Performance of a Pilot-Scale Air Sparged Continuous Flow Reactor and Hydrocyclone for Struvite Precipitation and Removal from Liquid Swine Manure

Timothy A. Shepherd  
*Iowa State University*

Robert T. Burns  
*Iowa State University*

Lara B. Moody  
*Iowa State University*

D. Raj Raman  
*Iowa State University, rajraman@iastate.edu*

Kenneth J. Stalder  
*Iowa State University, stalder@iastate.edu*

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ABSTRACT. The objective of this research was to test a pilot-scale air sparged tank reactor (ASTR) and the ASTR in combination with a hydrocyclone (called the pilot-scale ASTR-hydrocyclone system) on two swine manure slurries for struvite-based \((\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O})\) phosphorus removal and recovery. The pilot-scale ASTR system operated at flow rates of 80 to 115 L/min and was based on the bench-scale design from Shepherd et al. (2007). The ASTR effluent was processed using a hydrocyclone separator for struvite separation and total phosphorus (TP) recovery. The pilot-scale ASTR-hydrocyclone system provided a 92% reduction of dissolved reactive phosphorus (DRP) in manure slurry from a swine finishing facility concrete storage tank and a 91% reduction of DRP in manure slurry collected from a swine finishing facility deep-pit under floor collection system. The pilot-scale ASTR-hydrocyclone system removed 18% of TP in swine manure from a concrete storage tank and 9% to 14% of TP in swine manure slurry from a deep-pit under floor collection system. The low TP recovery was attributed to the hydrocyclones inability to provide effective struvite separation as operated. Full-scale economics and implementation of the tested struvite-based phosphorus removal is discussed. A case study of a typical Iowa deep-pit swine production facility (10,000 head/year) indicated that the annual cost of struvite-based phosphorus removal using this system would be approximately $8.88/finished pig or $0.035/L manure slurry treated ($0.134/gal). This cost often exceeds producer’s profit margins; this indicates that struvite-based phosphorus removal using this ASTR-hydrocyclone system in swine finisher manure slurries is not currently economically viable.

Keywords. Manure, Phosphorus, Struvite, Hydrocyclone, Swine.

Growing concerns about water quality and land management have resulted in requirements for the land application of manure, often in the form of legislation. These regulations can limit the application of manure based on crop nutrient needs, such as phosphorus (Shober and Simms, 2003). Phosphorus-based manure application rates can increase the land base requirements from three to eight times compared to nitrogen-based application rates in typical and worst case scenarios, respectively. (Burns et al., 1998). In addition to larger land requirements, application of supplemental nitrogen may be needed for optimal crop production. Removal and concentration of phosphorus from swine manure slurries as struvite \((\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O})\) has the potential to alleviate operational and environmental concerns associated with phosphorus over application. The development of an economical, robust, and flexible continuous flow struvite precipitation reactor for phosphorus removal could greatly benefit livestock operations.

Shepherd et al. (2007) developed a bench-scale continuous flow air sparged tank reactor (ASTR) for struvite precipitation in swine manure slurries. This system provided air sparging for pH adjustment and mixing, and MgCl\(_2\) injection for struvite precipitation. The bench-scale ASTR system provided dissolved reactive phosphorus (DRP) reductions of 78% and 93%; however, separation of precipitated struvite for total phosphorus (TP) reduction was not achieved with an up-flow clarifier operated in continuous flow mode. During those experiments, untreated and ASTR-treated manure slurries were evaluated to identify struvite removal and recovery possibilities. Phosphorus in ASTR-treated manures was concentrated during centrifuge tests when a G-Force larger than 80 was applied. Results from the bench-scale centrifuge tests indicated that a hydrocyclone, providing 80 G-forces or more may provide a continuous flow method to remove and recover precipitated phosphorus from ASTR-treated manure slurries.

Optimized struvite precipitation in manure slurries generally requires adding magnesium and increasing the slurry pH. Agitation of stored manure, which normally occurs prior to and in series with land application events, could increase the reaction and energy efficiency of a continuous flow struvite reactor. Agitation provides a homogenous manure mixture and has been shown to increase manure slurry pH (Zhu et al., 2001), thus reducing chemical and energy costs associated with pH adjustment; a reduction in magnesium requirements due to mixing is not expected however. The optimal time for the implementation of a...
continuous flow struvite treatment system would be in series with a land application event.

The most common manure slurry storage systems utilized in U.S. pork production include: under floor deep-pit storage, external storage tanks, holding ponds, and anaerobic lagoons. A nutrient removal system could be designed to treat manure over an extended period at low flow rates. However, this strategy necessitates a post-treatment storage system, increasing capital expenditures. Implementing a treatment system in series with field application events requires higher treatment flow rates, but reduces the need for a post-treatment storage system.

Additionally, deep-pit manure storage systems require specific management practices to minimize the release of hydrogen sulfide gas (H₂S) inside the production facility. To avoid increasing the amount of H₂S released from deep-pit stored manure slurry, agitation is avoided except during field application events. To minimize human and livestock exposure and risk to H₂S in deep-pit storage facilities, the most feasible treatment scheme should operate during land application events when agitation is necessary.

