Precipitating swine manure phosphorous using fine limestone dust

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Barrington, S.F., Kaoser, S., Shin, M. and Gélinas, J.B. 2004. **Precipitating swine manure phosphorous using fine limestone dust.** Canadian Biosystems Engineering/Le génie des biosystèmes au Canada **46**: 6.1 - 6.6. The effectiveness of fine limestone dust in precipitating swine manure total phosphorous (TP) and total solids (TS) was measured using 3-L volumes and a 1.30 m^3 volume. Respective duplicate limestone samples with particle sizes of 10, 17, 30, 12, 21, and 30 μm, (PULPRO 10, 17, and 30 and SW 12, 21, and 30) were mixed at levels of 0 and 6% into swine manure with a TS and TP of 7.4% and 1210mg/L, respectively. SW 21 was also mixed into the same swine manure at a 2% level. The 1.30 m^3 volume of 8.3% TS swine manure was treated with 6% PULPRO 30. The depth of sludge precipitation was monitored over time and the supernatant and sludge were analyzed for TP, TS, and pH after 12 and 30 days for the 3-L and 1.30 m^3 volumes, respectively. With 2 and 6% SW21, the supernatant liquid was analyzed a second time for TP after 60 days of settling. All limestone particle sizes and dosages performed similarly, by precipitating as much as 96 and 90% of the TP and TS into a sludge equivalent to 53% of the manure volume. Letting the supernatant settle for 60 days rather than 12 days further reduced its TP content by 40%. The 1.30 m^3 volume test further demonstrated that the sludge could be pumped despite its TS and density of 22% and 1.07 kg/L, respectively. The addition of 2% limestone improved TP and TS precipitation by 4% and greatly improved sludge density and sludge separation from the supernatant. **Keywords:** limestone, swine manure, TS and TP precipitation.

Des poussières fines de calcaire furent utilisées en laboratoire pour précipiter le phosphore total (PT) et les matières solides totales (ST) du lisier de porc. Dans le laboratoire, des volumes de lisiers brutes de 3-L furent traités alors que dehors, un volume de lisier brute de 1,30m^3fut traité. Les poussières de calcaire PULPRO 10, 17 et 30 et SW 12, 21 et 30 possèdent un granulométrie de 10, 17, 30, 12, 21 et 30 μm, respectivement. Chacune de ces poussières fut mélangée à un taux de 6%, dans du lisier avec une teneur en ST et PT de 7.4% et 1210 mg/L. Le SW 21 fut aussi mélangé à un taux de 2%. Le PULPRO 30 fut mélangé à l'extérieur et à un taux de 6%, dans du lisier de 8.3% de ST. L'épaisseur de boue formé en fonction du temps, et le volume et la qualité des boues et du surnageant furent déterminés après 12 jours, pour les essais en laboratoire, et après 30 jours, pou l'essai à l'extérieur. Le surnageant du mélange utilisant 2 et 6% de SW21, fut aussi analysé après 60 jours de précipitation. Toutes les poussières, aux deux taux de 2 et 6%, ont précipité sensiblement le même niveau de ST et de PT, soit jusqu'à 90 et 96%, dans un volume de boue équivalent à aussi peu que 53% de celui du lisier brute. Une période de précipitation de 60 jours, comparativement à 12 jours, produisait un surnageant avec une teneur en PT de 100 comparativement à 170 mg/L, soit 96% du PT initial. L'essai extérieur produisait des boues qui se pompaient et une qualité de surnageant semblable à celle produit dans le laboratoire. Quoique aussi peu que 2% de calcaire améliore le taux de précipitation de ST et PT de 4%, plus ou moins, ce traitement améliore la densité des boues et leur reprise sans le surnageant. **Mots clefs**: calcaire, lisier de porcs, précipitation du PT et des ST.

INTRODUCTION

Manure dewatering and solid/liquid separation can serve several purposes. In the collection of sludge from lagoons, a better balance in nutrient levels can be obtained for plant uptake, especially for total nitrogen (TN) and total phosphorous (TP), as reported by Worley and Das (2000). In getting manure to cropped land, dewatering can concentrate nutrients, thus reducing transportation costs (Barrington et al. 2002). In handling manure, the separation of solids can prevent the clogging of transfer pipes and the malfunction of pumping systems (Chastain et al. 2001a).

