

# Study of the recovery of phosphorus from struvite precipitation in supernatant line from anaerobic digesters of sludge

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## ABSTRACT

The goal of this work was to study the effective recovery of phosphorus from the supernatant of anaerobic digestion of sewage sludge by precipitation as struvite. The formation of struvite is envisioned as a promising process for nutrient removal and subsequent recovery, thus providing a strong incentive for its implementation, since the sewage is a renewable source of phosphorus. Struvite precipitation was obtained by controlled addition of  $Mg(OH)_2$  or  $MgCl_2$ . We evaluated the removal of ammonia and phosphate under equimolar conditions of magnesium and magnesium stoichiometric excess of 100 to 200% relative to the limiting reagent, under a stirring speed of 300 rpm at pH 8, 9 and 10. The best condition was  $MgCl_2$  in 1:1 molar ratio to phosphate, considering the stoichiometric ratio  $[PO_4^{3-}]:[NH_4^+]$  of 0.13 (presented by raw sample). The results show the best cost-benefit ratio, removal of phosphate of 90.6% and ammonium removal of 29%, resulting in  $23\text{ mg l}^{-1} PO_4^{3-}$  and  $265\text{ mg l}^{-1} NH_4^+$  concentration in effluent.

**Key words** | anaerobic digestion, phosphorus, recuperation, struvite

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## INTRODUCTION

The environmental protection agencies impose ever stricter standards related to the removal of nutrients, so contributing towards research and development of more efficient treatment processes to remove phosphorus and nitrogen. A consequence of the reduction of phosphorus concentration in the liquid phase during sewage treatment is the increase of phosphorus, nitrogen and magnesium concentrations in the sludge generated in wastewater treatment plants (WWTPs) (Van Haandel & Marais 1999; Doyle & Parsons 2002).

Anaerobic digestion is an alternative to the degradation of most organic compounds present in sludge, but nutrient enrichment in the liquid phase of the process is a difficult problem to solve (Obaja *et al.* 2003). In the process of anaerobic digestion, transference of phosphorus from the

solid phase (sludge) into the liquid phase (supernatant) occurs (Jordão & Pessoa 2011).

When the supernatant returns to the liquid phase of the WWTP, the phosphorus generated after digestion usually reaches values of up to 20% of the total crude influent charge, tending to impact on the biological treatment process. It is also known that the supersaturation of  $Mg^{2+}$ ,  $NH_4^+$ , and  $PO_4^{3-}$  ions under turbulent stirring and increase in pH can cause the natural formation and deposition of struvite, magnesium ammonium phosphate hexahydrate ( $MgNH_4PO_4 \cdot 6H_2O$ ). By the control of pH, temperature and the stoichiometric balance between the elements forming the struvite, effective recovery of phosphorus can be promoted by controlled application of  $Mg(OH)_2$  or  $MgCl_2$  in the anaerobic digestion supernatant

(Doyle & Parsons 2002; Uludag-Demirer & Othman 2009; Pastor *et al.* 2010).

The struvite is in the white crystalline orthorhombic form, composed of magnesium, ammonium and phosphate in a 1:1:1 molar ratio and may be recovered through solid–liquid separation due to its low solubility (Lee *et al.* 2003; Di Iaconi *et al.* 2010). The struvite is readily soluble in an acid medium, so it is generated in a neutral or alkaline medium. The precipitated struvite can be evaluated by analysis using X-ray diffraction (XRD) (Doyle & Parsons 2002).

Over 90% of dissolved phosphorus can be recovered from the supernatants of anaerobic digesters through the crystallization of struvite, achieving a recovery of 50 to 80% of the total phosphorus (Ueno & Fujii 2001; Von Münch & Barr 2001; Yoshino *et al.* 2003; Wu & Bishop 2004; Battistoni *et al.* 2005; Suzuki *et al.* 2005; Suzuki *et al.* 2007; Westerman *et al.* 2009; Jordaan *et al.* 2010).

Struvite can be used as a fertilizer in high application rates as it has slow release due to its poor solubility (Ohlinger *et al.* 1998), which is desirable to grassland or forest environments where high solubility fertilizers are not suitable (De-Bashan & Bashan 2004).