During land application events, manure slurry is typically applied at flow rates ranging from 2,300 to 6,800 L/min (500 to 1,500 gal/min) (Puck, 2008). The majority of struvite research with animal manures has focused on bench and pilot-scale reactors operating at low flow rates. The struvite research to date in the United States that is being developed commercially was conducted by Bowers and Westerman (2005a and b). Bowers and Westerman (2005a and b) tested a field-scale fluidized bed struvite reactor which treated manure slurries at 5.6 and 9.5 L/min (1.5 and 2.5 gal/min). While the Bowers and Westerman fluidized bed reactor system design is straightforward and could be scaled up, it was suggested to be operated at lower flow rates as a continuous process. A primary objective of the research effort presented in this article was to develop a struvite recovery process that could be easily operated at typical land-application flow-rates such that process could be utilized only during land-application events.

The three goals of this project were to develop a struvite-based phosphorus removal system which (1) utilizes aeration to reduce or eliminate chemical requirements for pH adjustment, (2) provides treatment at high flow rates to eliminate the need for post-treatment storage structures, and (3) recover small, nucleated struvite crystals to reduce the need to grow larger struvite crystals. The objective of this research was to test a continuous flow pilot-scale ASTR utilizing a hydrocyclone solids separator for struvite-based phosphorus removal and recovery.

**MATERIALS AND METHODS**

**PILOT-SCALE DESIGN**

A continuous flow pilot-scale ASTR was designed and constructed to precipitate phosphorus in liquid swine manure at flow rates up to 200 L/min (53 gal/min) at a 10-min hydraulic retention time (HRT). The ASTR-hydrocyclone system was operated at 80 and 115 L/min (21 and 31 gal/min) during continuous flow test runs. Design criteria and operational conditions for the pilot-scale ASTR were based on bench scale research conducted by Shepherd et al. (2007). A Hydrocyclone Separator (McLanahan Model S1.506 A20, McLanahan Corp., Hollidaysburg, Pa.) was implemented to provide phosphorus precipitate removal from the ASTR effluent. The ASTR-hydrocyclone system was constructed as a mobile, palletized system allowing for testing at various swine production sites (figs. 1a and b).

A 3,785-L (1,000-gal) cone-bottom, polypropylene tank (Den Hartog Inc., Hospers, Iowa) provided the reaction zone for the ASTR. Raw manure was pumped from the manure storage system to the top of the ASTR with a Vogelsang V100-90Q positive displacement pump (Vogelsang USA, Ravenna, Ohio); the ASTR effluent was recovered from the outlet of the cone-bottom tank and pumped to the hydrocyclone with a Vogelsang VX136-140Q positive displacement pump. The liquid discharge from the hydrocyclone overflow was considered to be the final effluent; a portion of the final effluent was re-circulated to the ASTR to control operational volume and hydraulic retention time (HRT). Separated solids from the hydrocyclone underflow were collected in a hopper. At an
PILOT-SCALE OPERATION

The ASTR-hydrocyclone system was tested using two manure slurries with four 50-min continuous flow treatments, performed in triplicate: 1) ASTR-hydrocyclone without aeration or MgCl₂ injection, 2) ASTR-hydrocyclone with aeration, but no MgCl₂ injection, and 3) ASTR-hydrocyclone with aeration and MgCl₂ injection. For each treatment, the flow rate to the hydrocyclone was set to achieve a pressure drop of 34.5 kPa (5 psi) across the hydrocyclone, as pressure drop across the hydrocyclone impacts its separation performance; approximately 50% of the hydrocyclone overflow was returned to the ASTR to maintain operational volume and HRT.

Each 50-min continuous flow treatment was divided into five 10-min increments for sample collection. Three sub-samples (300 mL) were collected for each 10-min increment at 2, 5, and 8 min; sub-samples were pooled for analysis. Collected samples were stored at 4°C until analysis. Influent, ASTR effluent, and hydrocyclone effluent were analyzed “as is.” Samples of the hydrocyclone underflow were allowed to settle for 48 h, after which the liquid fraction was decanted with a siphon. The decanted liquid and settled solids were then analyzed separately.

**Treatment 1 – ASTR-Hydrocyclone without Aeration or MgCl₂ Injection**

The ASTR was primed with 1,900 L of manure slurry so the ASTR was full upon initiation of the tests. Continuous flow operation was then initiated without aeration and MgCl₂ injection for 50 min. The system flow rate was approximately 115 L/min (31 gal/min).

**Treatment 2 – ASTR-Hydrocyclone with Aeration but without MgCl₂ Injection**

The ASTR was primed with 1,900 L of manure slurry so the ASTR was full upon initiation of the tests. Treatment then proceeded in two phases: a pre-aeration batch phase followed by a continuous-flow DRP-precipitation and separation phase. Pre-aeration consisted of applying diffused air at a flow rate of 566 L/min for 30 min to the 2000 L of untreated manure slurry. After the initial batch phase, continuous flow operation was initiated with air sparging at 566 L/min; MgCl₂ was not injected. The system flow rate was approximately 115 L/min (31 gal/min). Because aeration was necessary for mixing and pH adjustment, no tests were performed with MgCl₂ addition without aeration.

**Treatment 3 – ASTR-Hydrocyclone with Aeration and MgCl₂ Injection**

The ASTR was initially primed with 1,900 L of manure slurry. Treatment proceeded in two phases, with pre-aeration operated as previously described. Following the initial batch phase, continuous flow operation was initiated. During continuous flow operation, MgCl₂ was injected at a rate determined from laboratory analysis to achieve a Mg²⁺:PO₄³⁻ molar ratio of at least 1.6:1; air sparging proceeded at 566 L/min. The system flow rate was set at approximately 115 L/min (31 gal/min) for treatment of the deep-pit manure slurry and 79 L/min (21 gal/min) for treatment of manure slurry from the concrete storage system.