Techniques for the dewatering of swine manure have encompassed solid/liquid separation, the precipitation of nutrients using flocculants, and the aerobic/anaerobic treatment of manure. The total solids (TS) removal efficiency of mechanical separators was extensively reviewed by Zhang and Westerman (1997) who reported that gravity settling, screen separators, and centrifuge separators removed 60, 30, and 60% of the TS, respectively. Nevertheless, the removed solids had a moisture content exceeding 80% and a TP representing 30% of the initial manure TP. Chastain et al. (2001b) removed 45 and 16% of the TS and TP from swine manure using a screw press. Zhang and Lorimor (2000) used crop residues to filter swine manure and remove TS. Oat straw, soybean stubble, and corn cobs, chopped to respective lengths of 100, 300, and 40 mm, removed up to 50, 40, and 23% of the TS, on a dry mass basis, respectively.

The precipitation of solids from swine manure has been achieved using several flocculants. Vanotti and Hunt (1999) removed 46% of the TP in swine manure using PAM (cationic polyacrylamide polymer). Worley and Das (2000) reported removing 70 and 75% of the TS and TP using 0.5% alum by volume. Barrington et al. (1995) were able to precipitate 98% of the TP contained in swine manure at 5% TS by adding 33% kiln dust by weight. Although efficient in precipitating TP from swine manure, these chemical flocculants change the properties of the products. For example, manure Cl^- , SO_4^{2+}, and pH level can be significantly increased (Luo et al. 2001).

The aerobic treatment of swine manure was extensively researched during the 1970's to reduce its TN content by scrubbing. Lately, the biological treatment of wastewater, consisting of an anaerobic phase followed by an aerobic phase,

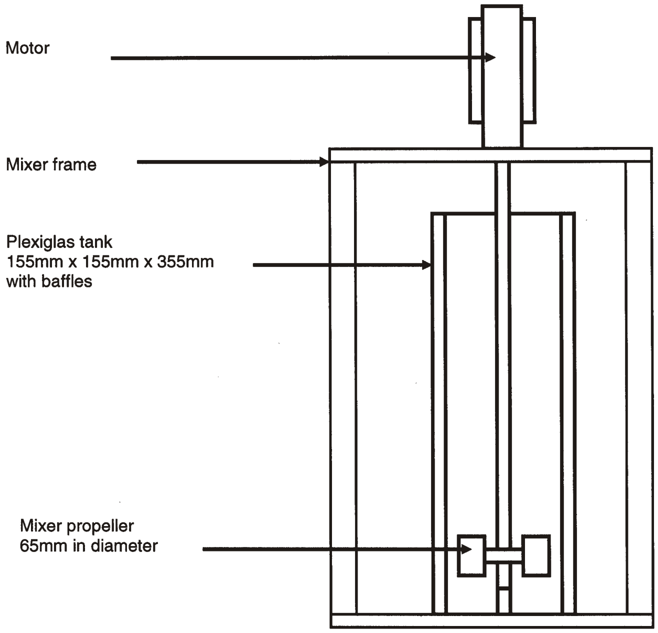

Fig. 1. Profile view of the laboratory mixer used to incorporate the limestone dust at a blade tip speed of 0.61 m/s.

has lead to significantly higher TP removal. Applied to swine manure, this treatment was found to remove 76% of the TP, while increasing the pH of the manure from 7.5 to 8.5 (Luo et al. 2001).

Maintaining the same manure pH throughout the dewatering process is important in achieving a closer TN and TP balance for manure sludge or separated solids. Increasing the pH of manure leads to the volatilization of ammonia produced by the transformation of NH_4^+ into NH_3 (Luo et al. 2001).

Limestone ($CaCO_3$) can precipitate phosphates to form calcium phosphate ($CaHPO_4$; $Ca(PO_4)_2$) and their complexes, which are both insoluble (Buckman and Brady 1969). As opposed to lime (CaO), calcium carbonate is an excellent buffer, precipitates before the pH exceeds 8.0, and thus has little effect on pH level. Using fine limestone dust, Barrington et al. (2002) removed 90% of TP from manure screened using a 0.1 mm sieve. The fine limestone had little effect on the pH of the treated manure supernatant and precipitated solids. The particle size distribution of the limestone dust had an impact on the level of TS and TP precipitated. Manure sieving was conducted to duplicate the effects of mechanical separation and this separation is an additional cost to the treatment process. Furthermore, many mechanical separators are not as efficient as the laboratory sieving process.

