



Struvite recovery options in conventional wastewater treatment plants (WWTPs)

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Abstract

In the face of increased demand for phosphorus recovery from waste and in particular from wastewater, the study deals with the opportunities offered by the sludge obtained in applying Chemicals Phosphorus Removal (CPR) and Biological Phosphorus removal (BPR). Two case studies of Bulgarian Municipal WWTPs are used as examples of CPR and BPR and sources of sludge generated in the corresponding plants. The high yield of struvite in treatment of sludge dewatering liquor from an activated sludge system with anoxic and oxic zones demonstrates the effectiveness of the application of sea water brine as a low cost magnesium source. The effects of CO₂ stripping and bio-electrochemical alkalinity generation on pH elevation through the process of struvite formation are shown for real sludge dewatering liquor in both laboratory and bench scale units. In addition, the potential of phosphorus mobilization from FePO₄ by microbial fuel electrolysis cell and the potential of such a process to be applied as a pre-step for struvite production from sludge developed in a CPR WWTP are revealed.

Keywords : Phosphorus Removal, struvite, dewatering,

1. Introduction

The issue of Phosphorus (P) attracts the world attention in two aspects, namely the eutrophication caused by the nutrient pollution (in particular P) and its depletion as an important nutrient element [1]. Currently, in many countries the problem of eutrophication of closed water bodies has not been resolved. Even in industrialized EU countries P released by human activities is not fully under control. Jarvie et al (2006) [2] based on a well-planned monitoring made the important conclusion that in UK diffuse inputs to surface waters are of no consequence related to waters eutrophication while the point discharges (such as streams from wastewater treatment work) should be of prime concern. As an example, according to HELCOM (2009) [3] the emissions from Municipal Wastewater Treatment Plants to Baltic Sea consisted 50 % of total P load in the whole catchment area. In order to answer the strict discharged standards imposed by EU to avoid eutrophication, P removal from wastewater is one of the main challenges of water utilities. Besides the important environmental implications based on non-proper wastewater treatment, a great nutrient potential for P recovery is found in municipal wastewater, which undergoes an enhanced P elimination in the treatment plants. In this sense, the wastewater works can be regarded as an important secondary source of P in extracting P concentrated within the sludge. With a potential of 56,700 t Pyr-1 (56% of all raw material fertilizers), the sewage sludge has the major role among the secondary raw materials in Germany [4].

Over 56 000 t dry sewage sludge was obtained in Bulgaria (the country of case studies in interest) in annually according to the national plan for management of sewage sludge from Municipal wastewater treatment plants [5], with a clear tendency with increasing wastewater volumes and advanced wastewater treatment processes, sludge production to be higher. Inside EU over 7 Mt dry solid (DS) municipal sludge is produced annually. EU estimates that the sludge amount will be 12,000,000 tons DS per annum (2015). Globally, 1.3 Mt P/year is treated in MWWTPs worldwide [6].

While techniques to remove P from centralized WWTP systems have been developed over several decades, the interest to recover and recycle P in a usable form is quite recent. There are various methods for phosphorus recovery at waste water treatment plants. These techniques differ by the origin of the used matter (wastewater, sludge, sludge liquor, sludge ash), the applied process (precipitation, wet chemical extraction, and thermal treatment [6, 7]). One of the most promising technologies is the phosphorus precipitation as ammonium magnesium phosphate (struvite) applied in sewage works. Commercial large-scale struvite production plants which precipitate struvite from digested sludge liquors are operating in the USA, Canada, and Belgium and recently in China (SCOPE Newsletter, 2012) [8]. However, in spite of the significant progress in struvite recovery technology, a huge room for struvite production optimization still exists.

It is reasonable to expect that P recovery depends on the method of its removal from the wastewater. The most common methods for P removal are the Chemical Phosphorus Removal (CPR) and the Enhanced Biological P removal (EBPR) [9-11]. The CPR has the disadvantage of increased sludge volume (by 26 % in activated sludge plants) and reduced P plant availability compared to EBPR. However, in several cases of municipal wastewater treatment, the less complicated approach than biological approaches, the CPR, is the only way to reach the stringent requirements for residual P because the EBPR can solely rarely ensure P removal efficiency higher than 60 %.

It is well recognized that the combination of EBPR and sludge digestion offers better opportunity for P extraction because during the anaerobic stage P is released in a high extent and makes the struvite precipitation efficient. In CPR based plants the recovery efficiency of P as struvite is only in the range of 10-50% of the total influent P load [12, 13] because part of P is metal bonded in the biomass.

