

# ENHANCED PHOSPHORUS REMOVAL FROM SWINE-NURSERY MANURE IN AERATED BATCH REACTORS

P. M. Ndegwa, J. Zhu, A. Luo, D. W. Hamilton

**ABSTRACT.** Phosphorus-based land application of swine manure slurry results in under-application of nitrogen (N), while nitrogen-based application leads to over-application of phosphorus (P). Significant amounts of P must be removed from swine manure to reduce P over-application, and at the same time, safely apply targeted amounts of N. This study compared three schemes of soluble orthophosphate (ortho-P) reduction from liquid swine nursery manure during batch aeration: (1) solids-liquid separation before aeration (pre-aeration solids-liquid, or PASL, separation), (2) solids-liquid separation in the middle of aeration (mid-aeration solids-liquid, or MASL, separation) and (3) no solids-liquid separation (control). Significantly more ortho-P was converted to non-soluble species with MASL separation (92%) than with either PASL separation (87%) or the control (84%). MASL separation also significantly improved the removal of total phosphorus (TP) in the sludge from the liquid after an overnight sedimentation process: 80%, 70%, and 66% for MASL separation, PASL separation, and control, respectively. Solids-liquid separation enhanced the maintenance of higher oxidation potentials (depicted by higher values of ORP in the separated manures than in the control) in the manure during the latter days of aeration, implying that substantial energy saving is possible for prolonged aeration of separated liquid manure. This is an important element in the post-aeration treatment for ensuring that P removed from the solution stays in the sludge and is not released back into solution, if removal of the sludge does not immediately follow scheduled aeration treatment.

**Keywords.** Batch aeration, Oxidation-reduction potential, pH, Phosphorus, Solids-liquid separation, Swine manure.

For sustainable animal agricultural practices, the norm is usually to encourage recycle of animal excreta, which is rich in nutrients and organic matter, for production of animal feeds or fertilizers. This indeed has been the traditional approach for many centuries. In modern times, however, economic factors have led to an increase in the size of animal facilities and regional concentrations of similar enterprises. These concentrations produce large amounts of liquid manure because of the methods of collection and handling used. Although it has been economically advantageous to collect and handle the manure as slurries, these systems have led to increased problems in manure utilization and recycling. In addition, because of introduction of more stringent legislation to protect the environment, land spreading of manure is becoming increasingly less attractive as a utilization method, and some kinds of pretreatments are needed to adjust levels

of organic carbon (OC), nitrogen (N), phosphorus (P), and odorous compounds.

This study addresses the problem of excess phosphorus in swine manure with respect to land application. Currently, regulations are being revised to replace the traditional N-based land application of swine manure by the P-content. The push towards P-based land application of swine manure is being rationalized from the fact that N-based land applications of swine manure slurries may lead to over-application of P. However, adopting P-based application without pre-treatment of manure to reduce the levels of P in the manure not only requires more land to apply the same volume of manure but also in the majority of cases results in under-application of N. This is because the N:P ratios common in swine manure are usually much smaller than the N:P ratios requirements of most crops and pastures (Sherman et al., 2000; Worley and Das, 2000; Powers and Van Horn, 2001). Significant amounts of phosphorus must be removed from swine manure to reduce P over-application, and at the same time, safely apply targeted amounts of N. The removed P, which is concentrated in a small volume of the sludge, can then be exported to remote areas where it can be utilized without adversely impacting the environment.

Considerably high removal of orthophosphates (ortho-P) in wastewater treatment has been accomplished by simultaneous upward adjustment of pH and addition of salts of iron, calcium, or aluminum to form sparingly soluble phosphates, which are subsequently removed by settling and a separation process (Fuhs and Chen, 1975; Kox, 1981; Maurer et al., 1999). On the other hand, substantial accumulation of phosphates in activated sludge has been observed without addition of such salts. Menar and Jenkins (1970) explained the latter observation by noting that calcium from naturally

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hard water formed insoluble phosphates on the activated sludge floc during aeration because of a rise in pH resulting from purging carbon dioxide from the activated sludge. Swine manure contains calcium ( $\text{Ca}^{+2}$ ) and iron ( $\text{Fe}^{+3}$ ) due to mineral supplements in feeds (Campbell et al., 1997). It follows, therefore, that similar aeration treatments of swine manure, even without addition of calcium or iron salts, are likely to have some effect on the amount of soluble ortho-P in the manure liquid.

