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Nutrient recovery from effluent of anaerobic digester treating swine wastewater via struvite precipitation: A case study in Hue City, Vietnam

Ho Thi Xuan Tuy[1], **Te Minh Son**[2], **Pham Khac Lieu**[2]*

[1] Institute for Global Health Innovations, Duy Tan University, 7/25 Quang Trung Street, Da Nang, Vietnam
[2] University of Sciences, Hue University, 77 Nguyen Hue Street, Hue, Vietnam

* Correspondence to Pham Khac Lieu <pklieu@hueuni.edu.vn>

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Abstract. Recently, the recovery of nutrients from nutrient-rich wastewater has attracted considerable attention as a sustainable alternative to contaminant removal. This study investigated the recovery of nitrogen (N) and phosphorus (P) from the effluent of an anaerobic digester treating swine wastewater in Hue City, Vietnam, through struvite precipitation. The effects of pH, reaction time, and $NH_4^+/Mg^{2+}/PO_4^{3-}$ (AMP) molar ratio on nutrient recovery efficiency and struvite formation were evaluated by using a Jar-Test system. The results reveal that increasing pH adversely affected N recovery because of NH_3 volatilisation, whereas P recovery and struvite formation improved slightly. The best struvite formation was observed at pH 9.0, the reaction time of 30 min, and the AMP molar ratio of 1:1.5:1.5 with a recovery efficiency above 90% and the N and P removal efficiencies of 70% and 57.1%, respectively. X-ray diffraction analysis confirmed that struvite was the primary crystalline product. Additionally, the struvite precipitate contained 4.6% N, 12.5% P, and 11.4% Mg under the most appropriate conditions. This study demonstrates that struvite precipitation efficiently recovered nutrients from anaerobic digestion effluents.

Keywords: anaerobic digester, nutrient recovery, struvite precipitation, swine wastewater

1 Introduction

The global expansion of livestock farming, including pig farming, is posing significant environmental challenges due to the discharge of large volumes of nutrient-rich wastewater [1]. A major challenge in managing this wastewater is to avoid excessive nutrient discharge into aquatic ecosystems, which can result in eutrophication of receiving water bodies [2, 3]. Anaerobic digestion is a common technology for reducing the organic load of swine wastewater by 40–80% and for biogas production [4]. However, effluent of anaerobic digester (AD) still contains a high content of nutrients, particularly excess ammonium (NH_4^+) and orthophosphate (PO_4^{3-}), and often exhibits low biodegradability, which may reduce the efficiency of nitrification and denitrification processes [5, 6]. Nutrient recovery from anaerobically treated swine wastewater offers an alternative that both removes nutrients and yields a product that can be used as a fertilizer [7].

Several technologies have been developed to recover nutrients from treated swine wastewater, each with varying levels of efficiency and applicability depending on the characteristics of the AD effluents [8]. Microalgae can effectively uptake nutrients from the wastewater, often with reported high removal rates of NH_4–N (58.9–90.2%) and PO_4–P (38.9–100%) under optimal conditions. Nonetheless, maintaining

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stable algal growth, as well as separating algal biomass from treated wastewater, remains a considerable challenge [9]. Membrane filtration technologies, such as nanofiltration [10] and reverse osmosis [11], can effectively concentrate both NH_4^+ (50–98%) and PO_4^{3-} (80–100%), but they require high-energy consumption and are prone to fouling, particularly at higher water recovery rates. Air stripping combined with acid absorption is another effective method for NH_4^+ recovery (up to 88.7%), especially under alkaline conditions (pH >10); however, it does not facilitate phosphorus recovery [12]. Recently, struvite precipitation ($MgNH_4PO_4 \cdot 6H_2O$) has gained increasing attention for its ability to recover both NH_4^+ and PO_4^{3-} simultaneously in the form of a slow-release fertilizer through a relatively simple chemical reaction