In both cases, CPR + BPR or EBPR, the struvite precipitation is usually performed on sludge that is digested anaerobically. The high concentration of P and ammonia in the digested sludge is a pre-requested for the efficient P precipitation as struvite. The precipitation can be conducted both before and after the dewatering process. The direct precipitated within the sludge has the advantage of improved separation during the dewatering process and lower maintenance because of preventing clogging of pipes and abrasion of centrifuges.

The addition of iron is a convenient way for removing phosphorus from wastewater, but this is often considered to limit phosphorus recovery [14, 13]. The alternative, EBPR is well accepted technology with good perspectives to be vastly implemented, however currently the majority of WWTPs are applying technological schemes with combined CPR/Bio P removal processes. Based on this reality, the present paper aims to clarify the potential of P recovery as struvite in applying the combination of CPR + Bio-removal and solely BPR.

Transforming the chemically “locked” phosphates in CPR sludge back into the soluble form is very important for the general yield during phosphorus recovery as struvite. From chemical point of view, the mobilization of ferric phosphate is possible by Fe^{3+} reduction. A newly discovered path for phosphorus recovery from sewage sludge is the mobilization of orthophosphates from iron phosphate (FePO_4) by microbial fuel cell (MFC) power [15,16] An electrical current is achieved in the microbial fuel cell by attaching electrogens (bacteria like *Escherichia coli*) to an anode which the bacteria use as electron acceptor. With a proton exchange membrane the anode cell is separated from the cathode cell. The electric flow provides reductive conditions next to the cathode. Thus dissolution of up to 82% of the FePO_4 has been achieved. This technique can be combined with

struvite crystallization by adding magnesium chloride and ammonium hydroxide in stoichiometric ratio [15]. Our aim is to prove this phenomenon in using real sludge dewatering liquor from the MWWTPs in interest.

2. Case studies

The studies are focused on optimization of phosphorus (P) precipitation as struvite using a sludge taken from BPR and CPR-BPR municipal wastewater treatment plants (MWWTP). Two case studies are under consideration, namely the MWWTP of Burgas (CPR + BPR) and MWWTP of Pomorie (BPR). Both plants are serving towns situated on the South-East Coast of Black Sea (Bulgaria). The main characteristics of the plants are given in Table 1. The plants are conventional activated sludge systems with D/N zones. Such technology configuration allows a partial bio-dephosphatation to occur. In the case of Burgas MWWTP, FeCl_3 is used as a source of ferric iron. FeCl_3 is added to a point before the activated sludge reactor. The plants P inlet/effluent concentrations and the corresponding P loads on a monthly base are given in Table 2.

Table 1: General characteristics of the MWWPs in study

Municipal WWTP	PE	P-removal	Stabilization	Dewatering
Burgas	200000	CPR/BPR	Anaerobic open	Centrifuges
Pomorie	40000	BPR	Anaerobic open	Centrifuge

Table 2: Monthly influent flow rate and influent/effluent P concentrations

2014	MWWTP Burgas		MWWTP Pomorie		
	Q_{av}^* m ³ /month	P_{av}^{**} mg/L Influent effluent	Q_{av} m ³ /month	P_{av} mg/L Influent / effluent	
January	1363306	2.9 0.2	153134	4.7	3.4
February	1124928	2.7 0.8	140327	3.8	2.2
March	1224029	2.2 0.4	166427	3.6	2.5
April	1036800	2.7 0.7	153560	4.9	3.2
May	1098144	2.5 0.8	170397	4.8	1.5
June	1031616	2.6 0.7	217304	4.4	2.9
July	1111536	2.4 0.4	263897	6.3	3.9
August	1004400	2.6 0.6	268726	4.9	2.6
September	1086048	2.4 0.5	214790	4.2	0.6
October	1248134	3 0.5	195350	6.6	2.3
November	1290816	2.5 0.4	186339	3.9	0.9
December	1885594	2.2 0.5	130945	3.9	0.9

* Q_{av} – average monthly inlet flow rate.