**Figure 2.** Air sparging diffusion platform and liquid level switches in the air sparged tank reactor (ASTR). 1 - Permacap5 Fine Bubble diffusers (Environmental Dynamics Inc., Columbia, Mo.) 2 - Liquid level switches (Madison Company, Branford, Conn.).
MANURE SLURRY SOURCES

The ASTR was tested on-site at two swine commercial finishing facilities near Ames, Iowa. One of the facilities utilized an under-floor deep-pit storage system and the other utilized an uncovered concrete storage tank. Treated manure slurry was returned to the storage system approximately 30 m from the extraction point. Variations in the hydrocyclone underflow and effluent flow rates required adjustment of the influent flow rate to maintain the operating volume of the ASTR.

The first manure slurry tested was extracted directly from the under-floor deep-pit storage system without agitation. The deep-pit manure was processed using Treatments 1, 2, and 3 of the ASTR-hydrocyclone system. The hydrocyclone flow rate was set at 277 L/min (73 gal/min), the system flow rate was approximately 115 L/min (31 gal/min), and the HRT was approximately 16 min.

The second manure slurry tested, from the concrete storage system, was agitated prior to and during testing; only Treatment 3 with MgCl2 addition and aeration was performed. The hydrocyclone flow rate was set at 300 L/min (79 gal/min), the system flow rate was approximately 79 L/min (21 gal/min), and the HRT was approximately 24 min.

SAMPLE ANALYSIS

Chemical analysis methods were as follows: dissolved reactive phosphorus - Standard Method 4500-P E (APHA, 1998); total phosphorus - Standard Method 965-17 (AOAC, 2002); total Kjeldahl nitrogen (TKN) - Standard Method 2001-11 (AOAC, 2000); ammonium (NH4+) - Standard Method 4500-NH4 B & C for (APHA, 1998); total solids - Standard Method 2540 B (APHA, 1998); Solution pH was determined with a pH electrode (Orion 4-Star pH/Conductivity probe, Thermo Fisher Scientific, Waltham, Mass.) calibrated with 7.0 and 10.0 standard pH solution prior to each treatment. Statistical analysis of phosphorus concentrations and total mass of phosphorus were accomplished using proc MIXED in SAS software (SAS, 2003).

RESULTS AND DISCUSSION

A continuous flow pilot-scale air sparged tank reactor (ASTR) was developed and tested for reducing phosphorus (P) in manure slurries from two different storage systems (under-floor deep-pit and concrete storage). Effluent from the ASTR was processed through a McLanahan Model S1.506 A20 Hydrocyclone Separator (McLanahan Corp., Hollidaysburg Pa.) for struvite-based phosphorus recovery. Table 1 provides the nutrient analysis of the untreated manure slurries utilized in the treatments. The ASTR-hydrocyclone system was initially tested on manure slurry from a deep-pit storage system at a flow rate of 115 L/min (31 gpm) with a 16-min HRT with Treatments 1, 2, and 3. While initial analysis indicated the slurry had an acceptable fraction of dissolved reactive phosphorus (DRP) for struvite recover, at the time of the test the fraction of TP available as DRP (3%) was significantly lower than initial tests had indicated. Therefore, additional tests were performed at another site. The second set of experiments was performed on manure slurry from a concrete storage system which had 31% of the TP available as DRP for struvite precipitation. Manure slurry from the concrete storage was only tested using Treatment 3. This decision was based on the fact that treatments without MgCl2 injection in the deep-pit slurry did not provide sufficient DRP reductions for struvite-based phosphorus removal.

The ASTR was designed to provide optimal conditions for DRP precipitation as struvite through pH adjustment, MgCl2 incorporation, hydraulic retention time, and mixing. The ASTR was not designed to remove phosphorus from the slurry. Therefore, ASTR performance is quantified by DRP reduction. The hydrocyclone was incorporated to provide separation of precipitated phosphorus from the ASTR effluent, and its performance was quantified as the concentration and removal of TP from ASTR-treated manure slurries.

DISSOLVED REACTIVE PHOSPHORUS REDUCTION

For the deep-pit manure slurry, 30 min of pre-aeration provided an average pH increase of 0.24 units from 7.83 to 8.07. Under continuous flow conditions, air sparging without MgCl2 injection maintained a minimum pH increase of 0.11 units, air sparging with MgCl2 injection maintained a minimum pH increase of 0.10 units.

For the concrete storage system, 30 min of pre-aeration provided an average pH increase of 0.25 units from 7.05 to 7.30 in the manure slurry. The addition of MgCl2 immediately decreased the pH to approximately 7.0 prior to continuous flow aeration; this suggests that the manure slurry from the concrete storage had less pH buffering capacity than the manure slurry from deep-pit storage. The pH was reduced to approximately 6.95 under continuous flow operation. Air sparging may not be capable of maintaining the desired pH adjustment for optimized struvite precipitation in manure slurries with low buffering capacities due to the injection of MgCl2, which is acidic. However, a caustic amendment system could be implemented for supplementary pH adjustment when aeration is insufficient.