A more recent study by Luo et al. (2001) showed that as much as 76% of soluble ortho-P can be removed using low-level aeration ( $0.067 \text{ L air} \cdot \text{L}^{-1} \text{ manure} \cdot \text{min}^{-1}$ ) of swine manure in one day. In the same study, it was also noted that, during the first day of aeration, the level of soluble ortho-P and organic-P decreased, while insoluble ortho-P increased. The level of insoluble inorganic P, however, declined with prolonged aeration while the organic P increased, presumably because of microbial biomass growth. The level of soluble ortho-P from which the microbes obtain the P to synthesize organic P, however, did not change appreciably during this prolonged aeration treatment. It can, therefore, be inferred that there is some form of equilibrium transformation between the forms of P such that, once some soluble ortho-P is absorbed into microbial biomass, an equal amount dissolves from the insoluble component into the soluble component to maintain the level of the dissolved portion.

It is hypothesized in this study that solids-liquid separation before aeration or after the first day of aeration could substantially enhance P removal in subsequent aeration treatments, not only from increased microbial absorption and immobilization of P because of increased microbial activity, but also from shifting of the equilibrium in favor of ortho-P precipitation. The overall goal of this study was to determine the effects of solids-liquid separation on the removal of soluble ortho-P in swine manure during aeration treatments. The specific objectives were to: (1) determine the effect of

pre-aeration solids-liquid separation on ortho-P removal, (2) determine the effect of mid-aeration solids-liquid separation treatments on ortho-P removal, and (3) explore the interrelationships of other pertinent parameters during aeration treatments.

## METHODS AND MATERIALS

### EQUIPMENT AND INSTRUMENTATION

A schematic diagram of one unit of the equipment and instrumentation used in this experiment is shown in figure 1. The two treatments (PASL and MASL) and the control were duplicated, resulting in a total of six such unit-reactors. The reactors, made of clear Plexiglas columns (91 cm tall and 15 cm diameter), were filled with liquid manure (14 L), leaving approximately 15 cm headspace to facilitate mixing and to provide room for frothing created by aeration. To aerate the manure, a positive pressure air pump (Model DOA-P104-AA, Gast Manufacturing, Benton Harbor, Mich.) was used to introduce air into the manure through a flexible rubber air diffuser (W107A, Won Brothers Inc., Fort Washington, Md.) placed at the bottom of each reactor. A variable airflow meter (Model P-32461-64, Cole-Parmer Instrument Company) was used to regulate and maintain the flow of aeration air in each unit. A low-level airflow rate of  $0.067 \text{ L air} \cdot \text{L}^{-1} \text{ manure} \cdot \text{min}^{-1}$  was maintained throughout the study period based on previous research (Luo et al., 2001). Oxidation-reduction potential of the liquid manure was determined electronically using a platinum band ORP probe and a voltmeter (Accumet 1003, Fisher Scientific). A handheld pH meter (Accumet 1003, Fisher Scientific) equipped with a temperature compensating probe was used to monitor the pH and the temperature in each reactor on a daily basis just prior to daily sampling of manure.

