Phosphate removal in agro-industry: pilot and full-scale operational considerations of struvite crystallisation

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Abstract Pilot-scale struvite crystallisation tests using anaerobic effluent from potato processing industries were performed at three different plants. Two plants (P1 & P2) showed high phosphate removal efficiencies, $89 \pm 3\%$ and $75 \pm 8\%$, resulting in final effluent levels of 12 ± 3 mg PO_4^{3-}-P/L and 11 ± 3 mg PO_4^{3-}-P/L, respectively. In contrast, poor phosphate removal ($19 \pm 8\%$) was obtained at the third location (P3). A noticeable difference in the influent Ca^{2+}/PO_4^{3-}-P molar ratio was observed between the test sites, ranging from 0.27 ± 0.08 (P1), 0.62 ± 0.18 (P2) and 0.41 ± 0.04 (P3). A negative effect on struvite formation occurred when a Ca^{2+}/PO_4^{3-}-P molar ratio of 1.25 ± 0.11 was obtained after initial pH increase in the stripper at P3. A full-scale struvite plant treating 90–110 m^3/h of anaerobic effluent from a diary industry also showed Ca^{2+} interference. Initially in this plant, influent phosphate levels ranging from 40 to 45 mg PO_4^{3-}-P/L were decreased to below 10 mg PO_4^{3-}-P/L, but no struvite was produced. A shift in Ca^{2+}/PO_4^{3-}-P molar ratio from 2.69 to 1.36 by an increased phosphate concentration resulted in average total phosphorus removal of $78 \pm 7\%$, corresponding with effluent levels of 14 ± 4 mg P_{total}/L(9 ± 3 mg PO_4^{3-}-P/L). Under these conditions pure spherical struvite pellets of 2–6 mm were produced.

INTRODUCTION

Although the advantages of anaerobic treatment are obvious, subsequent nutrient removal still remains an important issue. Readily biodegradable organic matter needs to be bypassed towards aerobic post-treatment in order to achieve the final nutrient effluent standards, thus reducing the potential biogas yield and increasing the waste sludge production. Nutrient removal by struvite ($MgNH_4PO_4 \cdot 6H_2O$) or magnesium ammonium phosphate (MAP) precipitation is an interesting alternative approach to address phosphorus removal (von Munch and Barr, 2001; Gonzalez and De Sa, 2007). MAP crystallisation can be applied for several purposes, such as to prevent scaling problems

(Doyle and Parsons, 2004) and to remove phosphate (Battistoni *et al.*, 1997) or nitrogen (Altinbas *et al.*, 2002; Laridi *et al.*, 2005). Recent publications show an increasing interest in struvite precipitation as a technology for phosphorus recovery taking into account the economic impact of increasing energy costs and limited natural phosphorus resources (Durrant *et al.*, 1999; Shu *et al.*, 2006; Carballa *et al.*, 2008; Forrest *et al.*, 2008). Consequently, integration of struvite formation as specific treatment of side-stream wastewaters is becoming a common practise (Caffaz *et al.*, 2008).

Increasing the operational pH and adjustment of the molar ratios of magnesium, ammonium and phoshate are the most important process parameters in MAP crystallisation (Ohlinger *et al*., 1998). In addition, presence of calcium has been shown to be determinative for both crystal size and purity (Le Corre *et al.*, 2005; Pastor *et al.*, 2007). When anaerobically processed wastewaters are treated, pH can be increased by simple air stripping (Williams, 1999). If air stripping does not suffice, additional alkaline reagents must be added. Alkaline addition can be combined with the required magnesium supplementation by using either MgO or $Mg(OH)_2$ (von Münch and Barr, 2001). Other magnesium sources include magnesium chloride, magnesium sulphate or seawater bittern waste (Li and Zhao, 2002). If ammonium removal is targeted, both magnesium and phosphate have to be supplied. Yet, to minimize reagent use, internal recycling of the magnesium and phosphate is possible by thermal decomposition of the recovered MAP (Stefanowicz *et al.*, 1992; He *et al.*, 2007).