Therefore, further research is required to test the performance of fine limestone without mechanical separation reproduced through laboratory sieving. The objective of the project was to investigate the precipitation of whole swine manure TS and TP following the incorporation of fine limestone dust of various particle sizes and types: one type was referred to as PULPRO limestone (particle size of 10, 17, and 30 µm); the second type was referred to as SW limestone (particle size of 12, 21, and 30 µm). The limestone's effectiveness in precipitating swine manure TS and TP was compared to a control, swine manure with no limestone addition.

MATERIALS and METHODS

Experimental materials

Fresh swine manure (7.4% TS and 1210 mg/L of TP) was obtained from the farrowing to finishing piggery of the Macdonald Campus farm of McGill University, located in Montreal, Quebec. The fresh manure was analyzed for pH, density, total solids (TS), and total phosphorous (TP). OMYA St. Armand (Montreal, Quebec) supplied the PULPRO and SW limestone dusts, which originated from limestone quarries located in St. Armand, Quebec and Perth, Ontario, respectively.

Precipitation apparatus

All laboratory tests were conducted using a rectangular Plexiglas® container measuring 155 mm by 155 mm by 355 mm in height (Fig. 1). This container was equipped with a variable speed propeller mixer. The centre of the mixer propeller stands 75 mm above the container floor and has a diameter of 65 mm. The rotational velocity of the mixer was adjusted to obtain a blade tip speed of 0.61 m/s, corresponding to that recommended for flocculation (Viessman and Hammer 1985).

Method

A large sample of swine manure was obtained, analyzed, and refrigerated to conduct all laboratory tests. When all tests were completed, the remaining sample was analyzed for TS. The remaining manure had the same TS as was tested initially, indicating that all tests were conducted with the same well mixed and uniform manure.

All tests were conducted by mixing 180 g (6% by mass) of limestone dust into duplicate 3L samples of swine manure. As control, duplicate samples of swine manure, without limestone, were also allowed to precipitate. As the mixture settled for 12 days, the sludge depth was measured regularly. After 12 days, the supernatant liquid and sludge were measured and removed to be sampled and analyzed for density, TS, TP, and pH. The sludge and supernatant mass and volume were calculated by multiplying their density by the depth and cross sectional area of the mixer.

The laboratory test consisted of duplicates using PULPRO 10, PULPRO 17, and PULPRO 30 and SW 12, SW 21, and SW 30, at a dosage of 6%. A subsequent test was conducted using 2% SW 21 on 3 L of the same swine manure.

Two batches of 650 L of 8.4% TS swine manure were treated by mixing into each batch 40 kg (6% by wet mass) of PULPRO 30. The manure and limestone were mixed in a container measuring 0.9 by 0.9 by 1.0 m in height, offering a capacity of 810 L and equipped with a mixer with a propeller measuring 0.61 m in diameter and turning at more than 1 m/s. The two manure and limestone mixes were pumped into a 1.5 m^3 rectangular tank and allowed to settle outside for 30 days under temperatures varying between –5 and 10ºC. After 30 days, both the supernatant liquid and sludge were measured and sampled. A 1.5 kW pump was used to transfer the sludge back to the pre-pit of the piggery to test its fluidity.

Analytical procedure

All manure fractions were analyzed using standard methods (APHA 1995). The moisture content was determined by drying at 103°C for 24 hours. The pH was determined using a pH probe

Table 1. Chemical composition of experimental limestone.

Component	SW (%)	PULPRO (%)
SiO_2	15.25	2.96
Al_2O_3	3.89	0.97
Fe_2O_3	0.77	0.27
MgO	1.42	0.88
CaO	42.7	52.63
Na_2O	0.14	0.07
K_2O	1.54	0.12
TiO_2	0.16	0.044
P_2O_5	0.05	0.012
MnO	0.004	0.004
Cr_2O_3	0.015	trace

(Orion Corporation, Model 7200A, Boston, MA). TP was measured on a digested sample using a colorimetric method (Hach Corporation, Loveland, CO). Density was measured using a density flask.

