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Effect of Seeding Materials and Mixing Strength on Struvite Precipitation

Jun Wang, Joel G. Burken, Xiaoqi (Jackie) Zhang

ABSTRACT: Struvite precipitation has increasing interest as a technology for removing and recovering phosphorus from wastewater streams. Many chemical factors have been studied, such as optimum pH values and component-ion molar ratios, yet, understanding of physical aspects is lacking. Two physical parameters were tested: (1) seeding material addition and (2) mixing. Objectives were to evaluate three seeding materials and to optimize mixing conditions for struvite-crystal precipitation, growth, and subsequent sedimentation. Results confirm that mixing strength and proper seeding materials increase crystal size and improve settleability. For unseeded solutions, optimum phosphorus removal was achieved at a mixing strength of $G = 76 s⁻¹$. Struvite crystals that were added as the seeding material provided the best performance with respect to phosphorus removal and crystal-size distribution. Overall, this study provided information to improve the practical application of struvite precipitation as a phosphorous-treatment technology for wastewaters, while generating a marketable slow-release fertilizer as a product. Water Environ. Res., 78, 125 (2006).

KEYWORDS: struvite precipitation, seeding materials, phosphorus removal, swine waste.

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Introduction

Phosphorous is a limited resource on earth. The resource base of phosphate rock, which can economically be exploited, could be depleted in as little as 50 years (Driver et al. 1999). If phosphorus can be recovered and recycled, not only could nutrient enrichment of rivers be prevented, but the availability of a finite resource could also be lengthened. Struvite precipitation process is a technology that not only achieves high phosphorus removal, but also recycles phosphorus by producing a slow-release fertilizer, struvite.

Precipitation of struvite (MgNH₄PO₄ \bullet 6H₂O), also known as magnesium ammonium phosphate (MAP), was first noted in the Hyperion wastewater treatment plant in Los Angeles, California, in the 1960s. Struvite crystals formed in the digested sludge lines and separating screens caused operational problems by clogging pipes or freezing valves (Borgerding, 1972). In wastewater treatment, struvite precipitation can lead to capacity loss by compromising process control and negatively affect the performance of the sludge and filtrate conveyance system (Benisch et al., 2002). Struvite has also been found in anaerobic sludge from liquid animal-production wastes (Booram et al., 1975). Although unintentional struvite formation has long been known to be a considerable nuisance in wastewater treatment plants (Munch and Barr, 2001), struvite precipitation can also serve as a process for removing and recovering phosphorus in wastewater streams if the precipitation can be controlled to the desired locations by optimizing physical conditions and chemistry. The struvite precipitation process has several advantages compared to other phosphorus removal technologies. First, the process removes two nutrients (phosphorus and nitrogen) simultaneously; second, this process is well-suited to wastewaters with concentrated nutrients (Dempsey, 1997); and third, the process produces a premium slow-release fertilizer by recycling phosphorus resources (Munch and Barr, 2001). The struvite process is essentially a ''waste–to-product'' conversion.

Many factors contributing to struvite formation have been reported, such as carbon dioxide release resulting from pressure decrease, pipeline roughness, high surface-area-to-volume ratio, metal fittings, and grit precipitation in flat pipelines (Booram et al., 1975; Borgerding, 1972). Although these factors may be variables, at lease four prerequisites are necessary for effective struvite precipitation: (1) all constituent ions must be at or above appropriate molar concentrations and ratios; (2) pH must be appropriate to allow supersaturated solution; (3) the existence of rough surface or seeding materials to provide crystal growth sites; and (4) mixing energy must be adequate to promote crystal growth. Operational pH values and component ratios have been investigated by numerous researchers, producing a range of values from 8.0 to 10.7 (Booker et al., 1999; Booram et al., 1975; Buchanan et al., 1994; Momberg and Oellermann, 1992; Ohlinger et al., 1998; Stumm and Morgan, 1996). One study on optimization suggested that pH values should be higher than 8.5 and an excess of ammonia (30 to 80 ppm) after a reaction would form relatively pure struvite (Stratful et al., 2001), while others found that pH influenced both the amount and composition of the precipitate and excess amounts of magnesium decreases struvite purity (Demeestere et al., 2001). Recent research predicts that phosphate-removal performance is optimal when the pH is 9.0 and the ratio of magnesium to total phosphorus is 1.6 (Burns et al., 2001). In similar research, a magnesium-to-phosphorus ratio of 1.3 and a pH value of 8.5 were the ideal conditions for struvite formation (Munch and Barr, 2001).