Different pilot and full-scale units using fluidized bed (Ueno and Fujii, 2001; Forrest *et al.*, 2008) or continuously stirred tank reactors (Mangin and Klein, 2004; Laridi *et al.*, 2005) have been operated. Most full-scale struvite plants do treat anaerobic liquor originating from primary and secondary sludge digestion (Ueno and Fujii, 2001; Battistoni *et al.*, 2005; Forrest *et al.*, 2008). This paper describes the successful use of a fairly straightforward stirred tank crystallizer for full-scale phosphate recovery by struvite crystallization treating anaerobic effluent from a dairy processing industry at an average flow rate of 100 m^3/h. It also points out the need of preliminary feasibility tests before full-scale application to exclude excessive calcium interference, which renders this technology inefficient.

MATERIAL AND METHODS

Pilot plant description

Figure 1 shows a scheme of the pilot plant used. The anaerobic effluent was continuously fed into the stripper through peristaltic pump 1. The total volume of the stripper was 180 L and air was supplied as coarse bubble aeration at 8 L/s.

The 200 L-crystallizer was equipped with a 3 blade top entry impeller (impeller diameter/tank diameter $= 0.5$) and $120°$ interval baffles. The crystals were retained by a transient quiescent settling zone. Online control was used to adjust pH with 29% NaOH (pump 2). $MgCl_2$ was used as magnesium source and Mg^{2+} dosage was controlled by setting the flowrate (pump 3) of $MgCl_2$ solution (30%, v/v) according to the influent flowrate. Under steady state conditions, pH was controlled between 8.50 and 8.70 and an Mg^{2+}/PO_4^{3-}-P molar ratio of 1 to 1.2 was maintained. Struvite crystals were removed by intermittent purging.
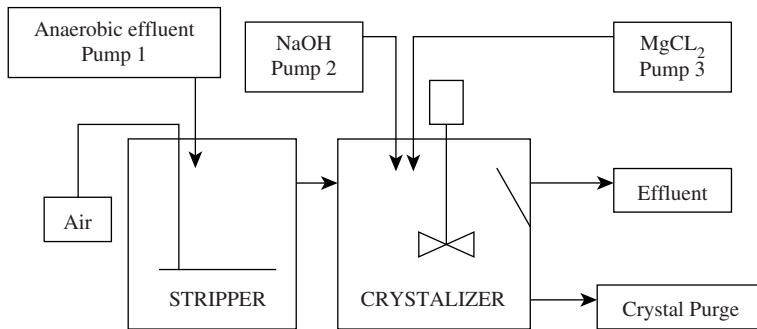

Figure 1. Pilot scale unit used in the struvite crystallisation tests.

Influent and effluent samples were taken 2–3 times a week for ammonium, phosphate and magnesium determinations. Recovered MAP was dried at ambient room temperature to avoid ammonia loss and then used for X-ray diffraction (XRD) analysis.

Anaerobic effluent characteristics

Pilot-scale tests were performed over a 2–3 month period at 3 different potato processing plants. On average, 5 m^3/day of anaerobic effluent coming from the full-scale upflow anaerobic sludge bed (UASB) reactor was treated in the struvite crystallisation. Table 1 shows the initial characteristics of the 3 different anaerobic effluents used.

Analytical methods

pH was measured with a pH meter (Consort C532). Ammonium was determined colorimetrically with Nessler reagent according to standard methods (APHA, 1992). Phosphate and magnesium were determined using a Metrohm 761 compact ion chromatograph equipped with a conductivity detector and flame atomic absorption spectrometry, respectively. Total phosphorus, dry matter

content and its mineral fraction were determined according to standard methods (APHA, 1992). XRD analyses were done by using a Siemens D5000 unit equipped with Cu K_α radiation at 1.54 Å.

Table 1. Characteristics of the UASB effluent (influent of struvite pilot plant) of the 3 test sites.