Table 2 provides the DRP concentrations entering and exiting the system; concentrations were averaged across replications for each treatment. During 50 min of continuous flow operation, Treatment 3 (with air sparging and MgCl2 injection) provided 91% reduction of DRP in manure from the deep-pit storage system and a 92% reduction of DRP in manure from the concrete storage system. For the deep-pit slurry, Treatment 1 (without air sparging and without MgCl2 injection) provided a 14% reduction in DRP, and Treatment 2 (with aeration but without MgCl2) did not provide a

<table>
<thead>
<tr>
<th>Table 1. Characteristics of the untreated swine manure sources.[a]</th>
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<tr>
<td>Manure Sources - Finishing Facilities near Ames, Iowa</td>
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<tr>
<td>Deep pit</td>
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<tr>
<td>Concrete storage</td>
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[a] Dissolved reactive phosphorus (DRP), total phosphorus (TP), total Kjeldahl nitrogen (TKN), ammonium (NH4), total solids (TS).
statistically significant reduction of DRP in the deep-pit manure slurry.

The ASTR-hydrocyclone system reduced the DRP concentration by 91% in the deep-pit manure slurry with an air sparging rate of 566 L/min, and MgCl2 was applied at a Mg2+:PO4 3− ratio of 7.3:1. The ratio of Mg2+:PO4 3− was 4.5 times greater than necessary because the Mg amendment was based on pre-experiment DRP analysis (at 100 mg/L). An initial deep pit slurry sample was collected and analyzed two days prior to the testing; however, during the tests the average DRP concentration was 22 mg P/L. The discrepancy was likely due to initial collection of a non-representative sample. For the deep pit slurry, the DRP concentration of the treated effluent was 2.0 mg P/L.

For tests using slurry from the concrete storage, the pre-experiment slurry and the tested slurry had a DRP concentration of 130 mg P/L. Air sparging occurred at a rate of 566 L/min and the MgCl2 was injected at an Mg2+:PO4 3− ratio of 1.6:1. Within the ASTR, a DRP reduction from 130 to 11 mg P/L was measured.

Effluent from the treatment of the deep-pit manure slurry had a significantly lower DRP concentration than the effluent from the treatment of the concrete storage manure slurry (P-value < 0.0005). This difference may be attributed to the higher struvite conditional solubility product created by over application of magnesium during the deep-pit manure treatment and the lower operating pH during the concrete storage manure treatment. Results indicate that the ASTR is capable of effectively and significantly reducing DRP at high flow rates when aeration and MgCl2 are applied. Further testing of the pilot scale system is needed to identify the optimal HRT’s, magnesium injection rates, and pH requirements which provide adequate treatment levels and minimize capital and chemicals costs.

**TOTAL PHOSPHROUS RECOVERY**

Table 3 provides influent, ASTR effluent, hydrocyclone overflow (effluent and ASTR return), and hydrocyclone underflow TP concentrations, averaged across the three replications of each treatment in the deep-pit and concrete storage manure slurries. Hydrocyclone underflow TP concentrations were found to be significantly higher than influent and effluent concentrations for all treatments in manure slurries from both the deep-pit and concrete storage systems (P< 0.0001).

Underflow TP concentrations were compared across treatments for the deep-pit manure, and no significant difference was observed between treatments without MgCl2 addition. However, introducing MgCl2 with aeration to the deep-pit slurry provided a statistically significant increase in underflow TP. The increased concentration in the underflow suggests that a portion of TP is being removed from the manure slurry. In the analysis of samples, statistics found that for all treatments there was no significant difference between the TP concentrations of Raw Influent, ASTR Effluent (which feeds the hydrocyclone), and Hydrocyclone Overflow (data in table 3). While the average influent concentrations shown in table 3 for all three treatments in the deep-pit manure are slightly lower than that of the effluent and hydrocyclone overflow, there was no statistical difference in these concentration values. Given the relatively large standard deviation on the influent TP concentration, we believe the fact that the reported influent TP concentrations are slightly higher than the ASTR effluent and the hydrocyclone overflow concentrations is due to sampling variability. Because the hydrocyclone underflow flow rate was very low compared to the system influent and effluent flow rates and the percentage of TP that was in the DRP form was also very low, a very small change in concentration between the influent TP concentrations and ASTR effluent

| Table 2. Dissolved reactive phosphorus (DRP) concentration entering and exiting the air sparged tank reactor hydrocyclone system (ASTR-hydrocyclone), averaged across replications for each treatment of the ASTR-hydrocyclone system. |
|---------------------------------|---------------------------------|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
|                                | Deep-Pit Storage                |                                | Concrete Storage                |                                |                                |
|                                | Treatment 1                      | Treatment 2                    | Treatment 3                      | Treatment 3                      | Treatment 3                      |
|                                | (mg/L DRP as P)                  | (mg/L DRP as P)                | (mg/L DRP as P)                  | (mg/L DRP as P)                  | (mg/L DRP as P)                  |
| In                              | 25 ± 3.4                         | 22 ± 1.7                       | 22 ± 3.2                         | 130 ± 13                         | 11 ± 1.6                         |
| Out                             | 22 ± 2.3                         | 21 ± 7.2                       | 2 ± 0.4                          | 11 ± 1.6                         | 92%***                          |
| % reduction                     | 14%*                            | Not Significant**              | 91%***                           | 92%***                           |                                 |
| pH                              | 7.83                            | 7.93                           | 7.92                             | 7.0                              |                                 |

