

Critical conditions of struvite growth and recovery using MgO in pilot scale crystallization plant

Nari Park, Hyangyoun Chang, Yeou Jang, Hyunman Lim, Jinhong Jung and Weonjae Kim

ABSTRACT

The struvite crystallization process can recover struvite crystals as a valuable slow-release fertilizer from the side stream of wastewater treatment plants (WWTPs). The purpose of this study is to demonstrate the crystal growth characteristics and determine the appropriate recovery criteria for a struvite crystallization pilot plant. A pilot plant (8.6 m³/d) was designed with a feeding system of MgO (magnesium oxide), a pH controller, and a hydrocyclone for recovering struvite; the plant was operated for 42 hours at a pH range of 8.25–8.5. The removal efficiencies for PO₄-P and NH₄-N were 82.5–90.7% and 13.4–22.9%, respectively. The struvite recovered from the hydrocyclone was sifted using standard sieves and analyzed by scanning electron microscopy (SEM) and X-ray diffraction (XRD). The dry weight fraction of the precipitate in the 300–600 μm range increased gradually from 7% to 74% in 18 hours. The XRD analysis revealed that the crystalline structure of the precipitate in the 150–600 μm range indicates struvite without any peaks of MgO, Mg(OH)₂, and MgCO₃. This indicates that the critical conditions for recovering struvite from the side-stream of WWTPs are an operation period of 18 hours and a crystal size greater than 300 μm.

Key words | crystallization, hydrocyclone, nitrogen recovery, phosphorus recovery, struvite (MAP)

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HIGHLIGHTS

- The time serial growth characteristics of struvite crystals were intensively investigated throughout the continuous operation of a pilot plant.
- The recovery criteria of struvite considering size distribution and crystalline properties were determined by the intermittent operation of a hydrocyclone in the pilot plant.
- An MgO feeding system with a pH controller was applied to the pilot plant, and its applicability was evaluated.
- Removal efficiencies of PO₄-P and NH₄-N were stably achieved 82.5–90.7% and 13.4–22.9%, respectively.
- Critical conditions for recovering struvite could be suggested as an operation time over 18 hours and a crystal size greater than 300 μm.

INTRODUCTION

Phosphorus (P) is an essential element that is present in plants and in animal and human bodies and is critical for growing crops, making it critical in the world food supply chain. However, current research suggests that global available phosphorus reserves may be depleted

by around 2300 (Daneshgar *et al.* 2018a, 2018b). Thus, it is necessary to recover phosphorus to meet future demand.

Enhanced biological phosphorus-removal wastewater treatment plants that use anaerobic digesters for sludge

treatment have high phosphorus concentrations in the side streams from their sludge dewatering equipment (Münch & Barr 2001). Phosphorus is released during anaerobic digestion via cell decay. By recovering phosphorus from the side stream, it is possible to simultaneously reduce phosphorus accumulation in wastewater treatment plants and recover phosphorus resources. Current phosphorus recovery technologies are mainly based on the crystallization of phosphorus as hydroxyapatite (HAP, $\text{Ca}_{10}(\text{OH})_2(\text{PO}_4)_6$) or struvite (MAP, $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) (Wang *et al.* 2005; Daneshgar *et al.* 2019).

Ammonia is also released from the hydrolysis of proteinaceous organic materials during anaerobic digestion (Nakashimada *et al.* 2008). Struvite crystallization is recommended as a method for removing and recovering ammonium and phosphate from the side stream. It consists of three components: Mg^{2+} , NH_4^+ , and PO_4^{3-} with equal molecular ratio (1:1:1) under alkaline conditions in the pH range, 8–10. The crystallization process is a simple chemical reaction, so it is easy to operate and maintain. Moreover, struvite can be commercialized for use as a slow-release fertilizer (Le Corre *et al.* 2009).

For applying the struvite crystallization process to the side-stream, it is necessary to adjust the pH and add magnesium. To increase the pH, NaOH is generally added and CO_2 is stripped from the solution (Fattah *et al.* 2008). As a magnesium source, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ is commonly used in both laboratory-scale and commercial-scale processes due to its high solubility. It can achieve a phosphorus removal efficiency of 90% (Moerman *et al.* 2009; Kataki *et al.* 2016a, 2016b). However, it is expensive and requires additional pH adjustment.

MgO is another magnesium source that can be simultaneously used for pH adjustment while being economical and convenient to feed. However, MgO has very low solubility; it should be added as a slurry and requires a longer retention time (Desmidt *et al.* 2015; Stolzenburg *et al.* 2015). Other researchers suggest simultaneously using MgO as a magnesium source and NaOH for pH adjustment to address these problems (Montag *et al.* 2007; Yu *et al.* 2013; Huang *et al.* 2014). The operating pH was set to 7.5–9.5 and MgO was continuously injected, maintaining a Mg/P ratio of 1.1–1.5; with this, a phosphorus removal efficiency of 80 to 90% was achieved. In this study, a new MgO feeding system is suggested where the MgO slurry is automatically injected coinciding with the target pH value, using a pH controller. This MgO feeding system with a pH controller was applied to a pilot plant, and its efficacy was evaluated.

Many struvite crystallization processes produce small crystals. This can affect the total suspended solid concentration of the effluent and result in low economic returns. In order to solve this problem, it was suggested that struvite be formed into pellets (Crutchik *et al.* 2017) and a cylinder reactor be designed with different diameters (Forrest *et al.* 2008; Iqbal *et al.* 2008). Shepherd *et al.* (2009) introduced a hydrocyclone to separate the crystals from the effluent. In this study, a hydrocyclone was installed for crystal growth and recovery. The crystal growth characteristics were demonstrated by operating a pilot plant continuously, and the appropriate recovery criteria were determined.

