Flocculation of Swine Manure: Influence of Flocculant, Rate of Addition, and Diet

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Abstract

Manure was collected from growing pigs (22–48 kg) fed one of 11 diets varying in total and available phosphorus content. Reduced phosphorus diets were amended with feed additives intended to improve availability of phytate phosphorus. Diets were replicated over three feeding periods resulting in 33 manure samples. Collected manure samples were diluted (final solids content approximated 2.4% total solids) and poured into Imhoff cones. Five flocculants plus a control were added at each of three concentrations and the diluted mixtures allowed to settle for 10 minutes followed by a second 10-minute settling period. Volume of settled material was recorded following each settling period. At the end of the second settling period (20 min of total settling) supernatant was poured off and frozen for future phosphorus analysis and settled solids were weighed and dried for total solids determination. Phosphorus and solids recovery was calculated. Results indicated that $Al_2(SO_4)_3$ and $FeCl_3$ were the most effective flocculants. Little improvement was observed when the flocculant was added at a concentration of 625 mg/l compared with 250 mg/L. Both flocculants recovered more than 85% of solids and more than ß 70% of the phosphorus. Addition of $Al_2(SO_4)_3$ at a concentration of 625 mg/l increased phosphorus removal to 90%. Diet and feeding period were significant influences on results. As the pigs aged, manure phosphorus was more soluble, possibly explaining the observation that less phosphorus was settled in feeding period 3 compared with periods 1 and 2. As producers consider nutrient management options, feed-to-field strategies should be implemented.

Keywords phosphorus, flocculation, manure

Introduction

Animal production facilities continue to expand and the public worries more and more about water and air pollution from these facilities (10). As the number of hogs per farm increases, so does the need for cost-effective treatment and storage of animal manure. Phosphorus (P) runoff from manure-applied land into surface waters can pose water quality concerns. Methods to bind manure phosphorus before to land application of the manure merit investigation. Solids separation can reduce odors and assist in nutrient management (11). Solids separation is not a new concept. It has been used in treating drinking water and municipal sewage for quite a while (5). The concentrated manure from solid separation also allows for easier transport of the solids off of the farm. Solid separation, before manure storage, reduces the buildup of the solids thereby increasing the life of the manure storage system (11). Phosphorus concentrations in wastewaters can be reduced through effective solid separation (7), however, the magnitude of reduction is often insufficient to address the P produced in surplus relative to available land and nitrogen production.

Flocculation can be an inexpensive form of solid separation. The addition of flocculants to wastes produces more sediment than sedimentation alone (3,7). In addition, calcium, iron, and aluminum salts effectively precipitate dissolved phosphorus and aid in the flocculation of suspended solids, which enhances settling (3,9). Ferric chloride has been shown to be the most effective metal salt at removing solids and nutrients through flocculation (10,11). As much as 92% of the P in simulated flushed dairy manure was sedimented with ferric chloride, with slightly lower performance by ferric sulfate (3). The goal in finding an effective flocculant is that it should not be too expensive and there should not be negative environmental effects that could cancel out any benefits. Many flocculants can be reasonably priced and some are fairly common. Also, there is no need for expensive equipment, which makes it possible for producers to use this in combination with solids separation techniques. Although some studies have indicated that flocculants are not economical (9), as removal of P becomes a greater limitation to spread manure in an environmentally sound manner, the economics may change.

The objective of this study was to evaluate the use of flocculants in effectively binding solids and phosphorus from liquid swine manure generated from pigs fed diets containing varying amounts of available phosphorus and dietary amendments aimed at improving phosphorus utilization. The relationship between diet fed and settling characteristics of resultant manure is yet undetermined. This project was intended to consider components (e.g., diet and post-excretion treatment) of a "feed to field" strategy to reduce phosphorus pollution potential.