* P-value = 0.0108.
** P-value = 0.6968.
*** P-value < 0.0001.

| Table 3. Total phosphorus concentrations (TP) of influent, air sparged tank reactor (ASTR) effluent, effluent, and hydrocyclone underflow, averaged across replications for each treatment of the ASTR-hydrocyclone system. |
|---------------------------------|---------------------------------|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
|                                | Deep-Pit Storage                |                                | Concrete Storage                |                                |                                |
|                                | Treatment 1                      | Treatment 2                    | Treatment 3                      | Treatment 3                      | Treatment 3                      |
|                                | TP (mg P/kg)                     | TP (mg P/kg)                   | TP (mg P/kg)                     | TP (mg P/kg)                     | TP (mg P/kg)                     |
| Influent                        | 780 ± 140                       | 930 ± 35                       | 680 ± 150                       | 420 ± 34                        |                                 |
| ASTR effluent                   | 900 ± 49                        | 990 ± 30                       | 860 ± 39                        | 360 ± 79                        |                                 |
| Hydrocyclone overflow           | 870 ± 60                        | 900 ± 20                       | 840 ± 58                        | 350 ± 93                        |                                 |
| Hydrocyclone underflow          | 1920 ± 370[a][b]                | 2000 ± 390[b]                  | 2650 ± 668[b]                   | 580 ± 47                        |                                 |

[a] No significant difference P = 0.3510, treatment without aeration and without MgCl2 vs. treatment with aeration but without MgCl2.
[b] Significant difference P < 0.0001, treatment with aeration and MgCl2 vs. treatment without aeration and without MgCl2; treatment with aeration and MgCl2 vs. treatment with aeration but without MgCl2.
and hydrocyclone overflow would be expected. It appears that this concentration change was less than the standard deviation of the concentration measurements.

To better quantify the TP reduction, a phosphorus mass balance was performed using influent and underflow flow rates and their corresponding TP concentrations. Table 4 provides the total mass of phosphorus entering and removed from the system in the hydrocyclone underflow; results were averaged across replications for each treatment.

Results from the deep-pit manure tests show Treatment 1 provided 12% TP recovery, Treatment 2 provided 14% TP recovery, and Treatment 3 provided 9% TP recovery. When no MgCl₂ was added, the difference between TP recovery for aerated and non-aerated treatments was not significantly different (P-value = 0.93). The treatments without MgCl₂ injection had significantly higher TP recoveries than the treatment with MgCl₂ addition (no aeration-no Mg inject P-value = 0.02, and aeration-no Mg inject P-value = 0.02).

Manure slurry from the concrete storage system had an 18% TP recovery when treated with MgCl₂ addition and aeration. Results from treatments in the deep-pit manure slurry were not expected, as treatment with MgCl₂ injection provided lower TP removal rates than treatments without MgCl₂ injection.

Precipitation of struvite through MgCl₂ injection should theoretically increase hydrocyclone phosphorus recovery when compared to treatments without MgCl₂ injection. However, due to the low availability of TP in the dissolved form for struvite precipitation (3% in the deep-pit manure), TP recovered as struvite may not be identifiable due to the variability between samples collected for analysis. Also, for the deep pit manure, the percent of phosphorus recovered exceeded the amount of phosphorus available for struvite precipitation. In other words, 3% of the TP in the slurry was available as DRP but between 9% and 14% was recovered from each of the treatments by the hydrocyclone. A possible explanation is that prior to treatment a portion of TP in the manure slurry was in the form of dense solids, most likely undigested feed or calcium-phosphorus precipitates, dense enough to be separated by the hydrocyclone.

Based on phosphorus available in the dissolved form in the concrete storage, the maximum achievable TP recovery rate was 31%; the rate achieved was 18% TP, as determined by the reduction in mass of TP when comparing the influent and the hydrocyclone underflow. Therefore, treatment of manure from the concrete storage system provided phosphorus removal rates lower than the theoretical struvite-based TP reduction. This indicates that the hydrocyclone was not able to capture all precipitated struvite particles. Hydrocyclone recovery efficiency is dependent on the particle size and density as well as liquid characteristics. Factors which may reduce struvite removal efficiency with a hydrocyclone include: precipitation of struvite particles too small to be captured, struvite precipitation onto particles with low densities such as organic matter, and hindered movement of struvite particles by low density solids.

A comparison of the total solids (TS) content for the influent and hydrocyclone underflow for each treatment on both manure types, averaged across replications, is provided in Table 5. Comparisons indicate that the hydrocyclone underflow effluent TS was significantly higher than the influent TS for all treatments in manure from the deep-pit storage system (P-value < 0.01). Hydrocyclone underflow effluent TS was significantly higher than the influent TS for the treatment in manure from the concrete storage system (P-value < 0.01).

