STRUVITE CRYSTALLIZATION IN A FULL SCALE PLANT AFTER AUTOTROPHIC NITROGEN REMOVAL (ELAN PROCESS)

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Abstract

A full scale struvite crystallization system is in operation in the Guillarei municipal WWTP (Northwest of Spain, influent flow 32500 m^3/d) for the treatment of the centrate obtained after anaerobic sludge digestion. The precipitation process is carried out in a two-phase fluidized bed reactor connected to a gravity settler. Industrial grade $Mg(OH)_2$ is added to the system as magnesium and alkali source.

In period I the centrate was directly fed to the crystallization plant; while in period II the centrate was pretreated by an autotrophic nitrogen removal process (the ELAN® process), the combination of the biological processes of partial nitrification and anammox performed in an SBR with granular biomass. This additional step removes around 80% - 90% of the ammonium and bicarbonate ions, reducing the needs of alkali addition for struvite crystallization. Phosphate concentration in the liquid effluent was around 15 mg $P\text{-}PO_4^{3-}/L$ when pH was controlled between 8.3-8.8, regardless of influent concentration. Struvite pellets with a size range of 0.5–5.0 mm, suitable to be used as agricultural fertilizer, were obtained.

Keywords

Anammox, autotrophic nitrogen removal, full scale, industrial magnesium hydroxide, phosphorus recovery, struvite.

Introduction

Nitrogen and phosphorus contained in wastewaters are the primary causes of environmental eutrophication in surface waters (E.P.A., 2007). However, eutrophication can be reversed by controlling the nutrient inputs to waterways (Smith and Schindler, 2009) and different legislation and normative were approved throughout the world to minimize the amounts of nutrients discharged in order to reverse eutrophication trends and to mitigate nutrient losses.

On the other hand, modern agriculture is highly dependent on phosphorus, which is derived from phosphate rock, a non-renewable resource, whose global reserves might be depleted in 50 years (Cordell et al., 2009), while other studies used a wider temporal period (Van Vuuren et al., 2010). Both problems, nutrients depletion and eutrophication, can be reduced at the same time by the application of technologies for recovering nutrients out of wastewater streams.

The recovery of phosphorus as a by-product can be a sustainable and economical alternative for recovering phosphorus from wastewater. It can be applied in separated collected streams (Morales et al., 2013) or in centralized wastewater treatment plants (WWTPs). Main option is the use of crystallization processes, where minerals with application in agriculture as fertilizers are produced: struvite ($NH_4MgPO_4 \cdot 6H_2O$) or hydroxyapatite [$Ca_5(PO_4)_3OH$]. Shu *et al.* (2006) indicated that struvite production is technically feasible and economically beneficial. In the other hand, uncontrolled struvite precipitation can cause operational problems in WWTPs (Jaffer et al., 2002).

However, even if struvite formation have been extensively studied (Rahman et al., 2014), only few struvite crystallization studies have been conducted at full scale (Ueno and Fujii, 2001, Moerman et al., 2009, Crutchik et al., 2016). Industrial systems requires high phosphorus concentrations (more than 50-60 mg PO_4^{3-}-P/L in order to reach the economic viability of the processes (Dockhorn, 2009, Rahman et al., 2014). These concentrations can be observed in the anaerobic liquor from primary and secondary sludge digestion in the urban WWTP. Anaerobic liquor from sludge digestion tends to have high ammonia and phosphorus concentrations because these compounds are released in the anaerobic digestion process, while the flow of these streams represents only around 0.5-1.0% of the plant influent flow. Ammonia concentrations usually exceed 500 mg N/L and can represent 15-25% of the total nitrogen load at a WWTP (Nifong et al., 2013), phosphorus concentrations usually exceed 75 mg P/L (Desmidt et al., 2015) and can represent around 5-35% of the overall P load at the WWTP.

