

**EFFECT OF ANAEROBIC DIGESTION ON STRUVITE PRODUCTION FOR
NUTRIENT REMOVAL FROM SWINE WASTE PRIOR TO LAND APPLICATION**

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Summary: Swine production facilities are becoming larger in size and are concentrated in smaller geographical areas. Additionally, there is an increasing trend to base manure land application rates on crop phosphorus requirements rather than crop nitrogen requirements alone. These factors have resulted in a need to find a feasible method for removing available phosphorus from swine waste prior to land application. Concentrating and transporting excess phosphorus to phosphorus deficient cropping areas may be economically viable. Forced precipitation of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is one option for phosphorus concentration and removal. Using raw swine manure, testing has demonstrated that up to 92% of the reactive phosphorus (PO_4^{3-}) can be removed by the addition of Mg^{2+} to the waste in the form of MgO and by heating to 35 °C to force the formation of struvite. Up to 98% of the reactive phosphorus (PO_4^{3-}) can be removed by using the same procedure with anaerobically digested swine manure.

Keywords: Struvite formation, Phosphorus removal, Swine waste, Anaerobic digestion

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Introduction

The increasing size of U.S. swine operations coupled with the encroachment of suburbia have decreased producers' abilities to land apply manure at environmentally sound rates. At the same time, economic pressures and labor constraints have encouraged producers to land apply manure at higher than agronomic rates (Sutton et al., 1995). In the past, regulations have required that manure application rates be based on nitrogen due to the mobility of nitrate-nitrogen in soil (Sutton et al., 1995). As a result of long-term land application practices for field fertilization and livestock waste disposal, elevated levels of phosphorus exist in many U.S. locations (Daniel et al., 1998). Elevated soil phosphorus levels may increase the potential for surface water eutrophication in some areas of the country. This concern has led to an increasing trend for state regulatory agencies to base their manure management planning requirements on nitrogen and phosphorus in an attempt to balance system inputs and outputs (Schmitt, 1998).

Land application regulations based on both phosphorus and nitrogen, rather than solely on nitrogen, can severely limit swine manure application rates. Switching from a crop uptake nitrogen based application rate, to a crop uptake phosphorus based application rate, could increase land area requirements by a factor of 4 - 8 times (Burns et al., 1998). A cost effective approach to reduce phosphorus levels prior to land application could greatly reduce land requirements for manure disposal.

One approach to removing phosphorus from swine manure prior to land application is the forced precipitation of magnesium ammonium phosphate hexahydrate ($\text{MgNH}_4\text{PO}_4\cdot 6\text{H}_2\text{O}$), commonly called struvite. Struvite is composed of equimolar concentrations of magnesium (Mg^{2+}), ammonium (NH_4), and phosphate (PO_4^{3-}). Struvite is an undesirable mineral frequently formed in recycle flush animal waste management systems. Formation of struvite in flush water recycle pipes has been problematic in liquid manure handling systems because it creates blockages (Booram et al., 1975; Buchanan, 1993; Westerman et al., 1985). Therefore, a large portion of struvite research has been directed towards removal and prevention of struvite formation rather than towards forced precipitation from solution. However, struvite has been found to be a good plant nutrient source for nitrogen and phosphorus since it releases them slowly and has non-burning features because of its low solubility in water (Buchanan, 1993; Salutsky et al., 1970).

Struvite formation occurs when a solution is supersaturated with the ions Mg^{2+} , NH_4^+ , and PO_4^{3-} . Buchanan (1993) hypothesized that availability of these ions in solution increases following the microbial digestion of livestock wastes. Therefore, anaerobic digestion as a precursor to formation of the mineral may enhance struvite formation by bringing more of the reaction limiting ion into solution. In animal waste, the limiting constituent is often Mg^{2+} .