Seeding materials and mixing energy are also important in struvite precipitation. Even when the pH and component-ion-molar ratios are appropriate, precipitation may cease before true equilibrium is reached because of poorly crystallized or formation of amorphous precipitates initially, resulting in increased solubilities. Reconstitution of such amorphous solids into crystalline solids occurs slowly and requires considerable energy (Sawyer et al., 2003). Crystals formed are uniformly small, ranging from 10 to 100 µm. Instances where larger crystals have formed (approximately $400 \mu m$) often require a considerable length of time to grow, generally on the order of days and weeks (Durrant et al.,1999). In designed struviteprecipitation processes, larger crystals are preferred, as finer solids require longer settling time. To optimize the process regarding crystal formation and growth in a reasonably sized reactor, effects of mixing intensity and seed material on struvite precipitation and settleability must be better understood.

To get a better understanding of the correlation between solids' diameter and their settling velocity in water, Stokes's law (1) and Newton's law (2) were used to calculate the theoretical settling velocity of particles in pure water, revealing an exponential relationship between the crystal size and its settling velocities. Crystals of 200 μ m diameter settle 16 times faster than crystals 50 μ m in diameter. Settleability and removal efficiency can be greatly improved by increasing precipitate size. However, such ''quick'' precipitation cannot be expected in a practical situation, because the calculation was based on several assumptions: (1) precipitation happens in pure water; (2) the process follows type I precipitation; and (3) the solids are spherical in shape. In reality, constructed systems deviate from these assumptions, to the detriment of settling efficiency. The dynamic viscosity coefficient of real wastewater is higher than that of pure water, and struvite crystal has a typical trapezoidal shape (Munch and Barr, 2001). Moreover, upflow velocity and turbulence caused by mixing in the reactors negatively effect settling. Therefore, the crystals should be orders of magnitude larger than the calculated values to enable efficient removal.

$$
V = \frac{g(\rho_p - \rho_f)d_p^2}{18\mu} \quad \text{(Stokes' law)}\tag{1}
$$

Where

- $V =$ terminal settling velocity for particle,
- $g =$ gravity constant,

 ρ_p = particle density,

- p_f = fluid density,
- d_p = particle diameter, and
- μ = dynamic viscosity.

$$
V = \left(\frac{4d_p g(\rho_p - \rho_f)}{3C_D \rho_f}\right)^{1/2} \quad \text{(Newton's law)}\tag{2}
$$

Where

 C_D = Coefficient of drag.

Table 2—Physical characteristics of three seeding materials used.

Table 3–-Operation parameters used in the experiment (experiments 1 to 6 were repeated for each seeding material; air mixing time and settling time are 10 and 30 minutes, respectively).

Crystal formation can be divided into two stages: nucleation and growth. Nucleation is the first stage, in which constituent ions combine to form crystal embryos. Crystal growth is the second stage, as crystals grow continuously until chemical equilibrium is reached (Ohlinger et al., 1999). Struvite will not precipitate without a nucleus; however, nucleation is unnecessary if a solution is already ''seeded'' (i.e., adding materials to serve as embryos for crystal growth). In the case of treated wastewater streams, because of lacking seeds in liquids, struvite can preferentially grow on surface of tanks, pipes, and equipment, which act as embryos (Benisch et al., 2002). Crystal growth rate on existing nucleation sites is primarily controlled by mixing energy (Ohlinger et al., 1999). Localized mixing energy differences lead to differences in local growth rates and control struvite accumulation. Such localized high growth rates lead to nuisance struvite formation on valves and flow meters, i.e., high mixing areas, in wastewater treatment plants. Although boosting energy input to the target solution is beneficial in accelerating crystal growth, there is a negative effect of high mixing strength. Excessive mixing has been found to break crystals and result in reduced particle size (Durrant et al.,1999).

