

EXTRACTION OF SOLUBLE PHOSPHORUS FROM SWINE WASTEWATER

M. B. Vanotti, A. A. Szogi, P. G. Hunt

ABSTRACT. Manure phosphorus (P) in excess of the assimilative capacity of land available on farms is an environmental concern often associated with confined livestock production. A wastewater treatment process was developed for removal of phosphorus from livestock wastewater. It includes nitrification of wastewater to remove ammonia and carbonate buffers, and increasing the pH of the nitrified wastewater by adding an alkaline earth metal-containing compound to precipitate phosphorus. Since ammonia nitrogen has been mostly converted to nitrate, increased pH does not result in significant gaseous nitrogen loss. The amount of phosphorus removed, and consequently the N:P ratio of the effluent, can be adjusted in this process to match specific crop needs or remediate sprayfields. In addition to the phosphorus removal aspect, the high pH used in the process destroys pathogens in liquid swine manure. The final product is calcium phosphate, which has the potential to be reused as fertilizer or processed to produce phosphate concentrates.

Keywords. Animal waste treatment, CAFO, Hog lagoons, Manure P, N/P ratio, Pathogens, Phosphorus removal, Sprayfield remediation, Swine wastewater.

Manure nutrients in excess of the assimilative capacity of land available on farms are an environmental concern often associated with confined livestock production. For the U.S. as a whole, in 1997 about 20% of the 929 million pounds of farm-level excess phosphorus (P) exceeded the assimilative capacity at the county level (Kellogg et al., 2000). This means that a substantial amount of manure P needs to be moved at least off the farms and that some needs to be transported longer distances beyond county limits to solve distribution problems of this nutrient.

One of the main problems in sustainability of animal production is the imbalance between nitrogen (N) and P in the waste (Cochran et al., 2000; USEPA, 2001). Nutrients in manure are not present in the same proportion needed by crops. For example, a typical N:P ratio (4:1) in manure is generally lower than the mean N:P ratio (8:1) taken up by major grain and hay crops (USDA, 2001). Therefore, when manure is applied based on a crop's nitrogen requirement, there is a P buildup in soil and increased potential for P losses through runoff and subsequent eutrophication of surface

waters (Edwards and Daniel, 1992; Heathwaite et al., 2000; Sharpley et al., 2000).

Past research efforts on P removal from wastewater using chemical precipitation have been frustrating due to the large chemical demand and limited value of by-products, such as alum and iron sludges, and to both the large chemical demand and large losses of ammonia at the high pH that is required to precipitate phosphorus with calcium (Ca) and magnesium (Mg) salts (Loehr et al., 1973; Westerman and Bicudo, 2000). Another method of removing P from wastewater is through struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) precipitation (Liberti et al., 1986; Wrigley et al., 1992; Burns et al., 2001). Struvite forms from 1:1:1 ratios of Mg, NH_4 , and PO_4 , compared to typical ratios of 1:16:1.2 for liquid manure (Nelson et al., 2000). Addition of MgCl_2 and NaOH (Nelson et al., 2000; Burns et al., 2001) or MgCl_2 and NaOH + phosphate (Liberti et al., 1986) have been used to balance the stoichiometric ratio and optimize struvite precipitation.

Other methods used for phosphorus removal include flocculation and separation of solids using polymer addition and filtration or settling (Vanotti and Hunt, 1999). Although polymer treatment is effective for removal of organic P forms in liquid manure, it is not effective for removal of the soluble P. This presents a problem in systems that use lagoons or other digestion or settling processes where P in the liquid effluent is mostly in the form of soluble phosphates.

Liquid swine manure is a mixture of urine and feces usually stored and treated in anaerobic lagoons. Livestock urine contains >55% of the excreted nitrogen (N); >70% of this N is in the form of urea (Sommer and Husted, 1995). Hydrolysis of urea by the enzyme urease produces ammonium and carbonate, as described in the following equation:



Therefore, a substantial part of the inorganic carbon is produced during decomposition of organic compounds in manure. Ammonia and carbonate alkalinity are the most

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important chemical components in liquid manure contributing to buffering capacity in the high pH range (Fordham and Schwertmann, 1977; Sommer and Husted, 1995), a necessary condition to form P precipitate with Ca and Mg compounds.

Biological removal of N through the process of nitrification is regarded as the most efficient and relatively low-cost means of removing ammonia from wastewater (Tchobanoglous and Burton, 1991). Nitrifying bacteria are present in almost all aerobic biological processes, but their numbers are limited. Many aerobic methods have been developed to promote nitrification. They can be separated into two main groups: suspended growth and attached growth (Grady et al., 1999). In suspended-growth nitrification, a nitrifying sludge composed of free bacteria is mixed with the wastewater liquid by the aeration or agitation of the liquid. The commonly used activated-sludge process is a suspended-growth process combining bacterial BOD and N removal treatments that are performed by separate bacteria in the same tank. In other cases, carbon oxidation and nitrification functions occur in separate tanks. In attached-growth nitrification, the nitrifying bacteria attach to the surface of various media; examples include trickling filters, rotating biological contactors, packed-bed reactors, overland flow, etc.

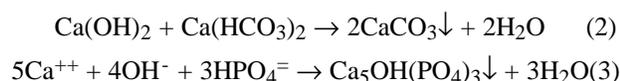
Another type of nitrification system is an intermediate between suspended growth and attached growth; it is a fluidized-bed biological reactor. In this type of reactor, nitrifying pellets remain suspended in the fluid, i.e., fluidized by the drag forces associated with the upward flow of air and water. The nitrifying bacteria are entrapped in polymeric porous materials made of polyvinyl alcohol (PVA) or polyethylene glycol (PEG). One advantage of using such nitrifying pellets is that the number of microorganisms in the reactor can be increased, thus removing the ammonia more quickly (Vanotti and Hunt, 2000). However, whether a faster fluidized-bed biological reactor or a slower suspended-growth process is used, the changes in water characteristics after nitrification are the same.

Nitrifiers are autotrophic microorganisms that consume ammonia and carbonate alkalinity. We hypothesized that elimination of these buffer compounds with a nitrification pre-treatment could enhance subsequent precipitation and removal efficiency of soluble P at high pH. The objective of this study was to evaluate the potential advantages and technical feasibility of soluble P removal using a nitrification-lime treatment process. Results from this study and others have been used in the filing of a U.S. patent on the process (Vanotti et al., 2001).