The generation of struvite also reduces operating costs of a WWTP, reducing the return of the supernatant back upstream to the beginning of the treatment. Thus, less sludge would be treated and disposed of in landfills. Consequently, maintenance costs associated with the incrustation of struvite in the WWTP system would be reduced.

This study aims to evaluate the removal of phosphorus from sewage by precipitation of struvite through the addition of magnesium hydroxide or magnesium chloride in the supernatant of anaerobic digestion of secondary sludge, treated by activated sludge.

## METHODS

The samples of supernatant from an anaerobic digester were preserved with acid and frozen to prevent the digestion process until used in the tests. The samples originated from an experimental WWTP of the Federal University of Rio de Janeiro, Brazil (UFRJ). The removal of ammonia and phosphate was evaluated, under equimolar conditions of magnesium, magnesium stoichiometric excess of 100 to 200% relative to the limiting reagent, and stirring speed of 300 rpm at pH 8, 9 and 10. Assays were performed in duplicate jar-tests during 1 h of reaction, using 300 ml of supernatant sample at ambient temperature of 25 °C. The pH was adjusted with NaOH 20% (w/v) solution. The reaction time of 1 h was assumed because

preliminary experiments indicated it to be sufficient to evaluate the reaction of struvite precipitation.

The addition of sodium hydroxide for raising the pH results in a slight increase in saturation of other magnesium precipitates that compete with the formation of struvite (bobierrite and magnesite). This results in extra consumption of magnesium, requiring the amount of magnesium to be larger than the stoichiometric value for struvite formation (Zeng & Li 2006).

The sample was packaged in a 600 ml beaker, under stirring at 50 rpm, where the reagents were dosed. When it reached the desired pH, stirring was increased to 300 rpm at the beginning of the experiments. After 1 h of reaction, the stirring was turned off and the sample was left to stand for about 1 h; an aliquot of the supernatant was filtered and preserved for subsequent analyzing, while the remaining volume in the beaker was taken to the greenhouse for approximately 1 week at 40 °C until evaporation of the liquid phase. Thereafter, the precipitate was scraped from the beaker, and its mass was measured and preserved for further analysis by XRD.

After addition of magnesium, the supernatant-filtered samples allowed the evaluation of the eventual composition of the precipitate and the supernatant. The phosphorus concentration was measured by the vanadomolybdophosphoric acid colorimetric method and ammonia concentration by the ammonia-selective electrode method, according to APHA (2011).

Following the previous methodology, experiments were performed to assess the formation of struvite in the equimolar ratio ammonium:phosphate (1:1), and the molar ratio of phosphate to ammonium (2:1) by adding monohydrogenated sodium phosphate. All the reagents used in this study were grade P. A. (analytical purity). The crystallization process of struvite clearly affects the final results (reactor configuration, position of the influent streams, initial seed). However, these aspects have not been evaluated in this study.

## Experimental design

The experiments were carried out using a factorial experimental design of order 3<sup>3</sup> (levels -1, 0, +1). This factorial design was chosen because it allows evaluation of the effect of three independent variables (pH, addition of magnesium and phosphorus supplementation) on two response variables (phosphate and ammonium). The independent variables were selected based on previous research (Doyle & Parsons 2002; Laridi *et al.* 2005; Schilling 2008). The

results were evaluated by the analysis of variance using Statistica 7 (StatSoft) program.

The pH was evaluated at 8, 9 and 10. The molar ratios of  $[\text{PO}_4^{3-}]:[\text{NH}_4^+]$  of 0.13, 1 and 2 were evaluated. These tests aimed to evaluate if the addition of phosphorus could lead to an increase in the kinetics of the reaction, providing better removal of phosphorus than previously obtained. The level (-1) of variable  $[\text{PO}_4^{3-}]:[\text{NH}_4^+]$  as 0.13 corresponds to the ratio found in the crude sample. The variable  $[\text{Mg}^{2+}]:[\text{Limitant}]$  was evaluated in a molar stoichiometric ratio and two and three times this molar ratio. Jaffer et al. (2002) studied the potential recovery of phosphorus in the form of struvite in a sanitary effluent and they obtained 97% of phosphorus removal by increasing the dosage of magnesium in a molar stoichiometric ratio. Lower dosages also resulted in the removal of phosphorus, but struvite was not the predominant form in the precipitate. Studies on laboratory scale showed that struvite can be formed at pH 9 and with molar ratios of magnesium: phosphorus in the range of 1.05:1.0.