Materials and Methods

Growing pigs were fed one of 11 dietary treatments containing different concentrations of available phosphorus with and without the addition of dietary amendments intended to improve the availability of dietary P. All pigs were fed throughout three 14-d feeding periods, housed in pens according to diet. Diets were reformulated during each feeding period. After a 1-wk dietary adjustment period, manure, primarily fecal material was collected from the pens such that each of the 11 diets was collected during each of the three feeding periods. Urine was able to drain away from the pen area. Total phosphorus content of the manure, as collected from the animal pen, was determined using a photometric method following digestion (1). Soluble phosphorus was determined using a water extraction procedure followed by acid digestion and photometric determination. Solids (TS; 2) and P content of this sample served as the reference point for determination of the extent of solids and P removed by flocculation and settling time.

Five flocculants were tested on each of the 33 diets (11 diets during each of three feeding periods), each at three rates of addition. A control (no flocculant) was run with each flocculation scheme (n = 594). The flocculants used were calcium carbonate ($CaCO_3$), ferric sulfate ($FeSO_4$), calcium oxide (CaO), aluminum sulfate $Al_2(SO_4)_3$ and ferric chloride ($FeCl_3$). Tested rates of flocculant addition were 40 mg/l, 250 and 625 mg/l.

To perform the flocculations, collected manure samples were diluted 100-fold to produce a dilute solution similar to that found in a deep pit (approximately 0.25–0.32% total solids, TS; 4). Each 10.0-g sample was transferred into a 1000 ml beaker and diluted to volume with deionized water. The beakers were then placed on a Phipps and Bird PB-900 Programmable Jartester (Phipps and Bird, Inc., Richmond, VA) and mixed for 10 min at 125 rpm. After 10 min, the Jartester was stopped and a flocculant was added to each beaker. Following, the solutions were mixed for 2 min at 125 rpm then poured into 1000 ml Nalgene Imhoff settling cones. A timer was set for 10 min and the solutions were allowed to settle. The level of sedimentation was read and recorded after 10 min. A second 10-min settling period was begun after which the level of sedimentation was again read and recorded. The liquid effluent in the top of the cones was subsampled and frozen for future P analysis. The solids that had settled in the bottom of the cones were weighed and oven-dried at $55^{o}C$ for 24 hours for TS analysis (2). The dry solids were saved. Phosphorus content of the liquid effluent was determined using an acid digestion followed by a photometric determination using the ascorbic acid Hach method 8190 (Hach Company, Loveland, CO).

Data were analyzed using the GLM procedures of SAS (8). A model containing diet, period, flocculant, and flocculant concentration served as the fixed effects for considering phosphorus and solids removal. Settling time was treated as a continuous independent variable.

Results and Discussion

Solids settling TS of manure collected from pigs fed each treatment diet during each feeding period did not differ ($P > .05$) and approximated 24% TS. Following dilution, TS content of the Imhoff cones was approximately 2.4% across all diets and feeding periods (n = 33). However, diet fed had a significant effect on TS settled in the collected manure ($P < .001$). Table 1 depicts the percentage of solids removed by gravity settling for each of the 11 diets by using each of the flocculants. Statistical probability levels indicate that dietary treatment influenced the degree of settling when $Al_2(SO_4)_3$, CaO, and $FeSO_4$ were added as flocculating agents. This same observation occurred when no flocculant (control) was added. The feeding period in which the manure samples were collected was also significant ($P < .001$; Table 2) perhaps due to digestive influences on manure characteristics as pigs aged. A significant interaction of diet and feeding period ($P < .001$) suggests that not all diets responded similarly across feeding periods. For example, more solids were settled during the first feeding period using manure from pigs fed diet 2 whereas, using manure from pigs fed diet 11 resulted in the great degree of solids settling during the third feeding period. Manure collected from pigs fed diet 7 resulted in similar percentage settling across all three feeding periods (Table 2).