$$Mg^{2+} + NH_4^+ + PO_4^{3-} + 6H_2O \rightarrow MgNH_4PO_4 \cdot 6H_2O$$

pH strongly affects the recovery efficiency of N and P via the struvite-forming reaction. It was reported that the interaction between pH and influent NH_4–N concentration plays a crucial role in this process. At lower NH_4–N concentrations (e.g., 105 mg/L), the nutrient recovery significantly increased at pH 10, whereas at higher concentrations (e.g., 1050 mg/L) the efficiency was less pH-dependent [13]. It was also found that no struvite precipitate formed at pHs below 7.5 under the conditions of limited N, Mg, and P availability [14]. However, the AD effluent is typically neutral to slightly alkaline (6.5–8.0), which may not be optimal for struvite formation and often requires pH adjustment [15]. Additionally, digestate effluents generally contain insufficient PO_4^{3-} (mainly present in particulate form) and Mg^{2+} ions, posing further challenges for effective struvite precipitation [16]. To overcome these limitations, the addition of phosphate salts (e.g., KH_2PO_4 and Na_2HPO_4) [17, 18] and Mg sources (e.g., $MgCl_2$ and MgO) [19, 20] is commonly required. Alternatively, the use of inexpensive materials that contain Mg sources (e.g., bittern and zeolite) is a more cost-effective option [21, 22]. Furthermore, determining the optimal $NH_4^+/Mg/PO_4^{3-}$ (AMP) molar ratio is essential, although the ideal stoichiometric ratio is 1:1:1. Several studies suggest that an AMP molar ratio with a slight excess of Mg to PO_4^{3-} (e.g., 1.0:1.2:1.0) often leads to improved NH_4^+ and PO_4^{3-} recovery (>90%) [17, 19, 23]. Conversely, recovery efficiency for PO_4^{3-} was reported as low as 6% when this ratio was high (1.0:1.3:0.8) [24]. Hence, the optimal AMP ratio needs to be determined through experiments depending on the type of AD effluents and operating conditions [16].

This study aimed to optimize struvite precipitation for nitrogen and phosphorus recovery from the effluent of an AD treating swine wastewater. The specific objectives were to determine the optimal reaction conditions, including pH, reaction time, and AMP molar ratio, for maximising nutrient recovery efficiency and to characterise the recovered struvite.

2 Materials and methods

2.1 Wastewater source

The anaerobic digester's effluent used in this study was collected from a pig farm in Hue City, Vietnam. The AD supernatant was screened through a 230-mesh sieve to remove large debris, allowed to settle for 12–16 h to reduce suspended solids, and then stored at 4 °C. A portion of the settled supernatant was filtered through a 0.45 μm glass fibre filter (Whatman, GE Healthcare, USA) for analysing initial chemical oxygen demand (COD), NH_4–N, PO_4–P, and Mg^{2+} concentrations (Table 1).

Table 1. Characteristics of anaerobic digester's effluent used for the recovery experiments

Parameter	Unit	Mean ± SD (*n* = 3)	Vietnam's discharge standard limits	
			S[1]	S[2]
pH	n/a	8.2	5.5–9.0	5.5–9.0
COD	mg/L	1540 ± 50	150	300
Mg^{2+}	mg/L	37 ± 9	n/a	n/a
NH_4–N	mg/L	750 ± 40	10	n/a
PO_4–P	mg/L	31 ± 3	n/a	n/a

[1] QCVN 40:2021/BTNMT: Vietnam National Technical Regulation on Industrial Effluent, B column.

[2] QCVN 62:2016/BTNMT: Vietnam National Technical Regulation on the Effluent of Livestock, B column.

n/a: not applicable.

2.2 Struvite precipitation experiments

A series of batch experiments was conducted to evaluate the influence of initial pH (7.5–9.0), reaction time (10, 20, 30, 40 min), and AMP ratios (original, 1:1:1, 1:1:1.5, 1:1.5:1.5) on struvite precipitation. All the experiments were performed in triplicate. The tests were carried out at ambient temperature with a Jar-tester (JLT4, VELP Scientifica, Italy). Before each test, 500 mL of AD effluent was transferred into a 1 L glass beaker, and the pH was adjusted by using a 1 N NaOH solution. $MgCl_2$ and KH_2PO_4 were added to adjust the Mg^{2+} and PO_4^{3-} concentrations, respectively. First, the mixture was stirred at 200 rpm, followed by 30 min of sedimentation. Second, 400 mL of the supernatant was collected and filtered for quality analysis. Finally, the settled portion was filtered through 0.45 μm glass fibre to recover the struvite precipitates, which were then dried at 40 °C for 48 h in an oven (Memmert UM500, Memmert GmbH + Co. KG, Schwabach, Germany).