The average influent TS content in manure slurry from deep-pit storage ranged from 2.2% to 3.0% and increased to 5.2% to 6.2% in the hydrocyclone underflow effluent. The average influent TS of manure slurry from the concrete storage system was 1.7% and increased to 2.3% in the hydrocyclone underflow effluent. Comparisons of TS content between the influent and hydrocyclone underflow effluent indicate that the hydrocyclone is partitioning a fraction of the TS content into the hydrocyclone underflow effluent. However, for the operational conditions of the hydrocyclone, the underflow effluent TS content was expected to range from 15% to 30% based on performance from initial tests with sand added to swine manure slurries. This suggests that the amount of TS, particle size, and density of materials in the manure slurries tested were not suitable for optimal hydrocyclone removal performance, decreasing the overall separation efficiency of the system. Manure with higher TS contents and larger particle sizes may provide increased hydrocyclone separation performance and efficiency.
X-RAY DIFFRACTION

Solids collected in the hydrocyclone underflow hopper from treatment with aeration and MgCl₂ were air dried and sieved prior to analysis with X-ray diffraction. The sieve analysis of solids recovered from the hydrocyclone is shown in Figure 3. X-ray diffraction results of sieve fractions passing 150 and 200 mesh (104 and 74 microns, respectively) were compared to the software database to identify the crystalline species present. X-ray diffraction analysis of material passing the 150 mesh (104 microns) indicated the presence of struvite, calcite (CaCO₃), and quartz (SiO₂). X-ray diffraction analysis of materials passing the 200 mesh (74 microns) indicated the presence of struvite, calcite, quartz, and dolomite (CaMg(CO₃)₂) (fig. 4).

The purity and amount of struvite, quartz, calcite, dolomite in the sieved samples was not quantified. The presence of quartz indicates sand, which was likely introduced to the system through dust, dirt, and construction

![Figure 3](image-url)

**Figure 3.** Sieve analysis of solids recovered from the hydrocyclone underflow collected during Treatment 3: ASTR-hydrocyclone with aeration and MgCl₂ injection. Solids passing a 150 and 200 mesh (104 and 74 microns, respectively) were analyzed with X-ray diffraction.

![Figure 4](image-url)

**Figure 4.** X-ray diffraction results of hydrocyclone solids passing a 200-mesh (74-microns) sieve showing correlating peaks of struvite, calcite, quartz, and dolomite.
materials. Calcite is a sparingly soluble solid (K_{sp} of 10^{-8.34}) and is common in water systems with high alkalinity (Snoeyink and Jenkins, 1980). In Iowa, groundwater is typically classified with high alkalinity levels due to the presence of calcium and magnesium hardness, and the presence of Mg^{2+} has been shown to inhibit the growth of calcite (Morse, 1983). Therefore, calcite was most likely present prior to treatment with the ASTR-hydrocyclone system. Dolomite is a calcium-magnesium complex with a slow precipitation rate (Mamalis et al., 1994). Calcium-magnesium carbonate species precipitate in solutions with high magnesium concentrations or from the supernatants of anaerobic sludge digesters (Snoeyink and Jenkins, 1980); therefore it is possible that dolomite was present prior to treatment or precipitated due to the over application of MgCl₂ during treatment.

Comparison of crystalline species found in the underflow solids, the specific gravities of quartz, dolomite, and calcite (2.65, 2.85, and 2.72, respectively) are significantly larger than that of struvite (1.7). If the hydrocyclone is capable of capturing a specific size of struvite particles, then materials of equal size which have higher specific gravities would also be captured.

Since this system was only tested with swine manures that had relatively low DRP:TP ratios, further testing should be performed on manure slurries with higher fractions of TP in the available DRP form for struvite precipitation. Additionally, the hydrocyclone should be tested under various operational flow rates and pressure drops to identify the optimal settings for improved struvite recovery (and hence TP removal) from typical swine manure management systems. Alternative solids separation methods should be investigated to determine their ability to separate struvite from ASTR-treated manure slurries.

**Phosphorus Recovery to Balance Crop Needs**

The maximum amount of struvite-based phosphorus removal and recovery is related to the availability of phosphorus as DRP. The ratio of DRP:TP can be used to identify the maximum theoretical TP removal rate for a specific waste. Theoretically, manure with DRP:TP ratios of 1:2 could achieve up to 50% TP removal via struvite precipitation and recovery. Manure slurry from the deep-pit storage system had an approximate DRP:TP ratio of 1:36 (22 mg P/L : 800 mg P/L), indicating only 3% of TP could be precipitated and recovered. Manure slurry from the concrete storage system had an approximate DRP:TP ratio of 1:3 (130 mg P/L : 420 mg P/L), indicating 31% of TP could be precipitated and recovered.

A typical corn crop in Iowa requires 20 to 21.6 kg N/metric tonne of grain produced (1.1 to 1.2 lb N/bu) and 6.8 kg P₂O₅/metric tonne of grain produced (0.375 lb P₂O₅/bu) (Iowa State University, University Extension, 2003). This suggests that manure slurries should have a N:P₂O₅ ratio of 3:1 for land application at a rate which balances both N and P for a typical Iowa corn crop. Manure slurry from the deep-pit system had an initial N:P₂O₅ ratio of 1.9:1; and if applied at nitrogen levels for a typical corn crop in Iowa, phosphorus would be applied in excess.

