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Manure sampling procedures and nutrient estimation by the hydrometer method for gestation pigs

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Abstract

Three manure agitation procedures were examined in this study (vertical mixing, horizontal mixing, and no mixing) to determine the efficacy of producing a representative manure sample. The total solids content for manure from gestation pigs was found to be well correlated with the total nitrogen (TN) and total phosphorus (TP) concentrations in the manure, with highly significant correlation coefficients of 0.988 and 0.994, respectively. Linear correlations were observed between the TN and TP contents and the manure specific gravity (correlation coefficients: 0.991 and 0.987, respectively). Therefore, it may be inferred that the nutrients in pig manure can be estimated with reasonable accuracy by measuring the liquid manure specific gravity. A rapid testing method for manure nutrient contents (TN and TP) using a soil hydrometer was also evaluated. The results showed that the estimating error increased from ±10% to ±30% with the decrease in TN (from 1000 to 100 ppm) and TP (from 700 to 50 ppm) concentrations in the manure. Data also showed that the hydrometer readings had to be taken within 10 s after mixing to avoid reading drift in specific gravity due to the settling of manure solids.

Keywords: Manure agitation; Nutrient estimation; Hydrometer; Linear regressions; Gestation pigs

1. Introduction

Carefully managed application of animal manure slurries on cropland will effectively utilize plant nutrients in slurry without threatening the environment. An integral part of this management is the estimation of the content of nutrients in slurry to determine safe and effective application rates (Chescheir et al., 1985). Past research has shown that there is a statistically significant correlation between the total solids (TS) and manure nutrient elements in pig slurries and there is also a correlation between TS and specific gravity of manure (Tunney, 1979, 1985; Chescheir et al., 1985). Such information has prompted research in developing fast, simple field tests that can be used by farmers to estimate the nutrient contents in the slurry (Dragun, 1978; Tunney, 1979, 1985; Chescheir et al., 1985; Piccinini and Bortone, 1991; Van Kessel et al., 1999). However, the progress in this area has not appeared to be very successful.

The problem that impedes the wide use of the rapid techniques to estimate the fertilizer elements in pig slurries may result from two primary factors, i.e., the quality of the manure sample and the accuracy of the estimation. It is commonly recognized that there is difficulty of obtaining representative manure samples for analysis due to the non-homogeneous properties of manure. Since a representative manure sample can only be secured when the bulk liquid is well mixed, the quality of agitation becomes a decisive factor. Reviewing literature indicates that there is little information available on agitation methods in terms of their capabilities of generating quality manure samples. More research is thus needed to produce such information that is certainly of significance to swine producers in helping them understand the advantages and disadvantages of different agitation techniques in order to get an optimal manure sample for nutrient estimation.

An inexpensive and robust measuring method that can be carried out on the farm is also important to

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guarantee the accuracy of manure nutrient content determination. According to the complexity of different on-farm quick testing methods for manure nutrients developed so far, the hydrometer method that measures the specific gravity of manure and relates the results to the nutrient contents is believed to be the simplest. Since specific gravity is a measure of the density of a liquid, which is determined by the solids content in the liquid, and there are reported linear relationships between the nutrient contents (total N and P) and the total solids (TS) content (Dragun, 1978; Tunney, 1979, 1985; Chescheir et al., 1985), the total N and P content in the liquid can be indirectly measured by a hydrometer. In addition, compared to other rapid methods such as use of conductivity meter, ammonia electrode, reflectometer, and quantofix-N-volumeter, the hydrometer method is the only one that can measure both total N and total P (Van Kessel et al., 1999), making it an appealing technique that deserves further research. Although some work was conducted in the past, more information would be necessary to improve the procedure to make it a better tool to serve swine producers.

The common practice of past researchers is to develop a universal equation for manure from pigs at all growing stages (gestation, nursery, and finishing). This research approach may provide a simple, one-size-fit-all type of solution but at the sacrifice of the accuracy of the estimation equation(s) developed due possibly to factors such as animal age, nutrition, and manure management and storage practices (Westerman et al., 1990; Kirchmann and Witter, 1992; Van Horn et al., 1994; Wilkerson et al., 1997). In order to improve estimation accuracy, reducing the number of influential factors becomes necessary. Therefore, this study will focus on manure from gestation pigs with the objectives being to (1) evaluate three different sampling procedures in terms of obtaining representative manure samples; (2) develop the relationships for manure total N and total P with specific gravity; and (3) determine the limitations and possible errors in using the hydrometer method to estimate the N and P contents for manure from gestation pigs.

