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Near-infrared Sensing of Manure Ingredients

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Abstract

The effectiveness of near-infrared (NIR) technology for quickly analyzing the nutrient content of three types of animal manure was evaluated. Swine lagoon effluent, liquid swine pit manure, and solid beef feedlot manure were tested. An NIRSystems 6500 scanning monochromator unit was calibrated against wet chemistry data. Total solids (TS), total Kjeldahl nitrogen (TKN), ammonia nitrogen (NH3-N), total phosphorus (P), and potassium (K) were measured. Correlation coefficients (r) ranged from 0.688 to 0.976, Ratios of data range:standard error of prediction (SEP) varied from 7.0 to 13.6 for the various chemical constituents and manure sources. Based on the individual ratios we conclude that NIR techniques will allow us to predict TS, TKN, NH3-N, and K in all three manure types. Further work will be required before P is predictable.

Keywords

Near-infrared, Animal waste, Water quality, Crop nutrient, Spectroscopy

Disciplines

Agriculture | Bioresource and Agricultural Engineering

Comments

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NEAR-INFRARED SENSING OF MANURE NUTRIENTS

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ABSTRACT. *The effectiveness of near-infrared (NIR) technology for quickly analyzing the nutrient content of three types of animal manure was evaluated. Swine lagoon effluent, liquid swine pit manure, and solid beef feedlot manure were tested. An NIRSystems 6500 scanning monochromator unit was calibrated against wet chemistry data. Total solids (TS), total Kjeldahl nitrogen (TKN), ammonia nitrogen (NH_3-N), total phosphorus (P), and potassium (K) were measured. Correlation coefficients (r) ranged from 0.688 to 0.976, Ratios of data range:standard error of prediction (SEP) varied from 7.0 to 13.6 for the various chemical constituents and manure sources. Based on the individual ratios we conclude that NIR techniques will allow us to predict TS, TKN, NH_3-N, and K in all three manure types. Further work will be required before P is predictable.*

Keywords. *Near-infrared, Animal waste, Water quality, Crop nutrient, Spectroscopy.*

The livestock industry is very important to the United States. Water pollution from land-application of manure, however, is a major environmental concern. Research shows that manure applied at proper rates poses little risk to either groundwater or surface water. Overapplication of either commercial fertilizer or manure nutrients, however, does pose a threat to water quality (Koelliker et al., 1971; Lorimor and Melvin, 1996; Prantner et al., 1999; Warnemuende et al., 1999). The 1998 EPA/USDA AFO/CAFO Strategy exemplifies the increasing emphasis on nutrient management planning. Many states have adopted regulations requiring permits and/or agency approvals of manure management systems and nutrient management plans for livestock operations (Council for Agricultural Science and Technology, 1996).

To properly manage manure nutrients, producers must know the nutrient concentration of the manure. Manure nutrient concentrations vary drastically from high-volume, low-concentration lagoon effluent, to low-volume-high, concentration liquid pit manure, to even more concentrated solid manure. Nutrient concentrations vary within individual storage structures. Because of the variability, sampling is best done during agitation at haulout time. Sampling is typically done then, and samples are sent to commercial chemistry labs that require several days to complete the analyses. The time delay makes good manure nutrient management very difficult since the land application may be completed before the sample results are known. If producers had a method to quickly determine manure nutrient concentrations, they could determine the correct rate of manure to apply to optimize crop production and minimize environmental risk. They could do a much better job of manure nutrient management.

NUTRIENT ANALYSIS METHODS

EXISTING TESTING DEVICES

A number of testing devices have been used to estimate manure solids and nutrient characteristics. Van Kessel et al. (1999) evaluated seven separate quick-test devices and found that different devices worked well for different types of manure. Several of them showed good agreement with lab results for NH_3-N. However, they concluded that while the devices provide useful estimates, they should not replace lab tests. Chescheir et al. (1985), Fleming et al. (1993), and Cross et al. (1996) evaluated several different devices including hydrometers, the Agros nitrogen meter, and electrical conductivity pens. They found that each device had its own advantages and disadvantages. Each tended to work for a specific constituent, or on a certain type of manure, but did not work as well on other types.

A significant disadvantage of the existing nitrogen-measuring devices is that they sense only ammonia, which is not a consistent percentage of total nitrogen. NH_3-N can account for as little as 8% of total nitrogen in solid manure. It accounts for approximately 67% in pit manure, and up to 80% or more of total nitrogen in lagoon effluent (Lorimor, 1999). Safley (1990) reported a mean of 63% and standard deviation of 19% for NH_3-N in swine pit manure,

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expressed as a percent of TKN. For swine lagoons the mean and standard deviation were 82%, and 10%, respectively, and for manure scraped from paved lots 50%, and 23%, respectively. In 174 samples from Iowa swine pits, average NH_3-N as a percent of TKN was 69% with a standard deviation of 11.5%.