Table 3 illustrates that flocculant concentration influenced quantity of solids removed with gravity settling. Choice of flocculant significantly influenced the percentage of solids removed. $FeCl_3$, CaO, and $Al_2(SO_4)_3$ settled more solids than the remaining flocculants across all rates of addition, particularly at concentrations of 250 and 625 mg/l. Little benefit was observed by adding $FeSO_4$ compared with the control, even at the 625 mg/l concentration (Table 3). Addition of $CaCO_3$ at the concentration of 625 mg/l did result in more solids settling than the control; however, addition at the lower concentrations did not improve solids recovery much, compared with the control (Table 3). Although economics of addition were not considered in this study, addition of flocculating agents at the highest concentration studied (625 mg/l) may not prove cost efficient given the relatively small gains, compared with 250 mg/l, in solids recovery for even the most promising flocculants [$Al_2(SO_4)_3$, $FeCl_3$, and CaO; Table 3].

Solids recovery in this study was similar to that observed by others. Ndegwa et al. (6) observed 83, 85, 91, and 93% TS sedimentation in 1 and 2% TS swine manure following addition of 0.5, 1.0, 1.5, or 2.0 g/l $Al_2(SO_4)_3$, respectively. This compares to 75% sedimentation in the control. Adding $FeCl_3$ at these same concentrations resulted in TS recoveries ranging from 86 to 93%, respectively. Slightly higher recoveries in studies conducted by Ndegwa et al. (6) likely reflect lower TS in the initial sample. Zhang and Lei (12) attribute performance differences of specific flocculants to manure characteristics (particle size and density) as well as solids content. It should be noted that numbers reported by Ndegwa et al. (6) reflect arithmetic means rather than least squares means and differences from the control may be numerical, only. Barrow et al. (3) reported TS recoveries in 1% TS dairy flushwater ranging from 71 to 89% when $FeCl_3$ was added at concentrations ranging from 69.5 to 278 mg Fe/l. The highest concentration of $FeCl_3$ used in this study (625 mg/L) corresponds to 217 mg Fe/l. Addition of $FeCl_3$ and a polymer to 0.5% TS dairy manure resulted in TS removal of up to 67% and greater than 80% TS removal when added to 2% TS swine manure (12). Sherman et al. (9) reported TS removal of up to 92 and 83% TS by using 317 and 106 mg Al/l from $Al_2(SO_4)_3$, respectively, compared with 72% for control samples. The 625 mg/l $Al_2(SO_4)_3$ addition used in our study is the equivalent of 99 mg Al/l. Addition of $FeCl_3$ resulted in TS recovery of 82 and 78% after adding 188 and 376 mg Fe/l, respectively (6).

Settling time. Greater than 80% of the solids that settled after 20 min had done so by 10 min of settling (Table 4). This is similar to that reported by Powers et al. (7). In the case of adding flocculants at a concentration of 40 mg/l no additional settling was observed in the second settling period, compared with the first 10 min of settling (Table 5). When flocculants were added at a concentration of 250 mg/L, only 75 to 85% of the final settling had occurred after 10 min settling time when $Al_2(SO_4)_3$, CaO, and $FeCl_3$ were added (Table 5). Flocculants added at a concentration of 625 mg/l resulted in 80 – 90% of the 20-min settling after 10 min when $Al_2(SO_4)_3$, CaO, and $FeCl_3$ served as flocculants (Table 5).

Phosphorus settling. Phosphorus composition of the diets fed to the growing pigs during each of the three feeding periods is depicted in Table 6. Available P represents that portion of the phosphorus that can be digested and potentially absorbed by the animal (non-phytate bound phosphorus). Soluble and total P composition of the fecal samples used in the flocculation study is shown in Table 7. Values in Table 7 reflect P content of the manure after drying (dry matter basis); before dilution of the excreted manure for the flocculation studies. Diet had a significant effect on total P composition of the feces (P = .01) but no affect on the soluble P content. As expected, feeding period had a significant effect on fecal total P due to the fact that P composition of the diet was reduced as the pigs aged and their requirement for P decreased. Consequently, fecal P decreased as the feeding periods progressed. However, the soluble fraction of the excreted P increased during the course of the feeding study, possibly reflecting digestive changes that resulted in more P hydrolyzed in the hind gut and therefore in a more soluble form upon excretion.