Salutsky (1970) determined that increasing the pH of the solution above 7 enhanced struvite formation and that the optimal pH for mineral formation was between 9 and 9.5. The elevated pH increases struvite formation because orthophosphate, the form of phosphorus necessary for struvite formation, does not exist in an acidic environment. However, if the pH exceeds 10, NH_4^+ is unavailable for reaction. Struvite formation can also be enhanced by supplementing the solution with the limiting ion, thereby keeping it supersaturated. In an attempt to better understand mineral formation prevention in recycle flush systems, Westerman et al. (1985) produced struvite with swine lagoon liquid effluent in their lab. They were able to form struvite by adjusting the pH and by adding either magnesium or phosphorus. The critical low nutrient limit for mineral formation was found by Westerman et al to be 30 mg L^{-1} of phosphorus and 15 mg L^{-1} of magnesium.

Enhancement of struvite formation to recover and concentrate nitrogen and phosphorus for transport has been used in some sewage treatment systems (Liberti et al., 1986; Salutsky et al., 1970). Salutsky et al. (1970) studied the feasibility of recovering phosphorus from anaerobic digestion supernatant sewage sludge to be sold as a fertilizer ingredient. They reported the solubility of struvite in water at 25°C to be 160 mg L⁻¹ (Salutsky et al., 1970). Salutsky et al., (1970) precipitated struvite by adding magnesia (a magnesium salt) and a soluble base for pH adjustment; they were able to recover greater than 90% of the phosphorus. Liberti et al. (1986) operated a mineral precipitating pilot plant using the RIM-NUT process to form struvite and thus recover phosphorus from solution. The RIM-NUT process used suitable ion exchangers to remove constituent ions selectively onto resins. The ions were then mixed and added with magnesium salts to obtain a stoichiometric ratio of Mg:NH₄:PO₄ = 1:1:1 and hence precipitated struvite.

For a swine producer to implement the precipitation of struvite prior to land application of manure slurry, the process must be economically feasible. A theoretical benefit/cost factor was calculated based on the stoichiometry of the reaction. This factor was calculated assuming that all of the magnesium needed to form struvite must be added as MgO at a cost of \$396 (metric ton)⁻¹ (\$360 (ton)⁻¹) (Speece, 1996). The struvite was assumed to have a fertilizer equivalency of 5.7 - 28.9 - 0 (N - P - K) and a value of \$206 (metric ton)⁻¹ (\$187 (ton)⁻¹) based on a nitrogen value of \$ 0.70 kg⁻¹ and a phosphorus value of \$ 0.57 kg⁻¹ (\$ 0.32 lb⁻¹ N and \$ 0.26 lb⁻¹ P). A benefit to cost ratio of 3.2 : 1 was calculated based on the moles of MgO required to produce one mole of struvite. This benefit cost factor does not include any cost of production other than the cost of MgO, but suggests that forced precipitation appears favorable enough economically to pursue. Making the assumption that all of the orthophosphate produced at a swine operation is force precipitated as struvite suggests that a swine facility could produce 113 kg struvite (1000 kg live animal weight-yr)⁻¹ (113 lb struvite (1000 lbs live animal weight-yr)⁻¹).

The objective of this research was to determine the feasibility of forced struvite production in swine waste as a viable phosphorus removal method. Additionally, the hypothesis that anaerobic digestion enhances the formation of struvite formation was tested. Several different reaction methods were tested in order to optimize the removal of reactive phosphorus in the PO₄³⁻ form. The variables in our process were pH, magnesium concentration, and waste pre-treatment (raw or anaerobically digested waste).

Preliminary experiments were performed to determine the chemical pH adjuster (sodium hydroxide, sodium bicarbonate, hydrated lime or magnesium oxide), the reaction temperature (25°C or 35°C), the reaction mixing time (15 min or 30 min), and the reaction pH (between 7 and 9.5). For the struvite formation reactions used to test the hypothesis in question, a mixing time of 15 min at a temperature of 35°C was decided upon based on the results of the preliminary tests. It was also determined that the addition of MgO was the most economical pH adjustment material, based both on cost and the fact that it provided magnesium for the reaction. The MgO proved to be effective at increasing the pH of the swine waste slurry to approximately 8.5. It was difficult to reach a pH greater than 8.5 with MgO due to its limited solubility. To test the effect of pH above 8.5, sodium hydroxide (NaOH) was added in addition to the MgO.