Experimental design and mass balance analysis

All tests were duplicated to measure the variation in sludge settling depth and the density and chemical properties of the sludge and supernatant liquid. The sludge settling depth varied by almost 10% during the first day, but by less than 2% thereafter. The density of the sludge and supernatant liquid varied by less than 3%. The TS and TP levels of the sludge and supernatant liquid varied by less than 2.5 and 5%, respectively. The significance among treatment was conducted using ANOVA and the Duncan's New Multiple Range Test (Steel and Torrie 1986).

A mass balance analysis was conducted between the limestone dust and raw manure TS and TP versus that of the precipitated sludge and the supernatant liquid to verify the accuracy of the results. To match these two levels, the sludge TS and TP were corrected by an additional value of ± 5%. It was logical to correct the value of the sludge TS and TP, since the sludge separation from the supernatant liquid was not perfect, and the sludge was not analyzed right after separation and further settling occurred in the mean time.

RESULTS and DISCUSSION

Limestone chemical characteristics

The PULPRO and SW limestone have different origins and therefore different chemical characteristics (Table 1). PULPRO has a higher CaO content while SW has a higher SiO_2 and Al_2O_3 content. Thus, PULPRO limestone is expected to be more active in precipitating swine manure P. SW had a higher nutrient value, in terms of P_2O_5, K_2O, and MgO content.

On a long-term basis, using either PULPRO or SW as swine manure TP precipitant will have little effect on a soil's chemical properties. First of all, either of the limestone dusts is of equivalent quality, if not better, than the coarse limestone normally applied to correct a soil's pH. Secondly, using the sludge precipitated with limestone can help maintain a soil's pH year after year. For example, a corn crop extracts 20 kg of P/ha per year and 7.5 kg of P/ha if applied in the planter as pop-up fertilizer. The rest of the P can be applied as swine manure sludge precipitated using fine limestone dust, at a rate of 12.5 kg P/ha per year. As will be seen later, this is equivalent to applying 6.25 m^3 of sludge/ha per year or 250 kg of limestone/ha per year. This limestone dosage is equivalent to the annual losses in Ca associated with soil mineral leaching (Gosselin 1986).

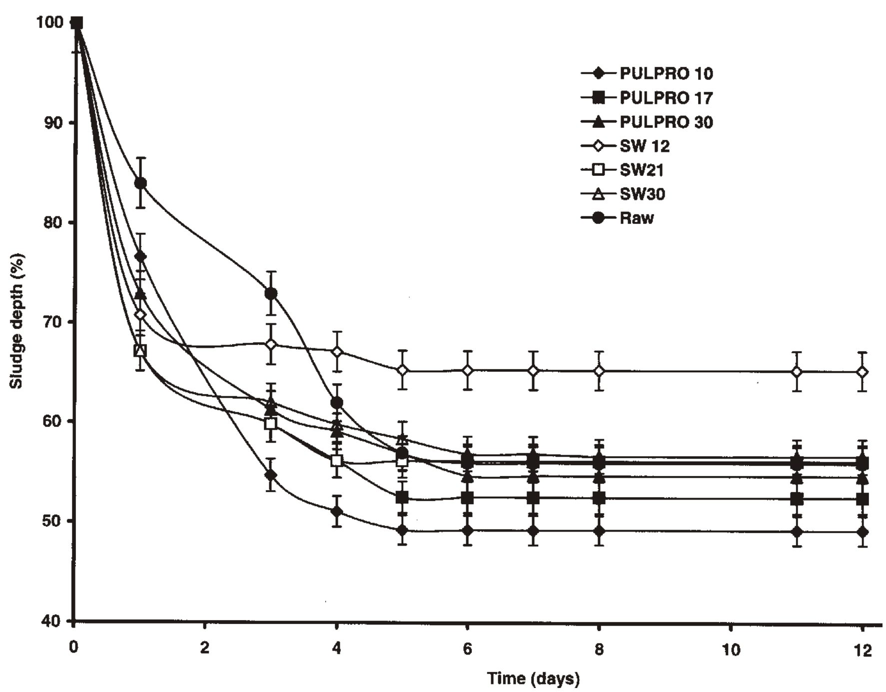

Fig. 2. Precipitation speed of limestone dusts incorporated into 7.4% TS swine manure at a wet mass of 6%.