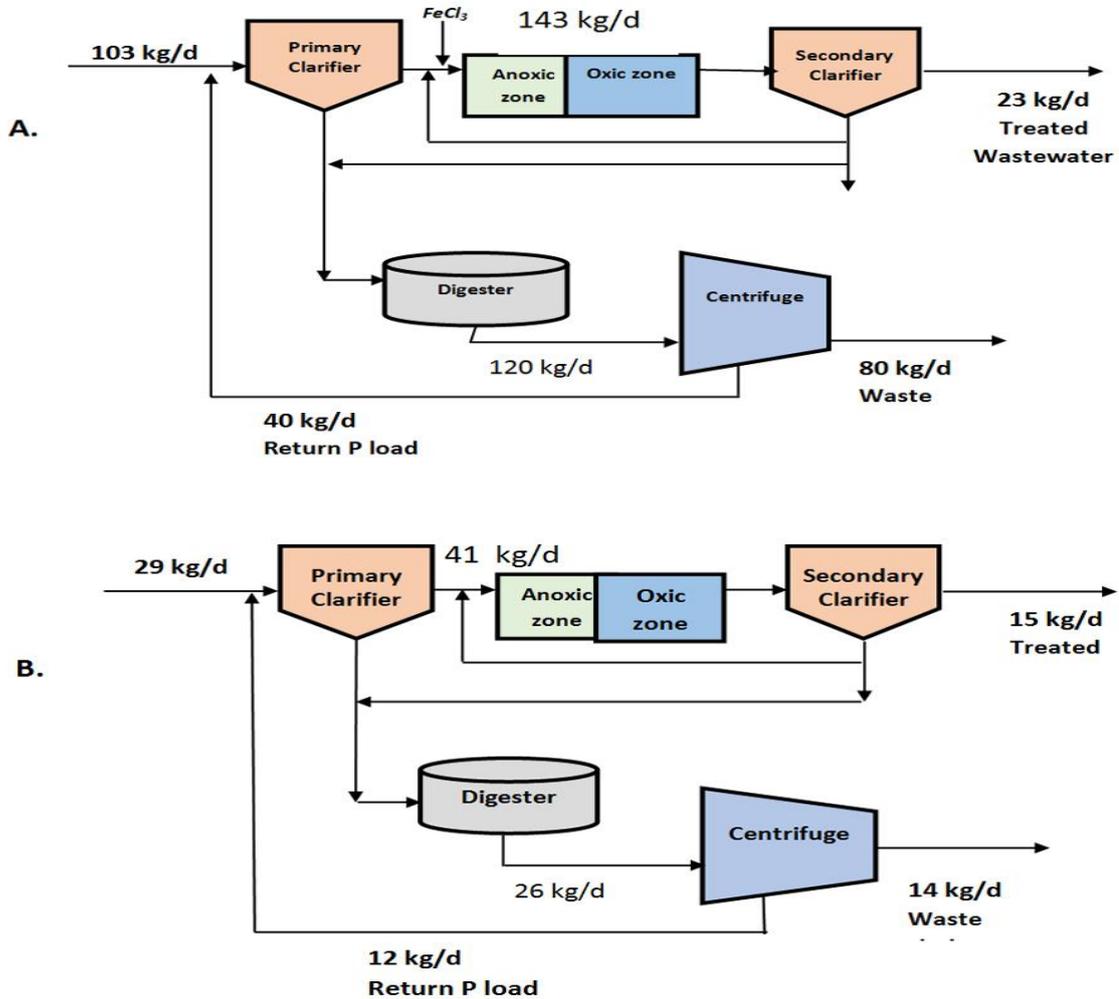
** P_{av} – average monthly concentration of total P.

The calculated doses of FeCl_3 per mass of P ($\text{mgFe}^{3+}/\text{mgP}$) based on the monthly flow rate and the used FeCl_3 (40% solution) are shown in Table 3.

Table 3: Doses of the applied FeCl_3

2014	MWWTP Burgas											
	Jan	Feb	March	April	May	June	July	Aug	Sept	Oct	Nov	Dec
$Q_{av} \cdot 10^3$ m ³ /month	1363	1125	1224	1037	1098	1032	1112	1004	1086	1248	1291	1886
FeCl_3 , m3/month	35.9	31	37.6	33.1	36.3	44.6	31.8	37.7	35.6	39	34.5	37.9
Dose, $\text{mgFe}^{3+}/\text{mgP}$	1.8	1.9	2.3	1.9	2.1	2	2.1	2	2.1	1.7	2.1	2.4

Based on the monthly values of wastewater flow rates, P removal efficiency and the parameters of the returned sludge dewatering liquor stream (1% of inlet flow rate, 100 mg /l phosphates) mass balance studies were performed. The mass balance features and the initial parameters for the corresponding WWTP are shown in Fig. 1.



A. MWWTP Burgas

Flow rate: 1208779 m³/d
 Influent P concentration: 2.6 mg/l
 Effluent P concentration: 0.6 mg/l
 Average Dewatering liquor concentration: 100 mg/l
 Ratio: (return dewatering liquor flow rate/inlet flow rate): 0.01

B. MWWTP Pomorie

Flow rate: 6188 m³/d
 Influent P concentration: 4.7 mg/l
 Effluent P concentration: 2.2 mg/l
 Average Dewatering liquor concentration: 200 mg/l
 Ratio: (return dewatering liquor flow rate/inlet flow rate): 0.01

Fig.1. P balance

3. Materials and methods

3.1. Sewage sludge samples

The sludge dewatering liquor samples represent a centrate from the corresponding dewatering stage of MWWTPs of Burgas and Pomorie. Immediately after sampling, the chemical oxygen demand (COD), phosphate and ammonia concentration of the sludge and sludge filtrates were measured by using *HACH Lange* cuvette tests (Product ID: *LCK 314*, *LCK 350* and *LCK 303* respectively) and *HACH Lange* DR 3900

spectrophotometer. The brine used as a Mg source was collected from salt evaporation ponds near Burgas – Bulgaria. The samples were tested for magnesium and calcium content by the Ethylene-diamine-tetra-acetic acid (EDTA) complexometric method [17] and Plasma atomic emission spectroscopy (ICP-OES).