	Plant 1	Plant 2	Plant 3
pH range	7.00–7.45	7.10–7.50	7.00–7.85
PO_4^{3-}-P (mg/L)	115 ± 13	43 ± 7	127 ± 8
NH_4^+-N (mg/L)	426 ± 45	208 ± 27	254 ± 36
Ca^{2+} (mg/L)	40 ± 8	36 ± 7	65 ± 7
Ca^{2+}/PO_4^{3-}-P molar ratio	0.27 ± 0.08[a]	0.62 ± 0.18[a]	0.41 ± 0.04[a]
			1.25 ± 0.11[b]
Average N/PO_4^{3-}-P molar ratio	8.22	10.76	4.54

[a]Influent of struvite plant (stripper inlet)
[b]Influent of crystallisation reactor (stripper outlet)

RESULTS

Pilot-scale tests

The anaerobic effluents from plants 1 and 3 were characterized by high phosphate levels (Table 1), with average PO_4^{3-}-P concentrations above 110 mg PO_4^{3-}-P/L. In contrast, plant 2 showed significantly lower phosphate levels, between 38 to 52 mg PO_4^{3-}-P/L. During the testing period, the operation of the full-scale UASB reactor was stable, resulting in effluent pH values of 7.00–7.85 and residual volatile fatty acid concentrations between 0.5 and 2.5 meq acetic acid/L. Therefore, besides normal full-scale fluctuations, the composition of the UASB effluent remained constant during the testing period (Table 1).

The results obtained in the struvite pilot plant are summarized in Table 2.

Table 2. Effluent phosphate levels and phosphate removal efficiencies at the 3 test sites.

	Plant 1	Plant 2	Plant 3
PO_4^{3-}-P (mg/L)	12 ± 3	11 ± 3	103 ± 11
Average PO_4^{3-}-P removal (%)	89 ± 3	75 ± 8	19 ± 8
Maximum PO_4^{3-}-P removal (%)	95	88	31
Minimum PO_4^{3-}-P removal (%)	79	49	5

High phosphate removal efficiencies were obtained in P1 and P2, with average values of 90% and 75%, respectively. Moreover, despite the initial phosphate levels differed by a factor of 2.67, similar concentrations were obtained in the effluent, around 10 mg PO_4^{3-}-P/L. However, while the elimination remained constant in P1, P2 showed occasionally a limited efficiency not exceeding 50%. Spherical self-retaining crystals of pure struvite, as confirmed by XRD analysis and with 98% of dry matter (after ambient temperature drying) were obtained in both plants. Heavy metal analysis only showed copper (21 mg Cu/kg dry matter) to be present in the struvite pellets of P1.

In contrast, low phosphate removal was noted in P3 (around 20%). In addition, no struvite formation was observed at any conditions tested. The reason was probably the formation of amorphous calcium and/or magnesium phosphate flocculent matter. Detailed analysis of Ca levels in the influent and effluent of P3 showed Ca elimination of 9–26%. A simple pH increase of the UASB effluent up to 8.30–8.50 was sufficient to initiate the amorphous sludge formation without $MgCl_2$ addition. Addition of $MgCl_2$ in the air stripping tank prior to pH adjustment in the crystallisation tank resulted in the formation of maiden orthorhombic pyramidal struvite crystals, which accumulated in the crystallisation tank. These smaller crystals, known as fines, could apparently not grow further, and thus not giving rise to spherical self-retaining crystals, such as those obtained in P1 and P2. Another difference was in the NaOH dose required to obtain the pH set-point. P3 needed a significantly lower amount (0.08–0.20 L/m^3) compared to the NaOH consumption in P1 (0.80–1.20 L/m^3) and P2 (0.50–1.00 L/m^3).