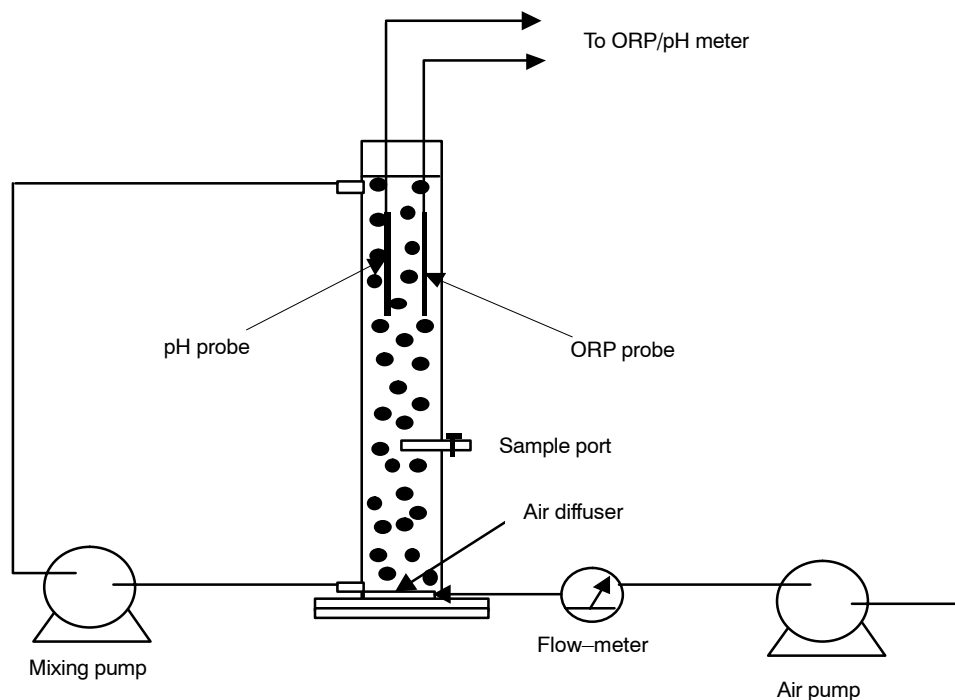


Figure 1. A unit of the equipment and instrumentation used in this study.

## MANURE COLLECTION, LOADING, AND SAMPLING

Swine manure from a nursery barn located at the Oklahoma State University Swine Research Facility in Stillwater, Oklahoma, was collected in plastic containers. The manure collection system in this facility is a pull-plug system operated either once or twice a week. The 8-week-old piglets in this barn were fed a corn-soybean diet. The characteristics of the manure are given in table 1. Prior to filling the reactors, the manure slurry was thoroughly mixed. To facilitate solids-liquid separation in two of the reactors for the pre-aeration solids-liquid (PASL) separation treatment, the manure was allowed to quiescently settle for 5 h. The liquid portion (supernatant) in two of the reactors was then decanted into separate containers. The two reactors were cleaned thoroughly of the solids before pouring back the separated liquid manure to fill up to the 14 L-mark. Before starting the aeration, the manure in all the reactors was again thoroughly stirred using a recirculation pump (Model 2E-38N, Little Giant Pump Co., Oklahoma City, Okla.) and a sample was drawn 30 cm from the bottom of each reactor for laboratory analyses. For the remainder of the nine days of the experiment, samples were taken every day from the same location.

A pair of the remaining four reactors with unseparated manure was chosen for the mid-aeration solids-liquid (MASL) separation treatment after four days of aeration. A procedure similar to that used for the PASL separation treatment pair of reactors was repeated for the randomly selected pair for the MASL separation treatment. For the purpose of control during this mid-aeration solids-liquid separation procedure, aeration treatments were also stopped in the other treatments (PASL and the control) and only restarted after the completion of this process for all the reactors. This precaution evened the impact of stoppage on the removal of P in all the treatments. Final TP concentrations were determined from supernatant samples taken at the completion of the aeration treatment after an overnight sedimentation process. Analyses were done immediately after the sampling whenever possible and if not, the samples were frozen for future analyses.

## LABORATORY ANALYSES

Using standard laboratory methods (APHA, 1998), the following parameters were determined for each collected sample: total solids (TS), total volatile solids (TVS), soluble ortho-P, TP, and total Kjeldahl nitrogen (TKN). To determine the soluble ortho-P, a sample was diluted and thoroughly mixed by shaking for 5 min. The diluted sample was then filtered using GF/A-1.6 µm Whatman filter papers. The soluble ortho-P in the filtrate was determined colorimetrically as the phosphomolybdate complex after reaction with ascorbic acid (APHA, 1998). TP was determined using the persulfate digestion method, by which all the species of P in a sample were first converted to ortho-P or PO<sub>4</sub><sup>-3</sup>. The samples were then filtered, and the P was measured colorimetrically using the ascorbic acid method. Throughout this article, therefore, both ortho-P and TP are reported in terms of PO<sub>4</sub><sup>-3</sup>. The volatile fatty acids (VFAs) in the filtrates were determined using an esterification method. This method is based on esterification of the carboxylic acids present in the sample followed by colorimetric determination of the esters produced by the ferric hydroxamate reaction. All VFAs are reported as their equivalent mg/L acetic acid (Hach, 1993).