MATERIALS AND METHODS

THEORETICAL BACKGROUND

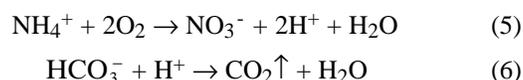
When an alkaline earth metal-containing compound is added to animal wastewater, it reacts with the bicarbonate alkalinity to form a carbonate, with ammonium to form ammonia, and with phosphate to precipitate phosphate compounds (Loehr et al., 1973; Tchobanoglous and Burton, 1991). Using calcium hydroxide as an example, the following equations define the reactions:



The reaction in equation 2 is complete above a pH of 9.5, while that of equation 3 starts above a pH of 7 but is very slow below a pH of 9 (Loehr et al., 1973). Phosphate removal improves with higher pH. As the pH value of the wastewater increases beyond 9, excess calcium ions will then react with the phosphate, as shown in equation 3, to precipitate calcium phosphate compounds. Not expressed in equation 2 is that in wastewater containing a high ammonia concentration, the lime dose required to elevate the pH is also increased due to the ammonium-ammonia reaction that neutralizes the hydroxyl ions (eq. 4):



Therefore, precipitation of phosphate in animal wastewater using an alkaline earth metal-containing compound such as lime is very difficult due to the inherently high buffer capacity of these wastes ($\text{NH}_4\text{-N} \geq 200 \text{ mg L}^{-1}$ and alkalinity $\geq 1200 \text{ mg L}^{-1}$). This buffer prevents rapid changes in pH when lime is added. However, this problem is solved using a pre-nitrification step that eliminates both ammonia (eq. 5) and bicarbonate alkalinity (eq. 6):



As a result, the buffering problem of ammonia (eq. 4) is eliminated with ammonia oxidation (eq. 5), and the problem of carbonate buffers (eq. 2) is eliminated by neutralization with the acid produced during nitrification (eq. 6). These reactions leave an unbuffered system that promotes phosphate removal (eq. 3) when smaller amounts of lime are added.

BASIC PROCESS CONFIGURATION

The basic configuration of the process evaluated in all experiments is shown in figure 1. Swine wastewater first received biological treatment in a nitrification reactor followed by chemical treatment with hydrated lime in a P reactor to precipitate and separate soluble P. Three experiments were done. The first experiment evaluated advantages of using nitrification versus no nitrification for the purposes of P removal. The second experiment used a different type of nitrification process and investigated P recovery in the precipitate in addition to P removal. Both experiments were done in the laboratory. The last experiment was a pilot evaluation done in the field at a larger scale to confirm laboratory observations and derive process recommendations.

LABORATORY EXPERIMENTS

Experiment 1

Lagoon wastewater from a swine operation received biological nitrification treatment followed by chemical lime treatment. Fluidized-bed biological reactors were used for the nitrification step. The nitrification reactors were constructed and operated as described by Vanotti and Hunt (2000) and contained acclimated lagoon nitrifying sludge (ALNS) immobilized in 3 to 5 mm polyvinyl alcohol (PVA) polymer pellets ($150 \text{ g pellets L}^{-1}$ reactor). The nitrifying pellets increased the microbial population in the vessel, which reduced total nitrification treatment time. Process temperature was set at 30°C using a heat regulator and a

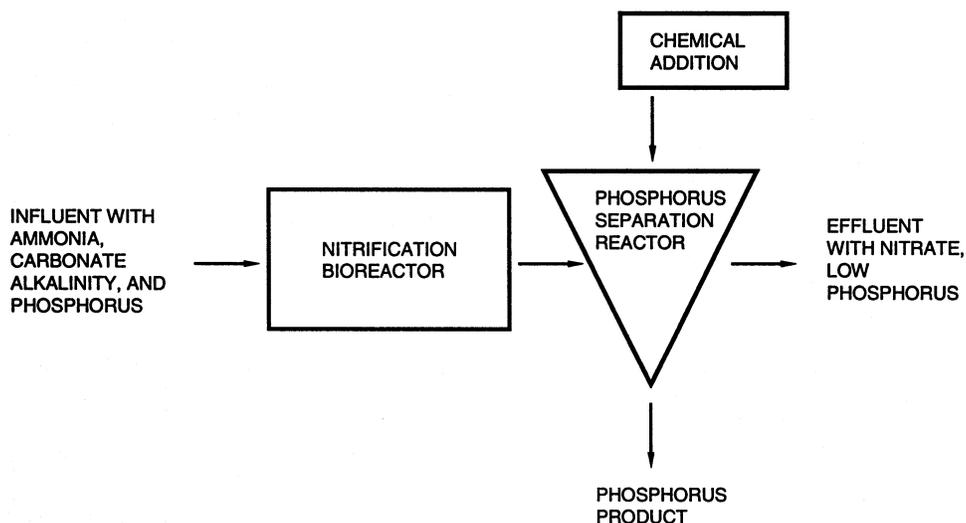


Figure 1. Schematic showing the basic configuration of the process used to remove phosphorus from swine wastewater.

recirculated water bath that accommodated up to six reactors (1.2 L volume). Fine-bubble aeration was provided at a flow rate of 0.5 L min^{-1} to fluidize the immobilized pellets; the resulting average dissolved oxygen concentration in the mixed liquor was 4.3 mg L^{-1} . Aeration was terminated at 16 h when pH reached a value <6.2 , an indication that natural alkalinity was exhausted (Vanotti and Hunt, 2000). Wastewater alkalinity was not supplemented during the nitrification step. The nitrification rate was $14.94 (\pm 0.14) \text{ mg N L}^{-1} \text{ reactor h}^{-1}$, or $359 \text{ mg N L}^{-1} \text{ reactor d}^{-1}$.

The nitrified effluent was transferred to glass reaction vessels where it was further treated with hydrated lime. Lime was applied in water suspensions at rates of 151, 753, and 1506 mg L^{-1} to obtain Ca:P molar ratios of 1.0, 5.0, and 10.0, respectively, based on initial orthophosphate-P concentration in the wastewater of 63 mg L^{-1} ($2.03 \text{ mmol P L}^{-1}$). Identical lime treatments were also applied to the control, but the nitrification step was omitted in the control. The liquid was mixed with a stirrer for 1 min, and the formed precipitate was allowed to settle for 0.5 h. After this gravity sedimentation period, the treated wastewater was sampled in the supernatant and analyzed for nutrients. Removal performance was determined by the difference between the phosphorus concentration in the effluent and that in the original wastewater sample using Standard Methods (APHA, 1998). The experiment was replicated twice.