The clear difference in the potential to produce struvite at the different tested plants can be probably explained by the varying Ca^{2+}/PO_4^{3-}-P ratios (Table 1). However, it should be noted that not only the Ca^{2+}/PO_4^{3-}-P molar ratio affects struvite formation, but also the absolute Ca^{2+} concentration. The Ca^{2+}/PO_4^{3-}-P molar ratio differed significantly between P1 and P2, while high phosphate removal efficiencies were obtained in both plants as well as similar struvite crystals. The reason is probably that no distinction in Ca^{2+} concentrations in the UASB effluent was noted, 40 $\pm$ 8 mg Ca^{2+}/L in P1 and 36 $\pm$ 7 mg Ca^{2+}/ L in P2. P3 had clearly higher Ca^{2+} influent levels of 65 $\pm$ 7 mg Ca^{2+}/L, which combined with the lower phosphate levels after the stripper (around 40 mg PO_4^{3-}-P/L), resulted in a Ca^{2+}/PO_4^{3-}-P molar ratio at the inlet of the crystallisation reactor of 1.25 (Table 1).

The NH_4^+-N concentrations were not limiting at any of the examined sites. The NH_4^+-N/PO_4^{3-}-P molar ratios in P1, P2 and P3 were 8.22, 10.76 and 4.54, respectively (Table 1). In terms of NH_4^+-N concentrations, no difference was

observed between P2 $(208 \pm 27$ mg NH_4^+-N/L) and P3 $(254 \pm 36$ mg NH_4^+-N/L), while higher levels were present in P1 $(426 \pm 45$ mg NH_4^+-N/L).

More detailed data at P1 revealed that most soluble phosphate removal occurred in the crystallizer (80%) and only a 20% decrease was noted in the stripper (Figure 2).
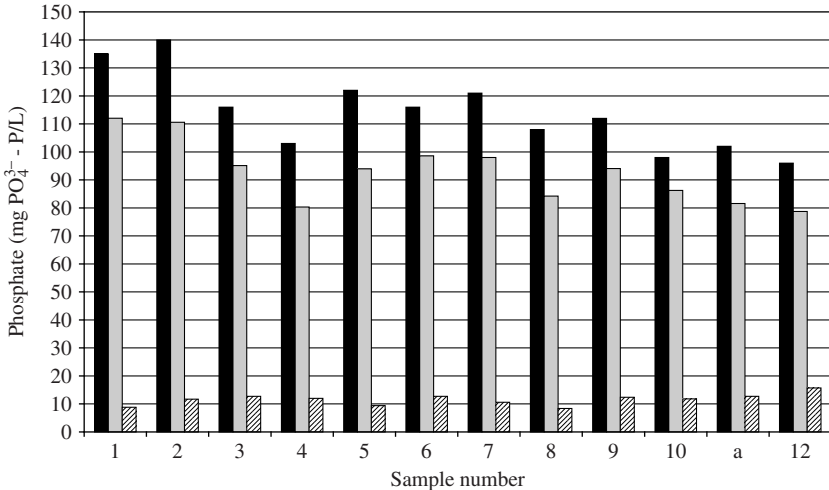

Figure 2. Orthophosphate levels in 12 samples taken during the pilot run in P1 Influent (black), air stripper (grey) and final effluent (striped).

The results shown in Figure 2 and Tables 1 and 2 refer to orthophosphate. After anaerobic treatment, around 90–95% of the total phosphorus was present as orthophosphate. Total phosphorus levels were analyzed once a week, approximately. In general, the total phosphorus effluent concentrations were 17 to 28% higher than the orthophosphate levels. Microscopic control of final effluent showed typical maiden orthorhombic fines in the effluent of P1 and P2.

Full scale unit

Based on the pilot tests, two full-scale units were designed and a process approach was developed, which is marketed as NuReSys® (Table 3). The main common features of both units are: air stripper, crystallisation reactor equipped with a top-entry mixer and a transient quiescent settling zone, pH control using NaOH and magnesium chloride as magnesium source.

The full-scale plant 1 contains a 200 m³ air-stripper and 125 m³ crystallisation reactor. The produced struvite crystals are retained in the tubular lamellae

Table 3. Main characteristics of the full-scale NuReSys plants.