Usually, these kind of wastewaters require the addition of magnesium in order to reach phosphate and ammonium concentration and promote struvite crystallization. Industrial processes commonly use high grade magnesium sources, such as $MgCl_2$, $MgSO_4$, MgO or $Mg(OH)_2$, which contribute to the operational costs of the phosphorus recovery systems. The recovering costs as precipitates can be higher than the current production costs derived from mineral extraction and can make the conventional struvite recovery process uneconomical (Ansari et al., 2016). The use of low cost magnesium sources, such as seawater, brine or industrial grade MgO or $Mg(OH)_2$, can significantly reduce this operating costs (Crutchik and Garrido, 2011, Lahav et al., 2013, Crutchik et al., 2016). In addition, struvite formation occurs by nucleation followed by crystal growth and requires substantially high pH conditions, which are usually reached by the addition of strong base chemicals in conventional precipitation processes (Ronteltap et al., 2007). $Mg(OH)_2$ is generally cheaper, and has the advantage of also raising the pH (Münch and Barr, 2001).

Regarding nitrogen, the removal of the high nitrogen load of centrate by conventional nitrification/denitrification processes can be highly energy intensive and may require supplemental carbon addition since these side streams have a low carbon to nitrogen ratio (C/N). Treating the reject water instead by a side stream autotrophic nitrogen removal process, provides energy savings and reduce supplemental carbon addition (Fux et al., 2003). The ELAN® process (acronym of *'ELiminación Autótrofa de Nitrógeno'*, autotrophic nitrogen removal in Spanish) is an anammox-based process performed in one single reactor which has stood out as an efficient way to remove nitrogen from highly loaded streams with a low C/N ratio (Vázquez-Padín et al., 2014). The process was developed by Aqualia with the know-how of the University of Santiago de Compostela. It combines the partial nitrification with the anammox process, based on the establishment of aerobic and anoxic zones within the granule (Morales et al., 2015a). Ammonia oxidizing bacteria (AOB) partially oxidize half of the ammonia to nitrite (Equation (1)), while anammox bacteria combine half of the ammonia and the nitrite to produce nitrogen gas, according to equation (2). The stoichiometry of the overall ELAN® process corresponds to equation (3).

$$NH_4^+ + 1.5\,O_2 + 2\,HCO_3^- \rightarrow NO_2^- + 3\,H_2O + 2\,CO_2 \qquad (1)$$

$$\begin{aligned} &NH_4^+ + 1.32\,NO_2^- + 0.066\,HCO_3^- + 0.13\,H^+ \rightarrow \\ &\quad 1.02\,N_2 + 0.26\,NO_3^- + 0.066\,CH_2O_{0.5}N_{0.15} + 2\,H_2O \end{aligned} \qquad (2)$$

$$NH_4^+ + 0.8\,O_2 + 1.11\,HCO_3^- \rightarrow 0.009\,C_5H_7O_2N + 0.028\,CH_2O_{0.15}N + 0.44\,N_2 + 0.11\,NO_3^- + 2.50\,H_2O + 1.04\,CO_2 \qquad (3)$$

The application of these processes to the side stream of WWTPs can save 7% of treatment costs, mainly by: reduction in aeration - increase in biogas production since organic matter is not needed for heterotrophic denitrification - reduction in greenhouse gas emissions (Morales et al., 2015b).

Taking into account that bicarbonate is the main counter ion of the ammonia in the centrate and that nitrification consumes alkalinity, the ELAN® process applied to treat reject water reduced its alkalinity. Consequently, the rise of pH needed for downstream struvite precipitation will be reached with less base addition when the autotrophic nitrogen removal process is applied prior to the struvite precipitation process.

In this study, the performance of a struvite precipitation system treating directly the supernatant of the sludge digester is compared with the results achieved when this supernatant is previously treated by the ELAN® process (Figure 1). Both plants were operated at full scale in in the Guillarei municipal WWTP.