### X-ray diffraction

The analysis of the chemical structure of the precipitate was done using a Rigaku Miniflex II model RIX 3100 diffractometer, with radiation  $\text{CuK}_\alpha$  range of  $5^\circ \leq 2\theta \leq 90^\circ$ , step  $0.05^\circ$  and scan speed of 4 seconds per step. The crystalline phases present in the samples were identified with the aid of the JCPDS (Joint Committee on Powder Diffraction Standards) to inorganic compounds and with the aid of the computer program MDI Jade 5 (<http://www.materialsdata.com>).

## RESULTS

### Precipitation of struvite

A supernatant sample of the digester used in the tests of struvite precipitation was analyzed and the characteristics are shown in Table 1.

The high content of carbonaceous alkalinity could lead to the precipitation of magnesium carbonate instead of the formation of struvite, according to Schilling (2008). The removal of carbonaceous alkalinity was achieved by adding  $\text{H}_2\text{SO}_4$  up to pH 4 and subsequent bubbling of air. After this step, a pre-established amount of magnesium hydroxide was added for the precipitation of struvite, according to the experimental design presented above.

Yokoyama et al. (2011) concluded that reactions of the precipitation of struvite at pH above 9 are not suitable

**Table 1** | Characteristics of the digester supernatant

pH	–	7.5
Carbonaceous alkalinity	$\text{mg l}^{-1}$	1135
Total chemical oxygen demand (COD)	$\text{mg l}^{-1}$	391
Soluble COD	$\text{mg l}^{-1}$	160
5-day biochemical oxygen demand ( $\text{BOD}_5$ )	$\text{mg l}^{-1}$	86
Total carbon	$\text{mg l}^{-1}$	290
Total inorganic carbon	$\text{mg l}^{-1}$	240
Total organic carbon	$\text{mg l}^{-1}$	50
$[\text{PO}_4^{3-}]$	$\text{mg l}^{-1}$	250
$[\text{NH}_4^+]$	$\text{mg l}^{-1}$	373
Total solids	$\text{mg l}^{-1}$	1309
Total suspended solids (TSS)	$\text{mg l}^{-1}$	299
Total volatile solids	$\text{mg l}^{-1}$	381
Volatile suspended solids (VSS)	$\text{mg l}^{-1}$	139

due to the evolution of ammonia gas. In order to investigate the claims mentioned above, quantitative analyses were conducted to evaluate the ammonia gas evolution. Experiments were conducted using the same test methodology, with no addition of magnesium in the beaker. The results obtained in the experiments indicated 5% of gaseous ammonia removal, which was not considered in the experiments. Table 2 shows the results with  $\text{Mg}(\text{OH})_2$  and  $\text{MgCl}_2$ .

**Table 2** | Removal of phosphate (%) in different pH conditions and molar ratio of  $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$