## DATA AND STATISTICAL ANALYSES

The objective of the study was to evaluate entire processes (solids separation and aeration) rather than stepwise P removals. Therefore, maximum percentage reductions in ortho-P and TP in the liquid manure were based on the initial concentration in the raw liquid manure and the minimum/final concentrations during the entire treatment process. The results, calculated using equation 1 are presented in tables 2 and 3 for ortho-P and TP, respectively.

$$\text{Maximum percent reduction (\%)} = \frac{[\text{Initial conc.} - \text{Minimum (or final) conc.}]}{[\text{Initial conc.}]} \times 100 \quad (1)$$

where "conc." refers to the concentration of either ortho-P or TP in mg/L.

Analyses of variances (ANOVAs) among the treatments and the control were performed on the reductions of both ortho-P and TP in the liquid manure based on duplicate data sets. The least significant difference (LSD) method was used to separate the means whenever there was significant difference in the reductions of either ortho-P or TP in the liquid manure (tables 2 and 3). Standard statistical software (SAS) was used for statistical analyses in this study, and test for significance between means is implied at the  $\alpha = 0.05$  level unless stated otherwise.

## RESULTS AND DISCUSSION

### SOLUBLE ORTHO-P AND TP

It is evident from the data in table 1 that soluble ortho-P was the largest component of TP in the raw manure, comprising approximately 86%, while the combined organic P and insoluble ortho-P comprised only 14% of TP. This

**Table 1. Characteristics of the manure used in this study.**

Parameter	Quantity
Total solids of raw manure (TS), g/L	15.59 ±0.81 <sup>[a]</sup>
Total solids (PASL), g/L	11.35 ±0.03
Total solids (MASL), g/L	11.93 ±0.43
Total volatile solids (TVS), g/L	10.28 ±0.64
Total phosphorus (TP), PO <sub>4</sub> <sup>-3</sup> mg/L	493.17 ±9.94
Soluble orthophosphate, PO <sub>4</sub> <sup>-3</sup> mg/L	424.97 ±16.16
Total Kjeldahl nitrogen (TKN), mg/L	2,391 ±59
pH	5.5 ±0.0
Oxidation-reduction potential, mV	-264 ±8

[a] "±" refers to one standard deviation from the mean ( $n = 6$ ).

**Table 2. Soluble ortho-P (SOP) in swine manure before and after treatments.**

Treatment	SOP in Raw Manure (PO <sub>4</sub> <sup>-3</sup> mg/L)	SOP in Separated Liquid after Treatment (PO <sub>4</sub> <sup>-3</sup> mg/L)	Maximum Reduction Achieved (%) <sup>[a]</sup>	Day on which Maximum Achieved
Control	424.97 ±16.16 <sup>[b]</sup>	67.02 ±14.12	84 a	8
Pre-aeration	424.97 ±16.16	57.63 ±2.28	87 a,b	4
Mid-aeration	424.97 ±16.16	34.81 ±0.46	92 b	8 <sup>[c]</sup>

[a] Values followed by the same letter are not significantly different at  $\alpha = 0.05$ .

[b] "±" refers to one standard deviation from the mean ( $n = 6$  for raw manure;  $n = 2$  after treatment).

[c] Four days after solids-liquid separation.