The graph in figure 5 was developed to illustrate the requirements to achieve a desired N:P₂O₅ ratio based on the initial DRP:TP ratio of the deep-pit manure tested and the combined DRP reduction and recovery efficiencies from treatment. Since this analysis has been made for deep-pit swine systems where 90% to 100% of the total N is in a plant available form (Iowa State University, University Extension, 2003), no adjustment for plant availability of the nitrogen has been made in this analysis. The combined DRP reduction and removal efficiency accounts for the ability of a treatment system to reduce DRP in the formation of struvite, and the ability of the system to remove struvite. Treatment with the ASTR has the potential to adjust the N:P₂O₅ ratio to 2:0:1 based on the DRP:TP limit of the deep-pit manure. To achieve the desired (balanced) N:P₂O₅ ratio of 3:1, manure slurry from this specific system would require at least 38% of the TP to be in the lower DRP reduction and recovery efficiencies necessitate a higher percentage of TP to be in the form of DRP to achieve the desired N:P₂O₅ ratio of 3:1.

Testing of the ASTR system at bench and pilot-scale indicated that a 90% reduction in DRP is achievable with a Mg₆PO₄ ratio of at least 1.6:1; however efforts to remove precipitated the DRP as struvite were unsuccessful. For treatment technologies implementing a struvite-based phosphorus reduction, an estimate of the required DRP reduction and removal efficiency can be identified from figure 5 for a specific DRP:TP ratio (and vice versa). For example, if a treatment system provided a DRP reduction and removal efficiency of 70%, the manure slurry from the deep-pit system would require approximately 55% of the TP to be available as DRP to achieve the desired N:P₂O₅ ratio of 3:1.

To further analyze the relationships shown in figure 5, three initial N:P₂O₅ ratios were identified to represent typical Iowa deep-pit facilities (table 5). Figure 6 shows the relationship between the achievable N:P₂O₅ ratio versus DRP:TP ratio, assuming a 70% DRP reduction and removal efficiency. For example, manure slurry with an initial N:P₂O₅ ratio of 1.9:1 would require approximately 55% of the TP to be available as DRP to achieve an effluent N:P₂O₅ ratio of 3:1. A manure slurry with a lower initial N:P₂O₅ ratio of 1.25:1 would require approximately 90% of the TP to be available as DRP to achieve the same treatment level with struvite-based phosphorus removal.

![Figure 5. Nomograph of combined DRP reduction and recovery efficiencies to determine achievable N:P₂O₅ ratio dependant upon the DRP:TP ratio for a manure with an initial N:P₂O₅ ratio (1.9:1). Source: Ames, Iowa deep-pit production facility (utilized in this study).](image-url)
Table 5. Initial Nitrogen to Phosphorus ratios (N:P2O5) of swine manure slurries representing typical Iowa deep-pit finishing facilities.

<table>
<thead>
<tr>
<th>N:P2O5 Ratio</th>
<th>Source</th>
<th>N:P2O5 Ratio</th>
<th>Source</th>
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<tbody>
<tr>
<td>High</td>
<td>1.90:1 Amens, Iowa producer (utilized in this study)</td>
<td>Medium</td>
<td>1.40:1 Nutrient content of finishing pig manure as excreted (MWPS, 1993)</td>
</tr>
<tr>
<td>Low</td>
<td>1.25:1 Manning, Iowa producer (independent lab analysis)</td>
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Figure 6. Achievable N:P2O5 ratio vs. DRP:TP ratio, assuming a 70% DRP reduction and removal efficiency.

TREATMENT ECONOMICS

An economic analysis was completed based on the assumption that the system was capable of reducing 90% of the DRP and 80% recovery. Note that during the tests conducted with the pilot-scale ASTR-hydrocyclone struvite recovery system a 91% DRP removal and only an 18% TP recovery were achieved with manure from a swine finishing facility. Clearly improved TP recovery performance from hydrocyclone, or other solids separation device must be achieved before the unit could be applied full-scale. The following analysis was conducted to provide insight into the economic feasibility of struvite-based phosphorus recovery with this system. Scaling equipment components from base cost to predicted cost was accomplished with equation 1. Each equipment component was scaled from defined pilot-scale size parameters (volume, flow rate, horsepower) to the required size for a full-scale system. For example, the full-scale cost for the reactor tank was calculated by multiplying the cost of the pilot-scale tank ($1,800) times the ratio of the required full-scale volume (28,500 L) to the volume of the pilot-scale tank (3,780 L). Based on crop nutrient requirements and facility information, if the land application regulations were changed from nitrogen based to phosphorus based, the producer would need to remove excess phosphorus via treatment or reduce the number of pigs fed per year from 10,000 to approximately 6,400.

Cost<sub>Custom</sub> = 0.0127<sup>TP<sub>removal</sub></sup> + 0.214

where

Cost<sub>Custom</sub> = custom applicator charge, $/1000 L slurry treated

TP<sub>removal</sub> = required reduction of total phosphorus (mg P/L)

Using the cost estimate of the ASTR-hydrocyclone system derived in equation 2, an economical analysis for a 10,000 head/year deep-pit pork production facility near Manning, Iowa was performed. This production facility produces approximately 6.0 million L of manure slurry per year (1.6 million gal/year). Independent lab analysis indicated that the slurry contained 5.31 kg N/1,000 L and 2.82 kg P<sub>2</sub>O<sub>5</sub>/1,000 L. Based on crop nutrient requirements and facility information, if the land application regulations were changed from nitrogen based to phosphorus based, the producer would need to remove excess phosphorus via treatment or reduce the number of pigs fed per year from 10,000 to approximately 6,400.