2. Methods

2.1. Experimental design for evaluating manure sampling techniques

2.1.1. Manure source and sampling procedures

Fresh manure was collected in a 55-gallon barrel (208 l) from the deep pit of a swine gestation building located in the vicinity of Waseca, MN. To ensure adequate representation, the manure was drawn off by continuously and steadily lowering and raising the sump pump vertically in the manure pit in a random manner.

Table 1
Feed ration for the gestation pigs in this study

Ingredient	Proportion in feed ration, %
Maize	81.45
Soya bean meal	15.0
Tallow	1.0
Dicalcium phosphate	1.0
Vitamin/mineral premix	0.25
$CaCO_3$	0.8
Salt	0.5

The experiment was carried out immediately after the manure was collected. Same manure was used for all three sampling procedures tested to avoid variations in manure characteristics that might affect the test results. The pigs were fed a standard corn-soybean based diet (Table 1).

Three manure sampling procedures were examined in this study. The experimental apparatus for manure agitation and sampling using procedure 1 is shown in Fig. 1. The purpose of this agitation procedure was to create a vertical, outward cycling fashion of mixing in the manure in the barrel using a Drum/Barrel Heavy-Duty mixer with two propellers installed on the shaft (Cole-Parmer Catalog Number: U-04318-15). The mixing speed was controlled at 1000 rpm. After mixing for about 5 min, manure samples of 100 ml each were taken at nine locations (as shown in Fig. 1) with 2.5 cm from the top and bottom ends and 5.0 cm from the barrel wall. The agitation remained in operation when sampling. This sampling plan generated three samples on each level (top, middle, and bottom) to accommodate statistical analysis.

For sampling procedure 2, the same barrel of manure was used after about 4 h of resting; however, the manure was agitated by a sump pump sitting at the bottom of the barrel with the outgoing stream shooting horizontally. This agitation scheme was to create a horizontal mixing pattern. Also, this method can be easily carried out on site. Similarly, nine samples were collected at the same locations as in the previous procedure.

The third procedure was to simulate the sampling situation for manure in a deep pit without agitation. It was found by past research that the solids in swine manure that settle would do so within 1.5–2 h. There-

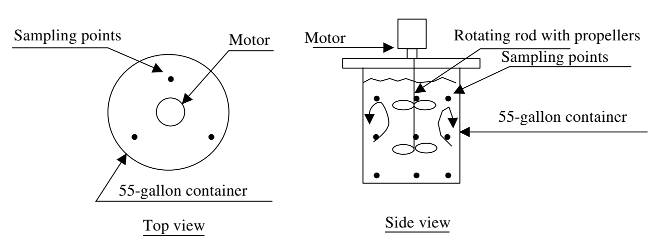

Fig. 1. Schematic of manure sampling device and locations ("●"—sampling point).

fore, after the sampling was completed for the first two procedures, the manure in the barrel was allowed to rest for about 5 h in order to reach relatively thorough sedimentation before executing sampling procedure 3. The sampling locations and the number of manure samples collected were the same as in the previous two methods.

In order to verify the equations derived using data from the diluted manure, additional sampling was conducted, in which one slurry sample was randomly collected from the pits of six gestation buildings at different depths. These raw samples were then analyzed without dilution. The pig buildings for the additional samples were located in the same areas as those in the previous sampling. The pigs from all the sampling sites were fed the same standard ration presented in Table 1.

2.1.2. Sample analysis

All manure samples were analyzed for TS, total volatile solids (TVS), total phosphorus (TP), and total nitrogen (TN). Standard methods recommended by the American Public Health Association for wastewater analysis were used in the sample analysis (APHA, 1998). TN was determined by the Kjeldahl method. TP was measured after digestion with H_2SO_4–H_2O_2 and dilution to 100 ml. Soluble orthophosphate was extracted by filtering 10-fold diluted manure using a Whatman filter paper. Orthophosphate was determined colorimetrically as the phosphomolybdate complex after reduction with ascorbic acid (APHA, 1998). Samples that were not immediately analyzed after collection were stored in a freezer at −20 °C and thawed prior to lab analysis.