**Table 3. Total phosphorus (TP) in swine manure before and after treatments.**

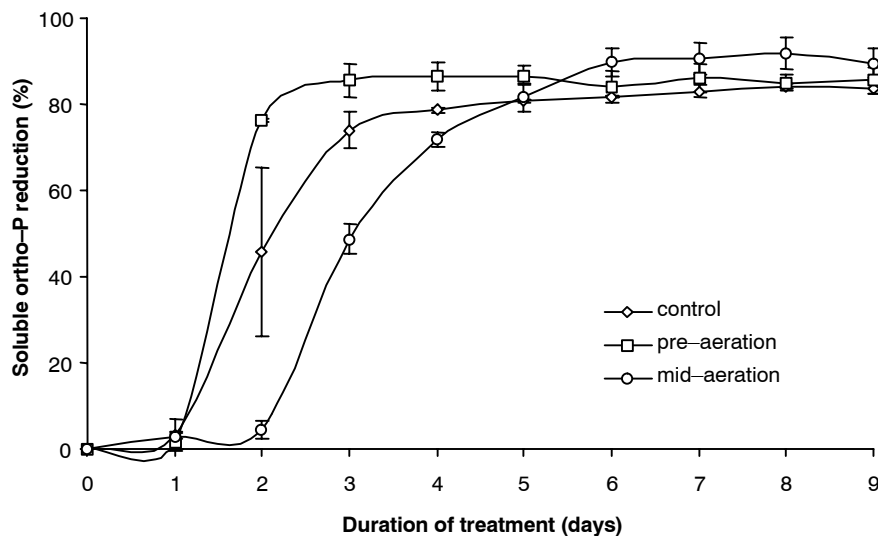
Treatment	TP in Raw Manure (PO <sub>4</sub> <sup>-3</sup> mg/L)	TP in Separated Liquid after Treatment (PO <sub>4</sub> <sup>-3</sup> mg/L)	Maximum Reduction Achieved (%) <sup>[a]</sup>
Control	493.17 ±9.94 <sup>[b]</sup>	167.54 ±0.91	66 a
Pre-aeration	493.17 ±9.94	148.86 ±14.57	70 a,b
Mid-aeration	493.17 ±9.94	99.91 ±1.82	80 b

<sup>[a]</sup> Values followed by the same letter are not significantly different at  $\alpha = 0.05$ .

<sup>[b]</sup> “±” refers to one standard deviation from the mean ( $n = 6$  for raw manure;  $n = 2$  after treatment).

observation suggests that a solids–liquid separation process on manure from a nursery pull–plug operated once or twice a week may not significantly solve the problem of higher P levels in the ensuing liquid manure since most of it is in solution already. This observation also makes a stronger case for separate collection of feces and urine because most of the P is usually held in the feces (Kroodsma, 1985). However, with current popular manure collection and handling systems (feces plus urine plus carrier water), it is quite clear that some form of treatment is necessary to reduce soluble ortho–P in the manure.

The changes in soluble ortho–P in the manure for the control and the two treatments are shown in figure 2. More detailed results of the percentage reductions and the respective statistical analyses are presented in tables 2 and 3 for both soluble ortho–P and TP, respectively. Large differences in ortho–P between the control and the MASL separation observed in figure 2 in days 2, 3, and 4 may be attributed to the large variation in the control within this time period. This large variation in the control, in our opinion, can largely be attributed to the kinetics of the microbial growth and hence substrate degradation. Within this period, a small time lag in bacterial growth in one reactor is amplified because the kinetics of growth in the others is fairly high. Hence, such variations are likely in reactors with the same substrate undergoing the same treatment. Low–level aeration followed by MASL separation has some advantages over PASL separation or the aeration of unseparated manure (control).



**Figure 2. Changes in soluble ortho–P in swine manure during aeration (error bars refers to one standard deviation,  $n = 2$ ).**

The MASL separation treatment leads to significantly more soluble ortho–P being converted to non–soluble species, which are amenable for removal by subsequent solids–liquid separation (84%, 87%, and 92% for the control, PASL separation, and MASL separation, respectively). This is evident in the data presented in table 3, which show a significantly higher overall removal of TP by subsequent removal of the sludge at levels of 80% for MASL, compared to 70% and 66% for the PASL and control, respectively.

### CHANGES IN pH AND RELATIONSHIP WITH ORTHO–P REDUCTION

A comparison of the change in pH and soluble ortho–P in the manure under the two treatments and the control is shown in figure 3. The general trends of pH change are exactly the reverse trends of soluble ortho–P in the manure liquid. Substantial changes in both pH and soluble ortho–P started between day 1 and 3 and slowed at approximately day 4, except for the MASL separation treatment, in which the two seemed to increase after the solids–liquid separation on day 4. In the first two days of aeration, the pH increase was less than 0.5 units. Previous studies by Zhu et al. (2001) and Ndegwa et al. (2001) indicated higher pH changes (1.0 and 1.5 units) in the first 1.0 and 1.5 days of aeration, respectively. The manure from these two studies came from deep pits under slat floors in finishing pig barns, while the manure for this study came from a nursery facility with a pull–plug manure management system. While the rapid pH changes observed in the Zhu et al. (2001) and Ndegwa et al. (2001) studies were believed to occur from purging of carbon dioxide from the manure slurry, this might not be the primary mechanism of raising the pH in this study. We believe that the rise in pH observed in this study is most likely due to oxidation of fatty acids and/or production of ammonia from ammonification of organic nitrogen. The latter postulation is well supported by the volatile fatty acids (VFAs) data obtained in this study and presented in figure 4. The strong coefficient of determination (0.9806) of the regression equation of the pH as a function of VFAs suggest that 98% of the increase in pH can be explained by the decrease in VFAs content in the manure.