Materials and Methods

The swine waste used in the study was collected from a shallow pit below a slatted floor farrowing house located at the University of Tennessee Knoxville Experiment Station swine unit.

Waste required for the experiment was collected from below the slatted floor using a 1-L dipper. No solids separation was performed on the waste prior to testing. The collected sample was analyzed for reactive phosphorus (PO_4^{3-}), chemical oxygen demand (COD), total solids (TS), and volatile solids (VS). PO_4^{3-} concentrations were determined using a *DIONEX* ion chromatograph (DIONEX Corporation; Sunnyvale, California). COD concentrations were measured using a *HACH* colormetric digestion method (HACH Company; Loveland, Colorado). TS and VS concentrations were analyzed using Standard Method 2540 C & E (APHA, AWWA, WEF).

Waste Pre-treatment

Seven liters of waste were anaerobically digested in a continuously stirred tank reactor (CSTR); this waste stream was considered digested swine waste. The CSTR consisted of a sealed container (gas outlet line submersed in water to maintain anaerobic conditions) mixed at 140 RPM on a stir plate within an environmental chamber held at 40°C. The swine waste was digested for 36 days. After 36 days, 34% of the initial COD concentration of 36,000 mg L⁻¹ was removed and 12% of the initial VS concentration of 14,300 mg L⁻¹ was removed. Struvite tests on the anaerobically digested waste were performed at the end of the 36-day digestion period.

Seven liters of waste were used for the struvite reaction testing of fresh waste upon return from the collection process at the swine unit; this waste stream was considered raw swine waste. The tests were performed immediately in order to preserve the integrity of the waste stream.

Struvite Formation Testing

The struvite formation reaction scenarios were performed in triplicate in 0.5-L glass beakers. Each of the reactions was performed in a randomized order determined by a random number table (Ott, 1992). Efforts were made during the tests to maintain the consistency of the waste by stirring the waste stream throughout the process, and thus prevent settling of solids. The experimental matrices used in this study are shown in Figure 1 and in Figure 2. Figure 1 shows the eight reaction scenarios tested in this experiment. Figure 2 shows the controls tested to help identify the affects the scenarios had on the struvite reactions. For each two tests, waste was poured from a mixed 20-L carboy into a 1-L beaker. The waste was stirred in the 1-L beaker for five min. While mixing, 0.3 L of waste was measured into a graduated cylinder and poured into a 0.5-L glass beaker for each test.

For each test, a specified amount of magnesium oxide (MgO) was added to the 0.3-L sample to increase the magnesium concentration and the pH. Magnesium treatment levels were 0.25 g or 0.50 g of added MgO. After the addition of MgO, the waste was placed on a stir plate and mixed until a stable pH measurement was recorded. As the MgO was added, not all of the compound went into solution due to the low solubility of the chemical. For tests with NaOH addition, the NaOH was added until the pH of the swine waste being tested reached 9.0. Finally, the temperature of the waste was increased to 35°C. Upon reaching the reaction temperature, mixing continued and a 15-min reaction period ensued. After the reaction, the contents were allowed to settle for 15 min. The supernatant was collected off the top of the sample for analysis.

