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Optimization of Phosphorus Partitioning in Dairy Manure Using Aluminum Sulfate with a Mechanical Solids Separator

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Abstract. *The over application of phosphorus as land applied animal manure is a problem on many livestock farms in the United States. The use of liquid aluminum sulfate (alum) to precipitate soluble phosphorus from dairy manure was investigated in conjunction with a mechanical screw-press solids separator. In the molar ratio optimization studies aluminum to soluble phosphorus ratios of 2.5, 5 and 7.5:1 were investigated in settling tests using Imhoff cones. The total suspended solids in the chemically amended supernatant were 56, 86 and 93% less than in the supernatant from the control. Also, the soluble phosphorus concentration in the alum-amended supernatant was 68, 95 and 98% less. For the screw press separator tests, alum was added at a aluminum to soluble phosphorus molar ratio of 5.5:1. The alum addition increased the press liquor flow rate by 35%, but it did not increase the dry mass capture efficiency. The soluble phosphorus concentration was immediately reduced in the screw press influent after the addition of chemical where it remained in the precipitate form throughout the separation process.*

Keywords. Dairy manure, screw press, aluminum sulfate, Imhoff cones, soluble phosphorus

Introduction

Nitrogen based land application of animal manure enhances phosphorus accumulation in the soil. Many agricultural lands in the US contain high levels of phosphorus. Over application of phosphorus in the soluble form increases the probability of phosphorus enrichment to surface waters during rain events. Reduction of soluble phosphorus animal manure prior to land application would reduce phosphorus runoff and help prevent eutrophication.

Soluble phosphorus can be reduced in animal wastewaters by the addition of chemicals that form insoluble precipitates between the dissolved, or soluble, ions in the waste, such as phosphate, and the metal ion added. Typical metal ions used for the amendment include calcium, iron and aluminum. Commonly used chemicals include the chloride or sulfate salts of these metals (Zhang and Westerman, 1997). While these chemicals form precipitates, they also enhance coagulation of suspended particles.

Many researchers have conducted experiments using chemical amendments to achieve phosphorus separation in low total solids (TS) manure (TS < 2) (Barrow, 1997; Chastain el al., 2001; Powers, 1995; Vanotti and Hunt, 1999; Westerman and Bicudo, 2000; Worley and Das, 2000; Zhang and Lei, 1996), but; little research has been performed using chemical amendments to achieve phosphorus separation in high TS manure (TS > 4). In the studies with low TS manure, phosphorus separation after chemical amendment was achieved through gravity settling.

Mechanical separation is an effective way to remove solids from manure slurries. A screw press separator is one type of mechanical separation device. The screw press uses a central screw conveyor within a cylindrical screen to remove solids. Research has shown that following solids separation with a screw press nutrients in the organic form are preferentially partitioned into the solids fraction while the soluble forms are partitioned into the liquid fraction (Burns and Moody, 2000). The addition of a metal salt prior to mechanical separation should increase screw press solids capture efficiency. In addition, the formation of an insoluble phosphorus precipitate should enhance the partitioning of soluble phosphorus into the recovered solids.

The objectives of this work were to 1) determine the optimal aluminum to soluble phosphorus addition ratio for soluble phosphorus precipitation and suspended solids reduction, and 2) determine the effect of liquid aluminum sulfate addition, using the optimal ratio determined in objective one on screw press solids capture efficiency and soluble phosphorus partitioning partitioning.

Materials and Methods

Dairy manure for the experiments was collected from a dry stack at the Cherokee Dairy Unit of the Knoxville Experiment Station at The University of Tennessee. During both the Imhoff cone settling tests and the mechanical screw press separator tests, fresh manure was collected before each experiment. The collected manure had an average TS concentration of 30%. Using tap water, it was diluted to ~8 %TS. The diluted manure was mixed in a 19 L bucket with a hand held electric paddle mixer for 5 minutes to obtain a homogeneous mixture.

Analyses performed during the experiments included TS, total suspended solids (TSS), soluble phosphorus (SP), total phosphorus (TP) and pH. Total solids and TSS were analyzed using Standard Method 2540 (APHA et al., 1998). Soluble phosphorus was analyzed using the Ascorbic Acid Method 4500-P E, and TP was analyzed using Persulfate Digestion Method

4500-P B5 (APHA et al., 1998). Both were analyzed by determining PO_4^{3-} on a Spectrophotometer 20.