Experiment 2

To illustrate that any nitrification process will work with the phosphorus precipitation process, a sequencing batch reactor (SBR) was used for the nitrification step as an example of a widely used suspended-growth nitrification process. Lagoon liquid was nitrified in aerated batch reactors containing nitrifying sludge (or free nitrifying bacteria as opposed to immobilized bacteria used in all other experiments). The sludge was acclimated to high ammonia concentration (ALNS) to prevent nitrification inhibition, using culture preparation procedures described by Vanotti and Hunt (2000). The concentration of nitrifying sludge in the filled reactors was $5.96 (\pm 0.13) \text{ g MLSS L}^{-1}$ or $4.73 (\pm 0.14) \text{ g MLVSS L}^{-1}$. Process temperature was set at 30°C using a heat regulator and a recirculated water bath that

accommodated up to six reactors (1.2 L volume). Air was supplied at a flow rate of 0.6 L min^{-1} ; the resulting average dissolved oxygen concentration in the mixed liquor was 4.1 mg L^{-1} . The nitrification rate was $3.96 (\pm 0.53) \text{ mg N L}^{-1} \text{ reactor h}^{-1}$, or $95.1 \text{ mg N L}^{-1} \text{ reactor d}^{-1}$. Alkalinity was monitored during the nitrification process, and aeration was terminated at 40 h when $>90\%$ of the natural alkalinity was consumed (alkalinity decreased from 1251 to 115 mg L^{-1}).

After a biomass settling period of 1 h, the nitrified effluent was decanted and collected in glass containers. The nitrification reactor was filled again with lagoon wastewater, and the react-settle-draw cycle was repeated to obtain sufficient nitrified effluent for the phosphorus precipitation step. The nitrified effluent was transferred to glass reaction vessels, and lime treatments were applied at rates of 148, 296, 445, 593, and $741 \text{ mg Ca(OH)}_2 \text{ per liter}$ (2, 4, 6, 8, and 10 mmol L^{-1}) and mixed with a stirrer for 1 min. Treated wastewater was sampled in the supernatant after a 0.5 h sedimentation period and analyzed for phosphorus to determine its removal efficiency. The precipitate was filtered with fiberglass filters (Whatman 934-AH, Whatman, Inc., Clifton, N.J.), dried and weighed to measure the amount of solids produced, and digested with acid to determine phosphorus content. The complete experiment was replicated twice.

FIELD EXPERIMENT

A pilot test was conducted at a swine production facility using lagoon liquid and the system shown in figure 2. The system used the same nitrification-phosphorus separation sequence investigated in the laboratory experiments. A fluidized-bed biological reactor was used for the nitrification step.

The pilot biological reactor consisted of a 0.34 m^3 contact aeration tank used to lower influent BOD, followed by a 0.18 m^3 sedimentation tank, and a 1.3 m^3 aerated fluidized tank for nitrification. Typically, $>80\%$ of the BOD reduction occurred in the contact aeration tank, and 99% of the nitrate production occurred in the fluidized tank (Vanotti et al., 2000). The contact aeration tank was packed with PVC crossflow media with $48 \text{ m}^2 \text{ m}^{-3}$ surface area (Accu-Pac CF-1900, Brentwood Industries, Inc., Reading, Pa.). The nitrification tank contained 130 L of polyethylene glycol nitrifying pellet cubes of 3 to 4 mm size (Hitachi Plant

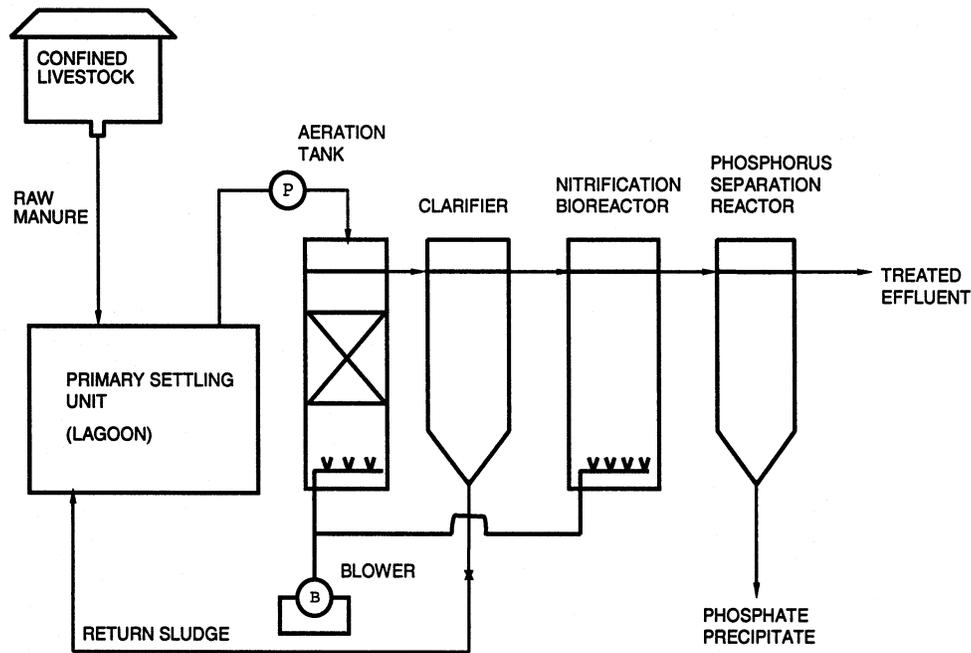


Figure 2. Schematic of the pilot unit used to remove phosphorus from swine lagoon liquid using a nitrification-lime treatment sequence.

Engineering and Construction Co., Tokyo, Japan). A wedge-wire screen (1.5 mm opening) installed at the nitrification tank outflow separated the pellets and liquid, and it retained the pellets inside the nitrification tank. Average airflow rates of 50 and 150 L min⁻¹ were applied to the contact aeration tank and nitrification tank, respectively. This airflow rate ensured appropriate fluidization of the pellets and maintained mixed liquor DO concentration at more than 3 mg L⁻¹. Aeration was provided with a common compressor and one fine-bubble air diffuser in each tank (FlexDisc, EnviroQuip, Inc., Cincinnati, Ohio), having an oxygen transfer efficiency (OTE) rate of 1.85% per ft or a total OTE of 8.5% at 1.4 m depth submergence in the tanks. Wastewater flow was 2.5 m³ d⁻¹, and hydraulic residence time of the nitrification tank was approximately 0.5 d. The pH in the nitrification tank was maintained between 7.5 and 7.8 using a pH controller to accomplish complete nitrification of ammonia. The biological unit had been operational for about 2.5 years before this experiment was conducted (Vanotti et al., 2000).