Struvite Formation Test Analysis

After performing the struvite formation reaction tests, collected samples were stored overnight at 4°C. Each sample was analyzed for PO_4^{3-} , Mg^{2+} , $\text{NH}_3\text{-N}$ (the $\text{NH}_3\text{-N}$ analysis method reports both NH_4^+ and NH_3 as NH_3), and TS. Triplicate analyses were performed on

each sample to determine PO_4^{3-} concentration. The results of the triplicate analyses for PO_4^{3-} were averaged for input into the SAS data set. Mg^{2+} concentrations were determined with a *HACH* test for determining water hardness, the calmagite colorimetric method (HACH Company; Loveland, Colorado). $\text{NH}_3\text{-N}$ concentrations were measured with a *HACH* test using the Nessler Method (HACH Company, Loveland, Colorado).

PROC MIXED was used to implement a fixed model for data analysis (SAS Institute, 1997). Fixed effects included in the model were: type of manure (raw or digested), level of MgO addition (0.25 g or 0.50 g), and NaOH addition (No NaOH addition, NaOH addition until pH of 9 was reached). Orthogonal comparisons were made to determine treatment mean differences.

Positive determination of struvite requires the use of x-ray diffraction techniques. While this method was not employed to positively confirm the formation of struvite by the reactions, a white flake-like precipitant was formed during the formation tests; it was assumed to be struvite. The main objective of the project was to measure the overall removal of phosphorus by the reaction, rather than to determine the exact composition of the precipitate. Future work to consider this process for a pilot- or full-scale application should include confirmation of the composition of the precipitate and an evaluation of the value of the precipitant as a fertilizer to provide a more accurate economic assessment of a full-scale application.

Results & Discussion

Results of Statistical Analysis

Pre-reaction Results

The least square means of the initial (before reaction tests) raw and anaerobically digested waste were compared using SAS (table 1). Statistically significant differences were tested for at the $\alpha \leq 0.05$ level and highly significant statistical differences at $\alpha \leq 0.01$. The digested waste had a mean Mg^{2+} concentration 72% greater than the raw waste stream. This difference was found to be highly significant, supporting the hypothesis that anaerobic microbial digestion aids in releasing bound nutrients into an available form. A more complete anaerobic digestion process would be expected to increase nutrient availability for struvite formation. A significant improvement was noted in this experiment with a digestion process that resulted in only a 34% COD removal rate. While the digested waste did have an increased PO_4^{3-} concentration, 21% greater than raw, the difference was not statistically significant. The increased PO_4^{3-} concentration however did support the idea that anaerobic digestion increased phosphorus availability. The difference between the raw waste mean NH_3 concentration and the digested waste mean NH_3 concentration was statistically significant (27% greater NH_3 concentration in the raw waste). This result indicated that more NH_3 volatilization occurred in the digested waste than occurred in the fresh waste. This result was as expected since the digestion process involved heating and stirring the waste. As expected, the digested waste resulted in a lower TS concentration, but the difference was not considered to be statistically significant.

Post-reaction Results

Table 2 shows results comparing the effect of the struvite reaction tests using raw waste and anaerobically digested waste. There was no statistically significant difference in Mg^{2+} or NH_3 concentrations between the reacted raw and reacted digested waste following the struvite formation reactions. The PO_4^{3-} concentration was 39% lower ($p < 0.01$) in the reacted digested waste as compared to the reacted raw waste (table 2).

Table 1. SAS analysis results - raw swine waste vs. anaerobically digested swine waste

	Least Squares Means		Difference Between Means	Standard Error of the Means	†Significance
	Raw	Digested			
PO ₄ ³⁻ (mg L ⁻¹)	1256.2	1591.2	-335.0	190.36	0.1533 NS
Mg ²⁺ (mg L ⁻¹)	88.3	313.3	-225.0	8.0	<0.0001 **
NH ₃ (mg L ⁻¹)	3240.0	2360.0	880	227.5	0.0180 *
TS (mg L ⁻¹)	25120	21417	3703.3	1762.7	0.1035 NS

† (NS) not significant, (*) significant $\alpha \leq 0.05$, (**) highly significant $\alpha \leq 0.01$

Table 2. SAS analysis results comparing post-reaction nutrient concentrations as affected by waste stream pre-treatment