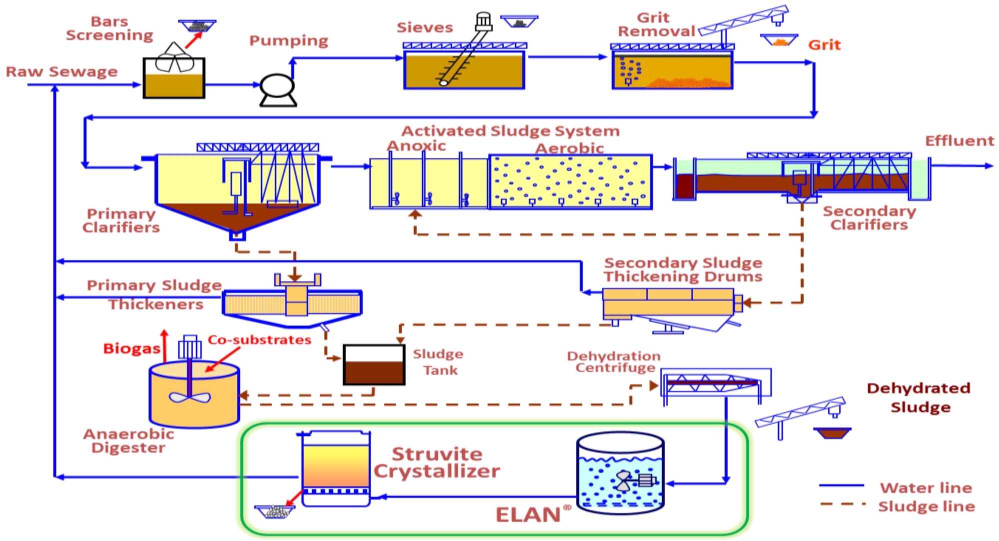

Figure 1. Phosphorus recovery by means of struvite precipitation after an autotrophic nitrogen removal (ELAN®) in a wastewater treatment plant.

Material and methods

The struvite crystallization plant at full scale was composed of a 125 L two-phase fluidized bed reactor (FBR), divided in two sections with different cross-sectional areas, and connected in series to a 3,260 L settler (Figure 2). A $Mg(OH)_2$ slurry tank was connected to the crystallization plant for the dosage of the magnesium, controlled by the pH in the settler. The settler was designed to promote the growth and recovery of fine crystals of struvite. A complete description of the plant and the key design factors can be found in Crutchik et al. (2016). $Mg(OH)_2$ was prepared by hydrating with water an industrial grade MgO at 75ºC in a batch process. A 20 m^3 tank was used to store the wastewater collected from the centrifugation of the supernatant of the sludge digester in period I or the wastewater treated by the full scale ELAN® reactor in period II and to homogenize inlet peak flows. The crystallization plant was operated continuously. The hydraulic retention time (HRT) was between 1.3 and 2.6 min for the FBR and 74.2–135.0 min for the settler. The overall HRT for the entire system was 75.5–137.6 min.

Struvite seed crystals were added to the FBR to promote the heterogeneous crystallization of struvite at the beginning of the operation.

Period I was divided in two sub periods (I-1 and I-2) where the phosphorus concentration was 34±6 mg-P/L and 68±28 mg-P/L, respectively. In period II the phosphorus concentration was 78.4±10 mg-P/L. Characteristics of the wastewater fed to the struvite are summarized in Table 1.

Table 1. Characteristics of the wastewater fed to the struvite precipitation plant.

Parameter	Period I-1	Period I-2	Period II
pH	8.2 ± 0.2	8.1 ± 0.1	7.2 ± 0.1
Conductivity (mS/cm)	7.7± 0.4	7.6 ± 0.2	1.9 ± 0.4
PO_4^{3-} (mmol/L)	1.1 ± 0.2	2.2 ± 0.9	2.4±0.3
NH_4^+ (mmol/L)	70.4 ± 3.1	70.0 ± 6.8	7.9 ± 2.4
Mg^{2+} (mmol/L)	0.5 ± 0.1	0.6 ± 0.1	0.7 ± 0.1
HCO_3^- (mmol/L)	67 ± 2.7	63.6 ± 2.5	8.7 ± 1.4