3.2. Struvite precipitation and characterization

Three different molar ratios of $Mg^{2+}: PO_4^{2-}$ were tested (1:1, 2:1 and 3:1). The influence of the pH on the precipitation process was studied at pH 8, 9 and 10. The products obtained during the precipitation experiments were characterized by: URD x-ray step scanning diffractometric (Cu K α monochromator - step size 0.030° and step time 1 s); Thermogravimetric by *NETZSCH STA 449 F3* TGA-DSC analyzer. The measurement of mass change was carried out at temperature range of 25-900 °C (heating rate 15 °C/min) and $\alpha-Al_2O_3$ as reference. The mass loss of MAP was plotted as a function of temperature; Plasma atomic emission spectroscopy (ICP-OES) The struvite samples and the sludge samples were dried in a vacuum oven from Salvis (Rotkreuz, Switzerland) for 24h at 40°C/105°C. In the next step the precise weighing scale XS 205 Dual Range (Mettler-Toledo GmbH (Giessen, Germany)) was used to measure the weight of the dried samples. Afterwards the dried the samples were pulverized with a ball mill MM 301 from Retch (Haan, Germany). The precipitated product was observed by optical microscopy (Microscope Optika B-150 (400 XS)).

MgCl₂ and sea water brine were selected as alternative sources of magnesium. Besides magnesium, the sea water brine contains calcium, potassium, sodium, chloride and sulphate. The high magnesium/calcium ratio is important for effective precipitation of struvite. In excess of calcium, the production of struvite could be suppressed due to the parallel synthesis of the calcium phosphate [18]. The density of the sea water brine sample collected from the evaporation ponds of Pomorie (Bulgaria) was 27°Bomme.

Before the first precipitation experiments, the concentration of magnesium and calcium ions in the brine were tested. The magnesium concentration observed (59.5 g/l) is significantly higher than the calcium concentration (3.5 g/l). The data of ICP analyses of both dried sludge and brine samples shown no detectable levels of the tested heavy metals. The concentrations of the other elements measured are presented in Table 4.

Table 4: Concentration of main elements of sea water brine and digested sludge

Element SAMPLE	Na	Mg	Al	P	S	K	Ca	Fe	Zn	Cl	Salt total
Sewage sludge [g/kg dry weight]	1.22	6.34	20.85	10.58	8.92	3.73	30.17	20.81	1.06	-	-
Brine [g/L]	11.34	59.5	-	-	19.17	16.03	3.5	-	-	161.97	248.5

The content of ammonia and phosphorous ($o-PO_4$) in the centrate is given in Table 5. The range of concentrations given in Table 5 is typical for the whole period of the study, 2013-2014. For the mass balance studies an average P concentration of 200 and 100 mg/l was accepted in the cases of Pomorie and Burgas WWTPs, respectively.

Table 5: Phosphate and ammonia levels in the centrate.

Supernatant	Average concentration of PO_4^{3-} , mg/l	Average concentration of NH_4^+ , mg/l
WWTP – Pomorie	138 ÷ 250	250 ÷ 600
WWTP - Burgas	86 ÷ 131	250 ÷ 500

The Microbial Fuel cell used in studying the FePO_4 mobilization was designed as a cylindrical reactor with two chambers separated by cation exchange membrane. The volumes of anodic and cathodic chambers are 20 and 10 ml, respectively. Each compartment had two ports for input and output flows. Both electrodes were made of carbon cloth with circular section. The anode compartment was filed by granular activated carbon to provide better biofilm formation and electron. The anode and cathode were connected with an external electrical circuit loaded with 100Ω resistor (Fig. 2).