Diet and feeding period had a significant effect on percentage of P settled (Table 8). A significant interaction of the two effects was observed as well (P < .001). The diet affect was perhaps due to the influence of feed additives in some diets, added with to improve P availability to the animal. If the additives were effective, dietary P would have been hydrolyzed better in the digestive tract and therefore, potentially more soluble when excreted. This explains the observation that as the pigs aged across feeding periods soluble P content of the manure P increased perhaps due to increased hydrolytic ability of the digestive tract (Table 7). The soluble component of excreted P may well have been less susceptible to the settling influences of the chemical amendments, explaining the reduced P removal in sedimentation as the feeding study progressed as well as explaining variation in P settling observed between diets (Table 8).

Flocculant selection impacted P sedimentation (P < .001; Table 9). Addition of $Al_2(SO_4)_3$, or $FeCl_3$ resulted in the greatest removal of P with sediment followed by CaO and, to a lesser extent, $FeSO_4$ (Table 9). Use of $CaCO_3$ resulted in no improvement in P settling compared with the control (P > .05). Although addition of $FeSO_4$ improved solids settling only moderately compared with the control (P = .04), $FeSO_4$ did improve P settling considerably (P < .001).

Similar to the results observed with solids settling, increasing the concentration of flocculant from 40 to 250 mg/L to 625 mg/l improved P settling to the greatest extent when $Al_2(SO_4)_3$ was the chemical amendment used (Table 10). Smaller gains in P removal efficiency were observed when CaO was used as the flocculant. Increased P settling was observed when the rate was increased from 40 to 250 mg/l for $FeCl_3$ and $FeSO_4$ but no further improvements were gained by increasing the concentration to 625 mg/L (Table 10). Although numerical differences were observed for $CaCO_3$ and the control, these settling percentages were not statistically different (Table 10). If P settling is the primary objective for the producer implementing flocculation schemes, addition of $Al_2(SO_4)_3$ at a 625 mg/l concentration may prove economically viable considering the improvement in P settling compared with use at a 250 mg/l concentration (91 vs. 74%, respectively) whereas addition at the higher rate may not be considered viable for solids removal only.

Phosphorus recovery in this study was similar to that observed by others. Ndegwa et al. (6) observed 77 and 85% P recovery in sediment following addition of 1.5 and 2.0 g/l $Al_2(SO_4)_3$, respectively, to 1 and 2% TS swine manure. This compares to 42% P recovery in the control samples. Adding $FeCl_3$ at these same concentrations resulted in P recoveries of 84 and 92%, respectively. Barrow et al. (3) reported P recoveries in 1% TS dairy flushwater ranging from 78 to 88% when $FeCl_3$ was added at concentrations ranging from 69.5 to 278 mg/l Fe. Only 40% of the P was recovered in the control samples. The highest concentration of $FeCl_3$ used in this study (625 mg/l) corresponds to 217 mg/l Fe. Addition of $FeCl_3$ in combination with a polymer resulted in P removal of up to 99% when added to manures ranging from 0.5% to 2.0% TS (12). Sherman et al. (9) reported P removal of up to 97 and 77% TS by using 317 and 106 mg Al/l from $Al_2(SO_4)_3$, respectively, compared with 37% for control samples. The 625 mg/l $Al_2(SO_4)_3$ addition used in our study is the equivalent of 99 mg Al/l. Addition of $FeCl_3$ resulted in P recovery of 82 and 89% after adding 188 and 376 mg Fe/l, respectively (9).

Conclusions

Diet fed to the pigs, and therefore composition of manure, did affect solids and phosphorus settling within the

Imhoff cones. Choice of flocculant and concentration of flocculant significantly impacted removal of solids with $FeCl_3$ and $Al_2(SO_4)_3$, demonstrating the highest percentage of solids removed. Most of the settling had occurred following 10 min of settling time. However, choice of flocculant did show some potential to remove additional solids when an additional 10 min of settling time was provided. Choice of flocculant also affected P removal. Overall, $Al_2(SO_4)_3$ proved most promising although some concerns with buildup in soil and related soil acidity management have been posed. Ferric chloride and calcium oxide also appeared promising as demonstrated by others. The dietary implications on solids and nutrient recovery potential did not influence the general conclusions but may warrant further investigation as producers look at whole-farm strategies for nutrient management.