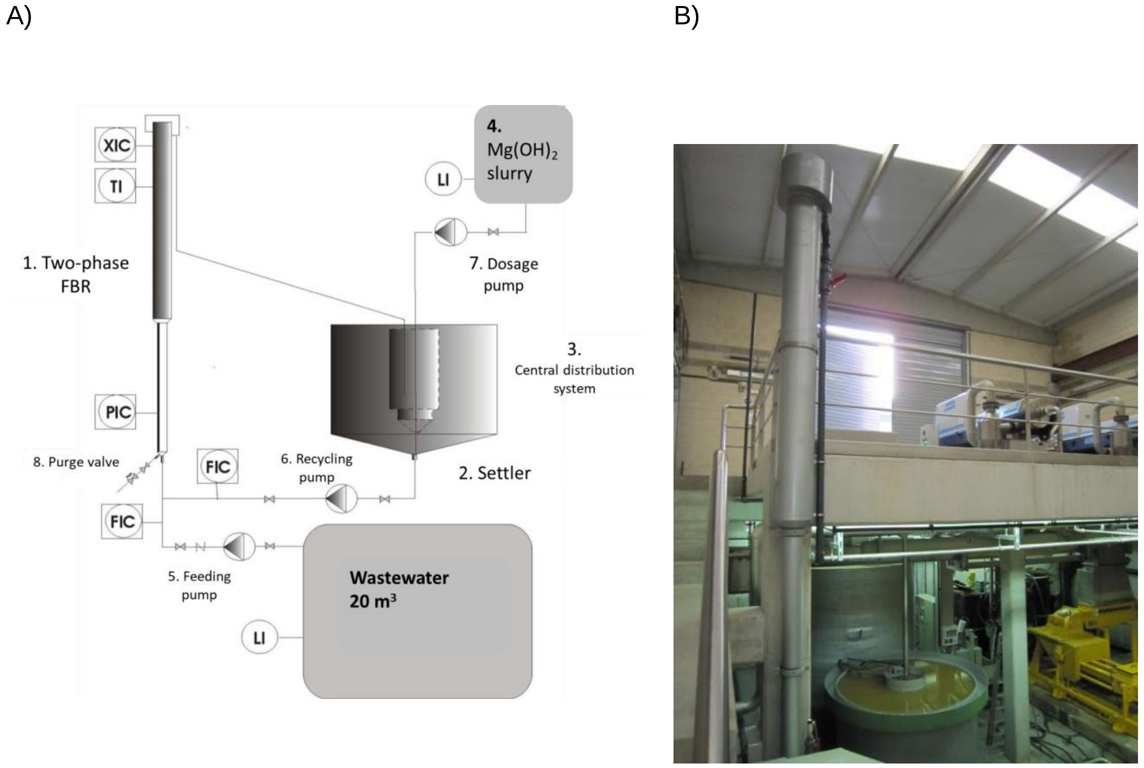

Figure 2. A) Diagram of the struvite crystallization plant: (1) two-phase FBR; (2) settler; (3) central distribution system; (4) industrial $Mg(OH)_2$ slurry unit. B) Photography of the full scale precipitation system installed in the Guillarei WWTP.

The ELAN® at full scale installed in the Guillarei WWTP consists in the following elements (Figure 3): A 90 m^3 underground homogenization tank, a 115 m^3 sequencing batch reactor (SBR) including 72 fine bubble air diffusers, an air blower, and an effluent decanter. The DO concentration, pH and conductivity were monitored with online probes. The operational cycle of the reactors was divided in feeding, reaction, settling and withdrawal stages. The operational strategy was based on the control of the hydraulic retention time (HRT) and the dissolved oxygen (DO) concentration in the bulk liquid in order to avoid ammonium and nitrite limitations and/or inhibitions as described by Vázquez-Padín et al. (2010), and following the "conductivity versus time slope" as method for reactor surveillance as detailed by Vázquez-Padín et al. (2014). This conductivity drop is mainly related to the conversion of ammonium and bicarbonate ions, both present in the anaerobic digester supernatant, into nitrogen gas, CO_2 and biomass, respectively.

Composition of the wastewater fed to the ELAN® reactor during the experimental period II is summarized in Table 2:

Table 2. Average composition of the influent and effluent of the ELAN® reactor, and removal efficiency (%) during the operational period II.

Parameter	Influent	Effluent	Removal
pH	8.00	7.06	-
Conductivity (mS/cm)	5.85	1.42	76%
PO_4^{3-} (mmol/L)	2.43	2.38	2%
HCO_3^- (mmol/L)	48.9	6.5	87%
NH_4^+ (mmol/L)	35.7	5.7	85%
NO_2^- (mmol/L)	-	0.7	-
NO_3^- (mmol/L)	-	3.1	-