Seeding materials and mixing strength are important in optimizing struvite precipitation. Research on these aspects is lacking, however, such as a comparison of struvite crystal growth on a variety of seeding materials and under variable mixing. This paper provides information that may lead to improved struvite precipitation.

Figure 1-Schematic of seeded experiment arrangement.

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Materials and Methods

The effect of mixing strength was studied for both solutions with and without seeding materials. Two mixing methods were used in the experiments: air mixing and mechanical mixing. Mechanical mixing is very easy to evaluate in experimental systems, as multiple G values can be quickly quantified and tested. Air mixing provides the benefit of no mechanical mixers in the solutions. Therefore, struvite precipitation has no effect on the mixing. Precipitation on impellers can change the rotation to mixing strength relationships.

Solution Preparation. Synthetic wastewater solution was formulated to mimic typical magnesium (Mg), calcium (Ca), ammonium (NH₄-N), and phosphate $(PO₄)$ concentrations as measured in the effluent of anaerobic lagoon treating piggery waste in central Missouri. Solution was prepared with magnesium sulfate heptahydrate (MgSO₄ \bullet 7H₂O), dehydrate calcium chloride (CaCl₂), ammonium chloride (NH4Cl), and ammonium phosphate monobasic $((NH₄)H₂PO₄)$. All chemicals were ACS grade (Fisher Scientific, Pittsburgh, Pennsylvania). The prepared solutions were equilibrated to room temperature in the laboratory for 24 hours before being used. Initial concentrations and molar ratios used in the experiments are listed in Table 1.

Seeding Materials Preparation. In the seeded experiments, three seeding materials were tested: commercialized white quartz sand (Fisher Scientific), granite chips (Encore Plastics, Bellevue, Ohio), and struvite crystals (prepared by crushing and sieving struvite crystals). The selected materials have low solubilities under alkaline conditions, which is necessary for struvite precipitation. Seeding materials were selected to cover a range of densities, to evaluate the effect on phosphate removal under variable mixing strengths. All of these materials were initially graded with a series of American standard sieves and an electrical shaker. Solids, with a diameter ranging from 0 to 75 μ m and 75 to 150 μ m, were sieved and collected respectively. Quartz sand and granite chips were acid

washed with 50% hydrochloride acid, rinsed with deionized water three times, and then dried at 105° C for 12 hours. Physical characteristics of the three seeding materials used in the experiment are listed in Table 2.

Analytic Methods. Magnesium and calcium concentrations were measured by atomic absorption spectrometric method (3500- Mg B) and (3500-Ca B), respectively (APHA et al., 1998). Ammonium concentration was measured by Hach Test' N Tube Method (Hach method 10031), phosphate level was assessed by Hach amino acid method (Hach Method 8178) (Hach Company, 1997). The deposits produced in the experiments were analyzed by X-ray diffraction (XRD) to determine crystal phase. Crystal sizes were measured with optical microscope (Leica Galen III, Exton, Pennsylvania); crystal pictures were taken with video adaptor and digital camera (Kodak, Rochester, New York).

Experiment Setup. In the mixing-strength experiments, a jar tester (Phipps and Bird PB-900, Richmond, Virginia) was used to investigate the effect of mixing strength and operation time on struvite crystal size and phosphate removal. There were no seed materials used to investigate mixing strength as the lone experi-

Figure 2—X-Ray diffraction analysis of deposit sample. A strong match of analysis and the database standard confirm that struvite is the lone crystal phase present.