Assuming that a custom applicator or cooperative operates a mobile struvite-based treatment system with a DRP reduction efficiency of 90% and removal efficiency of 80% (combined DRP removal and recovery efficiency of 72%), the number of pigs which could be placed back into the production facility can be determined. If 50% of the TP is available as DRP for struvite precipitation, treatment would reduce 90% of the TP to 17 L/h; the cost of diesel fuel used was $0.91/L. The market price for bulk MgCl<sub>2</sub> used was $0.95/kg Mg<sup>2+</sup>.

Energy cost for the full-scale ASTR-hydrocyclone system was estimated to be $0.045/1,000 L manure slurry treated ($0.172/1,000 gal). The chemical cost associated with treatment is directly proportional to the amount of phosphorus reduction required. Higher TP removal rates require larger chemical amendments which increases the operational cost, equation 2 is an estimate of the total cost per 1,000 L of deep-pit manure treated by a custom applicator or cooperative. A full description of the operating and chemical costs can be found in Shepherd (2007).
and offset the additional application of commercial nitrogen fertilizer required to maintain corn yields, approximately 11,400 kg N/yr (25,000 lb N/yr). Liquid swine manure is typically sold at 70% of their nutrient value, assuming that the separated solids can be sold for 70% of the commercial phosphate value, a revenue of $2,300/year could be realized (Phosphate $495/ton, Heartland Coop, Slater, Iowa, Fall 2007). A yearly fertilizer cost savings of $6,400 could be realized from avoiding the purchase of supplemental nitrogen (Anhydrous ammonia $510/ton, Heartland Coop, Slater, Iowa, fall 2007).

Based on the economical analysis of the full-scale ASTR-hydrocyclone system, avoidance of commercial nitrogen fertilizer, and sale of separated phosphorus, the yearly cost of treatment would be approximately $222,000. To offset the cost of treatment, a profit of at least $61.66/pig replaced would be required. For the entire 10,000 head production facility, the annual cost equates to $22.20/pig space ($8.88/finished pig, assuming 2.5 turns/year) or $0.0353/L of deep-pit manure slurry treated ($0.134/gal).

Custom feeding operations are contracted by large producers to finish pigs to market weight. The average custom feeding operation in western Iowa are currently paid $13.50 per finished pig for operational management, facilities, utilities, labor, and manure management (personal correspondent with a custom feeding operation near Manning, Iowa). A treatment cost of $8.88 per finished pig, representing approximately 66% of the total payment per finished pig, indicates that struvite-based phosphorus removal with the system tested is not currently economically viable for swine finishing facilities.

**CONCLUSION**

Field experiments with the ASTR-hydrocyclone treatment system demonstrated that it was possible to significantly reduce the quantity of dissolved reactive phosphorus (DRP) in swine manure slurry when aeration and MgCl₂ were provided. Average DRP reductions of 91% were observed at continuous flow treatment rates of 115 L/min (31 gal/min), producing an effluent with an average DRP concentration of 2 mg P/L in manure slurry from deep-pit storage. Average DRP reductions of 92% were observed at continuous flow treatment rates of 80 L/min (21 gal/min), producing an effluent with an average DRP concentration of 11 mg P/L in manure slurry from a concrete storage system.

A hydrocyclone separator was implemented to provide struvite-based total phosphorus (TP) reductions. The ASTR-hydrocyclone system provided TP removal rates of 9% to 14% in manure slurry from deep-pit storage and 18% in manure slurry from a concrete storage system. Comparisons of ASTR-hydrocyclone treatments and theoretical struvite-based TP removal rates versus actual TP removal rates indicate that the hydrocyclone did not provide sufficient struvite separation efficiencies as operated. Analysis of struvite precipitation efficiency and required phosphorus reduction levels in typical deep-pit manure slurries indicates that a feasible separation system should provide struvite removal efficiency of 70% to 80%. X-ray diffraction of solids collected from the underflow of the hydrocyclone indicated the presence of struvite as small particles. The quantity and purity of struvite collected was not determined. Further research should focus developing an alternative method to remove struvite from ASTR-treated manure slurries.

Prior to the application of struvite-based phosphorus recovery, manures should first be analyzed to determine if treatment can provide the desired phosphorus reduction levels. Achievable treatment levels of struvite-based DRP reduction and recovery for TP removal in a specific manure slurry is dependant on the relationship between the DRP:TP, N:P₂O₅, and the combined efficiency of DRP reduction and recovery. Manure slurries with high DRP:TP ratios have the potential to provide significant TP reductions dependant upon the initial N:P₂O₅ ratio and degree of separation efficiency required to achieve a manure slurry balanced in terms of N and P for a specific crop. Further testing of the ASTR-hydrocyclone system should be performed on manure slurries with significant portions (>50%) of TP available as DRP for precipitation as struvite.

An analysis of chemical, energy, capital, and depreciation operational costs for a full-scale (4700 L/min) ASTR-hydrocyclone unit indicated a high dependence on phosphorus removal requirements. The chemical cost of magnesium amendment is directly related to DRP reduction needed to achieve the desired treatment level. A case study of a typical Iowa deep-pit pork production facility feeding 10,000 head/year, could implement struvite-based phosphorus removal with an ASTR-separation system for an approximate yearly cost of $222,000. This annual cost equates to $22.20/pig space ($8.88/finished pig, assuming 2.5 turns/year) or $0.0353/L of deep-pit manure slurry treated ($0.134/gal) and indicates that the struvite-based phosphorus removal system tested in deep-pit swine manure is not currently economically viable.

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