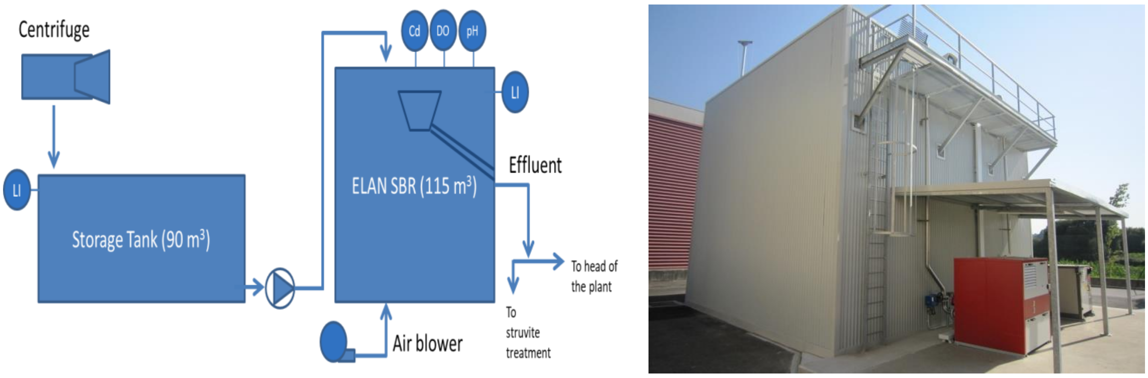

Figure 3. Scheme of the full scale ELAN® reactor. B) External view of the ELAN® full scale reactor including the air blower.

Both full scale plants were conceived to treat the nitrogen and phosphorus of the effluent of a sludge digester operated with co-digestion in the municipal WWTP of Guillarei (Galicia, NW Spain). The WWTP was designed for an influent flow of 32,500 m^3/d.

Results and discussion

MgO hydration

Conversion of MgO (a powdered product) into $Mg(OH)_2$ (a slurry of powdered product in water) was around 60% during the hydration process, following the methodology proposed by Castro et al., (2015), and the characteristics of $Mg(OH)_2$ slurry produced remained stable throughout the operation (Crutchik et al., 2016).

ELAN® operation

The ELAN® reactor at full scale was operated in the Guillarei WWTP since beginning of 2016, when it was inoculated with biomass from an ELAN® pilot scale reactor (1.2 m^3). Overall nitrogen removal efficiencies higher than 85%, with nitrogen removal rates higher than 0.4 kg $N/(m^3 \cdot d)$ were reached during the start-up of the reactor in approximately six months. Specific Anammox activity of the biomass formed in the reactor exceed 0.6 g $N/(g\ VSS \cdot d)$, verifying the success in the enrichment of the ELAN® biomass. From data of Table 2, it can be observed that the bicarbonate ion concentration was reduced simultaneously to the nitrogen removal (Figure 4D). Consequently, the alkalinity of the effluent fed to the struvite precipitation system during period II was significantly lower than during period I. Regarding the phosphorus, the concentration of this element was slightly reduced during the ELAN® process (Figure 4B).

Struvite precipitation plant

The phosphorus concentration in the effluent depends on the operating pH in the settler. Between 8.0 and 8.8 the percentage of P recovery increased with increasing pH, and did not depend on the phosphate concentration of the influent. The adequate operating pH was determined to be between 8.3 and 8.4 during Period I. The use of higher pH was not suitable, as higher set points implied the rise of the $Mg(OH)_2$ needed.

On the contrary, during Period II, as the alkalinity of the influent wastewater was reduced by the application of the ELAN® process, the operating pH were increased, with a moderate increase of the need of $Mg(OH)_2$ addition, and a range of 8.65-8.75 was used as set point for the addition of hydrated magnesium.

Despite the pH of the wastewater fed to the precipitation plant during period II (centrate treated in the ELAN® reactor) was significantly lower than the pH of the wastewater fed during period I (centrated) (Figure 4A), the pH setpoint of the precipitation plant was easily reached with the addition of $Mg(OH)_2$ during period II.

The consumption of $Mg(OH)_2$ slurry was affected by alkalinity level. Ammonium and bicarbonate ions were the species that contributed to wastewater alkalinity, and they were involved in other equilibrium reactions that release protons, when pH is raised, and consequently, it was necessary to add more $Mg(OH)_2$ slurry to achieve the optimal pH range for struvite crystallization in period I (Crutchik et al., 2016). This addition was moderated in period II, due to the reduction of alkalinity by the ELAN® process.