2.3 Analytical methods

pH was measured with a pH meter (ECO sense pH100A, YSI Incorporated, USA). Mg^{2+}, PO_4–P, and COD concentrations were analysed in accordance with the standard methods published by APHA [25], and NH_4–N was determined according to the OPP method [26]. A spectrophotometer (GENESYS 10S UV-VIS, Thermo Fisher Scientific, USA) was used for colorimetric analysis of NH_4–N, PO_4–P, and COD. Mg^{2+} concentration in water samples was determined by using atomic absorption spectroscopy (AAS) with a 240/280 Series instrument (Agilent Technologies, USA). The dried solid samples were ground into powder and dissolved in 0.1 N HCl, and the Mg^{2+} concentration was determined. The precipitates obtained from several experimental conditions were further characterised with an X-ray diffraction analyser (XRD, Smartlab, Rigaku, Japan) under the following conditions: CuKα radiation source; $\lambda = 1.5406$ Å; a scanning rate of $0.06°.s^{-1}$.

2.4 Calculations

The removal efficiency (*H*, %) of NH_4–N and PO_4–P from wastewater was calculated by using Eq. (1)

$$H = \frac{C_0 - C}{C_0} \times 100 \quad (1)$$

where C_0 and C are the initial and final concentrations of NH_4–N and PO_4–P (mg/L) in wastewater samples, respectively.

The nutrient recovery efficiency was calculated as the percentage of NH_4–N and PO_4–P recovered in the solid phase to the removed fraction from the wastewater, as shown in Eq. (2)

$$R_{\mathrm{h}} = \frac{m_S \times a}{C_0 \times 0{,}01 \times H} \times 100 \qquad (2)$$

where R_h is the nutrient recovery efficiency (for each N and P); m_S is the mass of struvite obtained from 1 L of wastewater (g/L); a is the nutrient (N, P) content of struvite (mg/g); H and C_0 are already defined in Eq. (1).

3 Results and discussion

3.1 Struvite precipitation reaction

Effect of initial pH

Crystallisation of struvite occurs when the concentration of its constituent ions exceeds the thermodynamic solubility [27]. As shown in Fig. 1, the nutrient removal efficiency from AD effluents generally increased with rising pH, reaching 55.8–61.8% for N ($p < 0.05$) and 55.6–58.3% for P ($p > 0.05$), and positively correlated with precipitate mass. However, the recovered struvite mass increased slightly with rising pH from 7.5 to 9.0 (7.5 ± 0.37 g/L to 7.9 ± 0.5 g/L) (Fig. 1a). The nutrient recovery efficiency, based on the proportion of N and P removed and recovered as struvite, was the highest at lower pH values, decreasing significantly from 97.8% at pH 7.5 to 78.3% at pH 9.0 for N (Fig 1b). The increased N removal efficiency at higher pH may be attributed to the volatilisation of NH_3. At higher pH, although less NH_4^+ participates in the struvite-forming reaction, other precipitates such as $Mg_3(PO_4)_2$ and $Mg(OH)_2$ may form, leading to a slight increase in the mass of precipitate obtained. Conversely, the P recovery remained consistently high (98.3% to 99.4%) across the pH range, indicating minimal sensitivity to pH variations (Fig. 1b). The amount of recovered struvite was about the same across all pH levels (Fig. 1a), which may be because the P/N molar ratio was limited on struvite precipitation. This is consistent with the findings reported by Wu and Zhou [13]. Although a pH greater than 9.0 is often considered optimal for struvite precipitation and minimising struvite dissolution [28], the higher pH can reduce struvite purity [29]. From the changing pattern of nutrient removal efficiency, recovery efficiency, and struvite purity with pH, as well as the practical application aspect, the pH of 9.0 was selected for subsequent experiments.

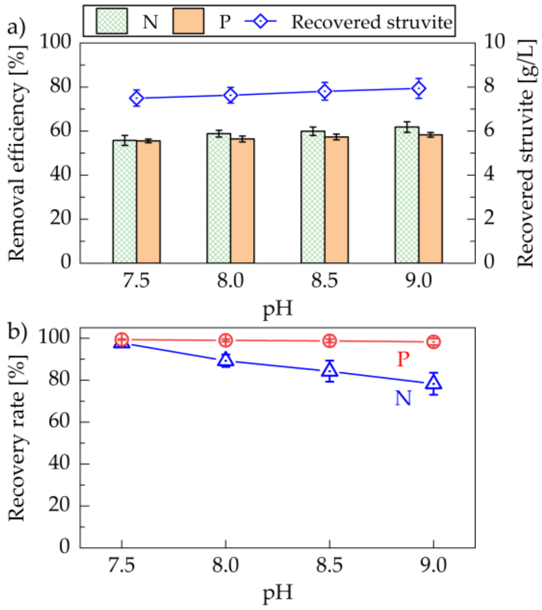

Fig. 1. (a) Removal efficiency of N and P from wastewater; (b) Recovery efficiency of N and P via struvite precipitation at different pH levels; Conditions: AMP molar ratio = 1:1:1, reaction time =20 min