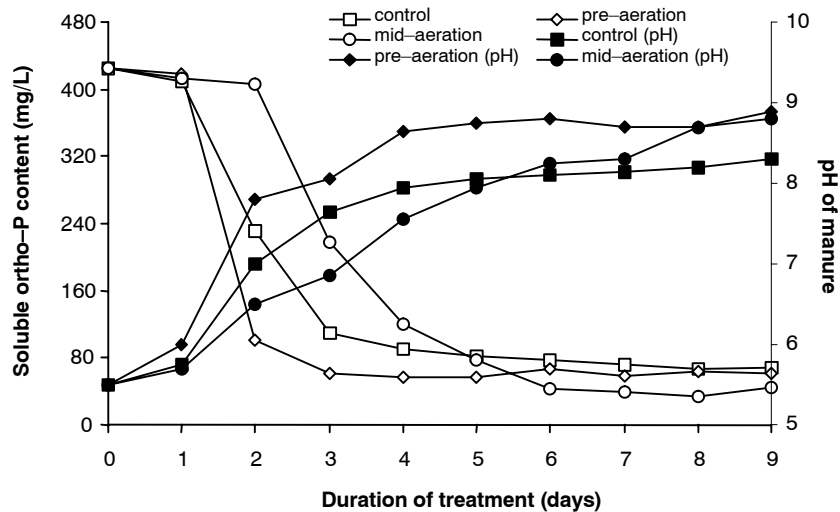


Figure 3. Comparison of pH change and ortho-P reduction in the manure during aeration.

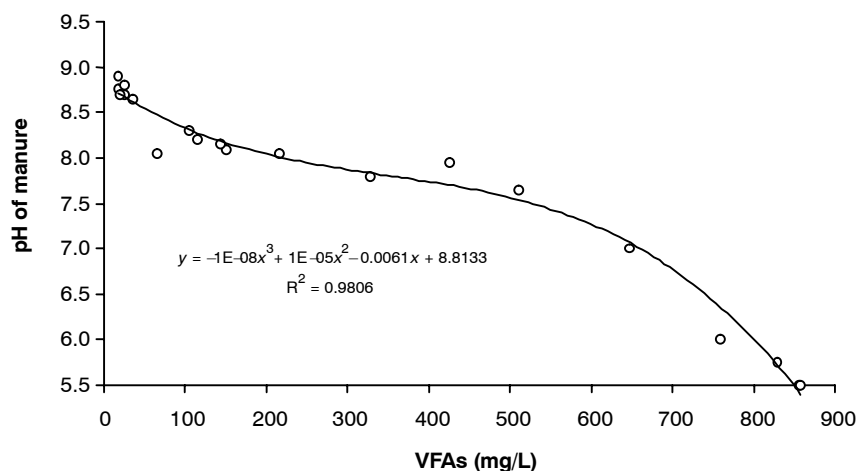


Figure 4. The pH of manure as a function of volatile fatty acids during aeration.

An increase in pH followed by a concomitant decrease in ortho-P, evident in figure 3, exhibits an inverse relationship between pH and the content of soluble ortho-P in the liquid manure. This phenomenon, in which soluble ortho-P precipitates into insoluble phosphates (iron phosphate, calcium phosphate, and aluminum phosphate) when pH increases, has recently been acknowledged as a major route in the removal of ortho-P from liquid manure (Zhu et al., 2001; Ndegwa et al., 2001; Luo et al., 2001). Moore and Miller (1994) also observed that the reactions between ortho-P and iron or calcium are highly dependent on pH. Within a pH range of 4.7 to 7.1, the reaction between iron and ortho-P predominates, while within the pH range of 7.2 to 12, the reaction between calcium and ortho-P predominates. On average, manure contains substantially more calcium ions than iron ions (Zhu et al., 2001); therefore, more ortho-P will be precipitated if pH moves towards the range in favor of the formation of calcium phosphate. This may explain the progressively higher removals of ortho-P in PASL and MASL compared to the control, because the pH increased progressively the same way.