NEAR-INFRARED TECHNOLOGY

Near-infrared (NIR) technology was first used to determine grain moisture 30 years ago. Since then it has been developed and refined to determine nutritional properties of ground grain samples, and most recently, to analyze samples of whole grains. Grain samples are routinely analyzed for moisture, crude protein, starch, fiber, and oil content (Hardy et al., 1996). Rippke et al. (1995) reported regression coefficients (R^2) of 0.996 and 0.980 for moisture and protein, respectively, in corn ($n = 120$ samples). Moisture ranged from under 12% to over 22%; protein ranged from under 7% to over 9%. For soybeans, R^2 values were 0.990 and 0.984 for moisture and protein, which ranged from 9% to 13% and 33% to 37%, respectively. The method has been accepted as a way to replace wet chemistry to provide accurate and stable compositional analysis of grains.

Nakatani et al. (1996) reported that NIR spectroscopy could be used to accurately measure total carbon, total nitrogen, ash, cellulose, hemicellulose, and lignin in cattle manure compost. NIR analysis of the C:N ratio yielded an R^2 value of 0.991. NIR analysis of cation exchange capacity (CEC) yielded an R^2 of 0.997. Nakatani and Harada (1995) were able to achieve correlation coefficients of 0.980, 0.791, and 0.890 for total nitrogen, ammonium nitrogen, and nitrate nitrogen, respectively, in solid manure compost. Their results showed that near-infrared spectroscopy analysis has potential for the quantitative analysis of solid animal waste.

There are several statistical procedures used to derive NIR calibrations including multiple linear regression, principal component analysis (PCA), partial least squares (PLS) regression, and neural networks. There is little consensus on the most appropriate method, so each constituent or product is evaluated separately, as was done with the manure in this study.

OBJECTIVES

The objective of this research project was to determine whether near-infrared spectroscopy (NIRS) calibrations could be developed to quickly and accurately analyze TS, TKN, NH_3-N, P, and K in three types of animal manure: liquid swine lagoon effluent, liquid swine pit manure, and solid beef feedlot manure.

MATERIALS AND METHODS

SAMPLES AND CONSTITUENTS ANALYZED

Iowa State University, Oklahoma State University, and University of Missouri at Columbia are the three universities that participated in this study. Iowa State University collected 174 liquid swine pit manure samples from throughout Iowa. To provide varying solids concentrations, samples from each storage pit were collected from the top, middle, and bottom, plus a composite sample of the entire vertical profile. The University of Missouri collected 100 swine lagoon effluent samples from commercial facilities in Missouri, and Oklahoma State University collected 100 solid manure samples from commercial open beef feedlots. The samples from Iowa and Missouri were immediately frozen in 125-mL Nalgene® bottles and those from Oklahoma were frozen in 250-mL HDPE containers. Samples were then transported to Iowa State University for NIR analysis.

Liquid pit manure and lagoon effluent samples were allowed to thaw overnight to room temperature in groups of 20 to 25. Once thawed they were shaken by hand, and approximately 70 to 80 mL of each sample were poured into an 8-cm × 15-cm, 6-mil Ziploc® bag. The temperature of each sample was determined just prior to scanning using a C-1600P TherMonitor manufactured by Linear Laboratories. Temperatures averaged 25.5°C with a standard deviation of 0.55°C. The temperature range was from 23 to 27°C. The samples (contained in the Ziploc bags) were individually scanned in the natural product cell of the NIRSystems 6500 unit.

Each solid cattle feedlot manure sample was mixed in the sample container with a laboratory spatula then transferred to a Ziploc bag with the spatula. Large clumps in the cattle manure samples were broken up by hand before analysis. Excess air was squeezed out of each bag by hand before it was sealed.

WET CHEMISTRY ANALYSIS

Wet chemistry analysis of the constituents (TS, TKN, NH_3-N, total P, and K) was conducted at the University of Missouri at Columbia. Moisture (total solids) was also run at Iowa State immediately after the NIR scans to insure that no changes would occur between the dates of the scans and wet chemistry analysis. All wet chemical analysis procedures were done in accordance with guidelines set forth by the American Public Health Association (APHA, 1992).