Imhoff Cone Optimization Tests

To select the optimal dose of alum addition for phosphorus and TS reduction, aluminum to soluble phosphorus ratios of 0:1 (the control), 4:1, 8:1 and 12:1 were tested in settling studies using Imhoff cones. To perform the experiments, manure diluted to 8% TS was analyzed for SP to determine the amount of aluminum required for each experiment. All experiments were replicated in triplicate; each replication included the four ratios. To perform a replication, the diluted waste was continuously mixed while 1 L quantities were distributed into four 2 L Nalgene beakers. The waste in the beakers was stirred for five minutes on magnetic stir plates. Then, the predetermined volume of alum was pipetted into the waste and allowed to mix for five minutes. After five minutes of mixing, the magnet was removed and the waste was poured into an Imhoff cone. The Imhoff cone settling tests duration was 240 minutes. Readings were taken at 2, 5, 10, 20, 40, 60, 120 and 240 minutes to monitor the settling layer. Following the tests, the supernatant and the settled solids were separated for analysis. The supernatant was analyzed for TS, TSS, SP, TP and pH. The settled solids were analyzed for TS, SP, TP and pH.

Screw Press Separation Tests

A Vincent CP-4 (*Vincent Corporation, Tampa, FL*) was used in the laboratory for the mechanical screw press alum amendment testing. The CP-4 has a 100 mm diameter stainless steel screw that is 343 mm in length; it turns at 30 rpm. The screen around the screw is 0.38 mm stainless steel wedge wire. A 4 kg weight was used placed on the unit to provide back pressure on the separator outlet cone.

For the separator tests, nine buckets containing 17 L of 8 %TS were prepared. The mixture was analyzed for SP so that the quantity of alum amendment could be determined. To start the separator tests, 17 L of waste were poured through the screw press. To enhance the building of the initial press cake, 2000 cm^3 of wood shavings were added at the beginning of the experiment. During the testing, four replicates were performed on the control and four replicates were performed using the chemical amendment. Each replication had a volume of 17 L, which filled the hopper on the CP-4 separator.

The control replications were tested first. For the fourth replication, six tablets of red fluorescent dye (*Formulabs, Escondido, CA*) were crushed and mixed in the container of waste and processed in the screw press. The result was red tinted press liquor and press cake. This test was followed by a container of alum-amended material. When the red tint was no longer in the press cake and the separation unit well was empty, the chemical amendment replications were started. For each chemical amendment replication, alum was added to the waste and mixed for five minutes before it was poured into the separator hopper and processed in the screw press.

For each replication, the process was timed and the press liquor and press cake were collected and weighed. Samples of each replication influent were taken before and after chemical addition, and samples were taken from the press liquor and press cake when half of the influent had been processed. The influent and press cake were analyzed for TS, SP, TP and pH and the press liquor was analyzed for TS, TSS, SP, TP and pH.

Results and Discussion

Imhoff Cone Optimization Tests

At mixing, the 8% TS slurry contained a SP concentration of 82 mg PO_4^{3-} (L)$^{-1}$. To achieve the defined aluminum to SP ratios of 4, 8 and 12:1, aluminum sulfate was added at 1.66, 3.32 and 4.98 mL (L waste)$^{-1}$. However, the average SP concentration of the control supernatant was 240 mg PO_4^{3-} (L^{-1}) when analyzed after the experiments. This concentration was ~3 times greater than the SP concentration when it was first mixed. This occurred because while the initial SP was being determined, phosphorus was still being water extracted from the collected manure. Also, the manure was stirred on the magnetic plate for an additional 5 minutes prior to being amended with chemical. In the future, to avoid this problem, the waste should be mixed for two hours prior to being tested for SP. This would mimic the SP extraction technique for solid manure. This means that instead of aluminum to soluble phosphorus ratios of 4, 8 and 12:1, the actual molar ratios were 2.5, 5 and 7.5:1.

Figure 1 shows the effect of alum addition on setteable solids. Adding alum increased the volume of settleable solids determined during Imhoff cone tests. The TSS of the supernatant was greatly decreased when alum was used. Figure 2a shows the average concentration of TSS as the molar ratio of aluminum to soluble phosphorus was increased. At the molar ratios of 2.5, 5 and 7.5:1, the average TSS is 56, 86 and 93% less than the average TSS concentration in the control supernatant, respectively. The volume of settleable solids in mL per L at any given time in the Imhoff cones increased with increasing aluminum addition. This is caused by the increase in voluminous precipitate forming with the addition of aluminum.

Both soluble and total phosphorus in the supernatant were reduced as aluminum addition increased. Figure 2b shows the average concentration of SP and TP as the molar ratio is increased. At molar ratios of 2.5, 5 and 7.5:1, the average SP is 68, 95 and 98% less than the average SP concentration in the control supernatant, respectively, and the average TP is 62, 81, and 88% less than the average TP concentration in the control supernatant, respectively. When alum was added, the TP in the settled solids increased, however; it did not increase with the increasing molar ratio. At a molar ratio of 2.5, 5 and 7.5:1, the TP was 44, 31 and 35% greater in the settled solids than in the control settled solids, respectively. The increased TP in the settled solids after alum addition is the result of the precipitation and coagulation reactions.