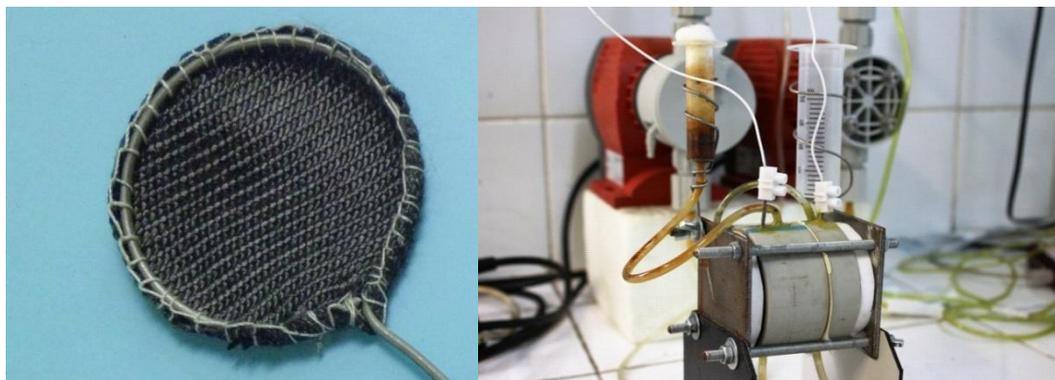


Figure 2: Design of the MFC reactor (A) and the carbon cloth electrodes (B) designed and used in phosphate dissolution experiments.

The electrogens were isolated from bottom sediment from “Yasna Polyana” Dam near Burgas. The enrichment of the mixed culture was performed in anaerobic conditions by inoculation of 0.5 ml sediment in 20 ml nutrient medium containing: glucose – 15 g/dm^3 ; tryptone - 10 g/dm^3 ; yeast extract - 5 g/dm^3 and NaCl – 5 g/l and pH -7. After 96 hours of cell growth the enriched culture was suspended in fresh nutrient medium to a microbial concentration of 10^7 CFU/ml and loaded in the anode chamber of the MFC. The process was conducted at the temperature range of $20\text{-}25^\circ\text{C}$. During operation of the MFC the potential difference between the electrodes was measured and recorded in 10 minutes intervals by *data acquisition system* connected to PC equipped with specific software.

Ferric phosphate used in this study was synthesized in our lab by imitating the coagulation procedure applied during conventional waste water treatment. The procedure includes phosphoric acid dilution and coagulation of phosphates as ferric phosphate by adding equimolar concentration of FeCl_3 . During the coagulation and synthesis the pH was adjusted to 3.2 by adding NaOH to the reagent mixture. The low pH values prevents formation of $\text{Fe}(\text{OH})_3$ which is parasitic reaction during this synthesis.

4. Results and discussion

4.1. P balance

The results obtained on removal efficiency show the capacity of CPR/BPR system to reach concentrations of residual P below $1 \text{ mg/l o-PO}_4^{3-}$ which is the limit for the most receiving water bodies in Bulgaria. The performed CPR/BPR case study clearly demonstrates that a considerable part of P is accumulated in the sludge. Based on the data given in Table3, the average Fe dose was calculated ($1.91 \text{ mg Fe/1 mg P}$). Such ratio is closed to the commonly applied mole ratio $1.0 - 1.5$ [22].

By accepting that all of the chemically bonded P remains in the sludge (120 kg/d) at the CPR/BPR plant and a monthly applied FeCl_3 of $1 \text{ m}^3/40\%$ solution, it can be easily calculated that 60% of P is chemically precipitated, while the remaining part of P is incorporating in bio-solids.

Table 6: Removal Efficiency and P separation in plant streams

Type of plant	Removal efficiency, %	Average residual P, mg/l	P waste sludge/P influent WW, %	P sludge dewatering liquor/P influent, %
CPR/BPR	77.6	0.54	77.7	39
BPR	51.7	2.24	48.3	41

4.2. Sea water brine application

In order to compare the effectiveness of both Mg sources, sea water brine and $MgCl_2$, the struvite precipitation process using phosphate- and ammonia- rich sludge dewatering liquor wastewaters was carried out at Mg:PO₄ 2:1 and pH = 9.5. The phosphate removal rates are shown in Fig.3. Better phosphate removal rate was obtained in applying sea water brine (92.1%) than the $MgCl_2$ (69.6%). This is mostly due to the Ca availability and the organic matter in the brine. The later may can have an incremental coagulation effect. In our further experiments sea water brine was used as a source of Mg.