The phosphorus reactor was placed after the nitrification reactor (fig. 2) to precipitate and separate the phosphorus from the nitrified effluent. It consisted of a 379 L (100 gal.) plastic tank with conical bottom made of XLPE resin (Nalgene, Nalge Co., Rochester, N.Y.) and a mixer with a 0.25 HP, 1725 rpm, 8.9 cm diameter impeller (Lightin, Rochester, N.Y.). A smaller 114 L (30 gal.) tank and mixer were used to prepare the lime milk (2% hydrated lime mixture in water). A peristaltic pump connected to a pH controller (Parts 57500 and 27003, Cole Parmer Instrument Co., Vernon Hills, Ill.) was used to transfer the lime milk into the P reactor. A magnetic flow meter (TigerMag, Sparling Instruments, El Monte, Cal.) measured the total amount of chemical added in each treatment. The chemical was a high-purity, commercial hydrated lime powder containing 98.5% Ca(OH)₂ and with a mean particle size of 3.5 micron (Codex Hydrated Lime, Mississippi Lime Company, Alton, Ill.).

For each treatment, hydrated lime was injected into the stirred reactor containing nitrified wastewater; the pH controller stopped injection when the pH of the mixed liquid reached a setpoint of 9.0, 9.5, 10, or 10.5 (treatments 1 to 4, respectively). Once the desired treatment pH was reached, the mixer was turned off and the liquid was allowed to settle for 30 min. After this settling period, duplicate liquid samples were collected from the supernatant for water quality determinations. The precipitated solids were removed by gravity from the bottom of the reactor and collected in 20 L plastic containers. Two samples of the precipitate were collected for each treatment and centrifuged, dried, and extracted with acid to determine solids and phosphorus content. A total of 16 batch runs were conducted, consisting of four pH treatment levels and four replicates.

LIQUID SWINE MANURE

The wastewater used in all experiments was a lagoon effluent from a swine operation near Kenansville, in Duplin County, North Carolina. This was a 4100 m³ single-stage anaerobic lagoon providing treatment and storage of flushed swine manure from 2600 pigs. The lagoon liquid contained, on average (1-year data, *n* = 80), pH 7.96, total solids 1810 mg L⁻¹, total suspended solids 456 mg L⁻¹, COD 1107 mg L⁻¹, BOD₅ 223 mg L⁻¹, TKN 419 mg L⁻¹, ammonia-N 331 mg L⁻¹, nitrate 0 mg L⁻¹, alkalinity 1846 mg L⁻¹, TP 81 mg L⁻¹, and PO₄-P 46 mg L⁻¹.

ANALYTICAL METHODS

Wastewater analyses were performed according to *Standard Methods for the Examination of Water and Wastewater* (APHA, 1998). Alkalinity was determined by acid titration to the bromocresol green endpoint (pH = 4.5) and expressed as mg CaCO₃ L⁻¹. This pH coincided with the inflection point of the titration curve. The orthophosphate (PO₄-P) fraction, also termed "reactive P," was determined by the

automated ascorbic acid method (Standard Method 4500-P F) after filtration through a 0.45 μm membrane filter (Gelman type Supor-450, Pall Corp., Ann Arbor, Mich.). The same filtrate was used to measure $\text{NH}_4\text{-N}$ by the automated phenate method (Standard Method 4500-NH₃ G) and $\text{NO}_3\text{-N}$ by the automated cadmium reduction method (Standard Method 4500-NO₃⁻ F). Total P and TKN were determined using the ascorbic acid method and the phenate method, respectively, adapted to digested extracts (Technicon, 1977).

The phosphorus content in the solid precipitate was determined with two methods. A concentrated acid digestion (Gallaher et al., 1976) was used to determine total P content, and a diluted acid extraction (1 N HCl) was used to determine inorganic P content. The diluted acid extraction method is recommended for removal of Ca-bound phosphate in soils (Olsen and Sommers, 1982) and gives quantitative recovery of a variety of phosphate compounds in calcareous sediments including $\text{Ca}_5(\text{PO}_4)_3\text{OH}$ (hydroxy-apatite), $\text{Ca}_3(\text{PO}_4)_2$ (tricalcium phosphate), and iron- and aluminum-bound phosphates (Hieltjes and Lijklema, 1980). For the diluted acid extraction, the following procedure was used: 5 mL of 1 N HCl was added to 100 mg of dry precipitate in a test tube, vortexed for 1 min, and allowed to sit for 1 h. The acid extracts in both methods were diluted to 75 mL with distilled water and analyzed for P with the automated method described before. Calcium, magnesium, and potassium concentrations were also measured in the HCl extract using inductively coupled plasma (ICP) analysis.

Mixed liquor suspended solids (MLSS) and volatile suspended solids (MLVSS) determinations were done by collecting liquor samples in 50 mL vials while continuously mixing the cultures with a bench stirrer. The residue retained on a glass-fiber filter was dried to 105°C for suspended solids determination and ignited to 500°C for volatile suspended solids.

RESULTS AND DISCUSSION

LABORATORY EXPERIMENTS

Experiment 1

Data in table 1 show changes in lagoon wastewater characteristics after nitrification treatment in a fluidized-bed reactor. During nitrification, there is a release of hydrogen ions, at a rate of 2 mol for each mole of NH_4^+ oxidized (eq. 5). The natural alkalinity concentration in the lagoon wastewater (1890 mg $\text{CaCO}_3 \text{ L}^{-1}$) was lower than 2140 mg L^{-1} estimated for complete oxidation of 300 mg $\text{NH}_4\text{-N L}^{-1}$, considering an alkalinity consumption of 7.14 mg CaCO_3 per mg $\text{NH}_4\text{-N}$ oxidized (Sherrard, 1976). Nitrification rate in the fluidized-

Table 1. Changes in swine lagoon wastewater characteristics after nitrification pre-treatment.^[a]

| Wastewater Parameter | Influent | After Nitrification |
|--|-------------|---------------------|
| pH | 8.05 (0.21) | 6.06 (0.12) |
| Alkalinity (mg $\text{CaCO}_3 \text{ L}^{-1}$) | 1890 (14) | 63 (7) |
| Ammonia (mg $\text{NH}_4\text{-N L}^{-1}$) | 300 (0.0) | 61 (1) |
| Nitrate (mg $\text{NO}_3\text{-N L}^{-1}$) | 0 (0) | 228 (10) |
| Orthophosphate (mg $\text{PO}_4\text{-P L}^{-1}$) | 63 (1.3) | 63 (0.1) |
| BOD ₅ (mg L^{-1}) | 192 (9) | 54 (5) |

^[a] Means (and SE), $n = 4$.