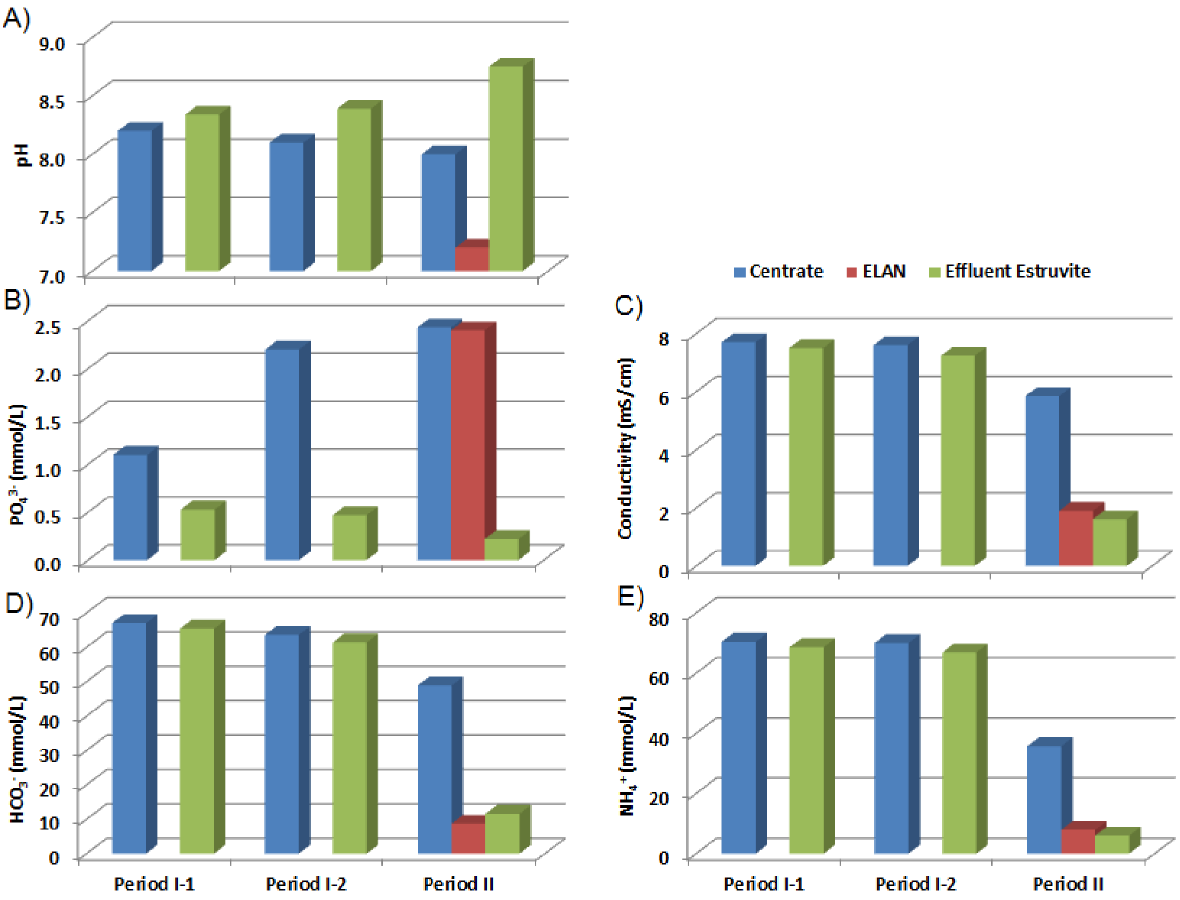

Figure 4. Values of the pH and conductivity (A and C), and concentration of phosphate (B), bicarbonate (D) and ammonia (E), in the effluent of the centrifugation process (the centrate), the ELAN® process and the struvite precipitation process.

Crutchik and Garrido (2016) developed a reversible struvite crystallization model which indicated that equilibrium conversion can be obtained in a few minutes. The phosphate concentration and struvite crystallisation follows a first-order kinetic model regarding the relative supersaturation. Consequently, the observed phosphate concentration of the effluent was independent of the phosphate fed, and it only depends on the operating conditions.

Magnesium concentration in the settler effluent was 1.2± 0.6 mmol Mg^{2+}/L and 1.5± 0.8 mmol Mg^{2+}/L during periods I-1 and I-b, respectively. During period II this value was around 1.9 mmol Mg^{2+}/L. Magnesium in the effluent was mainly associated with the magnesium from the industrial $Mg(OH)_2$ added, which did not react in the crystallization system.