	Average capacity	**Operational characteristics**	**Status**
1	100 m^3/h (192 kg P$_{total}$/d)	Dairy processing industry: HRT[*]: 3.25 h Footprint: 180 m^2	Operational
2	60 m^3/h (180 kg P$_{total}$/d)	Potato processing industry HRT[*]: 2.23 h Footprint: 92 m^2	Start-up in October 2008

[*]HRT: Hydraulic Retention Time.

settling zone located in the upper part of the crystallisation reactor and further purged towards a dewatering container by intermediate manual offset. These produced struvite crystals have been accredited for reuse in agriculture.

During the start-up of the plant, although a 70 to 75% decrease in phosphate was obtained, no struvite was produced due to similar Ca interference as observed during the pilot-scale tests at plant 3. The calcium and phosphate levels in the UASB effluent of the dairy plant averaged 140 mg Ca^{2+}/L and 40–45 mg PO$_4^{3-}$-P/L, resulting in an average Ca^{2+}/PO$_4^{3-}$-P molar ratio of 2.69. Moreover, no significant decrease of ammonia was observed during this period and a flocculent material was produced, as observed in the pilot-scale tests at P3. The produced amorphous matter contained mainly calcium, magnesium and phosphorus at a respective molar ratio of 2.50/0.75/1.80. Accordingly, Ca^{2+} concentrations decreased by 40–50%. The flocculent matter was also produced in the stripper unit suggesting its production to be driven by pH increase. Changing the position of the inlet ports of alkaline reagent NaOH and MgCl$_2$ to prevent the pH-driven formation of the amorphous matter was not successful.

A batch experiment was performed with 2 L of full-scale UASB effluent spiked with concentrated PO$_4^{3-}$-P solution to obtain a phosphate level of about 150 mg PO$_4^{3-}$-P/L. After phosphate spiking, pH was adjusted and Mg^{2+} was added. Phosphate was removed from 145 to 14.7 mg PO$_4^{3-}$-P/L. In addition, microscopic examination of the settled material clearly showed the formation of struvite crystals. Therefore, under these conditions corresponding to a Ca^{2+}/PO$_4^{3-}$-P molar ratio of 0.77, struvite formation was possible, but amorphous calcium/magnesium flocculent matter was still the main mechanism responsible for the phosphate decrease.

A gradual shift in UASB effluent due to an increase in phosphate concentrations up to an average of 69 mg P$_{total}$/L resulted in a decrease of the Ca^{2+}/PO$_4^{3-}$-P molar ratio from 2.69 to 1.36. The full-scale plant was operated under these conditions over a 23-week period and the results obtained are shown in Table 4.

Table 4. Average, minimum and maximum phosphorus levels in the influent and effluent of the full-scale unit (n = 169).

	Influent	Effluent		
	P_{total} (mg/L)	P_{total} (mg/L)	PO_4^{3-}-P (mg/L)	P_{total} removal (%)
Average	64	14	9	78
Stand. Dev.	13	4	3	7
Maximum	108	33	26	91
Minimum	38	7	4	38

After the increase of phosphate influent levels, the formation of the flocculent amorphous matter has been largely suppressed and replaced by the growth of 2–6 mm in diameter spherical crystals struvite crystals (confirmed by XRD). The average ammonium influent levels during this period were 110 ± 18 mg NH_4^+-N/L, resulting in a molar NH_4^+-N/PO_4^{3-}-P ratio of 3.45. Contrary to the start-up period, the phosphate removal was accompanied by an ammonium decrease of around 22 mg NH_4^+-N/L, which would account for an average 46 mg PO_4^{3-}-P/L immobilized as struvite. Taking into account the overall phosphate removal over the examined period, ca. 55 mg PO_4^{3-}-P/L, the amorphous matter was responsible for only 16% of the total phosphate removal.