Laboratory tests

The precipitation depth of the manure sludge, after limestone incorporation, is illustrated in Fig. 2. The smallest sludge depth or volume produces the most dewatering and the most effective volume of concentrated manure P to transport to distant cropped land.

PULPRO 10 produced the least amount of sludge. PULPRO 17 and 30 and SW 21 and 30 produced similar amounts of sludge, while SW 12 produced the most amount of sludge. As with previous work on screened swine manure (Barrington et al. 2002), sludge depth increases with limestone particle size for PULPRO. The inverse relationship is observed with SW. Figure 5 will later show that PULPRO 30 produced more sludge because it was more efficient in precipitating TS and TP.

The swine manure without limestone produced a sludge depth similar to that of SW21 and SW30. Thus, only the PULPRO limestone produced sludge depth smaller than that of the control, the

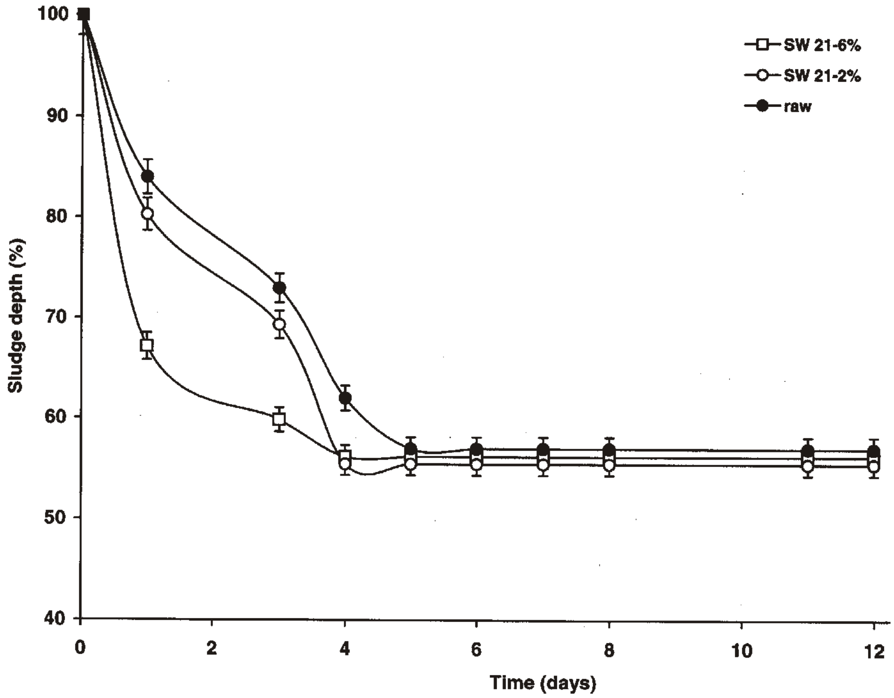

Fig. 3. Precipitation speed of SW dust incorporated into 7.4% TS swine manure at a wet mass of 2 and 6%.

swine manure without limestone. Nevertheless, the PULPRO limestone reduced the sludge volume by an additional 15%, as compared to no limestone.

Using 0 and 2% limestone rather than 6% limestone (SW 21) had an effect on the rate of precipitation rate but produced the same sludge depth after 5 days (Fig. 3). Furthermore, the precipitated sludge was quite similar for both limestone levels of 2 and 6%. The supernatant liquid had a slightly higher TP of 170 and 190 mg/L with 6 and 2% limestone, respectively, as compared to 220 mg/L with 0% limestone. The supernatants from the 6 and 2% limestone treatments were left to settle for an additional 60 days, and their TP levels dropped to 90 and 115 mg/L, respectively. Thus, longer settling periods lead to more TP precipitation.