Effect of reaction time

Reaction time did not significantly affect N and P removal efficiency via struvite precipitation, with no statistically significant observed differences ($p > 0.05$) (Fig. 2a). The average removal efficiency for N and P was 62.1 and 58.5%, respectively. Correspondingly, the amount of struvite formed per litre of wastewater shows minimal variation (7.82 to 7.94 g) across reaction time from 10 to 40 min ($p > 0.05$). Similarly, nutrient recovery shows only a slight increase with extended reaction time, with the N recovery increasing from 75.5 to 81.5% as reaction time increased from 10 to 40 min (Fig. 2b). In other words, both the nutrient recovery efficiency and the quantity of struvite produced increased only marginally with longer reaction time. As for practical applications for large volumes of wastewater, 30 min appeared sufficient to allow complete reaction and was selected for subsequent experiments.

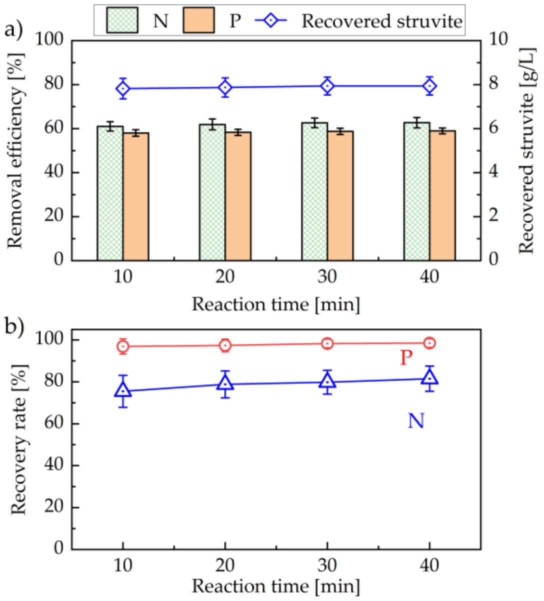

Fig. 2. (a) N and P removal efficiency from wastewater; (b) recovery efficiency of N and P via struvite precipitation at different reaction times; Conditions: AMP molar ratio = 1:1:1; pH 9

Effect of AMP molar ratio

The AMP molar ratio is an important factor related to the recovery efficiency of N and P and the physicochemical characteristics of struvite. The experiments with raw wastewater having a molar AMP molar ratio of 1:0.03:0.02 show virtually no struvite crystallisation (Fig. 3a), indicating that external supplementation of Mg and P is essential. The removal efficiency of N and P, as well as the amount of struvite formed, varied significantly with different AMP molar ratios ($p < 0.05$). At a molar ratio of 1:1:1, the N and P removal efficiency reached 62.3 and 58.8%, respectively, with a struvite yield of 7.89 g/L of AD effluent. Increasing the PO_4^{3-} molar ratio relative to Mg^{2+} and NH_4^+ (A/M/P = 1:1:1.5) did not significantly improve nutrient removal or struvite production ($p > 0.05$) (Fig. 3a). In contrast, simultaneously increasing the molar ratios of Mg^{2+} and PO_4^{3-} led to a substantial increase in struvite yield to 11.5 g/L, about 45% higher than at the 1:1:1 ratio ($p < 0.05$). Under the AMP molar ratio of 1:1.5:1.5, the average removal efficiency for N and P reached 70.0% and 57.1%, respectively.

The N recovery rate remained nearly unchanged between the 1:1:1 and 1:1:1.5 ratios, at around 79% but increased sharply when more Mg and P were added to maximise nitrogen recovery. The AMP molar ratio of 1:1.5:1.5 resulted in an N recovery rate of up to 91.0%. These results are comparable to those reported by Celen and Turker, indicating a higher N recovery efficiency at the molar ratio of 1:1.2:1.2, even at a low pH of 6.0 [30].