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Table 1. Least squares means of percentage solids settled using five chemical flocculants (plus control) and effect of dietary treatment on solids removal after 20 minutes of gravity settling time.

	Flocculant					
Diet	$Al_2(SO_4)_3$	$CaCO_3$	CaO	$FeCl_3$	$FeSO_4$	Control
1	74.8	55.3	75.8	82.9	52.3	48.6
2	78.1	62.9	79.5	83.6	56.5	55.3
3	83.4	66.8	78.8	89.2	62.6	59.7
4	77.8	59.6	76.7	81.8	52.5	48.2
5	80.0	56.7	72.0	71.9	50.9	47.6
6	78.1	62.0	78.3	84.7	58.0	52.9
7	75.5	66.9	75.4	82.8	57.8	56.3
8	78.3	58.0	73.0	79.8	53.8	48.5
9	76.4	58.4	74.2	80.8	53.8	46.1
10	74.0	59.7	71.9	79.6	51.5	47.6
11	77.6	58.8	70.0	81.2	53.0	49.5
P-value	0.0910	0.1315	0.0354	0.4186	0.0437	0.0053

Table 2. Least squares means of percentage TS settled with and without flocculants for each of 11 swine diets fed during each of three feeding periods.

	Feeding Period		
	1	2	3
Diet Number	Settled solids, %		
1	60.9	68.4	65.6
2	79.7	67.3	61.0
3	66.7	86.4	67.2
4	63.2	70.3	64.8
5	52.3	69.6	67.6
6	69.8	68.2	69.1
7	75.7	65.7	65.9
8	65.2	63.6	67.0
9	63.7	66.1	65.1
10	53.3	74.4	64.6
11	61.3	65.2	68.6

Table 3. Least squares means of percentage solids settled after 20 minutes of gravity settling time for three flocculant concentrations and effect of choice of flocculant.

	Flocculant Concentration		
Flocculant	40 mg/l	250 mg/l	625 mg/l
$Al_2(SO_4)_3$	53.6	86.5	92.8
$CaCO_3$	52.4	57.9	71.1
CaO	54.7	78.1	92.3
$FeCl_3$	62.2	90.9	91.9
$FeSO_4$	52.4	53.8	58.2
Control	52.6	50.1	50.2
P-value	<.0001	<.0001	<.0001

Table 4. Diet and feeding period effects on total solids settled following 10-minute settling time, expressed as a percentage of swine manure solids settled after 20 minutes settling time. Swine manure was collected from growing pigs fed one of 11 dietary treatments during each of three feeding periods.

	Feeding Period		
Diet Number	1	2	3
1	99.1	99.3	94.9
2	100.0	98.3	87.6
3	98.8	100.0	86.0
4	100.0	99.0	95.7
5	100.0	100.0	83.1
6	100.0	97.5	91.3
7	100.0	99.5	82.4
8	100.0	99.1	90.8
9	100.0	98.9	81.6
10	100.0	100.0	92.5
11	69.4	99.0	83.1

Table 5. Effects of flocculant and flocculant concentration on percentage total solids (TS) settled following 10 minutes settling time compared to 20 minutes settling time.

	Concentration of flocculant added		
Flocculant	40 mg/l	250 mg/l	625 mg/l
$Al_2(SO_4)_3$	100.0	79.8	79.7
$CaCO_3$	100.0	98.6	99.9
CaO	100.0	86.8	89.8
$FeCl_3$	100.0	76.6	83.3
$FeSO_4$	100.0	98.0	100.0
Control	100.0	99.2	100.0

Table 6. Phosphorus composition of diets fed to growing swine during each of three consecutive feeding periods.