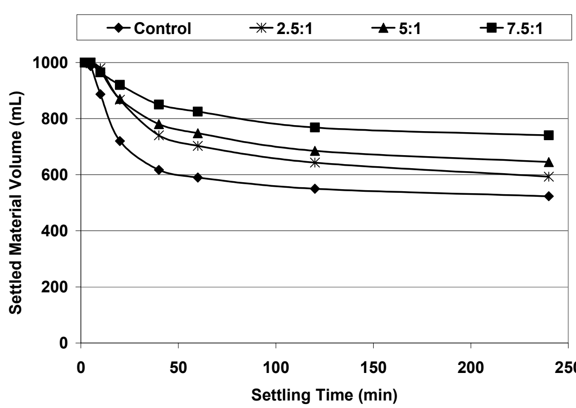
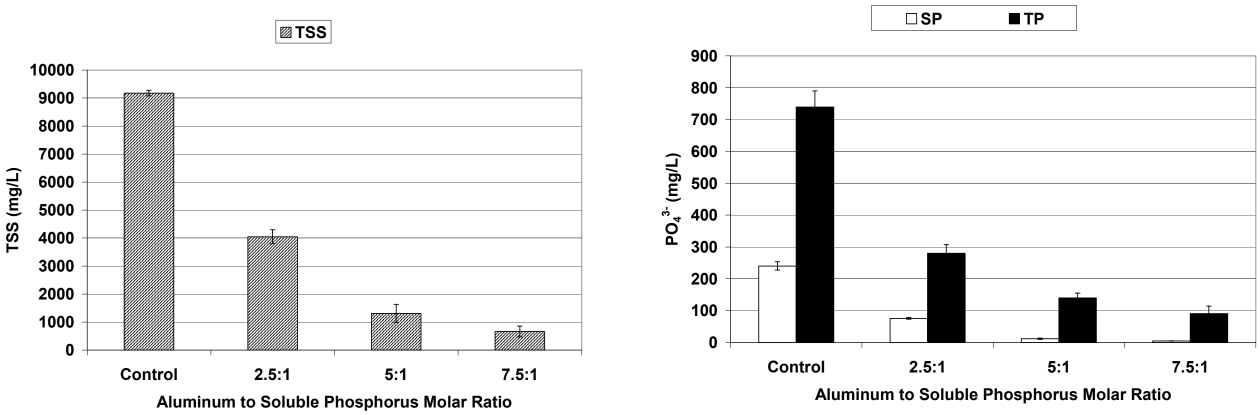


Figure 1. Average volume of material settled with respect to time during the Imhoff cone settling tests for each aluminum sulfate amendment ratio.



(a) (b)

Figure 2. Average constitute concentration in the Imhoff cone supernatant following 240 minutes of settling, (a) total suspended solids (TSS) and (b) soluble phosphorus (SP) and total phosphorus (TP). Error bars indicate the standard deviation in the results.

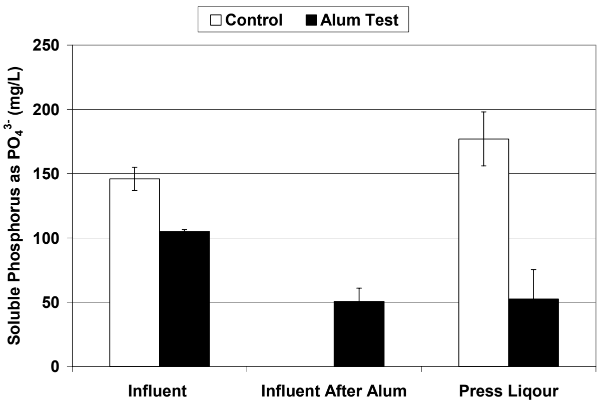
Screw Press Separation Tests

At mixing, the waste for the screw press test was intended to contain a TS of 8%, however; the actual mixture had a TS of ~4%. Alum was added at a rate of 2.94 mL (L waste)$^{-1}$; the aluminum to soluble phosphorus ratio was 5.5:1. During the tests with the screw press separator, the average press liquor flow rate was increased by 35% from 3.4 L (min)$^{-1}$ (± 0.08) to 4.6 L (min)$^{-1}$ (± 0.03) with the addition of alum. The dry mass capture efficiency was calculated using the testing protocol established by Burns and Moody (2001)(Equation 1). The average dry mass capture efficiency was 65% (± 3.3) for the control and 58% (± 3) for alum addition. While it appears that the dry mass-capture efficiency decreased with the use of alum, the standard deviations are too large to make this statement.