The mole ratio Mg:P and pH were studied as crucial parameters affecting the process of P precipitation as struvite. The ammonia concentration was not taken in consideration as it presented in a high excess. pH is one of the important parameters for struvite crystallization, nucleation and supersaturation. Many studies report that the minimum struvite solubility appears at pH range of 9.0 to 9.5 [19]. The results obtained at pH 9, 9.5 and 10 are given in Fig. 4. Efficiency up to 90 % at the highest molar ratio 3:1 and pH 10 was obtained. However, many authors report that at pH over 9.5 limits struvite precipitation process because other compounds rather struvite precipitate. For example, pH over 10 favors $Mg_3(PO_4)_2 \cdot 22H_2O$ formation which hinder struvite crystallization [20]. In addition, high pH values over 9.5 require higher costs for alkalization. Our results show that phosphate removal rates obtained at different molar ratios between Mg:PO₄ (1:1; 2:1; 3:1) and pH over 9 (precipitation rates of 83-90 %) were relatively close in respect of P removal effectiveness. Based on the above considerations, pH range of 9-9.5 and molar ratio 2:1 were chosen as optimal parameters for struvite crystallization from sludge dewatering liquor.

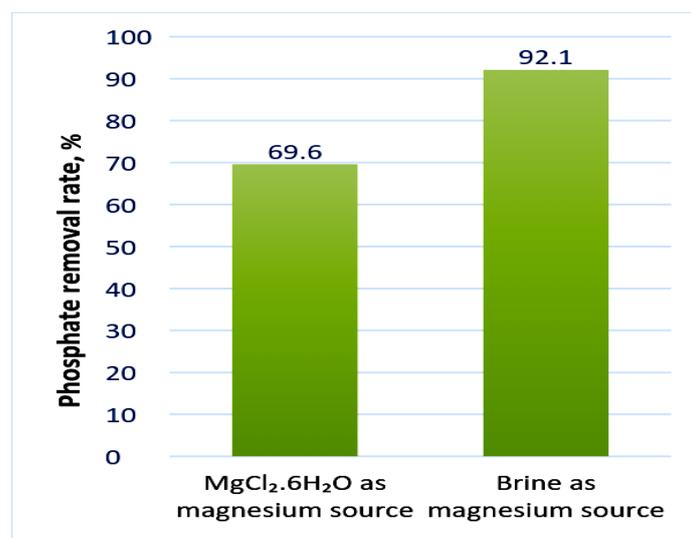


Figure 3: Phosphate removal rate at different magnesium sources (Initial concentration: PO₄³⁻, 181 mg/l; NH₄⁺, mg/l)

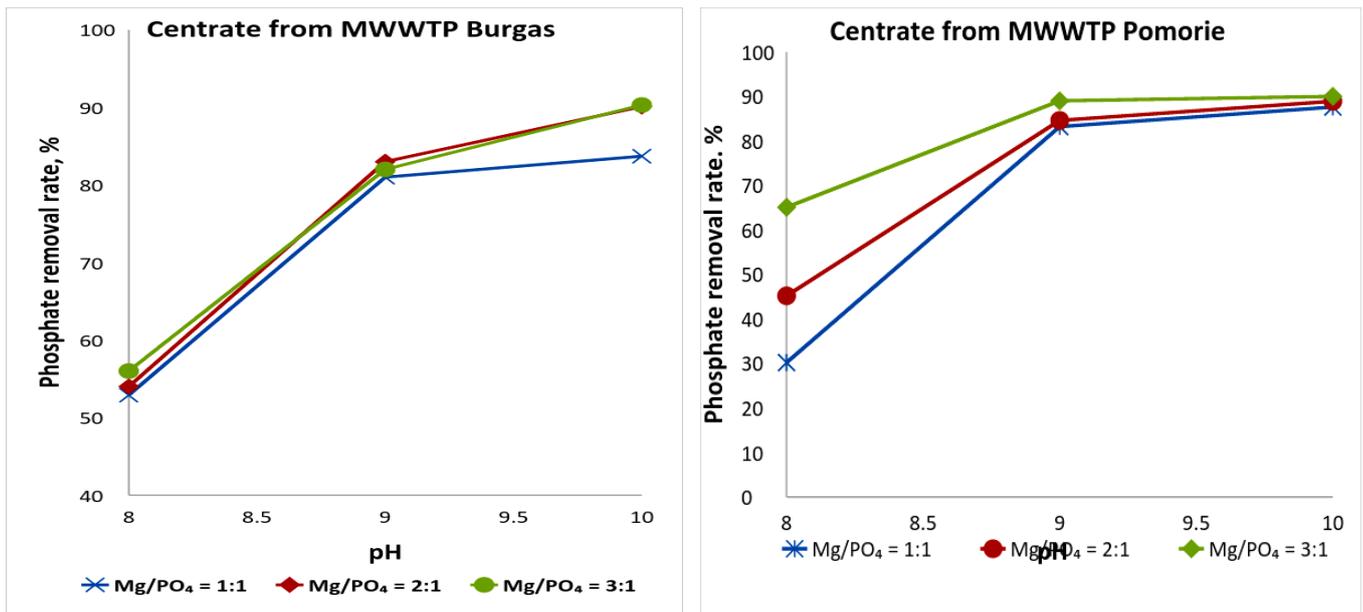


Figure 4: Phosphate removal rates [%] at different molar ratios and pH values

Fig.5 shows struvite crystals and their specific structures under a microscope and picture of the powder product. Typical orthorhombic crystals were observed at 400X magnification.