pH	$\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$	8	9	10
$\text{Mg}(\text{OH})_2$	1:7.87:1	$20.9 \pm 2.0$	$86.9 \pm 1.1$	$87.0 \pm 2.1$
	2:7.87:1	$46.2 \pm 1.1$	$90.9 \pm 0.7$	$92.8 \pm 1.0$
	3:7.87:1	$77.2 \pm 1.2$	$91.0 \pm 0.5$	$93.0 \pm 0.1$
$\text{MgCl}_2$	1:7.87:1	$72.4 \pm 0.0$	$90.6 \pm 2.6$	$94.7 \pm 0.0$
	2:7.87:1	$85.1 \pm 0.8$	$92.7 \pm 0.9$	$95.0 \pm 0.0$
	3:7.87:1	$87.8 \pm 0.4$	$93.4 \pm 0.0$	$95.1 \pm 0.6$
$\text{Mg}(\text{OH})_2$	1:1:1	$97.4 \pm 0.1$	$97.4 \pm 0.1$	$98.3 \pm 0.2$
	2:1:1	$98.0 \pm 0.1$	$99.0 \pm 0.6$	$99.0 \pm 0.0$
	3:1:1	$98.2 \pm 0.1$	$99.2 \pm 0.0$	$99.3 \pm 0.0$
$\text{MgCl}_2$	1:1:1	$94.2 \pm 0.2$	$98.8 \pm 0.1$	$99.3 \pm 0.0$
	2:1:1	$95.5 \pm 0.3$	$99.0 \pm 0.0$	$99.4 \pm 0.0$
	3:1:1	$97.1 \pm 0.1$	$99.1 \pm 0.0$	$99.4 \pm 0.0$
$\text{Mg}(\text{OH})_2$	1:1:2	$85.2 \pm 0.2$	$95.8 \pm 2.1$	$76.7 \pm 0.2$
	2:1:2	$94.8 \pm 0.2$	$99.6 \pm 0.3$	$75.9 \pm 0.2$
	3:1:2	$60.7 \pm 1.8$	$43.9 \pm 1.3$	$18.4 \pm 0.9$
$\text{MgCl}_2$	1:1:2	$45.8 \pm 0.4$	$52.7 \pm 0.3$	$85.3 \pm 0.4$
	2:1:2	$75.0 \pm 0.4$	$64.7 \pm 0.4$	$98.9 \pm 0.1$
	3:1:2	$34.3 \pm 0.4$	$98.6 \pm 0.0$	$99.6 \pm 0.0$

**Table 3** | Cost considerations

<b>Mg(OH)<sub>2</sub></b>					
Test	pH	[PO <sub>4</sub> <sup>3-</sup> ]:[NH <sub>4</sub> <sup>+</sup> ]	[Mg <sup>2+</sup> ]:[Limitant]	Daily cost (US\$)	Monthly cost (US\$)
1	9	0.13	1	5.35	160.43
2	9	1	2	14.46	433.70
<b>MgCl<sub>2</sub></b>					
Test	pH	[PO <sub>4</sub> <sup>3-</sup> ]:[NH <sub>4</sub> <sup>+</sup> ]	[Mg <sup>2+</sup> ]:[Limitant]	Daily cost (US\$)	Monthly cost (US\$)
3	10	0.13	1	1.57	47.09
4	9	1	1	10.68	320.35

Conversion of US\$1 = R\$2.30 (Brazil).

For the molar ratio  $[\text{NH}_4^+:\text{PO}_4^{3-}] = 7.87:1$ , using  $\text{MgCl}_2$  as source of  $\text{Mg}^{2+}$  ions, the removal of phosphate occurs more efficiently when compared with  $\text{Mg(OH)}_2$ , which may be attributed to its greater solubility in aqueous medium, making it readily available for the reaction of precipitation of struvite. At pH up to 8, with a dosage of equimolar magnesium and phosphate,  $\text{MgCl}_2$  presented very significant phosphate removal as compared with  $\text{Mg(OH)}_2$ . The increase of pH from 9 to 10 with  $\text{MgCl}_2$  showed more satisfactory results than at pH 9, obtaining removals above those represented by  $\text{Mg(OH)}_2$ .

For the molar ratio  $[\text{NH}_4^+:\text{PO}_4^{3-}] = 1:1$ , it was observed that at pH 8, phosphate removal with  $\text{MgCl}_2$  is inferior to that achieved with  $\text{Mg(OH)}_2$ . However, from pH 9 it becomes higher and then exhibits little variation with increasing magnesium dosage.

For the molar ratio  $[\text{NH}_4^+:\text{PO}_4^{3-}] = 1:2$ , lower phosphate removal was observed in some cases, when compared to the previous experiments. However, with  $\text{Mg(OH)}_2$  at pH 9, the results showed the highest values of efficiency of removal, reaching 99.9%, when the molar ratio of phosphate and magnesium was twofold higher than ammonia. It was not possible to view the same standard kinetic results reported previously, possibly because the reaction time should be increased for better results.