2.2. Experimental design for developing a rapid test method for TN and TP using a hydrometer

2.2.1. Manure source

In order to obtain representative manure samples to account for potential variations in livestock management techniques, manure from three gestation farms located in different areas in southern Minnesota was collected for the proposed research. Thick slurries were drawn at a level close to the bottom of the deep pit at each farm and the collected slurries were then diluted in the lab with clean water to generate liquid slurries with 10 different solids content as shown in Table 2. The pigs on the three farms were all fed on a standard corn/soybean ration (Table 1).

2.2.2. Hydrometer method

The prepared slurry samples were poured into a 1 l cylinder one after another and were thoroughly mixed before measuring specific gravity by a soil hydrometer having a scale of 1.000–1.070 (Cole Parmer Catalog Number: U-08292-16). The mixing was carried out manually using a brush to rigorously stir the manure in the cylinder to suspend all the solids. Subsequently, the hydrometer was placed in the cylinder to measure specific gravity immediately after stirring. The cylinder was rinsed clean between the measurements. Tunney (1979) observed that the hydrometer readings decreased with time due to the settling of the solids and recommended that the hydrometer readings be taken within one minute of mixing. In a later study by Chescheir et al. (1985), it was found that significant decrease in specific gravity (as great as 0.006) was observed within one minute, and thus, they suggested that hydrometer readings be taken within 15 s. In this study, the result turned out to be more stringent because it was found that stable hydrometer readings could only be obtained if the hydrometer was read within 10 s after mixing. Therefore, the specific gravity data presented in this paper were based on the hydrometer readings taken at 10 s after mixing in the cylinder.

Table 2
Manure solids (% DM) prepared by dilution for hydrometer experiments

Farm 1	Farm 2	Farm 3
6.35	6.54	5.89
5.40	5.12	4.65
4.52	4.42	3.88
3.81	3.05	3.27
3.08	2.35	2.38
2.36	1.87	1.84
1.70	1.24	1.21
1.09	0.88	0.97
0.53	0.49	0.64
0.26	0.22	0.32

2.2.3. Sample and statistical analysis

All manure samples (30 in total) were analyzed for TS, TP, and TN by using the same methods described in the previous section. Statistical *t*-test was performed on data collected from the above experiments if these data were used for comparison within each pig group and between different groups using the Microsoft Excel *t*-test package. A significance level of $\alpha = 0.05$ was applied to all comparisons.

3. Results and discussion

3.1. Total solids and total volatile solids

The means and standard deviations of TS concentrations for samples collected from the three barrels are presented in Table 3. There is no statistical difference in the TS concentrations between samples collected at different depths using procedures 1 and 2, suggesting that, for either procedure, no matter where the sample was taken (top, middle, or bottom), the sample would

Table 3
TS measurements by the three agitation and sampling procedures (g/l)

Sampling locations	Procedure 1	Procedure 2	Procedure 3
Top	$26.71 \pm 0.65^{a,x}$	$26.64 \pm 1.57^{a,x}$	$12.38 \pm 0.55^{b,x}$
Middle	$27.37 \pm 0.28^{a,x}$	$26.89 \pm 0.61^{a,x}$	$11.97 \pm 0.55^{b,x}$
Bottom	$27.02 \pm 0.30^{a,x}$	$26.85 \pm 0.55^{a,x}$	$40.73 \pm 2.67^{b,y}$

Note: Different letters indicate there is significant difference between numbers in different columns (a, b) and different rows (x, y). Sample number = 3 for all entries.

produce a representative TS concentration for the manure in the barrel. It is also observed that there is no statistically significant difference between procedures 1 and 2 in obtaining a representative sample for TS analysis. Therefore, it may be concluded that agitating manure with procedures 1 and 2 works well in homogenizing gestation pig slurry under the experimental settings in this study. With either of these two agitation methods, it is possible to obtain a representative manure sample from anywhere in the barrel without respect to the sampling location. Similar results were also observed for TVS concentrations (Table 4). Since it has been reported by many past researchers that there is a good correlation between TS and TVS for swine manure (Williams et al., 1984; Zhu et al., 2000; Chastain et al., 2001), it is not surprising to see that the TVS concentrations behave in the same fashion as those of TS. This may conclude that procedures 1 and 2, if used in taking manure samples, will also guarantee a representative sample for TVS analysis.