The struvite crystallization pilot plant was developed and operated at 'I' WWTP, in Gyeonggi province in Korea. The specific research objectives were (1) to apply a new MgO feeding method using a pH controller and (2) to investigate the growth characteristics of struvite crystals through continuous operation of a pilot plant developed for this method.

MATERIAL AND METHODS

Batch experiment

To determine the appropriate operating conditions for the new MgO feeding method, batch experiments were conducted in agitated jar-testers. Digested sludge filtrate from a dehydrator of the 'I' WWTP was used as the raw water for the process. The schematic diagram of the 'I' WWTP is shown in Figure 1. The MgO slurry (1%), made from 98% MgO (STARMAG[®]), was injected into 1 L of the digested sludge filtrate to adjust the molar ratio, (injected Mg)/P, to the range 0.6–1.5, and stirred at 120 rpm for 2 h. Three samples were taken after 30, 60, and 120 min, and the pH and Mg^{2+} , $\text{NH}_4\text{-N}$, and $\text{PO}_4\text{-P}$ concentrations were measured.

Operation of struvite crystallization pilot plant

The struvite crystallization pilot plant is located at 'I' WWTP and has a capacity of 8.6 m³/d. The pilot plant is composed of a pretreatment reactor, a crystallization reactor and a hydrocyclone. The pretreatment reactor (volume: 150 L, hydraulic retention time (HRT): 25 min) consists of two basins. In the first basin, the coarse sludge from the influent is removed by a scraper, and the residual sludge settles down in the second basin. The crystallization reactor (volume: 360 L, HRT: 60 min) is composed of a cylinder shaped

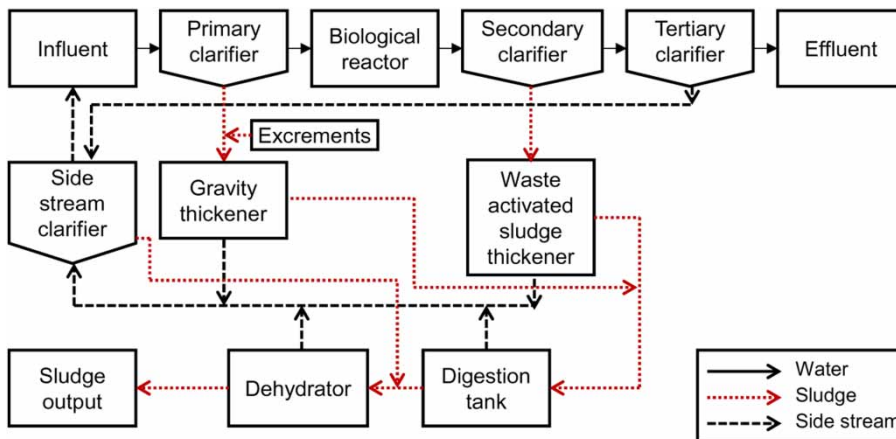


Figure 1 | Schematic diagram of '1' wastewater treatment plant.

upper part and a funnel shaped lower part. The cylinder has two aerated zones and a tranquil zone.

The influent and the MgO solution (1%) flow into the aerated zone #1. They are mixed in seconds to form crystal nuclei. Then, mixing water flows into the aerated zone #2. The mixing is continued for about 40 min, and the crystal nuclei grow. The grown crystal nuclei settle in the tranquil zone in approximately 20 min. Air flow rates in aerated zones #1 and #2 are 10 L/min and 20–30 L/min, respectively.

The grown struvite crystals are recycled intermittently by a hydrocyclone with dimensions 260 × 210 × 525 (L × W × H) (mm) and cone angle, 20°. The separation efficiency (loading rate of underflow/total loading rate) of the hydrocyclone was evaluated by using anthracite crystals (specific gravity 1.65)

with specific gravity very close to that of struvite (specific gravity 1.7). It was observed that the separation efficiency was greater than 77% for crystals larger than 150 μm, with an influent flowrate of 50 L/min. The underflow of the hydrocyclone was returned to the crystallization reactor as seeds of struvite while the upflow, including the sludge, was discharged into the drain. The conceptual diagram of the struvite pilot plant is shown in Figure 2.

The MgO slurry was injected coinciding with the target pH value of the crystallization reactor, using a pH controller. The pH controller consists of a pH probe and a peristaltic pump connected to the MgO slurry tank (MgO concentration: 1%). The pH probe is located outside the aerated zone #2 in the crystallization reactor to obtain a stable

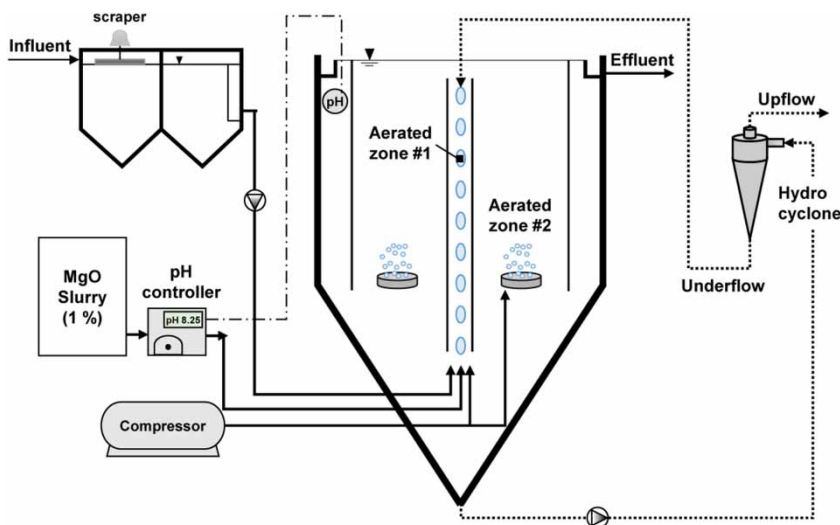


Figure 2 | Conceptual diagram of struvite crystallization pilot plant.

pH value. The peristaltic pump was automatically turned on and off to meet the target pH value.

