively. Each of them has been subjected to mixing, selected on the basis of the results of tests for synthetic wastewater. RW with Phoslock® was mixed for approximately 10 seconds after each sampling. The study was performed on the day of collection of the RW from wastewater treatment plant and after 24 hours of sedimentation. Sampling from each beaker was held 6 times, after certain times of contact of the solution with Phoslock®, respectively after 15, 75, 135, 1455, 1515 and 1575 minutes. All tests were conducted at room temperature approx. 23°C.

Physical and chemical analysis

Before measurement the parameters of synthetic wastewater, the samples were subjected to filtration to determine the total suspended solids. Content of total suspended solids was carried out on a very fine filters, which was calculated using the formula (2):

$$Z = \frac{(m_2 - m_1)}{V} * 1000 \qquad (2)$$

where: Z – total suspended solids concentration [mg /l], m_1 – mass of filter before filtration [g] m_2 – mass of filter after filtration [g], V – sample volume [l]

Temperature and pH were measured by using a pH meter WTW inoLab 720, conductivity was measured by conductivity meter HACH Lange HQ40D Multi. PO4-P concentration was define by cuvette tests HACH Lange LCK 350 (2.0–20 mg/l PO4-P) and LCK 349 (0.05–1.5 mg/l PO4-P).

Studies with synthetic wastewater and RW lasted approx. 24 hours and 26 hours respectively. Three series of repetitions were made. During a series of measurements collected 21 samples of synthetic wastewater (63 samples in total), and 25 samples for reject water (RW) generated during the mechanical dewatering of the digested sewage sludge (75 samples in total).

Results and discussion

Table 4 shows the average results of the experiments carried out with the synthetic wastewater (Table 4).

Table 4. Average results from using 100 g of Phoslock® after 24 hours contact time for each beaker for synthetic wastewater

Sample	pH	Conductivity	Concentration of PO_4-P	Quantity of adsorbed PO_4-P	
–	–	µS/cm	mg/l	mg/l	%
0	7.09	80.6	10	–	
Beaker 1	6.70	1216	0.079	9.921	99.2
Beaker 2	6.44	1151	0.057	9.943	99.4
Beaker 3	6.59	1171	0.066	9.934	99.3
Beaker 4	6.91	460	1.69	8.31	83.1

The following Figs. 1–6 shows the characteristics of the changes in individual parameters. During the test with the synthetic wastewater a rapid decrease of the phosphorus concentration was observed in each beaker (Fig. 1). In beaker 1 (no mixing) the concentration of PO4-P was reduced by more than 99% after almost 20 hours of sedimentation. In beakers 2, 3 (mixing after sampling) and 4 (continuous mixing) the concentration of PO4-P has dropped by more than 99% after 30 minutes of sedimentation. In beaker 4 after 20 hours of sedimentation, the release of phosphorus from the adsorbent was observed. On the basis of these observations, to the second part of the experiment was rejected both the continuous mixing, and no mixing.

Figure 2 shows the fluctuations of pH during the experiment. The pH level was 7.09, while after Phoslock® application slightly decreased to the value of approx. 6.50.

Conductivity of input (“0”) sample was 80.6 µS/cm. After the addition of the sorption material, conductivity significantly increased with contact time to the value of 1200.0 µS/cm and 24 (Fig. 3). In beaker 4 (continuous mixing) conductivity increased only to 460 µS/cm, which may be caused by substantial amount of non-sedimented suspension, which most likely adsorbed ions dissolved in the solution onto their extensive surface.

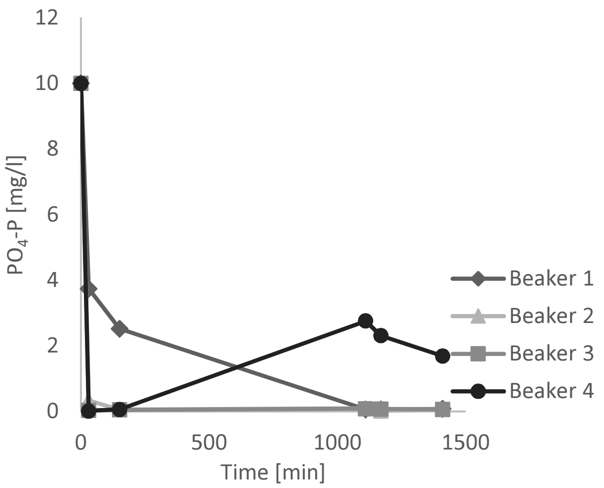

Fig. 1. Concentration of PO_4-P after using Phoslock® as a function of time for synthetic wastewater