TEST PLASTIC SAMPLE BAG FOR INTERFERENCE

To determine whether the Ziploc bags used to hold the manure samples for analysis would interfere with the scans, five wheat samples were scanned both with and without the plastic bags, over a wavelength range of 400 ηm to 2498 ηm. Average wavelength patterns of the five bagged samples were compared to average wavelength patterns of the unbagged samples. The samples exhibited very similar reflectance patterns throughout the spectrum with the exception of interference from 1700 ηm to 1875 ηm and from 2250 ηm to 2498 ηm (fig. 1). The plastic bag interference was evident at the same wavelengths for each type of manure.

CALIBRATION PROCEDURE AND STATISTICS

Once the samples were scanned and the wet chemistry analysis was complete, the NIR analysis was essentially a process of selecting the proper wavelengths, and developing regression equations from the pooled data, then testing the individual samples against the regression equations as shown in figure 2.

Calibrations were derived for each constituent by using a Partial Least Squares (PLS) Regression using The Unscrambler® data analysis software (The Unscrambler 6.11a, Camo A/S, Oslo, Norway, 1997). PLS regression is a bilinear modeling method that relates the variation in

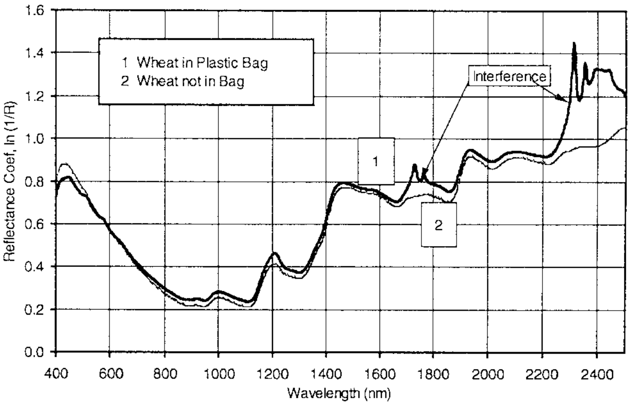

Figure 1–Interference from plastic bag used to hold manure samples.

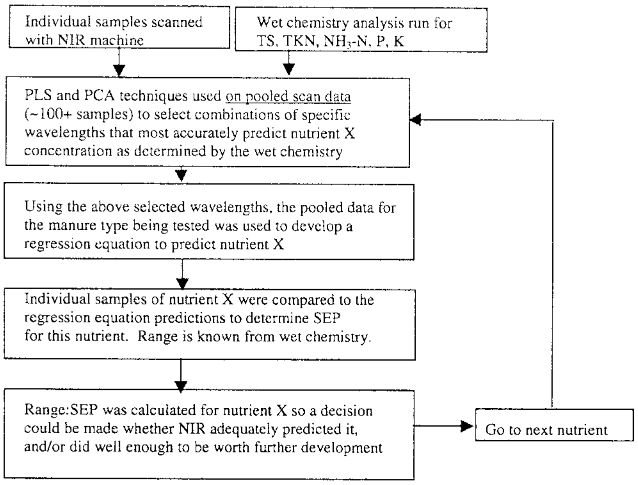

Figure 2–Process flow diagram.

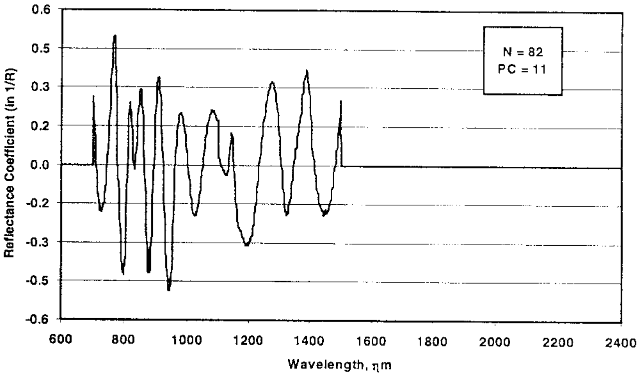

Figure 3–Example of spectrum truncated at 1500 ηm for lagoon TS analysis.

each response variable (Y-variable) to the variations of several predictors (X-variables). Principal Component Analysis (PCA) was used to eliminate outliers. PCA projects the information carried by the original variables onto a smaller number of underlying ("latent") or orthogonal variables called principal components. After scanning all samples of one manure type (lagoon, pit or solid), calibrations were developed between the reflectance patterns across the wavelength spectrum and each individual chemical constituent. The best correlations were sometimes found by using only a portion of the spectrum. In those cases the unused portion of the spectrum was truncated. An example is shown in figure 3.