The pH control algorithm is as follows: First, the target pH value and a surplus value are set in the pH controller. If the pH value is lower than the target pH, the peristaltic pump is turned on and the MgO slurry is injected until the pH value reaches (target pH + surplus); the pump is then turned off until the target pH is reached. In this pilot plant, the target pH values were set as 8.25 and 8.5, and the surplus value was set at 0.05. The pilot plant was operated for 42 hours at pH values of (8.25 + 0.05) and (8.5 + 0.05).

The applicability of the MgO feeding system with the pH controller was observed, and the PO₄-P and NH₄-N removal efficiencies were monitored for each pH condition. The removal efficiency was calculated as the ratio of the difference between influent and effluent concentrations (influent minus effluent) to the influent concentration.

Crystal growth in struvite crystallization pilot plant

The pilot plant was operated at a pH of 8.25 for 42 h. The mixture from the crystallization reactor passed through the hydrocyclone for 2 min every 6 h. The flowrate of the hydrocyclone was approximately 50 L/min. Two liters of sample slurry were collected from each underflow of the hydrocyclone after an operating time of 30 s to stabilize the separation efficiency and to drain the underflow abundantly.

The samples were sifted using standard sieves with mesh sizes 75, 150, 300, 600, and 1,200 μm and dried at 110 °C for 2 h in a dry oven. The dry weight fraction from each sieve was measured.

Analytical methods

The concentrations of Mg²⁺, NH₄-N, and PO₄-P were measured by Aquion (Thermo Fisher Scientific) ion chromatography (IC). The morphologies of the crystal samples were analyzed by S-4800 (Hitachi) scanning electron microscopy (SEM). The crystal structures of the samples were analyzed using the Empyrean (Panalytical B.V.) X-ray diffractometer (XRD).

RESULTS AND DISCUSSION

Batch experiment results

To investigate the optimal operating conditions for the pilot plant, batch experiments were performed in the range of 0.6–1.5 for Mg/P molar ratios. The raw water characteristics were pH of 7.6, 23.2 mg/L of Mg²⁺, 288.8 mg/L of NH₄-N, and 89.2 mg/L of PO₄-P. When the Mg/P molar ratios were adjusted to values ranging from 0.6 to 1.5, the ranges of effluent characteristics were: pH of 8.1–8.7, 17.0–45.8 mg/L of

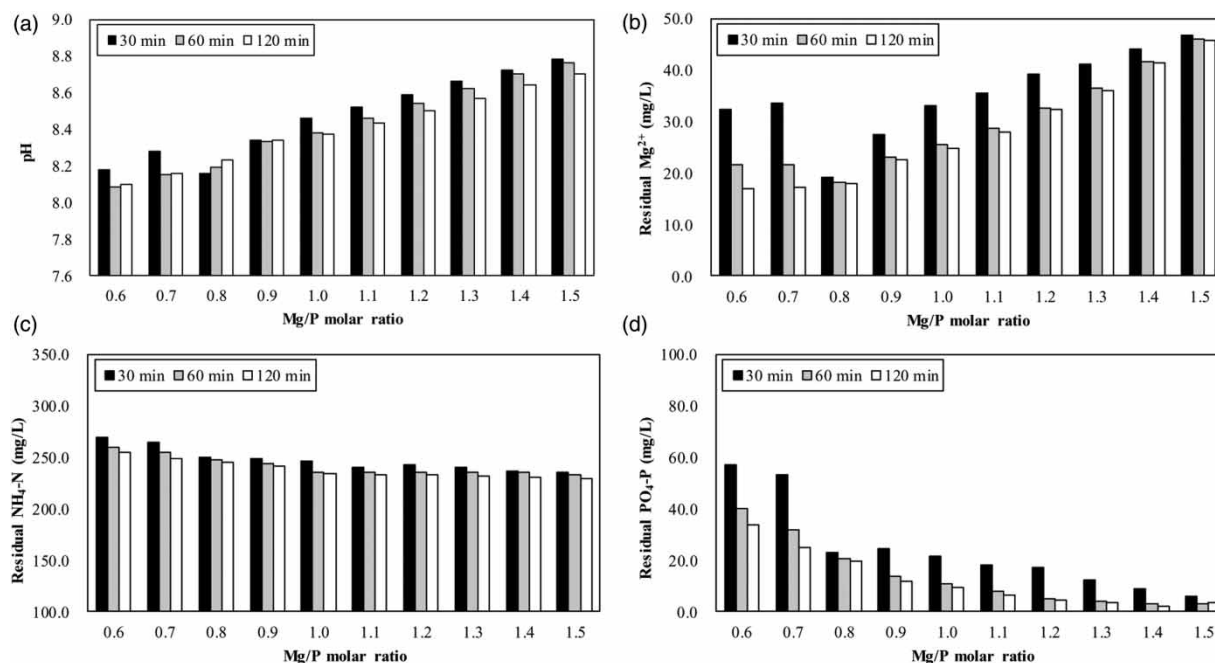


Figure 3 | Results of batch experiments at 30, 60, and 120 min. (a) pH, (b) Mg²⁺ concentration, (c) NH₄-N concentration, and (d) PO₄-P concentration.