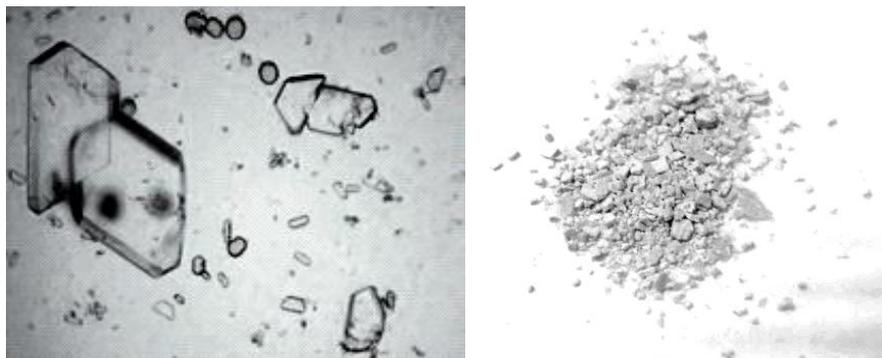


Figure 5: Microscopic observation of struvite crystals (400 XS) (left) and the dry precipitated product

4.3. Bio-electrochemical mobilization of FePO₄

In the first set of experiments the reductive conditions in the cathode chamber of the MFC was used toward model suspension of ferric phosphate with concentration of 240 mg/l. The initial concentration of *ortho*-phosphates in this solution left after the coagulation applied was 20 mg/l. As a result of electrochemical reduction the concentration of dissolved phosphates in the medium was increased to 184 mg/l in 96 hours (Fig. 6A). The maximal and average dissolution rates obtained are as follow: $Q_{\max} = 3.04 \text{ mgPO}_4 \cdot \text{h}^{-1}$ and $Q_{\text{av}} = 1.7 \text{ mgPO}_4 \cdot \text{h}^{-1}$.

In the next experiment the MFC was loaded with ferric phosphate suspension with higher concentration of suspended and dissolved phosphates. The idea was to apply concentrations which are closer to those observed in the real activated sludge after digestion. The results obtained are presented on Fig. 6B. As we expected the dissolution rates were higher - $Q_{\max} = 7.08 \text{ mgPO}_4 \cdot \text{h}^{-1}$ and $Q_{\text{av}} = 4.06 \text{ mgPO}_4 \cdot \text{h}^{-1}$. This is implication of a typical MFC behavior and correlation between the electric current obtained from the cells and the presence of electron acceptors. From the electrochemical point of view in such a system the Fe³⁺ plays a role of terminal acceptor of electron and higher concentrations of FePO₄ leads to generation of higher voltage, current and faster cathode reactions.

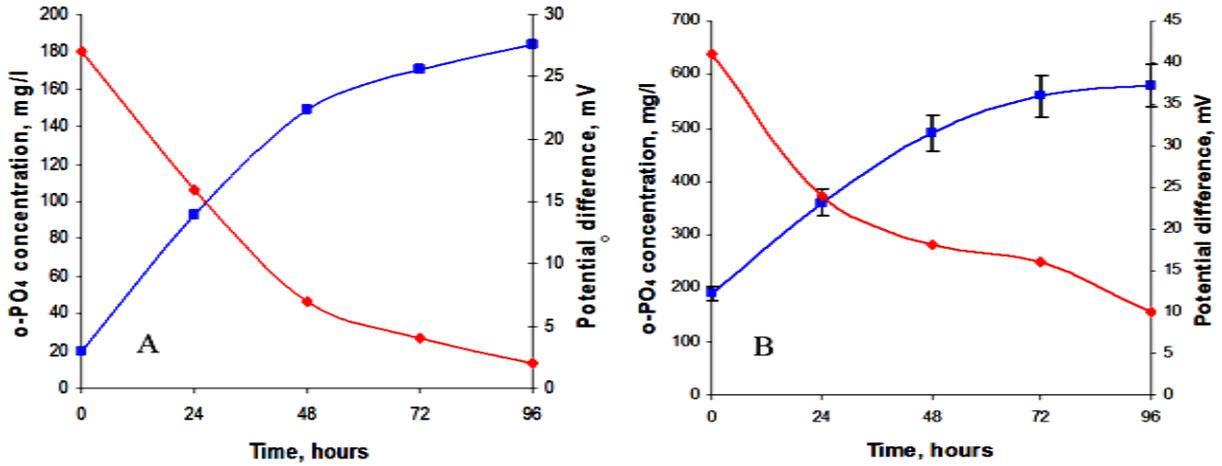


Figure 6: *Ortho*- phosphate concentrations (■) and potential difference (♦) in the MFC during the first sets of experiments. Initial dissolved and suspended phosphate concentrations in as follow: A – 20 mg/l and 240 mg/l; B – 190 mg/l and 450 mg/l;