	Feeding Period					
	1	2	3	1	2	3
Animal weight, kg	22–30	30–39	39–48	22–30	30–39	39–48
Diet Number	Diet total phosphorus, %			Diet available phosphorus, %		
1	.65	.51	.43	.43	.25	.22
2	.44	.44	.38	.18	.22	.16
3	.45	.38	.36	.19	.16	.14
4	.39	.32	.31	.14	.09	.09
5	.38	.33	.31	.15	.10	.09
6	.39	.34	.32	.15	.10	.09
7	.40	.33	.31	.16	.11	.10
8	.39	.33	.33	.15	.10	.10
9	.39	.33	.32	.16	.12	.10
10	.39	.33	.33	.16	.11	.11
11	.39	.34	.32	.14	.12	.09

Table 7. Total and soluble phosphorus content of swine feces collected from pigs fed one of 11 diets during each of three feeding periods.

	Feeding Period					
	1	2	3	1	2	3
Diet Number	Fecal total phosphorus, %			Fecal soluble phosphorus, %		
1	2.34	2.19	1.48	0.27	0.50	0.57
2	1.95	1.98	1.81	0.27	0.67	0.76
3	2.24	1.99	1.66	0.34	0.51	0.57
4	1.95	1.85	1.71	0.40	0.52	0.63
5	1.60	1.70	1.55	0.32	0.40	0.56
6	2.06	1.52	1.83	0.33	0.43	0.75
7	1.99	1.74	1.43	0.29	0.56	0.53
8	2.19	1.78	1.54	0.44	0.46	0.59
9	2.07	1.95	1.51	0.44	0.60	0.60
10	2.13	2.02	1.53	0.40	0.52	0.48
11	2.02	1.61	1.77	0.32	0.34	0.63

Table 8. Least squares means of percentage P settled with and without flocculants for each of 11 swine diets fed during each of three feeding periods.

	Feeding Period		
	1	2	3
Diet Number	Settled P, %		
1	50.0	31.4	46.9
2	54.8	29.6	31.4
3	45.0	57.4	32.3
4	36.5	43.7	42.1
5	45.7	32.3	28.6
6	49.4	35.8	35.0
7	64.0	47.4	31.7
8	33.3	47.3	37.0
9	58.6	51.4	33.6
10	54.2	47.5	28.9
11	39.6	29.5	31.2

Table 9. Least squares means of percentage of phosphorus settled using five chemical flocculants (plus control) and effect of dietary treatment on phosphorus removal after 20 minutes of gravity settling time.

	Flocculant					
Diet	$Al_2(SO_4)_3$	$CaCO_3$	CaO	$FeCl_3$	$FeSO_4$	Control
1	65.3	14.4	53.3	66.2	42.4	15.1
2	55.9	15.5	52.0	59.6	32.5	16.2
3	61.5	25.3	55.9	63.0	41.4	22.2
4	61.4	17.6	51.6	62.5	37.9	14.5
5	60.0	10.2	38.8	61.4	30.1	12.6
6	65.0	14.3	50.7	66.9	31.8	11.8
7	68.1	27.3	54.7	68.2	44.1	23.8
8	61.7	15.2	47.1	63.2	33.3	14.5
9	64.2	27.7	55.3	58.6	47.2	34.0
10	64.8	22.5	45.6	65.9	42.7	19.7
11	53.5	7.5	40.5	64.3	29.5	5.3

Table 10. Least squares means of percentage of phosphorus settled after 20 minutes of gravity settling time for three flocculant concentrations and effect of choice of flocculant.

	Flocculant Concentration		
Flocculant	40 mg/l	250 mg/l	625 mg/l
$Al_2(SO_4)_3$	21.0	73.9	90.9
$CaCO_3$	15.0	17.6	21.2
CaO	26.7	55.4	66.7
$FeCl_3$	30.0	82.7	78.2
$FeSO_4$	21.8	41.7	49.1
Control	19.0	17.8	14.6