Mg²⁺, 229.7–254.8 mg/L of NH₄-N, and 3.2–33.5 mg/L of PO₄-P. The results of the batch experiments at 30, 60, and 120 min are shown in Figure 3. The pH as well as the concentrations of all ions tended to decrease slightly as reaction time increased. For every condition, the pH values (pH 8.1–8.7) were appropriate for the formation of struvite crystals. The removal efficiencies of NH₄-N and PO₄-P improved with increasing Mg/P molar ratios. Removal efficiencies of NH₄-N and PO₄-P at 120 min were 11.8–20.4% and 62.5–96.4%, respectively, with the removal

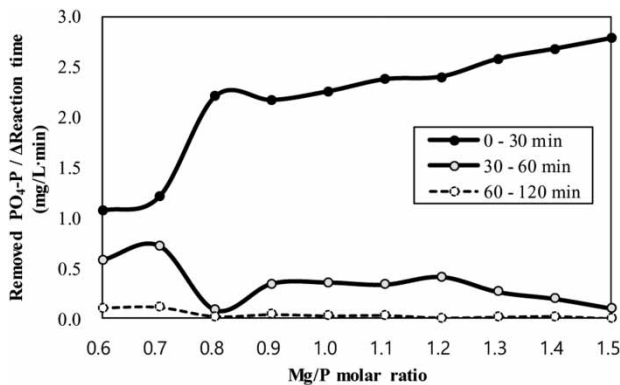


Figure 4 | Removed PO₄-P considering the reaction time in batch experiments.

Table 1 | Characteristics of digested sludge filtrate

Parameter	Concentration range
pH	7.4–7.8
T-Alkalinity, mg/L as CaCO ₃	945–1195
SS, mg/L	153–300
Mg ²⁺ , mg/L	17.8–38.9
NH ₄ -N, mg/L	251.4–324.3
PO ₄ -P, mg/L	77.6–144.7

efficiency of PO₄-P reaching about 90% at an Mg/P ratio of 1.0, HRT of 120 min and pH value of 8.37.

Figure 4 shows the PO₄-P removal rate (mg/L·min) against reaction time (0–30, 30–60, 60–120 min). When Mg/P ratios were 0.6–0.7, both residual Mg²⁺ and PO₄-P were high at 30 min; because dissolved Mg²⁺ was not enough to match the PO₄-P, the reaction rate was slow. The removal efficiencies of PO₄-P were 62.5–72.2%. When Mg/P ratios were in the 0.9–1.2 range, PO₄-P was removed quickly within 60 min and the removal rate then slowed down until 120 min; removal efficiencies of PO₄-P reached the range of 87.1–95.0%. When the Mg/P ratios were 1.3–1.5, the removal efficiencies of PO₄-P were satisfactory and residual Mg²⁺ was higher than 35 mg/L. The optimal operating conditions were observed to be Mg/P molar ratios of 1.0–1.2, pH of 8.25–8.5, and HRT greater than 60 min. Under these conditions, it was possible to remove more than 90% PO₄-P and maintain residual Mg²⁺ concentrations lower than 35 mg/L.

Operation results of the struvite crystallization pilot plant

The influent was the digested sludge filtrate typically containing 17.8–38.9 mg/L of Mg²⁺, 251.4–324.3 mg/L of NH₄-N, and 77.6–144.7 mg/L of PO₄-P. Table 1 shows the characteristics of the digested sludge filtrate.

For the purpose of evaluating the applicability of using a pH controller with the MgO slurry feeding system, pH values and pH controller on/off conditions were monitored every minute during the operation of the pilot plant. The raw water flowed into the pilot plant at 6.0 L/min (HRT: 1 h), and the MgO slurry (1%) flowrate was 0.5 L/min. Figure 5 shows pH monitoring results at pH values of 8.25 and 8.5. Although it took longer to reach the pH 8.5 set point, the

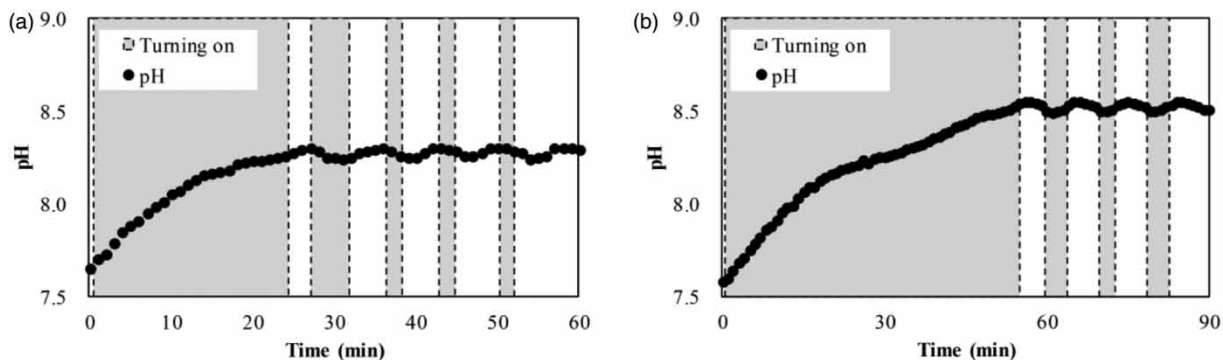


Figure 5 | pH variation and pH controller on/off patterns. (a) pH 8.25, (b) pH 8.5.

pH controller was turned on and off approximately every 5 min repeatedly under both pH conditions.