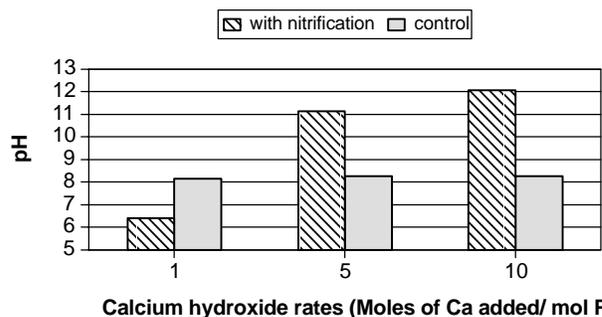


Figure 3. Effect of nitrification and hydrated lime addition on pH of swine lagoon wastewater. The control is lagoon liquid without nitrification. Initial wastewater characteristics are shown in table 1, and the corresponding phosphorus (P) removal data are presented in figure 4. Data are the mean of two replications. $\text{LSD}_{0.05} = 0.38$ pH units applies for comparison between any two means.

bed reactor was 14.94 (± 0.14) mg N L^{-1} reactor h^{-1} ; thus, the natural alkalinity was essentially consumed after a 16 h batch treatment. Only an alkalinity concentration of 63 mg $\text{CaCO}_3 \text{ L}^{-1}$ remained, and the pH decreased from about 8.1 to approximately 6.1 (table 1).

The pH of the wastewater was initially lowered with the acid produced by the nitrifying bacteria. However, because the resulting nitrified wastewater was low in ammonia and carbonate buffers, the total amount of chemical needed to increase the pH above 9 was substantially reduced (figs. 3 and 4). We were thus able to effectively obtain the pH higher than 9 needed to optimize precipitation of soluble phosphorus using Ca-based alkali compounds (eq. 3).

The pH in the control without nitrification pre-treatment was not significantly increased by lime treatment; it varied little from 8.05 to 8.27 (fig. 3). As a consequence, phosphorus removal rates were low (<34%, fig. 4). This illustrates that precipitation of phosphate in animal wastewater using an alkaline earth metal-containing compound, such as hydrated lime, is very difficult due to the inherently high buffer capacity of these wastes ($\text{NH}_4\text{-N}$ and alkalinity), which prevents rapid changes in pH, as shown in the control treatment in figure 3. This contrasts with results obtained in the nitrification/phosphorus removal sequence; the pH increased significantly up to about 12 and phosphorus removal increased to about 100% using the same lime rates as the control. Thus, once ammonia and carbonate alkalinity concentrations are substantially reduced with a nitrification

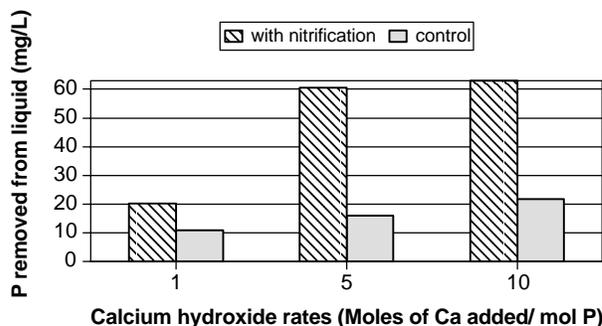


Figure 4. Effect of nitrification and hydrated lime addition on phosphorus (P) removal from swine lagoon wastewater. The control is lagoon liquid without nitrification. Initial $\text{PO}_4\text{-P}$ concentration was 63 mg L^{-1} . Other initial wastewater characteristics are shown in table 1 and corresponding pH changes are shown in figure 3. Data are the mean of two replications. $\text{LSD}_{0.05} = 2.1$ mg L^{-1} applies for comparison between any two means.

Table 2. Phosphorus (P) removal and recovery from swine wastewater using nitrification pre-treatment with free nitrifiers followed by lime precipitation.^[a]

| Ca(OH) ₂ Applied | | pH | Alkalinity (mg L ⁻¹) | Suspended Solids (mg L ⁻¹) | NH ₄ -N (mg L ⁻¹) | NO ₃ -N (mg L ⁻¹) | Total P (mg L ⁻¹) | P Removal (%) ^[b] | P Recovery (%) ^[c] |
|--|-----------------------|--------------------------|-------------------------------------|---|---|---|----------------------------------|---------------------------------|----------------------------------|
| (mmol L ⁻¹) | (mg L ⁻¹) | | | | | | | | |
| Influent (lagoon liquid) | | | | | | | | | |
| 0 | 0 | 7.9 (0.2) ^[d] | 1251(26) | 180 (7) | 212 (1) | 0 (0) | 62.9 (4.1) | -- | -- |
| Effluent after nitrification pre-treatment | | | | | | | | | |
| 0 | 0 | 6.5 (0.1) | 115(13) | 118 (10) | 55 (1) | 158 (4) | 63.5 (3.4) | 0 | -- |
| Effluent after phosphorus precipitation | | | | | | | | | |
| 2 | 148 | 8.0 (0.1) | -- | 78 (5) | 52 (2) | 162 (2) | 30.0 (2.3) | 52 | 88 |
| 4 | 296 | 9.2 (0.0) | -- | 63 (10) | 42 (2) | 163 (4) | 4.0 (4.0) | 94 | 101 |
| 6 | 445 | 10.2 (0.1) | -- | 70 (3) | 35 (2) | 161 (4) | 0.0 (0.0) | 100 | 105 |
| 8 | 593 | 10.9 (0.1) | -- | 70 (9) | 33 (2) | 162 (7) | 0.0 (0.0) | 100 | 100 |
| 10 | 741 | 11.4 (0.0) | -- | 69 (5) | 32 (2) | 161 (5) | 0.0 (0.0) | 100 | 99 |

^[a] Sequencing batch reactor (SBR).

^[b] P removal from liquid fraction relative to influent lagoon concentration.

^[c] P recovery = percent ratio of P recovered in precipitate and P removed from liquid by process.