	Least Squares Means		Difference Between Means	Standard Error of the Means	†Significance
	Raw	Digested			
PO ₄ ³⁻ (mg L ⁻¹)	120.5	46.5	74.0	11.9	<0.0001 **
Mg ²⁺ (mg L ⁻¹)	227.4	225.0	2.4	66.1	0.9712 NS
NH ₃ (mg L ⁻¹)	2250.8	2206.7	44.2	102.7	0.6363 NS

† (NS) not significant, (*) significant $\alpha \leq 0.05$, (**) highly significant $\alpha \leq 0.01$

Table 3 shows the SAS results from testing the effect of two MgO treatments, 0.25 g and 0.50 g additions. Based on the initial raw swine waste, 0.25-g MgO addition was calculated to provide a stoichiometrically sufficient amount of Mg to form enough struvite to remove all of the PO₄³⁻ in the waste stream. The 0.50-g addition of MgO was also tested to determine if Mg additions above the calculated stoichiometric needs would provide enhanced PO₄³⁻ removal as struvite. It was thought that some of the supplemented Mg²⁺ could bind with other compounds and would become unavailable to the struvite reaction. The difference in the post-reaction concentration of PO₄³⁻ comparing the addition of 0.50 g MgO and 0.25 g MgO was statistically significant with the 0.50 g MgO treatment providing greater PO₄³⁻ removal. As expected, the Mg²⁺ concentration following the 0.50-g MgO addition reactions was significantly higher when compared to the concentration following the 0.25-g MgO addition reactions. Concentrations of NH₃ following both levels of MgO addition were not significantly different. The SAS results shown in Table 3 indicate that the addition of MgO at both treatment levels significantly enhanced PO₄³⁻ removal.

While equimolar parts of PO₄³⁻, Mg²⁺, and NH₄⁺ are required to form struvite, no statistically significant reduction in NH₃ or Mg²⁺ was noted, though a significant decrease in PO₄³⁻ occurred. However, we believe that reductions in PO₄³⁻ were due to the formation of struvite. The fact that no statistically significant reduction in NH₃ or Mg²⁺ occurred can be explained by the fact that while equimolar concentrations of each nutrient are required to form struvite, stoichiometrically far more PO₄³⁻ than NH₃ or Mg²⁺ is required on a mass basis (5.3 and 4.0 times more by weight, respectively). This, coupled with the fact that the waste stream had a much higher pre-reaction NH₃ concentration than PO₄³⁻ concentration explains the non-significant reduction in NH₃ during PO₄³⁻ removal as struvite. While Mg²⁺ is removed by the struvite formation, the addition of MgO was sufficient to offset this removal, so no significant difference in pre and post reaction Mg²⁺ concentrations was seen.

Table 3. SAS analysis results comparing post-reaction nutrient concentrations as affected by two treatment levels of MgO addition (0.25-g and 0.50-g MgO addition)

	Least Squares Means		Difference Between Means	Standard Error of the Means	†Significance
	0.5 g MgO	0.25 g MgO			
PO ₄ ³⁻ (mg L ⁻¹)	70.1	96.8	-26.7	10.7	0.0219 *
Mg ²⁺ (mg L ⁻¹)	305	147.4	157.6	66.1	0.0278 *
NH ₃ (mg L ⁻¹)	2151.7	2305.8	-154.2	130	0.1092 NS

† (NS) not significant, (*) significant $\alpha \leq 0.05$, (**) highly significant $\alpha \leq 0.01$