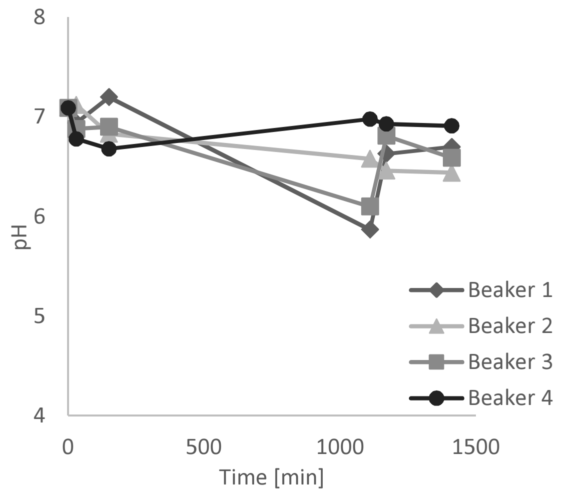

Fig. 2. Characteristic of pH changes during the investigation for synthetic wastewater

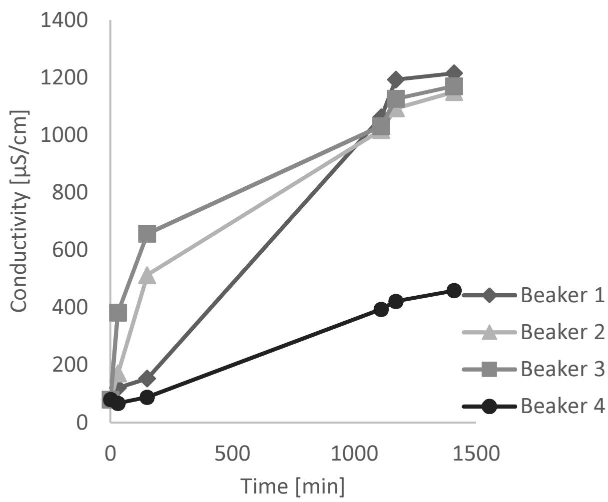

Fig. 3. Characteristic of conductivity during the investigation for synthetic wastewater

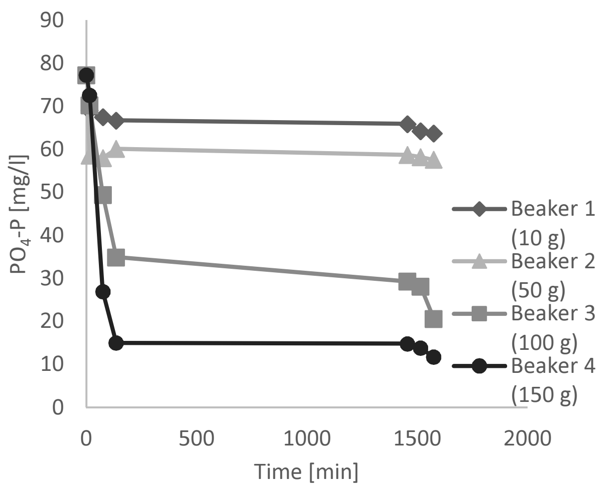

Fig. 4. Concentration of PO_4-P after using Phoslock® as a function of time for reject water (RW) generated during the mechanical dewatering of the digested sewage sludge

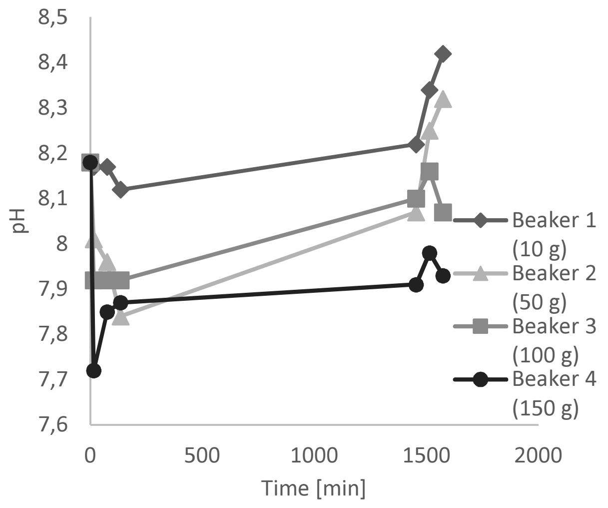

Fig. 5. Characteristic of pH changes during the investigation for RW

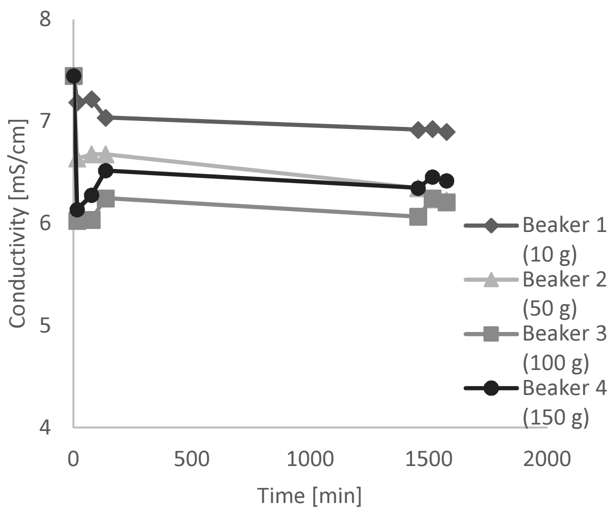

Fig. 6. Characteristic of conductivity during the investigation for RW

During the experiment carried out with RW, there were used 4 different doses of Phoslock® and the same method of mixing (few times mixing with glass rod after taking the sample). The average results of the observation after 26 hours contact time in each of the three series are presented in Table 5.