^[d] Data are the average of two replicates. Values in parentheses are standard errors.

pre-treatment, the subsequent addition of Ca(OH)₂ rapidly increases the pH of the liquid above 9, thereby promoting formation of calcium phosphate precipitate with small amounts of chemical added.

Experiment 2

The nitrification rate obtained with the SBR used in this experiment was 3.96 (±0.53) mg N L⁻¹ reactor h⁻¹, which is about three times slower than rates obtained with the pellet-immobilized bacteria in experiment 1. Nevertheless, the data in table 2 demonstrate that, independently of the nitrification pre-treatment used, once ammonia and carbonate alkalinity concentrations are reduced with a nitrification pre-treatment, the subsequent addition of Ca(OH)₂ rapidly increases the pH of the liquid, thereby promoting formation of calcium phosphate. Nitrifiers are autotrophic microorganisms that consume ammonia, oxygen, and carbon dioxide and produce oxidized nitrogen (nitrate and nitrite) and acidity. In the present system, the nitrification process is used to remove ammonia and carbonate buffers and prevent substantial ammonia loss with high pH treatment. In general, most nitrification methods will produce similar results, provided that the bacteria are adapted to operate at inhibitory high ammonia concentrations (Anthonisen et al., 1976) typical of livestock wastewater.

Reaction of lime and P was fast. Clear precipitate flocs, an indication that the reaction was complete, were evident in the mixed liquid after <1 min of stirring. Phosphorus removal efficiencies of 94% and 100% were obtained with lime application rates of 4 and 6 mmol L⁻¹ and corresponding pH values of 9.2 and 10.2, respectively (table 2). Significantly, using nitrified wastewater, about half of the P (52%) was removed with 2 mmol L⁻¹ treatment and a relatively low pH of 8. This observation contrasts with the lower P removal efficiency obtained using high alkalinity wastewater and a similar pH (control in fig. 3), and it supports the case of removing bicarbonate and NH₃ competition (eqs. 2 and 4) to enhance phosphate precipitation.

Another potential advantage of the process is the remarkable clarity observed in the treated effluent. As can be seen in table 2, the concentration of suspended solids in the lagoon liquid was significantly reduced. Low suspended solids concentration is often an important consideration in the use

of subsurface or surface irrigation equipment for land application of livestock effluents.

The P removed from the liquid fraction was quantitatively recovered in the analysis of the precipitated solids (table 3). The amount of precipitate solids increased with lime treatment applications in the study. Excessive applications (≥593 mg L⁻¹) did not increase P recovery and resulted in solids with a lower P grade (9% to 10% P or 21% to 23% P₂O₅). However, when chemical rates were in the responsive range in terms of P removal (≤445 mg L⁻¹), the P content in the solids was consistently higher and ranged from about 12% to 14% P (27% to 32% P₂O₅). As a comparison, the composition of phosphate rock deposits (in the U.S.) used in superphosphate manufacture is 30.5% to 36.5% P₂O₅ in Florida fields and 27.5% to 37.9% in western fields (USDA-TVA, 1964). Therefore, the calcium phosphate precipitate produced with this process has potential for use as a fertilizer or can be treated to produce concentrated phosphorus products such as superphosphates or ammonium phosphates.

FIELD EXPERIMENT

Phosphorus Removal Performance

Lime treatments were based on various endpoint pH values that were selected based on the responsive range found in the laboratory experiments to avoid excessive chemical applications. Automation was provided with a pH controller connected to the injection pump to maintain a selected treatment pH of the liquid.

Table 3. Phosphorus (P) content in solids produced from swine lagoon liquid using the nitrification-lime precipitation process in the laboratory.^[a]

| Ca(OH) ₂ Applied (mg L ⁻¹) | Precipitate Solids Produced per L of Liquid Treated (mg) | P Recovered in the Precipitate (mg) | Phosphorus Content | |
|---|--|---|-----------------------|---------------------------------|
| | | | % P | % P ₂ O ₅ |
| 148 | 227 (47) | 27.0 (1.9) | 11.9 | 27.2 |
| 296 | 421 (56) | 59.2 (7.4) | 14.1 | 32.2 |
| 445 | 543 (26) | 65.8 (5.9) | 12.1 | 27.7 |
| 593 | 637 (69) | 62.7 (8.0) | 9.8 | 22.5 |
| 741 | 719 (53) | 64.8 (5.1) | 9.0 | 20.6 |

^[a] Water quality characteristics of clarified effluent shown in table 2. Data are the mean (and SE) of two replicates.

Table 4. Performance of field prototype used to remove and recover phosphorus (P) from lagoon swine wastewater in Duplin County, North Carolina.

| Treatment ^[a] | pH | Ca(OH) ₂ Applied (mg L ⁻¹) | Ca:P Molar Ratio ^[b] | Alkalinity (mg L ⁻¹) | Suspended Solids (mg L ⁻¹) | NH ₄ -N (mg L ⁻¹) | Total N (mg L ⁻¹) | PO ₄ -P (mg L ⁻¹) | Total P (mg L ⁻¹) | N:P Ratio ^[c] |
|--|------|---|---------------------------------------|-------------------------------------|--|---|----------------------------------|---|----------------------------------|-----------------------------|
| Influent (lagoon liquid) | 7.7 | -- | -- | 1738 (90) ^[d] | 327 (41) | 278 (4) | 320 (9) | 43.7 (6.2) | 71.9 (4.7) | 4.45 |
| Effluent after nitrification pre-treatment | 7.7 | -- | -- | 532 (28) | 194 (37) | 10 (5) | 300 (15) | 46.0 (6.1) | 65.4 (11.1) | 4.59 |
| Effluent after phosphorus precipitation | | | | | | | | | | |
| 1 | 9.0 | 141 (5) | 0.82 | 511 (20) | 83 (8) | 8 (4) | 303 (14) | 14.7 (3.5) | 25.5 (2.8) | 11.9 |
| 2 | 9.5 | 271 (28) | 1.58 | 570 (41) | 94 (7) | 8 (4) | 301 (14) | 5.4 (2.4) | 11.1 (0.2) | 27.1 |
| 3 | 10.0 | 344 (2) | 2.00 | 557 (42) | 85 (5) | 6 (3) | 299 (15) | 2.2 (1.5) | 3.3 (0.9) | 90.6 |
| 4 | 10.5 | 433 (14) | 2.52 | 545 (22) | 89 (13) | 5 (3) | 299 (15) | 1.2 (0.9) | 1.6 (0.7) | 186.9 |

^[a] Treatment: Hydrated lime liquid [Ca(OH)₂] was injected into a stirred tank containing nitrified lagoon wastewater; a pH controller stopped injection when the pH of mixed liquid reached a setpoint of 9, 9.5, 10, or 10.5 (treatments 1 through 4, respectively). Once the desired treatment pH was reached, the precipitated solids were removed from the bottom of the tank after a 30 min settling period. The precipitated solids were analyzed for phosphorus content (table 5).