The real fluids obtained from activated sludge digestion, which will be the potential target of this technology, are characterized by very high organic content. It is known that the variety of organic molecules can affect the electrochemistry of the fuel cells by providing alternative acceptors and shifting the cathode reactions to different “parasitic” processes. To evaluate the influence of organics in the activated sludge on the above described process we performed experiment for phosphate mobilization in model fluid prepared by suspending the FePO₄ in activated sludge filtrates with COD 1500 mgO₂·l⁻¹. The dynamic of phosphate dissolution and the comparison between the mobilization rates in fluids with and without organic contents are presented on Fig. 7.

The results obtained shown the negative influence of the organic matter on the process kinetics. This is probably a result of deterioration of electrochemical conditions leading to decreasing the cathode potential in presence of biodegradable organics in the cathodic space. It has to be point out that even at these conditions the electrochemical reduction and mobilization is possible; dissolution of 111 mg/l phosphates in 168 hours was observed. The ferric phosphate mobilization test show that considerable part of the “locked” P can move into the liquid phase. The data obtained allows accepting that the soluble phosphate can be increased at least three times in treating free of organic matter liquor (Fig.6, case B) and 1.5 times in treatment real sludge dewatering liquor (Fig.7) by MFC. Regarding the MWWTP Burgas in which around 13 tons of FePO₄ is formed monthly, the potential for bio-electrochemical P recovery is high.

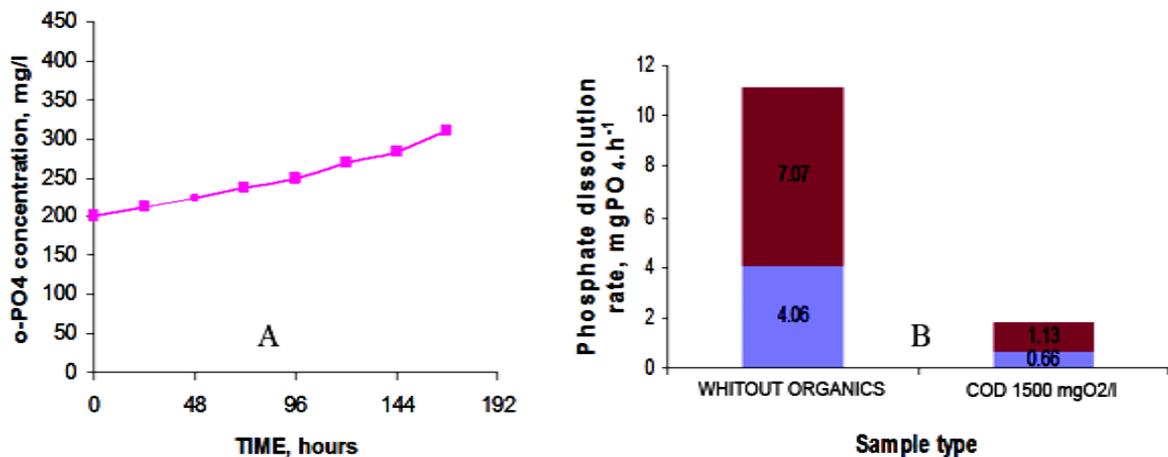


Figure 7: Electrochemical phosphate dissolution in the presence of high organic content in the medium (A) and the effect on the process kinetics (B) – Maximal Dissolution Rates in purple; Average Dissolution Rates in Blue.

Conclusion

The studies related to the usages of sea water brine as a source of Mg proved that such application can be viewed as another step for more economical production of struvite from sludge dewatering liquors originated from MWWTPs. The P balance studies show that in case of CPR/BPR plant the prevailing part of P is chemically bonded, i.e. the chemical precipitate can be considered as an important secondary source of P. Such a conclusion makes promising the application of technologies for P extraction which enable mobilizing the phosphate constituents and their subsequent conversion into valuable chemicals with fertilizing properties. In this sense, the results obtained with the MFC show a way for low energy mobilization of phosphates locked in FePO₄ precipitates. The results from the lab scale MFC experiments illustrated the general principle of bio-electrochemical methods and specifically the Microbial Fuel Cell (MFC) technology in phosphorus mobilization and recovery. This approach was successfully applied with a model solutions of ferric phosphate and real fluids obtained from sewage sludge containing FePO₄.

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