In contrast to procedures 1 and 2, the problem with procedure 3 in getting a uniform manure sample for both TS and TVS analysis is clearly shown (the third column in Tables 3 and 4). There is significant difference between data collected from the top two sampling locations and the bottom location using this sampling method. The bottom sample has significantly higher average TS concentrations than the top two samples, which conforms to the general natural settling characteristics of manure solids. The data clearly indicate that using this method to randomly take a manure sample in the barrel, it is impossible to secure a representative sample for TS and TVS analysis.

Table 4
TVS data by the three agitation and sampling procedures (g/l)

Sampling locations	Procedure 1	Procedure 2	Procedure 3
Top	$18.95 \pm 0.65^{a,x}$	$18.73 \pm 0.54^{a,x}$	$7.68 \pm 0.38^{b,x}$
Middle	$19.36 \pm 0.17^{a,x}$	$18.05 \pm 1.21^{a,x}$	$7.45 \pm 0.55^{b,x}$
Bottom	$18.97 \pm 0.27^{a,x}$	$18.13 \pm 0.43^{a,x}$	$29.14 \pm 2.16^{b,y}$

Note: Different letters indicate there is significant difference between numbers in different columns (a, b) and different rows (x, y). Sample number = 3 for all entries.

3.2. Total nitrogen and total phosphorus

Comparisons of the means and standard deviations for TN and TP among the three sampling procedures are presented in Figs. 2 and 3. Apparently, there is no statistical difference in TN measurements between samples collected from the three sampling depths using agitation procedures 1 and 2. Similar observation is present for TP data (Fig. 3). Although there are some variations as indicated by the error bars, there is no difference between using procedures 1 and 2 to obtain a representative sample. These data thus indicate that using either procedure 1 or 2 can produce a reliable manure sample for TN and TP analysis, regardless of the sampling location in the barrel.

The performance of agitation procedure 3 is again disappointing as observed in the scenario for the TS and TVS analysis. The bottom samples contain significantly higher concentrations of TN and TP than the top two

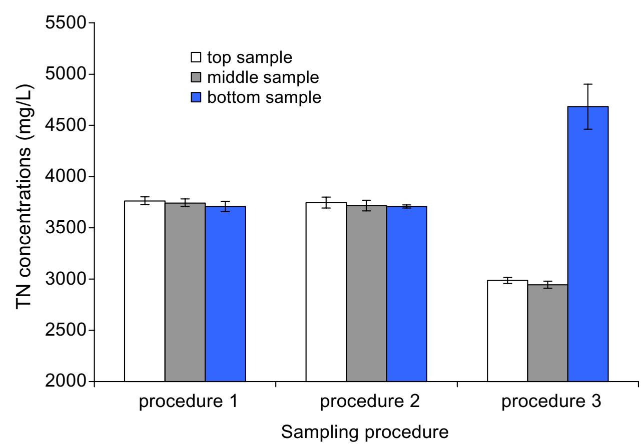

Fig. 2. Comparisons among the three sampling procedures in TN concentrations (sample# = 3 for each calculation).

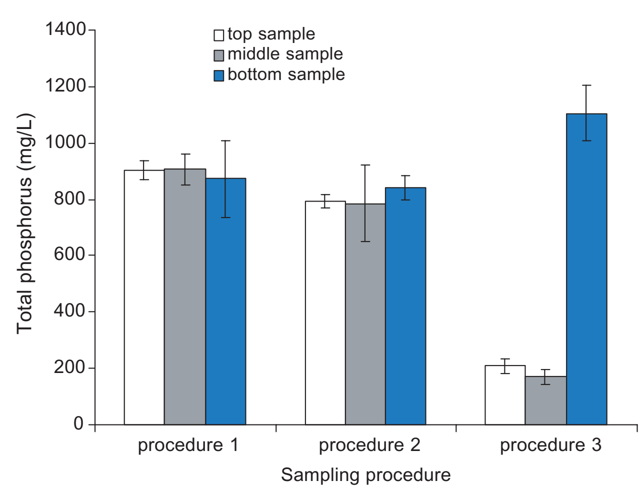

Fig. 3. Comparisons among the three sampling procedures in TP concentrations (sample# = 3 for each calculation).

samples. Clearly, the natural sedimentation process has a tremendous impact on TN and TP concentrations for samples collected at different depths. According to the above discussion, it can be concluded with confidence that procedure 3 is not suitable for manure sampling, and thus, should not be used.