DISCUSSION

Results obtained at pilot-scale in plant 3 and during start-up of the full-scale plant 1 showed a negative interference of calcium. Pastor *et al*. (2007) observed a similar effect treating 2 anaerobic digestion liquors with Ca^{2+}/PO_4^{3-}-P molar ratios of 0.37 and 2.34, respectively. The high Ca^{2+}/PO_4^{3-}-P molar ratio liquor only removed 35% of the phosphorus as struvite, whilst the low Ca^{2+}/PO_4^{3-}-P molar ratio liquor achieved 73%. Overall phosphorus removal efficiencies were as high as 83% (ratio Ca^{2+}/PO_4^{3-}-P = 2.34) and 91% (ratio Ca^{2+}/PO_4^{3-}-P = 0.37), indicating that phosphorus removal was partially attributed to amorphous calcium phosphate. Similarly, Le Corre *et al*. (2005) reported that Ca^{2+}/Mg^{2+} molar ratios between 0.5 and 1.0 affected significantly struvite formation, and values exceeding 1 excluded nearly completely the formation of crystalline struvite. These findings are in accordance with the results obtained in this study, where Ca^{2+}/Mg^{2+} molar ratios are nearly equal to the Ca^{2+}/PO_4^{3-}-P ratios since the Mg^{2+}/PO_4^{3-}-P molar ratio used was 1–1.2. Full-scale results obtained at the average Ca^{2+}/PO_4^{3-}-P molar ratio of 1.36 are not in accordance with the

limited struvite formation observed during the batch phosphate spiking test at Ca^{2+}/PO_4^{3-}-P of 0.77. However, in order to compare these results rigorously, the differences in concentration profiles and mixing, and the presence of secondary nucleation surfaces need to be considered.

The use of top-entry mixers has been shown to be an alternative to fluidized bed systems for struvite formation (Pastor *et al.*, 2008; Kim *et al.*, 2008). The results obtained in this work confirm that this operational approach can be successfully applied, even at considerable high flow rates of 100–125 m³/h. Furthermore, adjustable mixing rotary speed and flexible selection of reagent injection points are major advantages of this system (Mangin and Klein, 2004).

From mass balance calculations, it becomes obvious that not all of the phosphate is removed as struvite. Moreover, not the entire quantity of immobilized orthophosphate, either as struvite or as amorphous phosphate, is retained within the crystallisation reactor. The washed-out immobilized phosphates will be determinative in achieving the final effluent standards in terms of total phosphorus. Sperandio *et al.* (2008) have shown that the pH of activated sludge systems, influenced by nitrification/denitrification and aeration-driven stripping effect, is a major factor affecting the formation and conservation of crystalline phosphate compounds, and thus contributing to the overall phosphate removal efficiency.

The produced full-scale crystals were shown to be mainly composed of pure struvite. This fact confirms earlier reports related to the production of a high quality end product suitable for agricultural reuse (Miles and Ellis, 2001; Shu *et al.*, 2006; Forrest *et al.*, 2008).

CONCLUSIONS

1.   Phosphate recovery by struvite formation is a high value added technique, mainly after anaerobic treatment as typically applied in the agro-industrial sector.
2.   The interference of calcium is an important factor to be addressed, which could exclude the use of this technology.
3.   Since the final Ca^{2+}/Mg^{2+} molar ratios are a direct consequence of the initial Ca^{2+}/PO_4^{3-}-P molar ratios, the latter should be used as a determinative parameter instead of the Ca^{2+}/Mg^{2+} molar ratios. The Ca^{2+}/PO_4^{3-}-P molar ratio should be low, preferably below 1.0.
4.   Formation of amorphous calcium and magnesium phosphates may contribute to overall phosphate removal, but as a non-recoverable product.

5. Feasibility of phosphate removal via struvite at high flowrates of 100–125 m^3/h has been demonstrated without affecting the high efficiency.
6. The high-quality of the obtained struvite crystals indicates that minimal processing is required prior to reuse.

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