The struvite pilot plant was operated for 42 h at pH 8.25 and pH 8.5. The influent and effluent were analyzed

every 6 h. The measured parameters were pH, T-alkalinity, and T-P, PO₄-P, T-N, NH₄-N, and Mg²⁺ concentrations. During this period, the pH was adjusted in the range of 8.1–8.4 at the set point of pH 8.25, and 8.3–8.7 at the set

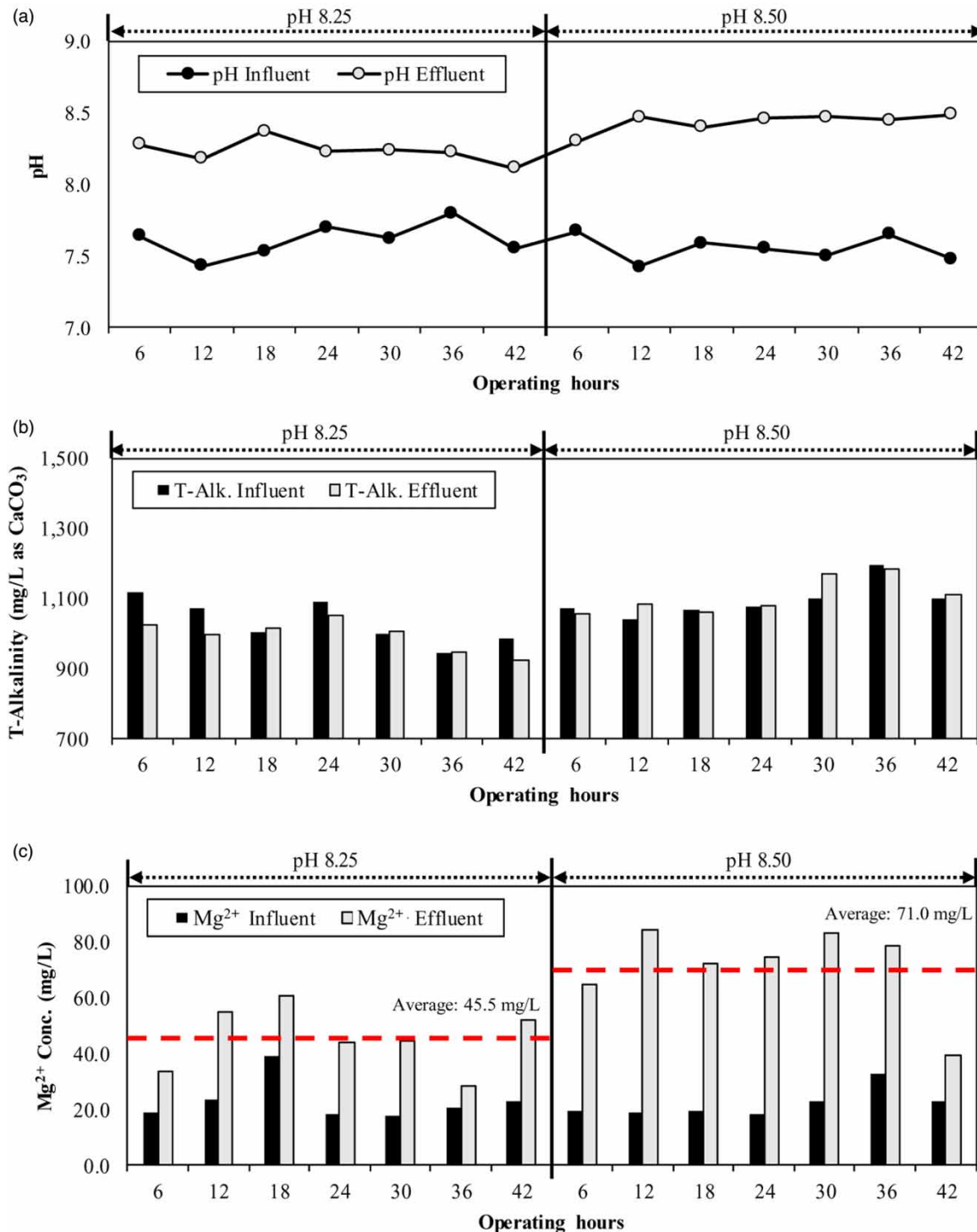


Figure 6 | Operation results at pH 8.25 and pH 8.5. (a) pH, (b) T-alkalinity, and (c) Mg²⁺ concentration.

point of pH 8.5. pH values in the effluent could be adjusted appropriately within a fluctuation range of 0.2 (Figure 6(a)). For reference, other reported commercial plants operating within a pH range of 8.0–9.0 by injecting NaOH or stripping CO₂ (Müller *et al.* 2007; Remy *et al.* 2013) were considered.

The high T-alkalinity of the influent originates from the high bicarbonate concentrations in the digestion process, which is the reason why the T-alkalinity values of the influent and effluent were similar (Figure 6(b)).

The solubility of MgO increases in proportion to the presence of acidic species that offer H⁺ ions. The digested sludge filtrate contains bicarbonate (HCO₃⁻) and ammonium (NH₄⁺) ions as acidic substances that affect the solubility of MgO. It is difficult to maintain a constant residual Mg²⁺ concentration because the bicarbonate and ammonium concentrations in the influent fluctuate. The Mg²⁺ concentration of the effluent was 28.4–60.9 mg/L at pH 8.25 and 35.8–86.3 mg/L at pH 8.5 (Figure 6(c)). Although Mg²⁺

concentrations in the influent fluctuated, the Mg²⁺ concentrations of the effluent were maintained at less than the maximum values of 60.9 and 86.3 mg/L at each pH condition during the continuous 42 h operation. Therefore, the pilot plant was judged to be considerably stable. In addition, this implies that the new MgO feeding method using a pH controller can be a suitable magnesium source for the struvite process. However, residual Mg²⁺ concentrations in the effluent were higher than those in the batch experiments; with the high pH values in the effluent, losses of MgO cannot be avoided. Further studies that consider slurry flowrate and economic feasibility are needed to develop MgO feeding techniques that can maintain precise residual Mg²⁺ concentrations.