#### CHANGES IN ORP DURING THE AERATION TREATMENTS

The variations in ORP in the manure during aeration for each of the two treatments and the control are presented in figure 5. During the first three days of aeration, the oxidation level remained higher in the unseparated manure than in the separated manure. A possible explanation for the former observation is that microbial action was enhanced more in the separated manure than in the unseparated manure because of the improved contact between the microbial mass and the dissolved substrate. However, prolonged aeration treatments seems to favor solids-liquid separated manure in terms of maintaining higher oxidation status at the same level of aeration. This is quite evident in figure 5 after the third day of aeration, where PASL separated manure stabilized at a much higher value (-15 mV) than the unseparated manure (-200 mV). Moreover, after the MASL separation at day 4, the ORP in the two reactors for this treatment immediately reversed and steadily increased, finally stabilizing at approximately the same ORP value as that of the PASL separated manure. However, even with prolonged aeration, the ORP status of the control reactors containing the unseparated manure still stabilized at the same lower ORP levels of approximately -200 mV. These observations suggest that the oxygen transfer efficiency is significantly improved by

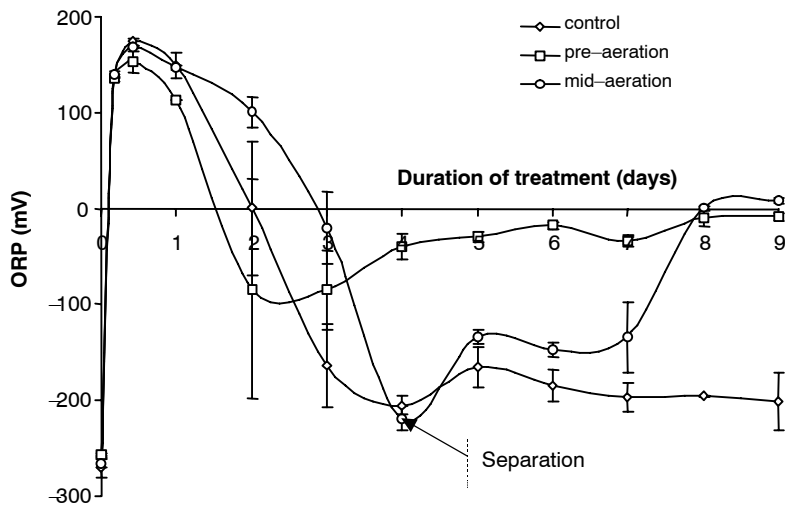


Figure 5. Oxidation–reduction potential (ORP) status in the manure during aeration (error bars refers to one standard deviation,  $n = 2$ ).

solids–liquid separation treatments. The consequence of this postulation is that it is possible to use only a fraction of the energy to maintain adequate ORP level, especially in the latter period of the treatment. This is especially important for ensuring that P already removed from the liquid stays in the sludge and is not released back into solution if there is delay in complete removal of the sludge. Charpentier et al. (1987) proposed maintaining an ORP level of above  $-300$  mV in wastewater to prevent release of P back into the liquid, but such a value has not been established for swine manure. This observation also has an important economic dimension in aeration treatment of manure for control of odor generation, where prolonged aeration treatments may be necessary.

#### RELATIONSHIP BETWEEN pH AND ORP

The changes in pH and ORP with time of aeration are presented in figure 6 for the control, PASL separation, and MASL separation. Based on these results, the increase in pH to some extent was followed by a reduction of ORP in the manure. These observations are consistent given the fact that an oxidizing environment is characterized by the ability of a

system to accept electrons, while a reducing environment is characterized by its ability to donate electrons. A rise in pH generally means a reduction in  $H^+$  ion concentration. Hydrogen ions are potential acceptors of electrons; hence, a reduction in their concentration translates into a decrease in the oxidation potential. For the control treatment and within the range of pH change observed in this study, the ORP decreased as long as the pH was increasing. However, a different scenario was observed for the two treatments (PASL and MASL). For the PASL separation treatment, the ORP initially decreased with the rise in pH between pH 5.5 and 7.8, after which further increase in pH was followed by an increase in ORP status in the manure (between pH of 7.8 and 8.8). For the MASL separation, beside the difference in the ranges of pH in which specific trends were observed, the variation of ORP with pH was found to approximately resemble that of the PASL separation treatment, in general.