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Figure 3—Phosphorus removal percentage at various operation times and mixing intensities. All solutions were unseeded.

mental variable. Two liters of synthetic wastewater was transferred into each of four jars. Solutions were adjusted to pH 8.7 with 1.0 M sodium hydroxide (NaOH) (ACS grade, Fisher Scientific). The pH value used in the unseeded experiments was 8.7, as determined by MINEQL+ version 4.5 (Environmental Research Software, Hallowell, Maine). MINEQL required thermodynamic data input regarding struvite precipitation. Input data included the following: struvite pK_{sp} = 12.6, carbon dioxide partial pressure = $10^{-3.5}$ atm, ionic strength = 0.15 M, temperature = 25° C. Mixing strength was adjusted by controlling the blade-rotation rate. Initially, the solution was quickly mixed at high G value $(240 s⁻¹)$ for 30 seconds. Mixing was lowered to a designated value, and the solutions were mixed for 0 to 120 minutes. Following the reaction periods, solutions were filtered with 5-µm membrane filter paper (Millipore Corporation, Billerica, Massachusetts). Filtered deposits were weighed after drying at room temperature for 24 hours. Using optical microscopy, 15 crystals of each sample were randomly selected, and crystal sizes were measured directly under the microscope.

Two methods were applied to determine the deposit composition: elemental analysis and XRD. In the elemental analysis, deposit samples were diluted in 200-mL volumetric flasks; 1 mL of hydrochloric acid (ACS grade, Fisher Scientific) was added to dissolve the solid phase. Then, magnesium, NH_4-N , and PO_4 concentrations were determined with the analytic methods outlined above. The crystals were further analyzed via XRD analysis using a Scintag XDS 2000 diffractometer (Cupertino, California) with a copper (Cu) Kalpha radiation source and a liquid-nitrogen-cooled germanium (Ge) detector. Phase identification used Scintag DMSNT software (version 1.37) in the Material Research Center at the University of Missouri-Rolla.

For each seeding material, the experiments were conducted according to the parameters in Table 3 (experiments 1 to 6). For nonseeded control experiments, the parameters listed in experiments 7 to 9 of Table 3 were adopted. In the experiments, 60 mL of synthetic solution and 0.5 g of seeding materials were transferred to a 125-mL glass-separatory funnel. An Orion 230A plus digital pH meter (Orion Research, Inc., Beverly, Massachusetts) was used to monitor solution pH values. The solution was adjusted to $pH = 9.0$ with 0.1 and 1.0 N NaOH (ACS grade, Fisher Scientific). The pH value maintained in the seeded experiment was 9.0, as published in earlier literature (Burns et al., 2001). The mixture of synthetic solution and seeding materials was mixed with compressed air for 10 minutes with a predetermined air flowrate (Table 3). Air was introduced to the funnel from the bottom (Figure 1). Air mixing ensured material was continually suspended, even at low mixing rates. The mixing strength (G value) was not determined, however.

Figure 4—Microscopic picture of struvite crystals formed at a G value of 0 s⁻¹ and 1 hour of reaction.

Figure 5—Microscopic picture of struvite crystals formed at a G value of 76 s⁻¹ and 1 hour of reaction.

The mixing strength in the funnel was controlled by adjusting air flowrates, which was monitored and controlled with a gas flowmeter (Cole-Parmer, Vernon Hills, Illinois). In all the experiments, the settling time was 30 minutes. After the settling, 15 mL of supernatant were withdrawn, and 5 mL were filtered with a 0.45 - μ m syringe filter, 5 mL were filtered with 0.20 µm, and the reminder was unfiltered. The filtered and unfiltered samples were transferred to 100-mL volumetric flasks, respectively. The flasks were filled to 100 mL with deionized water. After vigorous shaking for 1 minute, magnesium, calcium, NH_4-N , and PO_4 concentrations were analyzed separately (Figure 1).