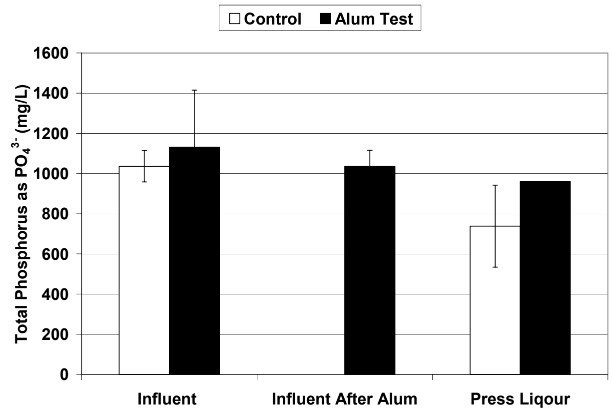
$$Eff.\ (\%)_{Capture} = \frac{m_{in}(\%TS_{in}) - m_{PL}(\%TS_{PL})}{m_{in}(\%TS_{in})} \times 100 = \frac{m_{PC}(\%TS_{PC})}{m_{in}(\%TS_{in})} \times 100 \qquad \text{(Equation 1)}$$

Eff. $(\%)_{Capture}$ is dry mass capture efficiency, m_{in} is influent mass, m_{PL} is press liquor mass, m_{PC} is press cake mass, $\%TS_{in}$ is percent total solids in the influent, $\%TS_{PL}$ is percent total solids in the press liquor, and $\%TS_{PC}$ is percent total solids in the press cake.

The addition of alum did have an effect on the SP concentration in the press liquor and the press cake. Without the addition of alum (the control), the SP concentration increased in the press liquor by 18% as compared to the influent slurry (Figure 3a). However, with the addition of alum, the soluble phosphorus in the press liquor was 50% less than the SP in the influent slurry. In the press cake, the percent of TP as SP decreased with the addition of alum. In the control, 6.4% of the TP in the press cake was soluble. However, with the addition of alum, only 3.3% of the TP in the press cake was soluble. No significant decreases were observed in the press liquor or the press cake in TP (Figure 3B). One goal of amending dairy manure with alum was to partition phosphorus from a soluble form in the press liquor to an insoluble form in the press cake in the form of an aluminum phosphate precipitate. While amendment with alum was very effective at reducing phosphorus in the soluble form by forming an insoluble precipitate, this precipitate was not partitioned into the captured manure solids. The fact that no appreciable decrease in TP was noted in the press liquor indicates that the precipitate was not effectively captured by the manure solids separator. As shown in Figure 4 alum addition had no effect on TS levels in the press liquor or press cake.



(a)



(b)

Figure 3. Average constituent concentration in the Control and Alum Tests in the influent and the press liquor, (a) soluble phosphorus and (b) total phosphorus.

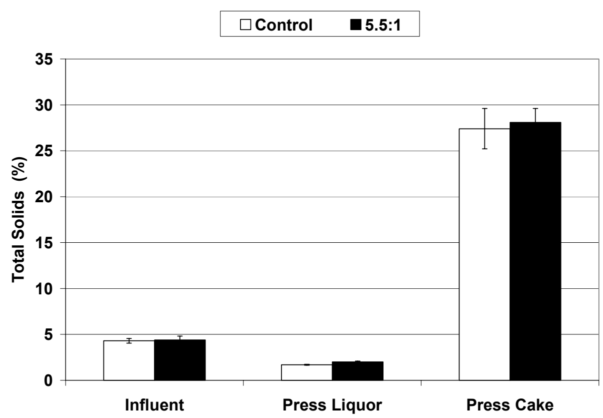


Figure 4. Average total solids concentration in the Control and 5.5:1 molar ratio tests at various phases of the solid separation process.

Conclusion

In the Imhoff cone tests, aluminum sulfate amendment to dairy manure effectively precipitated soluble phosphorus (SP) from solution and reduced the total suspended solids (TSS) in the supernatant. An aluminum to phosphorus ratio of 5:1 removed 95% of the soluble phosphorus in the supernatant. In screw press testing dairy manure amended with aluminum sulfate increased the press liquor flow rate by 35%, but did not increase the dry mass capture efficiency. Amendment of dairy manure with alum was very effective at reducing phosphorus in the soluble form by forming an insoluble precipitate. However, this precipitate was not partitioned into the captured manure solids. The addition of alum can effectively reduce SP levels in dairy manure. A reduction in manure SP before land application may be an effective method of limiting the risk of phosphorus runoff from manure application areas.

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