3.3. The relationship of manure TS with TN and TP

The results describing the relationships for TS with TN and TP based on manure samples collected from the three different gestation buildings are presented in Fig. 4. The two linear regression equations (TS vs. TN and TS vs. TP) prove highly significant with correlation coefficients (R) of 0.988 and 0.994, respectively. This information indicates that there is no substantial variation in terms of manure characteristics among the 30

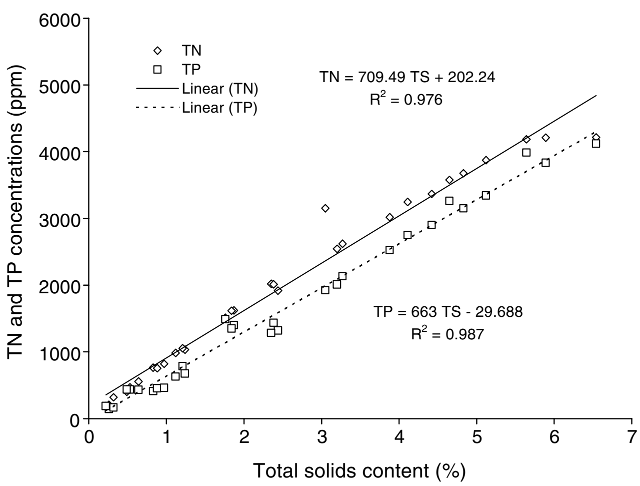

Fig. 4. The relationships between manure TS and TN, TP concentrations.

samples, despite that the manure for this study was collected from three farms located in different areas, a case to be perceived as very likely to bring about variation due possibly to feed ration, management, and manure handling. Therefore, it may be inferred that, for gestation pigs that are fed on similar materials, the manure properties may not vary much with the site location. As a result, the manure nutrient contents (N, P) can be estimated with good accuracy by using the linear equations in Fig. 4 based on the TS measurements.

It is interesting to note that the linear regressions obtained in this study are probably the best among those reported by a number of other researchers. A comparison between regression equations from this study and the others is presented in Table 5. Apparently, none of the past researchers have achieved R^2s as high as the ones reported here. One explanation could be the large variations present in their data possibly caused by the difference in rations and other operating factors between farms. Such comments were also made by Chescheir et al. (1985) in which they stated that variability was likely due to differences in such factors as rations, management, animal breeds, and climate. However, this postulate could not be verified because no ration information was reported in the past publications. The second explanation may be perceived by reviewing the published work of past researchers (Dragun, 1978; Tunney, 1979, 1985; Chescheir et al., 1985; Piccinini and Bortone, 1991; Van Kessel et al., 1999). The equations presented by them were developed based on data from manure samples taken from all kinds of pig units including gestation and farrowing, nursery, and finishing. As indicated early, the intention of lumping all the data together is to develop a universal equation that can be applied to all pig manures without regard to pig's

Table 5
Regression equations for TS and SG versus TN and TP from literature data and this study

Related parameters[a]	Regression equation	R^2	Source
TN vs. TS	TN = 1194 + 424 (TS)	0.84	Tunney (1979)
	TN = 362 + 598 (TS)	0.91	Dragun (1978)
	TN = 2433 + 396 (TS)	0.77	Chescheir et al. (1985)
	TKN = 109.5 + 600 (TS)	0.81	Piccinini and Bortone (1991)
	TN = 202.2 + 709.5 (TS)	0.98	In this study
TP vs. TS	TP = −117 + 232 (TS)	0.85	Tunney (1979)
	TP = 112 + 239 (TS)	0.89	Dragun (1978)
	TP = 32 + 312 (TS)	0.77	Piccinini and Bortone (1991)
	TP = −29.7 + 663 (TS)	0.99	In this study
TN vs. SG	TN = −103,580 + 105,591 (SG)	0.86	Chescheir et al. (1985)
	TN (g/kg) = −67.4 + 69.19 (SG)	0.56	Piccinini and Bortone (1991)
	TN = −102,806 + 102,609 (SG)	0.98	In this study
TP vs. SG	TP = −79,958 + 79,795 (SG)	0.60	Chescheir et al. (1985)
	TP (g/kg) = −46.61 + 46.88 (SG)	0.56	Piccinini and Bortone (1991)
	TP = −95,802 + 95,564 (SG)	0.97	In this study