Results and Discussion

Deposit Composition Determination. Results of elementary analysis of the deposit showed that the measured values are very close to the theoretical ones. An approximate ratio among magnesium, NH_4 -N, and PO_4 concentrations was $1.0:0.7:1.0$ (Table 4). According to the MINEQL + model, struvite is the only precipitate that has a $1:1$ ratio of magnesium and PO_4 in all possible deposit forms in the experiment setting. The samples were further analyzed with XRD (Figure 2). Results overlaid with the database standard revealed that the sample is qualitatively single phase, and struvite is the only crystalline phase. Because of incomplete grinding of the samples, some preferred orientation effects were observed. Combining results from two assay methods, the deposit is confirmed as high-purity struvite crystals. Sample purity is closely tied to operational pH also. At different pH values, by products other than struvite may be formed from ions in solution, such as brucite $[Mg(OH)_2]$, hydroxylapatite [Ca₅(PO₄)₃OH], octacalcium phosphate [Ca₄H(PO₄)₃ \bullet 3H₂O], and

dicalcium phosphate dihydrate [CaHPO₄ \bullet 2H₂O]. The analytic findings were in agreement with thermodynamic modeling. Ion concentrations in Table 1 were input into MINEQL. Under this condition, struvite precipitation potentials reach its maximum and other precipitates are still undersaturated.

Unseeded Experiment. From the results of the jar test shown in Figure 3, mechanical mixing had a clear affect on phosphorusremoval efficiencies. After a 1-hour reaction time, without mixing, only 7.7% of phosphate was intercepted by 5-µm filter paper. With increased mixing energy, a higher phosphorus removal percentage was achieved (80.2 and 91.9% for 33 and 76 s^{-1} , respectively). No significant increase in phosphorus removal was obtained when the mixing strength was greater than 76 s^{-1} .

Microscopic examination of the deposits revealed that the crystals formed at G values of 76 s^{-1} or lower have considerably larger sizes than the others (Figures 4 to 7). This phenomenon is consistent with crystal-growth theory: mixing energy causes suspension of particles and increases the collisions; hence, increasing crystal size and resulting in enhanced settling and increased sedimentation. Excessive mixing strength exposes larger crystals to the shear force caused by strong mixing. The large crystals are broken down, and the settleability of the precipitate and phosphorus removal efficiency is decreased. From the experiment results, the optimum mixing strength is approximately 76 s^{-1} for unseeded synthetic animal lagoon wastewater. Under these conditions, struvite crystals can grow to an average size of $350 \pm 32 \,\text{\mu m}$ (95% confidence interval) in 1 hour, as shown in Figures 4 to 7.

Operation time was important to phosphorus removal at low mixing strengths. Phosphorus removal increased 152 and 11.5%, when the operation time increased from 1 to 2 hours and the G values were 0 and 33 s^{-1} , respectively. At higher mixing intensities,

Figure 6—Microscopic picture of struvite crystals formed at a G value of 100 s⁻¹ and 1 hour of reaction.

however, the precipitation process reached equilibrium quickly. Microscopic analysis revealed no significant crystal growth after 1 hour, when G values were higher than 76 s^{-1} (Figures 5 to 7). The results suggested two important points: (1) appropriate mixing strength can significantly reduce time needed for struvite crystal growth and cut down the footprint of the reactor needed; (2) an operation time of 1 hour or less is adequate, as no notable improvement regarding crystal growth was observed beyond it.

Seeded Experiment. In the seeded experiments, mixing strength also affected phosphate-removal efficiency for all three seeding materials studied and unseeded solutions (control). All seeding materials increased precipitation and phosphorus removal. Solutions with seeding materials achieved 22.9 to 82.6% phosphorus removal, while those without showed only 8.1 to 13.9 % phosphorus removal under the same operation conditions (Figure 8). For the unseeded solutions, the phosphorus removal efficiency was 10.5% under low mixing strength (mixing-air flowrate 200 mL/min), increased to 13.9% with medium mixing (300 mL/min), and declined to 8.1% at high mixing (400 mL/min). Experimental results of the seeded solution experiments showed a similar trend.

The medium mixing strength (300 mL/min) was optimum for the seeding materials less than $75 \mu m$ (Figure 8). Greater mixing improves seed materials suspension; however, the risk of crystal breakage and smaller resultant crystals increases. The implication is that each specific seeding material performance can be optimized with mixing intensity for the enhanced phosphorus removal and low energy consumption. Combined seed-and-mixing optimization is critical in the practical operation of struvite crystallization processes.