The operating results for T-P and PO₄-P are described in Figure 7. The effluent T-P and PO₄-P concentrations were 18.0–49.3 mg/L and 11.1–17.3 mg/L at pH 8.25. Removal efficiencies were 59.2–86.0% and 84.6–90.2%, respectively. At pH 8.5, the effluent T-P and PO₄-P concentrations were

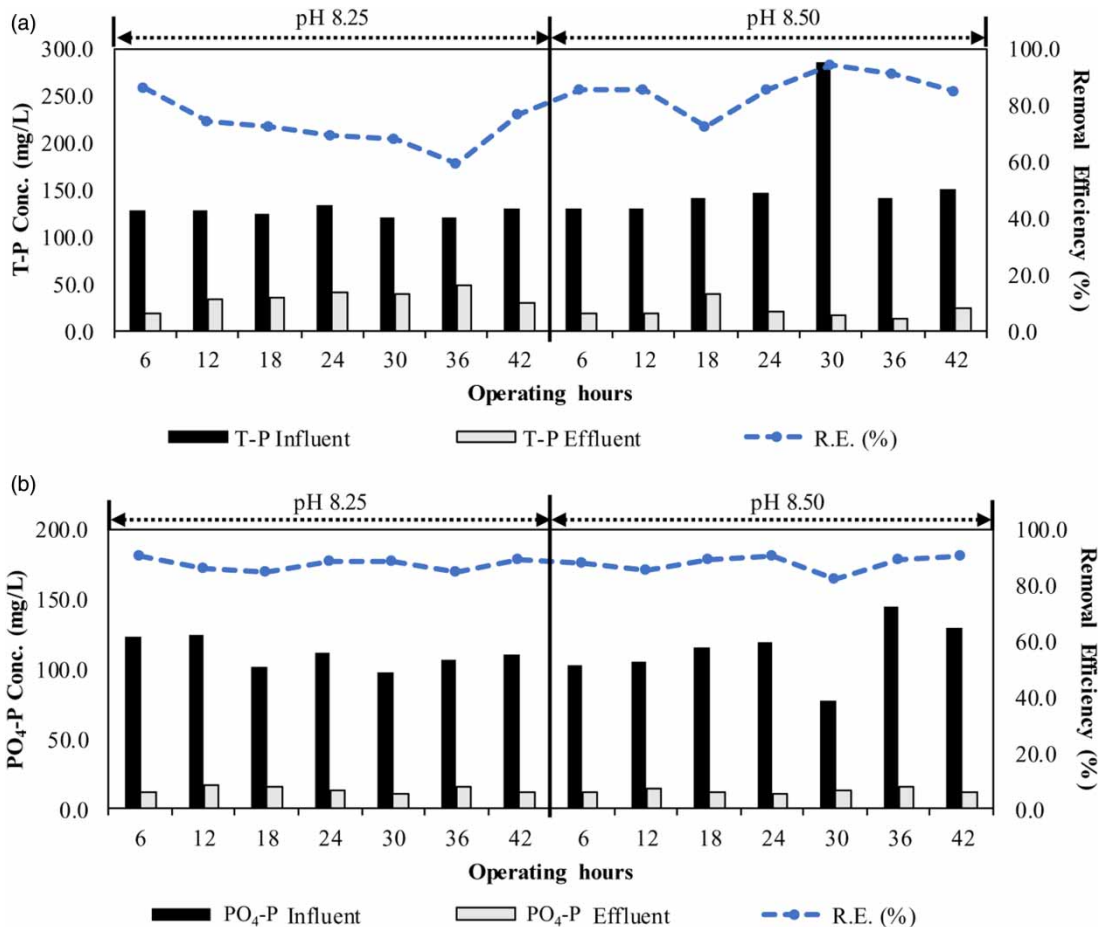


Figure 7 | Operating results at pH 8.25 and pH 8.5. (a) T-P, (b) PO₄-P.