[a] Units: TS (%), TN and TP (ppm, unless otherwise indicated).

age. Such operation has obviously increased variability in data because of the differential in manure properties between gestation and farrowing, nursery, and finishing pigs. It may therefore be inferred that the concept of using a universal equation for all pig species needs to be revisited. The results from this study actually suggest that different equations should be developed to calculate manure nutrients (N, P) for manure from pigs at different growing stages. In this way, the TS method, as well as the hydrometer method to be discussed in the next section, may be significantly improved, and relatively accurate estimates of manure N and P contents can be made, as demonstrated by the data from this study. This postulate, nevertheless, is waiting for verification by data for manure produced by pigs at other growing stages (nursery and finishing).

3.4. The relationships for manure specific gravity with TN and TP

The results for manure specific gravity versus TN and TP with linear regression lines fitted are shown in Figs. 5 and 6. The two regression equations are listed below for the convenience of discussion.

$$\mathrm{TN} = 102,609\ \mathrm{SG} - 102,806 \quad R^2 = 0.98 \tag{1}$$

$$\mathrm{TP} = 95,564\ \mathrm{SG} - 95,802 \quad R^2 = 0.97 \tag{2}$$

where TN = total nitrogen (ppm); TP = total phosphorus (ppm); SG = specific gravity.

The regressions are highly significant with correlation coefficient $R = 0.991$ and 0.987, respectively. These correlations are found to be higher than those reported by past researchers (Table 5), indicating that true values of manure TN and TP contents can be approached accurately by a linear relationship with specific gravity. The information obtained from this study apparently provides better evidence than that of past studies in supporting this concept. In addition, this conclusion appears to be supported by the additional data, although limited, generated from the undiluted samples from six gestation buildings (the solid symbols in Figs. 5 and 6).

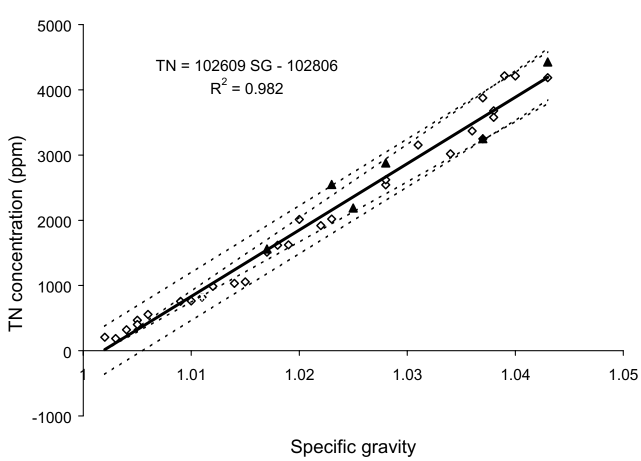

Fig. 5. The relationship between manure specific gravity (SG) and TN concentration (◇—dilution series; ▲—additional sampling data from six different gestation buildings).

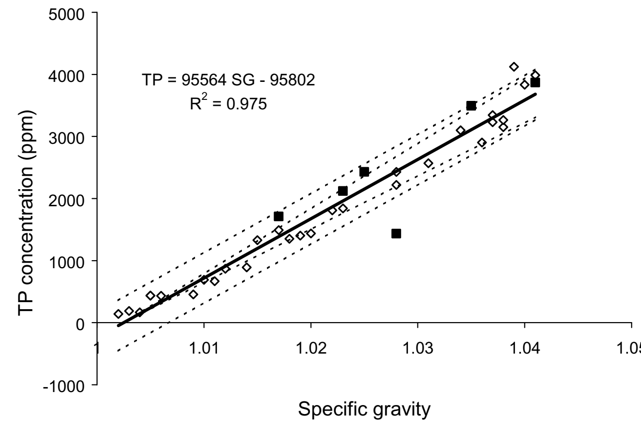

Fig. 6. The relationship between manure specific gravity (SG) and TP concentration (◇—dilution series; ■—additional sampling data from six different gestation buildings).