We believe that the combined effects of pH and microbial oxygen consumption on the ORP outweighed the oxygen addition in the initial stages of aeration in both the treatments and the control. In the latter stages of aeration, however, with

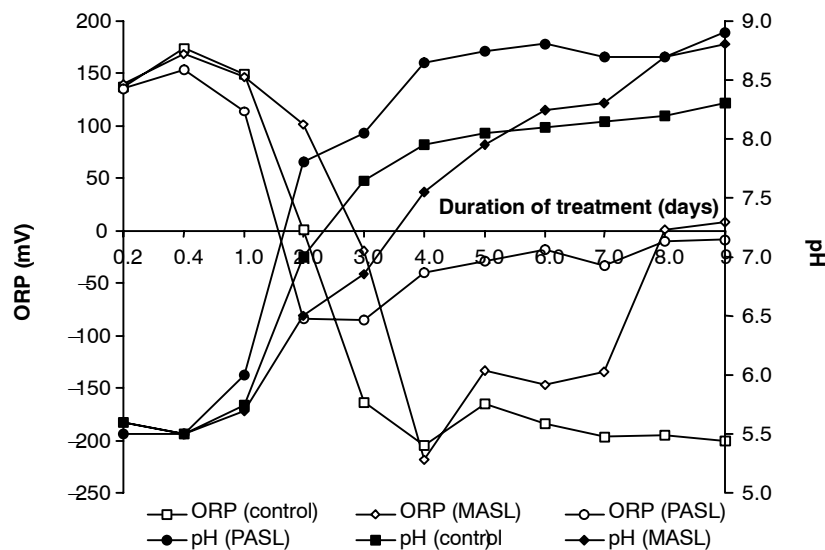


Figure 6. Changes in pH and oxidation–reduction potential (ORP) in the manure during aeration.

manure possibly stabilized (reduced microbial activity), it was only the effect of pH acting against oxygen addition. Because of the possibly improved oxygen transfer efficiency in the separated liquid manures (PASL and MASL treatments), the oxidation potential due to oxygen levels were adequate to cancel the effect of pH in the latter stages of aeration. The poor oxygen transfer efficiency in unseparated manure, however, limited the counteracting effect of oxygen-induced oxidation potential against the decrease in oxidation potential due to increasing pH in the unseparated manure (the control).

## SUMMARY AND CONCLUSIONS

In the raw swine nursery manure from a pull-plug house used in this study, the soluble ortho-P was the largest component of TP, comprising approximately 86%, while the combined organic-P and insoluble ortho-P comprised only 14% of TP. Given that even a 100% removal of solids would not remove this entire 14% because of the soluble organic P component, solids-liquid separation of this nursery manure without some kind of pre-treatment will not significantly solve the problem of higher phosphorus levels in liquid manure.

MASL separation resulted in significantly more soluble ortho-P being converted to non-soluble species, which are amenable for removal by subsequent solids-liquid separation: 84%, 87%, and 92% for the control, PASL separation treatment, and MASL separation treatment, respectively. The MASL separation similarly improved the overall removal of TP in a subsequent removal of the sludge: 80%, 70%, and 66% for MASL separation, PASL separation, and control, respectively. Clearly, the removal of soluble ortho-P is significantly higher than the overall removal of TP. An improvement in solids-liquid separation is therefore necessary to realize the gains achieved in the soluble ortho-P removal during the low-level aeration, whether solids are separated before or during aeration.

Solids-liquid separation enhanced the maintenance of higher oxidation potentials in the manure (shown by higher values of ORP in the separated manures than in the control) during the latter days of aeration, implying that significant energy saving is possible for prolonged aeration of separated liquid manure. This is an important element for post-aeration treatment for ensuring that P already removed from the solution stays in the sludge and is not released back into solution if removal of the sludge does not immediately follow scheduled aeration treatment.

## ACKNOWLEDGEMENTS

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