Among the seeding materials tested, struvite crystals had the best phosphorus removal performance compared with granite and quartz

sand, likely because of physical characteristics. Struvite has a low specific gravity relative to the other seed materials and, thus, has more particles and greater surface area than the other two at the same total mass addition. The results in all experiments are consistent with this explanation: quartz sand has a higher phosphorus-removal efficiency than granite, which has higher specific gravity. Struvite is also better suspended under lower mixing strength; therefore, optimum mixing intensity is lower for struvite than others. Thus, the risk of crystal breakage is reduced at the lower mixing.

Precipitation and settling was enhanced by seed addition, as was revealed in filtration analysis (Figure 9). For the seeded samples (1 to 6), phosphorus removal enhancement ranged from 3.7 to 8.9% and 7.3 to 20.9% after filtering through 0.45- μ m and 0.20- μ m pore-size filter paper, respectively, showing that almost all struvite crystals were larger than $0.45 \mu m$. For the unseeded sample (7) , the enhancement is ten times greater: 129 and 309%, respectively. These results testified that, without seeding materials, the struvite crystals formed in 10 minutes of mixing and 30 minutes of settling mostly exist as colloidal particles, even though the chemical equilibrium was achieved. According to the calculation, the theoretical settling velocities of particles of 0.45 µm and 0.25 µm in diameter are 8.7×10^{-8} and 2.7×10^{-8} m/s, respectively. Such low settling velocities make sedimentation of these particles negligible, and filtration of such fine particles is also infeasible in practical application. These results suggest that struvite formed without proper seed material will not be removable via sedimentation.

Comparison between Seeded and Unseeded Experiments. The different methods and parameters used (shown in Table 5) make quantitative comparison of all results from unseeded and seeded experiments difficult. However, results provide evidence that seeding materials clearly improved the settling characteristics to make phosphorus removal via struvite precipitation more efficient.

Figure 7—Microscopic picture of struvite crystals formed at a G value of 145 s^{-1} and 1 hour of reaction.

For a reactor of 5.0 m height, particles would theoretically need to be larger than $100 \mu m$ to enable extraction within 30 minutes of settling. Considering the discrepancies of the calculation assumptions and the real conditions mentioned above, struvite sizes must be orders of magnitude larger than the minimum sizes calculated to enable successful extraction. Without seeding materials and optimizing the crystal-growth condition, producing such large solids is likely infeasible within reasonable operation time. There-

Figure 8—Phosphorus removal comparison of seeded and non-seeded solutions at various air mixing rates. Experimental parameters of seeded solutions refer to experiments 1 to 3 in Table 3, and seeding materials are less than 75 μ m. Unseeded experiments refer to experiments 7 to 9 in Table 3.

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Figure 9—Comparison of phosphorus-removal efficiency of filtrated (0.45- and 0.20- μ m) and unfiltered samples. Solutions were seeded with struvite crystals, and experimental parameters refer to experiments 1 to 6 and 7 in Table 3.

fore, seeding material is a crucial factor in making struvite precipitation process viable for treating wastewater.

Conclusions

Seeding materials can greatly help increase crystal size in struvite precipitation process. Among three seeding materials tested, struvite powder has the best performance in increasing crystal size and phosphorus removal efficiency. In this study, solution seeded with struvite powder (0 to 75 µm in diameter) achieved 5 times higher phosphorusremoval efficiency than the unseeded solution, under the same conditions. These results also suggest that self-seeding methods may provide sustainable operational methods for practical application.

Mixing strength has a significant effect on struvite crystal sizes and efficient settling. For unseeded-solutions-batch experiments, a mixing strength of 75 s^{-1} provided the optimum condition for crystal growth. The struvite crystal grew to 350 ± 32 µm (95%) confidence interval) in 1 hour. Operation time beyond 1 hour has no apparent effect on phosphorus removal. Appropriate seeding materials plus optimized mixing strength can greatly help struvitecrystal growth and reduce the reaction time for optimizing designs and practical applications.

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Table 5–-Comparison between unseeded and seeded experiments.

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