^[b] Ca:P molar ratio = applied calcium (Ca) : P in the influent.

^[c] N:P ratio = total nitrogen (N) concentration : total P concentration. Total N of treated effluent is mostly nitrate-N.

^[d] Data are the average of four batch runs. Values in parentheses are standard errors.

Phosphorus removal performance of the pilot system under field conditions was consistent with results obtained in the laboratory (table 4). Phosphorus concentration was significantly decreased with treatment. Phosphorus removal efficiencies of 95% and 98% were obtained with pH treatment levels of 10.0 and 10.5, respectively, and corresponding Ca:P molar ratios of 2 and 2.5. In practice, the level of treatment added will depend on the degree of phosphorus removal desired and should preferably be added in the minimum quantity necessary to balance the N:P ratio of crops or to remediate sprayfields.

Since ammonia nitrogen has been converted to nitrate, increased pH does not result in significant gaseous nitrogen loss. Therefore, the amount of phosphorus removed, and consequently the N:P ratio of the effluent, can also be adjusted in this process to match the N:P ratio needed by the growing crop to which it will be applied. For example, using data from Edwards and Daniel (1992), a final N:P ratio of 10.7:1 and 13.4:1 would be needed to match wheat and coastal bermudagrass specific nutrient uptake needs, respectively, which can be delivered with about 1 Ca:P molar ratio treatment (table 4).

Higher N:P ratios would be prescribed to clean phosphorus-polluted sprayfields. This remediation uses a negative mass phosphorus balance between phosphorus applied and phosphorus removed by harvestable plant materials that is achieved with irrigation of treated wastewater having N:P ratios >15 (table 4). The higher this ratio number, the shorter

the time needed by a succession of crops to deplete soil phosphorus and bring back soil phosphorus test values to environmentally acceptable levels.

Other cases are possible in which sufficient land for effluent disposal is not available or land application of phosphorus and nitrogen is not desirable. Vanotti et al. (2001, 2003) incorporated a denitrification unit into the system shown in figure 2 to convert the nitrate-nitrogen into nitrogen gas (N₂) and provide an effluent virtually free from N and P. The treated effluent is poorly buffered, and the high pH decreases readily once in contact with the CO₂ in the air. For example, Vanotti et al. (2003) showed that short-term (2.5 h) aeration treatment of the effluent can create enough acidity to lower the pH from 10.5 to 8.5.

In addition to the phosphorus removal aspect, the high pH reached in the process destroys the pathogens in liquid swine manure. Results obtained with this process showed that a pH setpoint of 10.5 in the P reactor produces a sanitized effluent free of *Salmonellae*, enterococci, and other pathogen indicators evaluated (Vanotti et al., 2002).

Phosphorus Recovery in Solids

More than 90% of the total P removed from the liquid was recovered in the solids, as shown in the solids analyses (table 5). The precipitate settled readily, and it was concentrated to a volume that ranged from 32 to 62 L per m³ of liquid treated. The precipitate was characterized using the sludge volume index (SVI) with values of 73 to 91 mL g⁻¹ MLSS (table 5). The precipitate was also characterized for volatile

Table 5. Phosphorus content and recovery in solids produced from swine lagoon liquid using the nitrification-lime precipitation process (Duplin County, North Carolina).

| Treatment pH | Precipitate Volume Produced per m ³ of Liquid Treated (L) | SVI ^[a] (mL) | Dewatered Solids Produced per m ³ of Liquid Treated (g) | Total P Recovered in the Precipitate per m ³ of Liquid Treated (g) | P Recovery (%) ^[b] | Phosphorus Content (% P ₂ O ₅) | Calcium Content (% Ca) |
|-----------------|--|----------------------------|--|---|-------------------------------------|---|------------------------------|
| 9.0 | 32.3 ^[c] | 73 | 560 | 43.0 | 93 | 17.5 | 22.5 |
| 9.5 | 48.5 | 84 | 750 | 56.6 | 93 | 17.2 | 22.4 |
| 10.0 | 60.0 | 91 | 910 | 64.0 | 93 | 16.1 | 25.7 |
| 10.5 | 62.0 | 80 | 970 | 66.4 | 94 | 15.7 | 27.9 |

^[a] SVI = Sludge volume index: volume (in mL) occupied by 1 g of a suspension after 30 min settling (APHA, 1998).

^[b] P recovery = percent ratio of total P recovered in precipitate and total P removed from liquid by nitrification - lime precipitation process. Water quality characteristics of clarified effluent are shown in table 4.

^[c] Data are the average of four batch runs.

solids (VS) content after ignition to 500°C. The VS content was consistent among treatments and averaged 37.5% ($\pm 1.8\%$) of the dry precipitate.

Although the amount of precipitate substantially increased with lime application, the phosphorus and calcium content of the precipitate changed little (16% to 17% P_2O_5 and 22% to 28% Ca), indicating that most of the chemical applied was selectively used to precipitate P. The phosphorus grade of the product obtained in the field experiment was lower than that obtained in the laboratory (table 3). In the laboratory, we used a settling period between the nitrification step and lime application, while in the field experiment, the nitrified effluent went directly into the P reactor. This slight difference in procedure resulted in a nitrified effluent from the laboratory tests with lower suspended solids, which undoubtedly affected the composition of the precipitate. Thus, to obtain a high-purity material, the liquid after the nitrification step should be passed through a clarifier (settling unit, membrane filtration unit, etc.) before reaction with lime.

The diluted acid extraction method (1 N HCl) was effective in redissolving the freshly precipitated inorganic phosphate; it extracted 95.5% ($\pm 3.1\%$) of the total P (table 6). Thus, most of the P obtained in the precipitate was mineral, and only a small fraction (<5%) was organic. Analyses of this extract indicated that calcium phosphate compounds were the predominant form in the precipitate (table 6). The precipitate contained relatively small amounts of potassium (K) and magnesium (Mg), and no ammonia-N, an indication that struvite was not present. The resulting molar ratio was approximately 1.0:2.7:0.1:0.2 for P:Ca:K:Mg, respectively.