However, these linear regression equations (as well as all other linear equations) cannot be used without limitations, regardless of the goodness of the correlation coefficients. Piccinini and Bortone (1991) first used error evaluation to examine the goodness of these equations in terms of the precision in calculating the nutrient contents (N and P) in pig slurries. Precision in this case refers to the repeatability of the measurement calculated on the basis of the ratio between the standard deviation of the estimated variable (TN) and its average value. This means that when the equation of the straight-line regression is used to estimate the TN (or TP) content in the pig slurry, it is based on only one determination of specific gravity. Based on this concept, the data calculated using the linear equations derived by Piccinini and Bortone (1991) were liable to a minimum error of ±25% for TN and ±46% for TP. Errors at such levels appear to cause concerns in using those linear equations.

In order to examine regression equations (1) and (2), the error analysis used by Piccinini and Bortone (1991) is also applied to the data obtained from this study. The results show that using the two equations to estimate TN and TP may lead to a minimum error of ±10.2% and ±11.0%, respectively (the two external dotted lines in Figs. 5 and 6 representing the 95% confidence limits). This means that these equations show a precision no better than ±10.2% for TN and ±11.0% for TP. If the error of estimation is desired to be limited within ±10% for all estimates by using the regression equations, an error range needs to be established so that the actual data points obtained can be checked against the average value to see if these data fall within the error range. Such

Table 6
The lowest levels of TN and TP for different error ranges

Error ranges	TN (ppm)	TP (ppm)
±10%	1000	700
±20%	500	300
±30%	100	50

operation produces two internal dotted lines in Figs. 5 and 6 which indicate the error ranges of ±10% when using these equations to estimate manure TN and TP content. As can be seen in Figs. 5 and 6, under low TN and TP conditions, the estimating errors of using these equations will be larger than ±10% because the likelihood for the observed data points to fall outside the two internal dotted lines becomes quite obvious. Specific to the linear regression equations in this study, when the TN and TP concentrations in manure fall below about 1000 ppm (1.0 g/l) and 700 ppm (0.7 g/l), the chance to control the measurement error within ±10% appears to be remote.

The above discussion implies that even if a linear regression equation is characteristic of a good correlation coefficient, significant errors in estimation still can occur. This, however, does not mean that this equation is defective and cannot be used. The critical guideline is to understand that there is an error range associated with the equation. The error ranges for the lowest concentrations of TN and TP of gestation pig slurry in this study when Eqs. (1) and (2) are used are given in Table 6. Obviously, the accuracy of using Eqs. (1) and (2) to estimate the TN and TP contents in gestation pig manure is contingent upon the existing concentrations of these two nutrient elements. The error in estimation will increase with the decrease in nutrient concentrations. Practically in most cases, gestation pig manure contains TN and TP at higher concentrations than 1000 and 700 ppm, respectively (corresponding to a TS content of 1.2%, see Fig. 4). Unless the manure is extremely diluted, the precision of using Eqs. (1) and (2) to calculate the TN and TP contents in the manure can thus be accurate within ±10%. For diluted manure with TN and TP contents less than 100 and 50 ppm, respectively, the estimation error by using the above equations can be larger than 30%. Also, the equations discussed in this paper only apply to gestation manure.

4. Conclusion

Data from this study have shown that representative manure samples cannot be obtained without agitation. The two agitation procedures examined in this project are capable of producing a representative manure sample for TS, TN, and TP analysis. And there is no statistical difference between these two procedures.

The linear regressions are highly significant between manure specific gravity and TN, TP contents with correlation coefficients of 0.991 and 0.987, respectively. It may therefore be concluded that relatively accurate information about manure TN and TP contents can be obtained using these linear regression equations together with the manure specific gravity measured by a soil hydrometer. Also, the study shows that the hydrometer readings have to be taken within 10 s after mixing to avoid reading drift caused by the settling of solids.

The precision of using linear regression equations to estimate manure nutrients may be compromised by low concentrations of TN and TP in the liquid manure. According to the data from this study, the error in estimation will increase, from ±10% to ±30%, with the decrease in TN (from 1000 to 100 ppm) and TP (from 700 to 50 ppm) concentrations. Therefore, for diluted manure ($TS < 1.2\%$), using regression equations generated from this study may not be appropriate to estimate the TN and TP contents in the manure.

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