The addition or non-addition of NaOH resulted in no significant difference between post-reaction PO₄³⁻ and Mg²⁺. The pH in the struvite reactions utilizing MgO additions ranged from 8.3 – 8.8 depending on which MgO treatment was being tested. A pH of 9 – 9.5 is considered to be optimum for struvite formation (Salutsky et al., 1970). For the NaOH treatment tests, NaOH was added until a pH of 9 was reached in the reaction solutions. The purpose of the NaOH treatment was to determine if improved PO₄³⁻ removal in the 0.50-g MgO treatment as compared to the 0.25-g MgO treatment was the result of increased Mg²⁺ concentrations or of increased pH in the 0.50 g MgO treatment level tests. The SAS analysis results shown in Table 4 indicate that the pH increase gained from the addition of NaOH did not enhance PO₄³⁻ removal. This indicates that the improved PO₄³⁻ removal noted at the higher MgO treatment as shown in Table 3 was due to increase in the Mg²⁺ concentration. However, a significant difference in NH₃ concentrations occurred with or without the addition of NaOH. The increased pH associated with the NaOH treatment may explain the significantly lower NH₃ concentrations, caused by the volatilization of NH₃, while PO₄³⁻ and Mg²⁺ concentrations were unchanged by this treatment. The vapor pressure of NH₃ would be expected to increase due to the conversion of NH₄⁺ to NH₃ with increased pH. The Henry's Law constant, in mole fraction form, for ammonia gas in water increases five fold from a pH of 8 to 9 at 38°C (Thibodeaux, 1979). This provides one possible explanation for the significant decrease in NH₃ with no increase in struvite removal.

Table 4. SAS analysis results comparing post-reaction nutrient concentrations as affected by two treatment levels of NaOH addition (NaOH addition and no NaOH addition)

	Least Squares Means		Difference Between Means	Standard Error of the Means	†Significance
	NaOH	No NaOH			
PO ₄ ³⁻ (mg L ⁻¹)	89.3	77.6	11.7	15.1	0.2855 NS
Mg ²⁺ (mg L ⁻¹)	247.5	204.9	42.6	66.1	0.5273 NS
NH ₃ (mg L ⁻¹)	2125	2332.5	-207.5	91.9	0.0359 *

† (NS) not significant, (*) significant $\alpha \leq 0.05$, (**) highly significant $\alpha \leq 0.01$

Phosphorus Reductions

Table 5 and Figure 3 show the removal of PO₄³⁻ under all of the reaction scenarios. In the struvite formation reactions, phosphorus removals of greater than 90% were achieved in all but one case following the addition of MgO. Across all tests, reactions in the raw swine waste were not as effective at removing PO₄³⁻ as reactions in the digested swine waste. This was attributed to the facts that Mg²⁺ concentrations were significantly higher following anaerobic digestion, and that Mg²⁺ was the nutrient limiting struvite formation in this swine waste stream. It should be noted that settling alone removed 28% and 45% of the measured PO₄³⁻ in the raw and anaerobically digested waste streams, respectively.

Table 5. Mean PO₄³⁻ concentrations and percent PO₄³⁻ reductions in swine waste

	Mean Resulting [PO ₄ ³⁻] (mg L ⁻¹)	Percent PO ₄ ³⁻ Removal
Raw		
Initial	1256.2	N/A
Stirred and Settled	900.7	28 %
Heated, Stirred, and Settled	734.2	42 %
0.5 g MgO / NaOH	127.7	90 %
0.5 g MgO / No NaOH	97.9	92 %
0.25 g MgO / NaOH	151.1	88 %
0.25 g MgO / No NaOH	105.1	92 %
Digested		
Initial	1591.2	N/A
Stirred and Settled	872.4	45 %
Heated, Stirred, and Settled	868.2	45 %
0.5 g MgO / NaOH	27.9	98 %
0.5 g MgO / No NaOH	26.9	98 %
0.25 g MgO / NaOH	50.5	97 %
0.25 g MgO / No NaOH	80.5	95 %