ECONOMIC CONSIDERATIONS

The chemical and electrical cost to precipitate phosphorus from liquid manure using the nitrification-lime treatment process was calculated on the basis of treating the lagoon effluent from a 4,000-head finishing operation in North Carolina growing pigs from 22.7 to 100 kg (50 to 220 lb). Treatment parameter and target values used in these calculations are based on this study, except for effluent volume and nutrient concentration, which are typical values developed for North Carolina and reported by Barker and Zublena (1995), with the following conditions:

- Anaerobic lagoon liquid effluent of 169 L per 1,000 kg live mass d^{-1} (Barker and Zublena, 1995).

Table 6. Percent and molar composition of the solid precipitate produced from swine lagoon liquid using the nitrification-lime precipitation process (Duplin County, North Carolina).^[a]

| Constituent | Percent Composition (g per 100 g) | Molar Ratio Relative to Phosphate-P |
|--------------------------------------|-----------------------------------|-------------------------------------|
| Total phosphorus (P) ^[b] | 7.27 (0.28) | 1.05 |
| Inorganic phosphate-P ^[c] | 6.94 (0.25) | 1.00 |
| Calcium (Ca) ^[c] | 24.61 (1.35) | 2.74 |
| Potassium (K) ^[c] | 0.49 (0.04) | 0.06 |
| Magnesium (Mg) ^[c] | 1.21 (0.01) | 0.22 |
| Ammonia-N ^[c] | 0.00 (0.00) | 0.00 |

^[a] Data are the average of four pH treatments (table 5). Values in parentheses are standard errors.

^[b] Concentrated acid digestion extract. Total P $\times 2.29 = P_2O_5$ (table 5).

^[c] 1 N HCl extract.

- Liquid containing 563 mg TKN L^{-1} , 456 mg $NH_4-N L^{-1}$, and 98 mg TP L^{-1} (Barker and Zublena, 1995).
- TKN removal efficiency of 96% and BOD₅ reduction during nitrification treatment from approximately 200 to 50 mg L^{-1} (this study).
- Oxygen requirements in biological reactor of 4.57 kg $O_2 kg^{-1} N$ removed for ammonia oxidation, 2.29 kg $O_2 kg^{-1} N$ for biomass respiration, and 0.45 kg $O_2 kg^{-1} BOD_5$ removed (design values used in pilot plant).
- Oxygen transfer efficiency of 24.2% at 4 m diffuser submergence (1.85% OTE ft^{-1}) and density of air = 1.202 kg m^{-3} .
- Chemical requirement of 0.34 kg $Ca(OH)_2 m^{-3}$ and 95% TP removal (treatment 3, table 4).

On average, each pig weighs 61.4 kg (135 lb); therefore, the quantity of lagoon liquid effluent for a 4,000-pig operation is 41.5 $m^3 d^{-1}$, and the amount of hydrated lime required is 14.1 kg d^{-1} . The cost of prepared hydrated lime slurry (30% hydrated lime, 3.01 lb gal⁻¹) in tote tanks in North Carolina is approximately \$0.2039 kg^{-1} dry hydrated lime (\$185 ton^{-1}). This results in a daily chemical cost of \$2.87 (\$1,049 per year).

Electric power needs are those required for aeration used in the biological reactor, and pumps and mixers used throughout the total process. Based on the conditions listed above, an aeration capacity of 1.61 $m^3 air min^{-1}$ (56.8 $ft^3 min^{-1}$) at 4 m water depth (5.7 psi) would be required to remove 22.4 kg TKN d^{-1} and 6.2 kg BOD d^{-1} in the nitrification pre-treatment. This requirement can be met with a positive displacement blower with 2.7 bhp (brake horse power) rating. The power consumption for 24 h d^{-1} blower operation and 90% motor efficiency (ASCE, 1988) is 53.7 kWh d^{-1} , which results in an annual electric power cost of aeration of \$1,115 (unit cost = \$0.0569 kWh⁻¹). Pumps and mixers used in the process add approximately \$465 (22.4 kWh d^{-1}) to a total annual electrical power cost of \$1,580.

For an operation that grows 2.8 groups of pigs per year, the annual chemical and electrical cost for the 4,000-head operation is \$2,629, and the cost per finished pig is \$0.23. With a 95% process efficiency, the amount of phosphorus potentially recovered in the solids produced from the same operation is 1,410 kg per year (7,113 lb P_2O_5 per year) with an estimated fertilizer value of \$1,778 (\$0.25 $lb^{-1} P_2O_5$). A complete economic analysis of the technology would need to consider annualized capital and labor costs, as well as other benefits such as remediation of sprayfields, reduction of land area required to dispose P in liquid manure effluents, and sanitation of these effluents.

CONCLUSIONS

We found that soluble phosphorus can be easily removed from animal wastewater, which has had a nitrification pre-treatment, in the form of an alkaline earth metal-containing phosphate. In the process, carbonate and ammonium buffers contained in liquid waste must be at least reduced or eliminated during the nitrification pre-treatment. This substantially reduces the overall chemical demand needed for optimum phosphorus precipitation and removal. With our process (Vanotti et al., 2001), the amount of phosphorus removed, and consequently the N:P ratio of the effluent, can

also be adjusted to precisely match the N:P ratio needed by the growing crop or to remediate a sprayfield having excessive soil phosphorus levels by increasing the N:P ratios above the present crop requirement. This technology not only solves current problems with excessive accumulation of phosphorus in soils receiving liquid manure, it also produces a valuable phosphorus fertilizer material and reduces the presence of infectious microorganisms due to the high pH in the process. The aspect of reuse is important because, unlike nitrogen, world phosphorus reserves are limited. Operational cost to remove 95% of phosphorus from lagoon wastewater effluent was estimated to be \$0.23 per finished pig.

Collectively, our findings indicate:

- Once ammonia and carbonate alkalinity concentrations in swine wastewater are substantially reduced with a nitrification pre-treatment, the subsequent addition of lime rapidly increases the pH of the liquid, thereby promoting formation of P precipitate with small amounts of chemical added.
- Since ammonia nitrogen has been mostly converted to nitrate, increased pH does not result in significant gaseous nitrogen loss.
- The amount of phosphorus removed, and consequently the N:P ratio of the effluent, can be adjusted in this process to match specific crop needs or remediate sprayfields.
- The final product is calcium phosphate, which has the potential to be reused as fertilizer or processed to produce phosphate concentrates.

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