Precipitates characteristics

Characteristics of obtained precipitates are influenced by the reactor hydrodynamics and the phosphate loading rate, while the efficiency of the crystallization process is mainly affected by the wastewater composition and operating conditions (Crutchik et al., 2016).

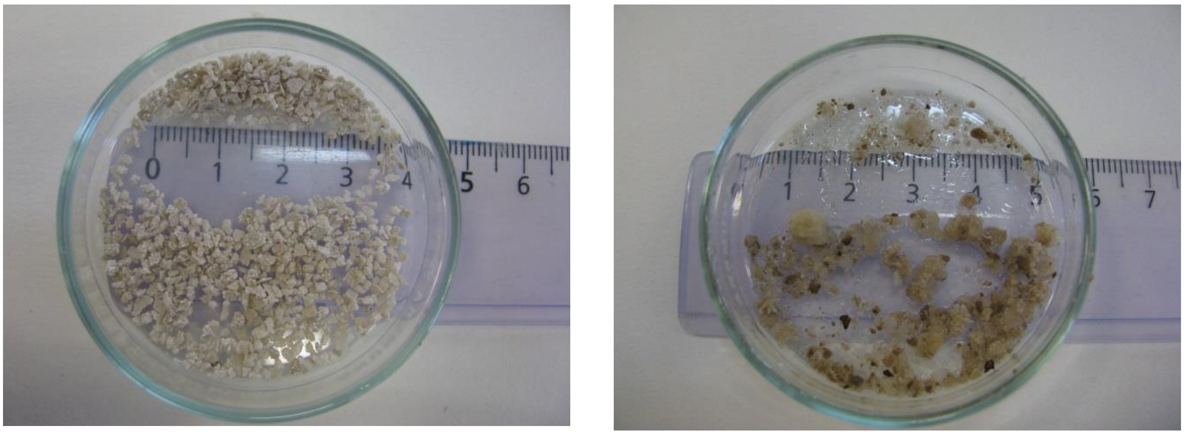

Figure 5. Pictures of the struvite recovered in the precipitation plant. A) Dry sample of the pellets recovered during period I. B) Wet sample of the solids recovered during start-up of the plant in period II.

Fine precipitates grew during the operation of the crystallization plant in all periods, and subsequently these precipitates agglomerated, especially in the FBR, to form pellets. The pellets grew by assimilating remaining crystals of lower size. While in period I-1 most of the precipitates had a size less than 0.5 mm and solid phase was mainly a powder, in period I-2 and period II the presence of larger particles increased, which can be associated with the increase of the influent phosphate concentration. A dried sample of the struvite recovered during period I is shown in Figure 5A. Figure 5B display the presence of pellets of struvite combined with small crystals of struvite observed in the two phase FBR during first days of operation in period II.

Preliminary cost analisys

Taking into account the costs associated with the preparation of the $Mg(OH)_2$ slurry (€ 350 per ton) (Crutchik et al., 2016), the reductions in the need of dosage of the slurry between periods I-a and I-2 resulted in a decrease of the operational costs of the process. The beneficial effects of the introduction of the ELAN$^{\circledR}$ process in the precipitation process in period II, due to the reduction in the base addition, further reduced the operational costs of the process, as the dose of magnesium added to the precipitation system was closer to the stoichiometric values. The estimated costs were around € 4,700·t/$P_{precipitated}$ in Period I-1 (Crutchik et al., 2016) while this value was lower than € 980·t/$P_{precipitated}$ in period II, when ELAN$^{\circledR}$ was included. These values are much lower than the values obtained in other researches, when $MgCl_2$ and NaOH were used for struvite crystallization, around €4,000·t/P precipitated (Carballa et al., 2009), or when the conventional processes are used for the removal (and not recovery) of the phosphorus in WWTPs, around €2,000 and €3,000·t/$P_{removed}$ (Dockhorn, 2009).

In future research, the operation of the struvite precipitation plant will be optimized for phosphorus recovery, save of industrial magnesium reactive and for the improvement of the distribution size of the struvite pellets, in order to recover most of the phosphorus as large struvite pellets, reducing the percentage of fine crystals produced in the precipitation plant.

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