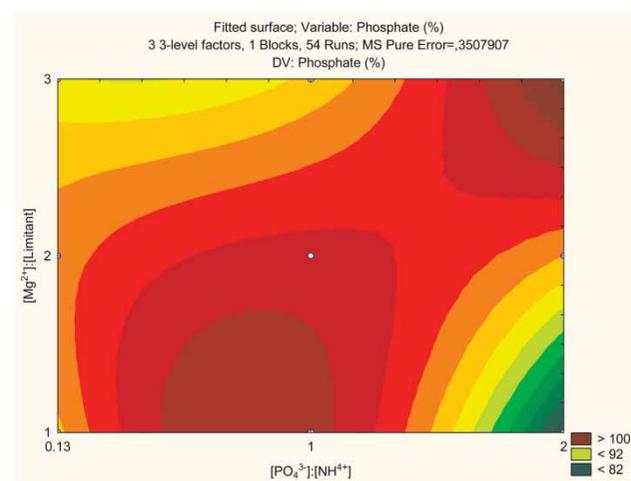
In all tests carried out, the lowering of the pH was observed throughout stirring, requiring pH adjustment. Also, the formation of a white crystalline precipitate in the bottom of the beaker was observed for all experiments, suggesting the formation of struvite.

From the data of the statistical design, it was possible to suggest good operating conditions for analysis with and without the addition of chemical reagent, due to its cost and greatest reductions achieved for ammonium and phosphate.

Thus, we defined the two best conditions for each reagent (Table 3), with and without phosphorus supplementation. The phosphorus supplementation allows greater removal of ammonia and reduced phosphate concentrations, and can contribute to an increase in the mass of precipitate formed.

Based on the results, it can be seen that Test 3 presented the best cost-effective relation, as it requires no addition of phosphorus and lower dosage of  $\text{MgCl}_2$  relative to  $\text{Mg(OH)}_2$ .

Figure 1 shows the surface response graph for Test 3. It can be observed that pH 10 and the molar ratio  $[\text{PO}_4^{3-}]:[\text{NH}_4^+]$  equal to 0.13 presents good efficiency by the dosage of  $[\text{Mg}^{2+}]:[\text{Limitant}]$  equal to 2, probably due to the formation of other compounds with magnesium in excess, reducing the formation of struvite. When the molar ratio  $[\text{PO}_4^{3-}]:[\text{NH}_4^+]$  is 1, increasing the dosage of magnesium implies lower yields for phosphorus removal, which might be related to the

**Figure 1** | Contour plot for Test 3 at pH 10.

previous case. When the molar ratio  $[\text{PO}_4^{3-}]:[\text{NH}_4^+]$  is 2, a gradual increase in the dosage of magnesium is necessary for optimum performance in phosphorus removal.

In Test 3, the quantity of precipitate was 0.743 g to 300 ml of supernatant. Thus, for 100 l of supernatant were formed 247.7 g of precipitate per day and 7.43 kg of precipitate per month. Thus, struvite has commercial value as a fertilizer and could be offered to the agricultural market for economic gain.

In this work, removals of 90.6% and 29.0% were obtained for phosphorus and ammonium, respectively,

thus resulting in  $23 \text{ mg l}^{-1} \text{ PO}_4^{3-}$  and  $265 \text{ mg l}^{-1}$  of  $\text{NH}_4^+$  concentration in a supernatant for Test 3, according to Table 4. The current return of supernatant to the feed alimentation of a WWTP system would present a concentration of  $250 \text{ mg l}^{-1} \text{ PO}_4^{3-}$  and  $373 \text{ mg l}^{-1} \text{ NH}_4^+$ , increasing the organic load to the process.

Figure 2 shows the analysis by XRD of the precipitate obtained. The peaks of the upper line (red line in online version) show the struvite pattern standard and peaks of the lower line (green in online version) are the sample, confirming the presence of struvite in the sample.