Figure 4 summarizes the properties of the precipitated sludge. The sludge density ranged from 1.03 for the control (manure without limestone) to 1.10 for the PULPRO 30 sludge. Thus, the sludge density was significantly higher for PULPRO 30 because of its higher rate of precipitation (Fig. 5) and because of its larger particles. All fine dusts produced sludge masses running between 0.50 and 0.66 of the initial manure mass, for PULPRO 10 and SW12, respectively. The TS and TP content of the sludge ranged between 18 to 23% and 1.6 to 2.1 g/L, respectively, when the manure without limestone gave a sludge TS and TP of 11% and 2.6 g/L. Thus, the sludge TP and TS were significantly different only between PULPRO 10, SW12, and the non-treated manure (95% confidence level). The higher PULPRO efficiency is explained by its higher CaO content (Table 1). As compared to the non treated manure, the lower TP level of the limestone sludge results from the dilution effect of the limestone solids.

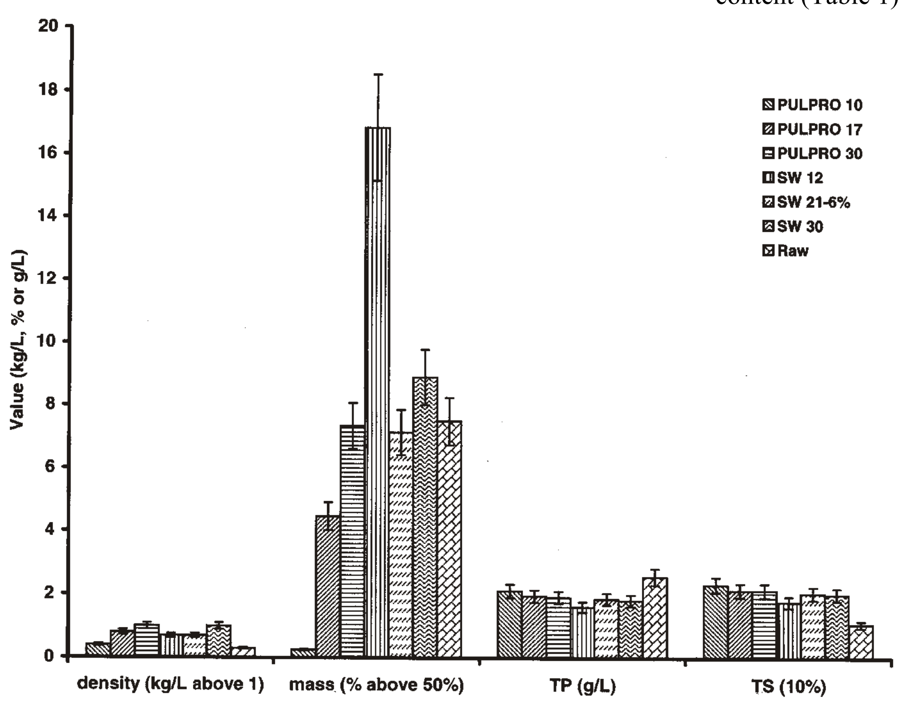

Fig. 4. Sludge properties resulting from the incorporation of limestone dust to 7.4% TS swine manure.

The properties of the supernatant liquids are presented in Fig. 5. The lowest TP and TS fractions, based on the initial raw manure content, were obtained with PULPRO 30. This is consistent with previous tests conducted on screened swine manure (Barrington et al. 2002). Similarly, PULPRO 30 produced the lowest supernatant TS and TP levels. As compared to the control, PULPRO 30 significantly reduced the supernatant TS and TP fraction from 14.5 to 10.5% and from 7.7 to 3.9%, respectively. Even if it only improves TS and TP precipitation by up to 4%, the limestone dust greatly improves the sludge density for easier separation from the supernatant liquid.

To illustrate the mass balance obtained from the results, the mean sludge and supernatant density, pH, and TP levels for PULPRO 30 are illustrated in Table 2, as compared to that of raw manure before treatment. The PULPRO dust precipitated 96 and 90% of the TP