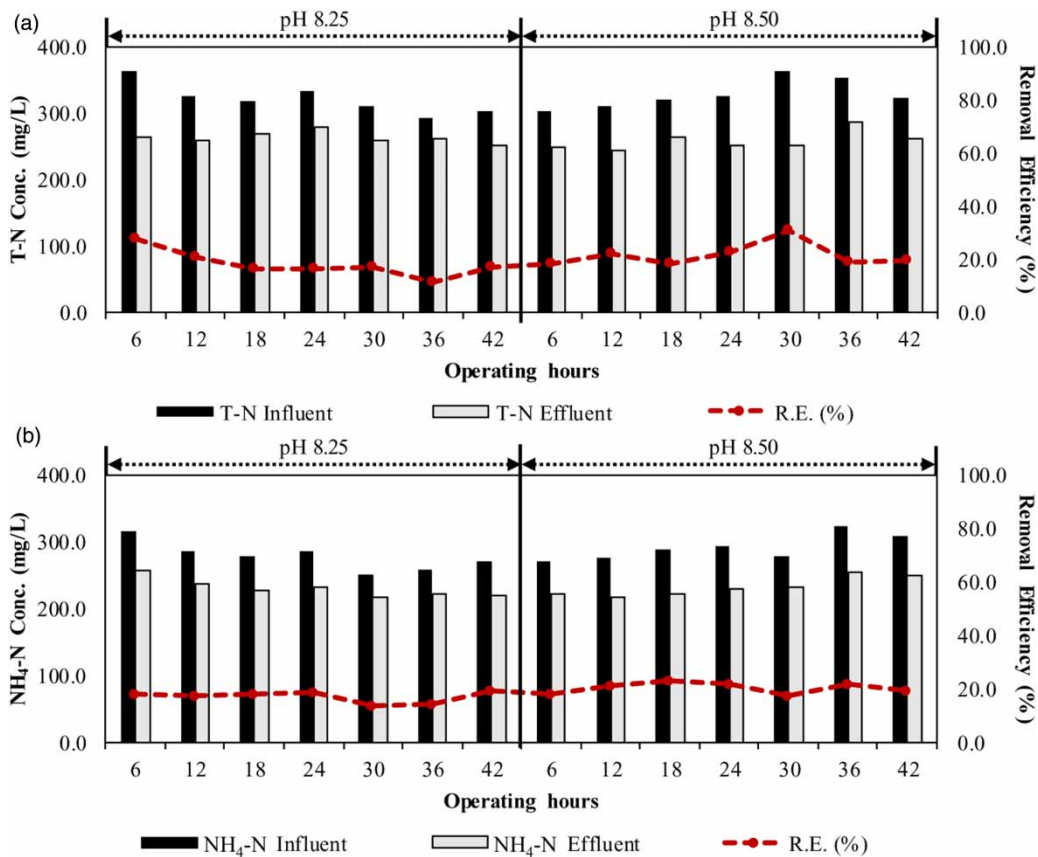


Figure 8 | Operating results at pH 8.25 and pH 8.5. (a) T-N, (b) NH₄-N.

12.3–39.0 mg/L and 11.3–15.7 mg/L, respectively. The removal efficiencies were 72.3–94.4% and 82.5–90.7%. It showed a similar performance to global struvite full-scale plants that showed 80–90% removal efficiency of phosphorus (Ueno & Fujii 2009; Kataki et al. 2016a, 2016b).

The removal efficiencies at pH 8.5 were higher than those at pH 8.25 because more MgO slurry was injected in order to meet the higher pH value, which resulted in more magnesium getting dissolved; the reaction rate and removal efficiencies also increased with high magnesium concentrations.

The T-N and NH₄-N operating results are shown in Figure 8. The effluent T-N and NH₄-N concentrations were 252–279 mg/L and 218–258 mg/L at pH 8.25. Removal efficiencies were 11.0–27.2% and 13.4–19.2%, respectively. At pH 8.5, the effluent T-N and NH₄-N concentrations were 244–287 mg/L and 218–255 mg/L, and the removal efficiencies were 17.8–30.9% and 17.4–22.9%, respectively. When the pH increased to 8.5, the removal efficiencies of nitrogen increased with those of phosphorus due to the rise of the reaction. In contrast, the removal

efficiencies of nitrogen were lower than those of phosphorus because the molar concentration of ammonium (average 0.0198 M) was about 5 times higher than that of phosphate (average 0.0036 M) in the influent. If it is necessary to increase ammonium removal efficiency, additional treatment would be required.

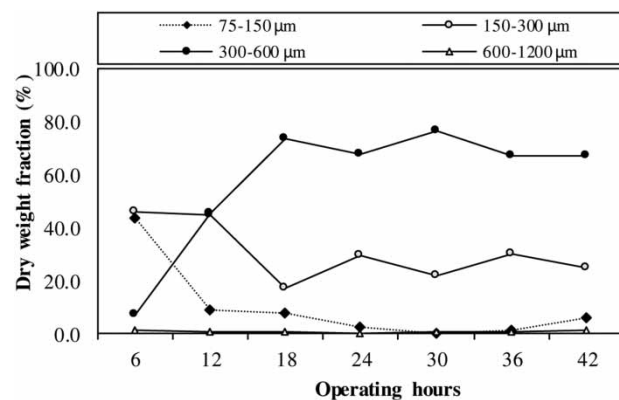


Figure 9 | Dry weight fractions at pH 8.25.

Crystal growth characteristics

To demonstrate the crystal growth characteristics, the pilot plant was operated for 42 h at a pH of 8.25. The dry weight fractions according to sieve sizes are shown in Figure 9. The dry weight fraction of the precipitate in the

300–600 μm range increased gradually from 7.3% to 73.7% within 18 h, and remained consistently above 70% while that of the precipitate in the 150–300 μm range remained above 40% for 12 h and decreased by less than 20%. The fraction of the precipitate in the 75–150 μm range remained above 40% for 6 h and decreased by less than 10%.