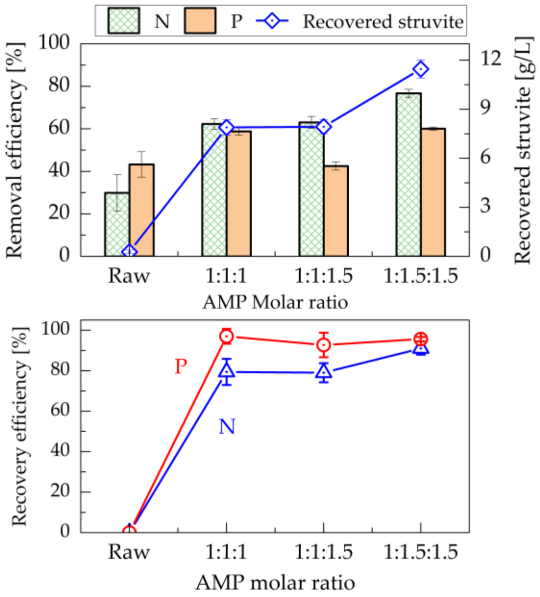

Fig. 3. (a) Removal efficiency of N and P from wastewater; (b) Recovery efficiency of N and P via struvite precipitation at different AMP molar ratios; Conditions: reaction time = 30 min, pH 9.0

The phosphorus recovery rate was highest at the 1:1:1 ratio and slightly decreased when more phosphate was added because a large amount of P was supplemented; however, the P recovery remained consistently high, above 90% (Fig. 3b).

To summarise, the recovery of NH_4^+ and PO_4^{3-} from AD effluents as struvite precipitates necessitates the addition of magnesium and phosphate salts to achieve an ideal stoichiometric AMP ratio. However, increasing the molar ratio of PO_4^{3-} relative to Mg^{2+} and NH_4^+ (A/M = 1:1) had no significant effect on P recovery, while the N recovery efficiency and struvite yield remained nearly unchanged (Fig. 3a). In contrast, simultaneously increasing the molar ratios of Mg^{2+} and PO_4^{3-} relative to NH_4^+ (1:1.5:1.5) resulted in positive effects on nitrogen recovery and struvite production. Nevertheless, excessively high ratios may raise concerns about excess phosphorus and magnesium remaining in the treated effluent, and the increased chemical cost warrants careful consideration. Future studies on the AMP ratio and economic analysis would benefit from optimising the removal of P and enhancing the mass of struvite product.

3.2 Characterisation of the struvite precipitates

XRD analysis (Fig. 4) was used to compare the solid materials recovered at different AMP ratios with standard struvite. The patterns of precipitates confirmed that struvite is the dominant crystalline phase, exhibiting characteristic 2θ peaks at 15.92° (002), 20.99°(111), 21.56°(012), 27.18°(103), 30.7°(211), 32°(120), and 33.3° (022) [31, 32].

The nutrient analysis (Table 2) revealed that recovered struvite consistently contained lower N and higher Mg content than that in standard struvite ($MgNH_4PO_4{\cdot}6H_2O$). For instance, at pH 7.5, recovered struvite contained 54.8 mg-N/g and 113.8 mg-Mg/g, compared with 57.1 mg-N/g and 99.0 mg-Mg/g in standard struvite. This suggests that the presence of an additional Mg amount in the recovered materials, such as co-precipitated compounds (e.g., $Mg(OH)_2$) [13]. Notably, increasing the pH from 7.5 to 9.0 resulted in a slight decrease in both P and Mg content in the recovered struvite. At pH 7.5, P was 122.8 mg-P/g, and Mg was 113.8 mg/g, while at pH 9.0, these values dropped to 120.3 mg-P/g and 109.1 mg/g, respectively. This could also be attributed to the formation of competing precipitates at higher pH and may decrease the purity of the recovered struvite [33]. In brief, the struvite precipitate obtained under the most appropriate conditions in this study (pH 9.0; AMP = 1:1.5:1.5; reaction time = 30 min) contained 4.6% N, 12.5% P, and 11.4% Mg.