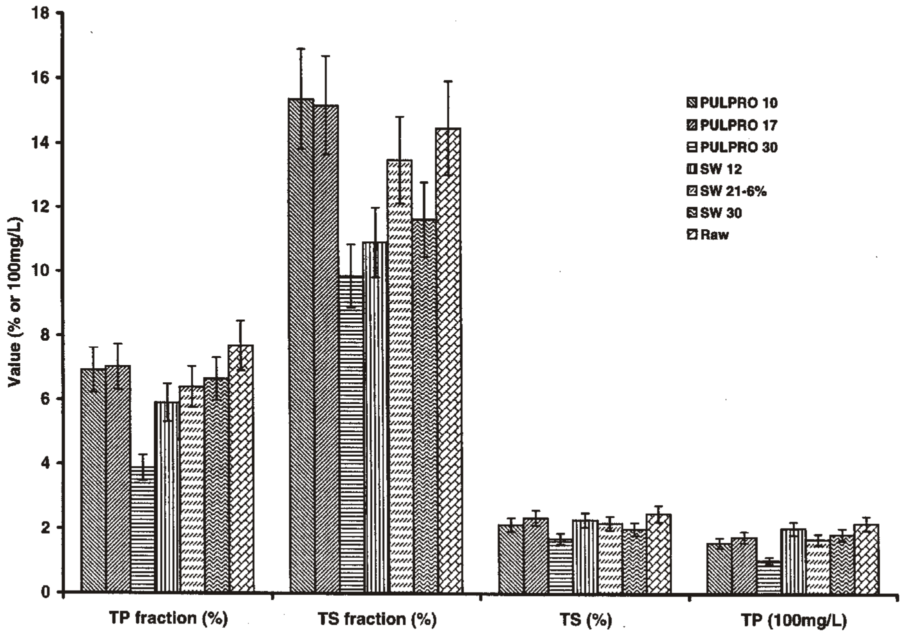

Fig. 5. Supernatant liquid properties resulting from the incorporation of limestone dust to 7.4% TS swine manure.

and TS, with a slight change in pH. Thus, applying limestone dust to unscreened swine manure can lead to just as efficient a TS and TP precipitation process as with screened manure (Barrington et al. 2002) as compared to screened manure, most likely because the large particles of unscreened manure settle by themselves, without assistance of the limestone.

PULPRO 30 test

The exterior test using 6% PULPRO 30 produced a sludge volume of 45% and a supernatant liquid TP of 180 mg/L, after 30 days of settling. The supernatant liquid retained 8% of the initial TP. The sludge had a TS of 22% and a density of 1.07 kg/L. It was easily pumped with a 1.5 kW centrifuge pump back to the piggery pre-pit, using a soft hose, 50 mm in diameter and 20 m in length. This ease of pumping implies that the sludge can be handled as a liquid.

Table 2. Supernatant and sludge properties after PULPRO 30 treatment.

Property	Raw manure	Supernatant	Sludge
TS (%)	7.4	1.71	21.3
TP (g/L)	1.21	0.104	1.91
pH	6.6	7.1	6.8
Density (kg/L)	1.00	1.0	1.1
Mass (%)	100	42.7	57.3
Volume (%)	100	45	55
Percentage of TP	94	3.9	96.1
Percentage TS (manure)	94	10.5	89.5
Mass balance			
TS (g/L)	69.6	7.3	122.1-60*=62.0
TP (g/L)	1.14	0.044	1.094
Equation for TS	TS (g)	TS (g/L) × mass fraction	TS × mass fraction - 60
Equation for TP	TP (g)	TP (g/L) × mass fraction	TP × mass fraction

* Corresponds to limestone added

CONCLUSION

Fine limestone dust can effectively precipitate up to 96 and 90% of the TP and TS of high TS (7.4%) swine manure, respectively, without significantly altering its pH. These nutrients are precipitated in a sludge mass representing 50 to 67% of the original mass. The best results were obtained with PULPRO 30, because of its larger particles producing a denser sludge and its higher Ca content, compared to the SW limestone, therefore precipitating more TS and TP.

High TS swine manure can also settle without limestone to produce a sludge containing 92 and 86% of the TP and TS and representing 58% of the initial mass. Nevertheless, fine limestone can marginally improve swine TP and TS precipitation by 4%, but can greatly improve sludge density allowing it to separate more easily from the supernatant liquid. The sludge produced from the limestone treatment can still be pumped despite its density of 1.07 kg/L and TS content of 22%.

A limestone dosage of at least 2.0% (wet mass) and a minimum settling time of 5 days were required to optimize the precipitation of swine manure TS and TP. Furthermore, the fine limestone dust has the same properties as that applied to lime soils and therefore the soil application of precipitated swine sludge can prevent drops in soil pH resulting from nutrient leaching.

ACKNOWLEDGEMENT

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