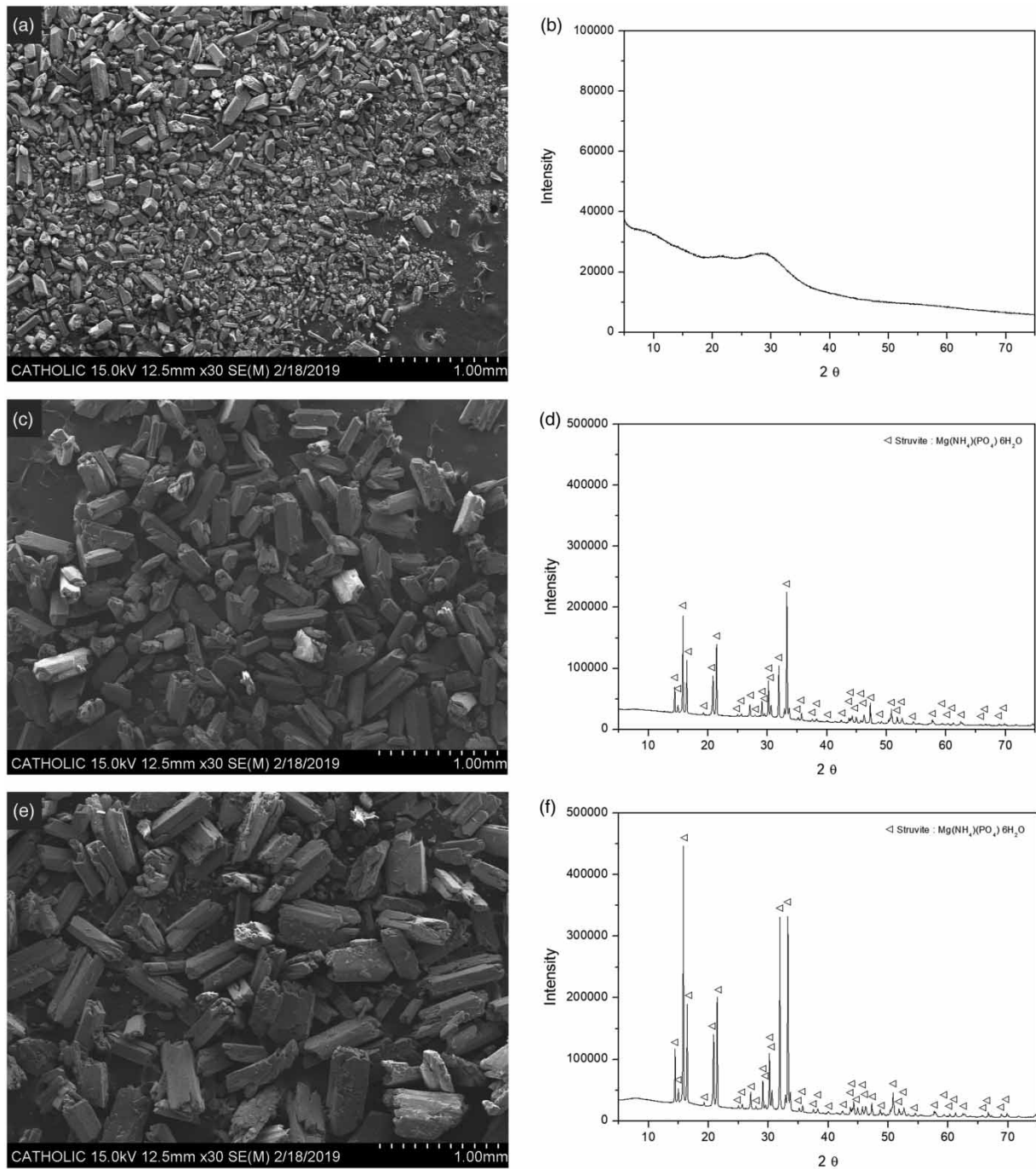


Figure 10 | SEM and XRD results at pH 8.25. (a), (b) 75–150 μm , (c), (d) 150–300 μm , (e), (f) 300–600 μm .

Final precipitates of sizes 75–150, 150–300, and 300–600 μm were analyzed by SEM and XRD (Figure 10). In the SEM image, tiny crystalline structures in the range of 75–150 μm (Figure 10(a)) were observed. However, the XRD results of this range showed 100% amorphous structures (Figure 10(b)). This implies that they were composed predominantly of amorphous particles rather than struvite crystals and hence commercially unviable due to the high percentage of amorphous solids.

When the crystals grew larger than 150 μm , the SEM images showed that the orthorhombic crystalline structures became considerably formed in both size ranges of 150–300 μm and 300–600 μm (Figure 10(c) and 10(e)). There were clear struvite peaks in the XRD results (Figure 10(d) and 10(f)) and peaks of MgO , $\text{Mg}(\text{OH})_2$, and MgCO_3 were not observed. In the range of 300–600 μm , the intensity of struvite was higher than in the 150–300 μm range. Quantitative analysis of the crystals showed that the struvite fraction of crystals was 100% in size ranges of both 150–300 μm and 300–600 μm , and that when the size was larger, the purity of the crystal was higher. Consequently, this investigation indicates that when struvite crystals larger than 300 μm are recovered, high-purity fertilizer can be ensured. To attain the conditions that satisfy this criterion, it takes longer than 18 hours.

CONCLUSIONS

In this study, a struvite crystallization pilot plant was installed and operated with an MgO (magnesium oxide) slurry feeding system combined with a pH controller. The growth characteristics of struvite crystals were investigated intensively throughout the continuous operation of the pilot plant.

First, we conducted batch experiments in order to determine the appropriate pH for the pilot plant, considering the Mg/P molar ratios when the MgO slurry was injected. When the MgO slurry is injected based on the phosphate concentration, the pH can be adjusted and the magnesium supplied appropriately. The optimal MgO injection conditions were approximately a pH range of 8.25–8.5 and a molar ratio Mg/P of 1.0–1.2. Under these conditions, the removal efficiency of $\text{PO}_4\text{-P}$ reached 90%.

The pilot plant was operated for 42 h at pH 8.25 and pH 8.5. The incorporation of the pH controller to the pilot plant along with the MgO supply made it possible to adjust the pH and inject the magnesium accurately. The plant successfully achieved stable phosphorus and nitrogen removal

efficiencies under both pH conditions. The removal efficiencies of $\text{PO}_4\text{-P}$ and $\text{NH}_4\text{-N}$ were 82.5–90.7% and 13.4–22.9%, respectively.

In order to demonstrate struvite crystal growth, dry weight fractions of the precipitates were analyzed according to sieve sizes. It was observed that the precipitate of 300–600 μm was the dominant fraction after 18 h and consistently remained above 70%. The SEM and XRD analysis showed that when the size of crystals became greater than 150 μm , those with orthorhombic crystalline structures became more predominant, showing clear struvite peaks. Consequently, it proves that when struvite crystals larger than 300 μm are recovered after 18 h, high purity of fertilizer can be ensured.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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