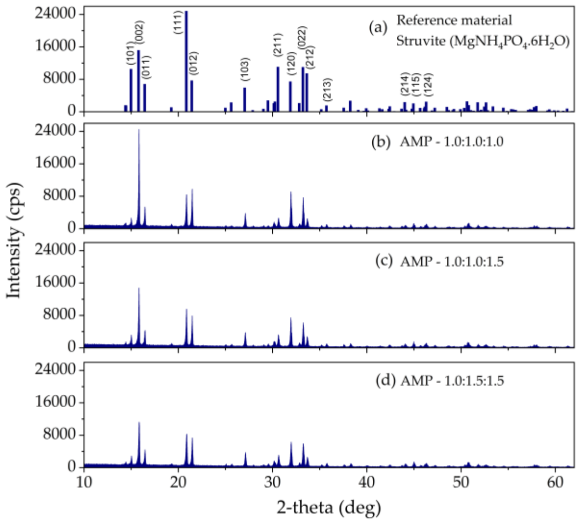

Fig. 4. XRD patterns of the reference of (a) standard struvite and recovered materials at different AMP ratios; (b) 1.0:1.0:1.0, (c) 1.0:1.0:1.5; (d) 1.0:1.5:1.5

Table 2. Composition of struvite precipitate samples compared with standard struvite ($NH_4MgPO_4{\cdot}6H_2O$)

Struvite sample	NH_4–N (mg-N/g)	PO_4–P (mg-P/g)	Mg (mg/g)
pH 7.5	54.8 ± 3.2	122.8 ± 1.5	113.8 ± 1.1
pH 8.0	51.8 ± 2.5	122.2 ± 0.8	111.5 ± 0.5
pH 8.5	48.6 ± 4.2	120.9 ± 0.3	109.5 ± 0.7
pH 9.0	45.8 ± 4.0	120.3 ± 0.1	109.1 ± 1.0
AMP 1:1:1	47.2 ± 2.2	120.5 ± 2.7	113.0 ± 3.2

Struvite sample	NH_4–N (mg-N/g)	PO_4–P (mg-P/g)	Mg (mg/g)
AMP 1:1:1.5	47.2 ± 1.7	123.2 ± 3.9	113.1 ± 2.4
AMP 1:1.5:1.5	45.9 ± 2.4	124.6 ± 1.4	113.5 ± 1.6
Standard struvite*	57.1 ± 1.2	126.2 ± 0.8	99.0 ± 1.7

*Source: [34, 35]

3.3 Remarks and implications

The remaining NH_4–N concentration in wastewater ranged from 175 to 525 mg/L. Although N recovery exceeded 70% under the conditions of pH 9.0, the post-treatment NH_4–N concentration was 17 times higher than the national effluent standard (QCVN 40:2021/BTNMT) of 10 mg/L. The average PO_4–P concentration after struvite formation was approximately 300 mg/L, significantly lower than the added concentrations (713 mg/L for 1:1:1 ratio and 796 mg/L for 1:1.5:1.5 ratio) but still 10 times higher than the initial concentration in raw wastewater. To improve cost efficiency and reduce excess PO_4–P content in the effluent, future studies should focus on optimising phosphate dosing, recirculating the effluent, or investigating alternative low-cost phosphorus sources.

Furthermore, the struvite reaction contributed to the removal of organic matter, with an average COD reduction of 34.6% under the most appropriate conditions and a final COD concentration of approximately 1000 mg/L. The average Mg^{2+} concentration after treatment was low (8.5 mg/L) compared with raw AD effluent, reducing concerns related to water hardness. However, this low residual Mg^{2+} concentration may limit the complete removal of NH_4^+ and PO_4^{3-}, suggesting that the Mg^{2+} dosage still fell short of the stoichiometric requirement. Thus, more precise control of Mg^{2+} input or the use of natural Mg-rich additives (e.g., bittern and dolomite) can enhance struvite crystallisation without significantly increasing operational costs.

4 Conclusion

The recovery of N and P from the effluent of an anaerobic digester treating swine wastewater was conducted with struvite crystallisation. In struvite reaction experiments, the pH value was the main factor causing a loss in N recovery efficiency. N and P recovery efficiency increased slightly with reaction time. The AMP molar ratio significantly affected the recovery efficiency. Under the most appropriate conditions (pH of 9.0, AMP molar ratio of 1:1.5:1.5, and reaction time of 30 min), the average removal efficiency of NH_4–N and PO_4–P was 70.0% and 57.1%, respectively. The obtained materials were proven to be a struvite phase of $MgNH_4PO_4{\cdot}6H_2O$ via XRD analysis with a precipitated mass value up to 11.5 g/L. The excess concentrations of NH_4–N, PO_4–P, and COD in the effluent after recovery presented a limitation of the study, suggesting the need for further optimisation of the conditions for struvite precipitation.

Declaration of conflict of interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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