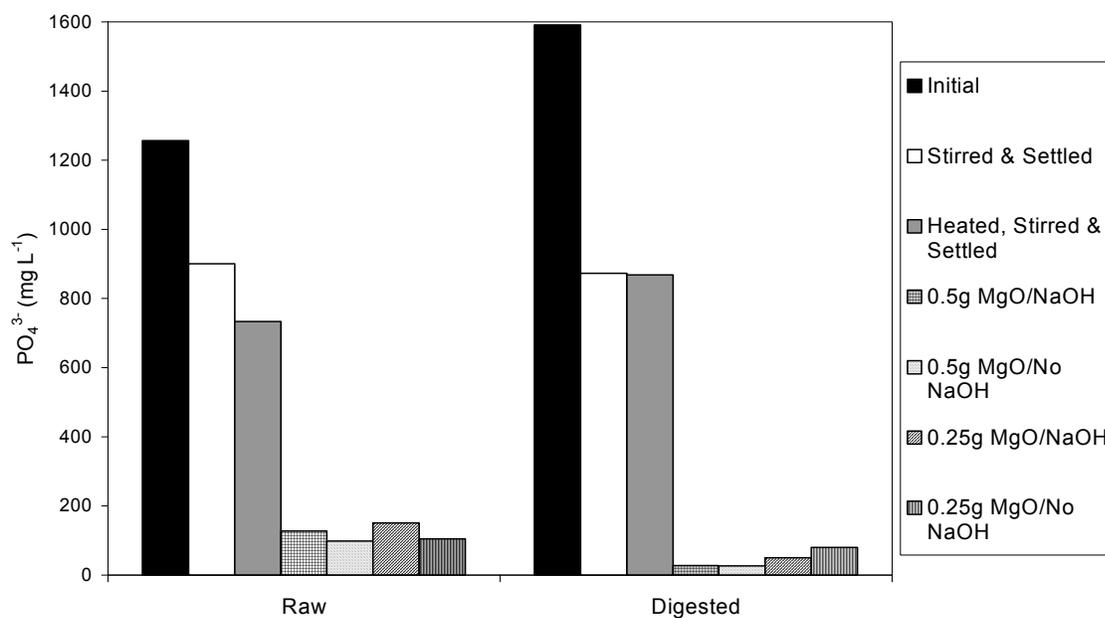


Figure 3. Mean PO₄³⁻ concentrations in swine waste before and after precipitation reactions.

Conclusions

The forced precipitation of struvite is an effective method of concentrating and removing available phosphorus from swine manure prior to land application. This laboratory test demonstrated that at least 88% of the reactive phosphorus could be removed by forcing struvite formation after the addition of MgO. Anaerobic digestion of swine waste prior to the struvite formation reactions enhanced PO_4^{3-} removal. The most effective removal method tested was the addition of 1.67 g L^{-1} of MgO ($0.50 \text{ g (300 ml)}^{-1}$) to anaerobically digested swine waste heated to 35°C . This magnesium addition rate was calculated to be twice the stoichiometric requirement for the waste stream tested. This reaction method yielded a 98% reduction in available phosphorus. The same reaction process using a 0.83 g L^{-1} ($0.25 \text{ g (300 ml)}^{-1}$) addition of MgO provided a 95% available phosphorus reduction. The 3% PO_4^{3-} removal increase gained at the higher MgO addition level was achieved at twice the chemical input cost. For this reason the most feasible scenario tested was considered to be the 0.83-g L^{-1} ($0.25 \text{ g (300 ml)}^{-1}$) addition of MgO to digested swine waste. The 0.83-g L^{-1} ($0.25 \text{ g (300 ml)}^{-1}$) MgO addition represents a chemical cost of $\$0.33 (1000 \text{ L})^{-1}$ ($\$1.25 (1000 \text{ gal})^{-1}$) of swine waste treated. This equates to a recovery cost of $\$0.22 (\text{kg PO}_4^{3-})^{-1}$ ($\$0.10 (\text{lb PO}_4^{3-})^{-1}$) based solely on the cost of MgO at the 0.83-g L^{-1} ($0.25 \text{ g (300 ml)}^{-1}$) addition level.

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