Table 5. Average results from using Phoslock® after 26 hours contact time for reject water (RW) generated during the mechanical dewatering of the digested sewage sludge

Sample (mass of adsorbent)	pH	Conductivity	Concentration of PO_4-P	Quantity of adsorbed PO_4-P	
–	–	mS/cm	mg/l	mg/l	%
0	8.18	7.45	77.3	–	
Beaker 1 (10 g)	8.42	6.90	63.7	13.6	17.6
Beaker 2 (50 g)	8.32	6.23	57.6	19.7	25.5
Beaker 3 (100 g)	8.07	6.21	20.6	56.7	73.4
Beaker 4 (150 g)	7.93	6.42	11.7	65.6	84.9

The initial concentration of PO4-P was 77.3 mg/l. The amount of adsorbed phosphorus ions increased with the amount of added Phoslock® and contact time (Fig. 4). The largest decrease in the concentration of PO4-P was observed after 75 minutes, in following samples concentration also fell, but not ran rapidly. With dose of 10 g after 26 hours of contact with adsorbent, the amount of adsorbed PO4-P was 13.6 mg/l, with dose of 50 g maximum quantity of removed phosphorus was 19.7 mg/l, with doses of 100 g and 150 g the amount of adsorbed PO4-P was 56.7 mg/l and 65.6 mg/l respectively. The maximum efficiency of phosphorus removal up to 85%, was achieved for the highest dose of Phoslock® (150 g) but there was lower by 14% than the result achieved for synthetic wastewater. This difference arise from the initial concentration of PO4-P, which, in comparison with the synthetic wastewater was nearly 8 times higher. Factors which could also effect on effectiveness of the phosphorus reduction may be composition of RW and presence of chemicals compounds which could interact with sorption process.

Results from field test presented by Haghseresht *et al.* 2009, shown higher effectiveness about 98% of phosphorus removal from real wastewater. A similar efficiency 87% of phosphorus reduction shown research performed by Zamparas (Zamparas *et al.* 2015), which was reached at the similar to our temperature of 25°C. According to Zamparas (Zamparas *et al.* 2015) efficiency of phosphorus removal increases with increasing of temperature from 5°C (73%) to 25°C (87%). Our laboratory study was conducted at room temperature approx. 23°C, therefore the conditions for the pursuit of research may be considered as optimal.

The pH level during the experiment, similarly for the synthetic wastewater, has been subject to slight fluctuations (Fig. 5). Its value was oscillated between 7.7–8.4 with an initial pH equal to 8.18. The pH value of the wastewater during the test for synthetic wastewater and RW fluctuated at a level of 6.5–8.5, therefore remained within the optimal pH value for maximum efficiency of phosphorus removal equal 5.0–9.0 (Ross *et al.* 2008). Phoslock® did not shown the ability to change the pH of the solution, which can be considered as an advantage. For the initial pH exceeding these values, must be adjusted to the optimal conditions of the adsorption material.

Characteristics of conductivity indicates a slight decrease over contact time from the value of 7.45 mS/cm to a level of 6.90 mS/cm in a beaker 1 and approx. 6.20 mS/cm in beakers 2,3,4 (Fig. 6) for RW. Conductivity values are significantly higher than the values for synthetic wastewater.

Conclusions

During the experiment, a very high efficiency of phosphorus ions removal was achieved both for synthetic wastewater (99%) and reject water generated during the mechanical dewatering of the digested sewage sludge (85% for the largest amount of Phoslock® – 150 g).

Experiment did not show significant effect of Phoslock® on the pH level of the solution, which is an extremely important advantage in wastewater treatment.

After application of Phoslock®, conductivity of the synthetic wastewater has increased with the contact time of the value 80.6 µS/cm to over 1200.0 µS/cm. In the case of RW, the conductivity was 7.45 mS/cm and ranged to the value of approx. 6.50 mS/cm, therefore Phoslock® caused a slightly decrease in conductivity. This means that the analyzed absorbent material does not significantly affect the conductivity of the real wastewater.

The study showed a high adsorption capacity of Phoslock®. The concentration of phosphorus compounds in synthetic wastewater was too low to exhaust the sorption capacity of the amount of material. Due to the duration of the experiment of 24–26 hours, there were not obtained exhaustion of sorption capacity of adsorption material for RW. The prospect for further research is to carry them until to obtain exhaust of sorption capacity of Phoslock® and to further investigate the conditions of the process to optimise it.

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