**Table 4** | Characteristics of the supernatant before and after the precipitation of struvite

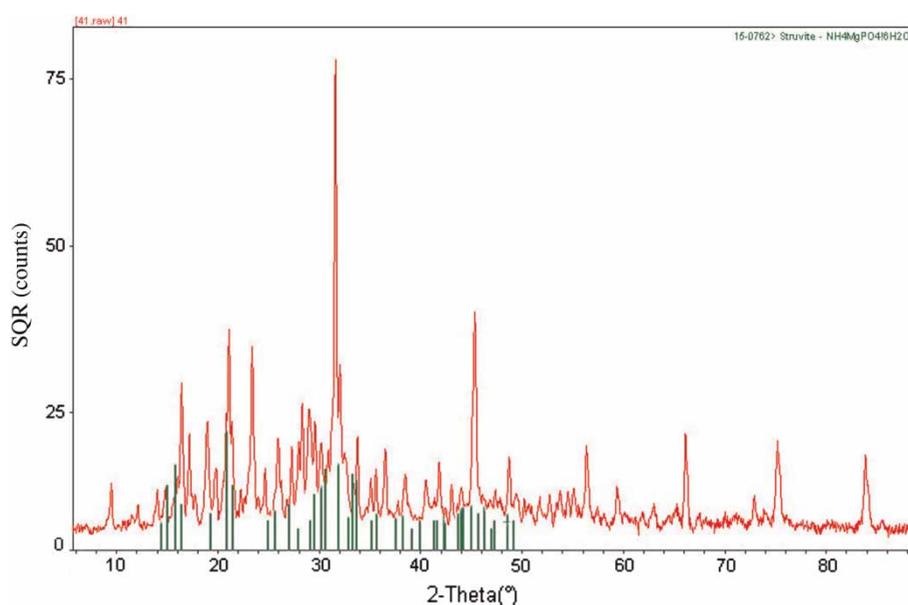
Parameters	Unit	Before precipitation	After precipitation	Removal (%)
pH	-	7.5	10	-
Carbonaceous alkalinity	$\text{mg l}^{-1}$	1135	20	98.2
Total COD	$\text{mg l}^{-1}$	391	350	10.5
Soluble COD	$\text{mg l}^{-1}$	160	231	-44.4
BOD <sub>5</sub>	$\text{mg l}^{-1}$	86	68	20.0
Total organic carbon	$\text{mg l}^{-1}$	50	48	4.4
Phosphate	$\text{mg l}^{-1}$	250	23	90.6
Ammonia	$\text{mg l}^{-1}$	373	265	29.0
TSS	$\text{mg l}^{-1}$	299	218	27.0
VSS	$\text{mg l}^{-1}$	139	79	43.2

## CONCLUSIONS

It was possible to recover phosphorus as struvite – ammonium magnesium phosphate hexahydrate – by the addition of  $\text{MgCl}_2$  and  $\text{Mg}(\text{OH})_2$  to the supernatant line of anaerobic digestion of sludge.

The addition of  $\text{MgCl}_2$  to phosphate in a 1:1 molar ratio, considering the stoichiometric molar ratio  $[\text{PO}_4^{3-}]:[\text{NH}_4^+]$  equal to 0.13 (presented by crude sample) at pH 10, is the best cost-effective condition for implementation on a larger scale, requiring lower dosage of  $\text{MgCl}_2$  in relation to  $\text{Mg}(\text{OH})_2$ .

The required pH can readily be achieved with the addition of sodium hydroxide, which has a relatively low cost. The fact that the pH of these experiments was performed in the alkaline range requires the continuous



**Figure 2** | Diffractogram of XRD analysis. (The full colour version of this paper is available online at <http://www.iwaponline.com/wst/toc.htm>).

adding of a base. However, the formation of struvite could compensate for the investment, because the compound of struvite presents an important function as a soil conditioner, and could be marketed.

In this work, removals of 90.6 and 29.0% were obtained for phosphorus and ammonium, respectively, thus resulting in a supernatant concentration of  $23 \text{ mg l}^{-1} \text{ PO}_4^{3-}$  and  $265 \text{ mg l}^{-1}$  of  $\text{NH}_4^+$  of the sample selected. Returning this to the feed alimentation of a WWTP system at a concentration below the crude sample would have a smaller impact on the treatment process of the WWTP. The analysis by XRD confirmed the presence of struvite.

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First received 10 June 2013; accepted in revised form 9 January 2014. Available online 4 February 2014