Wavelengths where the Ziploc bags had strong responses (1700-1875 ηm, and 2250-2498 ηm) were omitted unless a higher correlation (r) was attained by not removing these wavelengths.

A separate regression equation was developed from the pooled NIR scan data (independent variable) to predict each nutrient (dependent variables) in each type of manure. Analysis of the data was based primarily on standard error of prediction (SEP), which are frequently used in NIR analyses. The SEP is a measure of scatter, or dispersion, of the actual nutrient concentrations about the regression line. The smaller the SEP, the closer the estimate is likely to be to the actual value of the dependent variable. The SEP was then used to calculate the ratio of range:SEP for each nutrient and each type of manure. The range was the difference between the high and low nutrient concentrations. Since a small SEP indicates the likelihood of good estimates, a large range:SEP indicates accurate predictions can be expected over a large data range. A ratio of range:SEP between 4 and 8 indicates a possibility of distinguishing between high and low values. With a range:SEP between 8 and 12, there is a possibility of predicting quantitative data. A ratio of range:SEP greater than 12 indicates good predictability (Williams and Hurburgh, 1999).

RESULTS AND DISCUSSION

REPEATABILITY OF SCANS

Scans of several Iowa manure pit samples with similar solids contents illustrates that peaks occur at the same wavelengths, and are of approximately the same relative magnitude. Figure 4 is an example of scans of three individual pit manure samples with similar TS content, ranging from 9.4 to 10.0%. Figure 5 is an example of scans of four individual pit manure samples with more widely varying solids concentrations (2.6-12.7% TS). It illustrates that peaks still occur at similar wavelengths, but for samples with lower solids concentrations the relative magnitudes of the peaks are less pronounced. We would thus expect that a scan of a lagoon effluent sample would

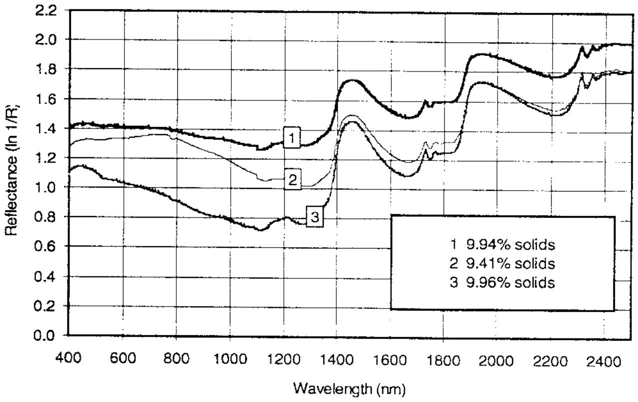

Figure 4–Scans of three liquid swine pit samples with similar solids content.

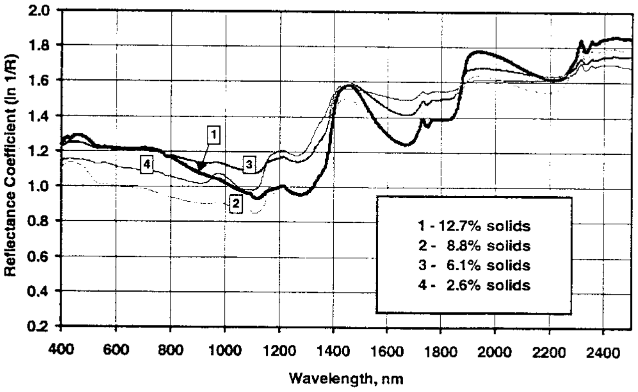

Figure 5–Scans of four liquid swine pit samples with widely varying solids content.

result in a flatter curve than one of pit manure or solid manure with higher solids concentrations.

Swine lagoon effluent responded only in the lower end of the near-infrared spectrum for constituent analysis, while beef feedlot manure used the entire spectrum. Swine pit manure samples used the lower end of the spectrum for some constituents and the entire spectrum for others.

Comparisons of plots for total solids between the three different types of manure reveals that peaks occur in the same range of wavelengths for each type of manure, but intensity of the peaks varies between manure types. Peaks representing water content occurred around 1100 ηm and 2000 ηm. Figure 6 shows a representative scan for each of the three different types of manure utilized in this study (swine pit manure, swine lagoon effluent, and dry beef feedlot manure).

The PLS and PCA analysis showed that TKN correlated best with peaks around 900 ηm and 1300 ηm, and NH_3-N correlated best with peaks near 700 ηm, 800 ηm, and 1300 ηm.

Phosphorus, or organic structures associated with it, correlated best with peaks at different wavelengths for the different manure types. In swine pit manure, peaks occurred around 1100 ηm and 1500 ηm. In swine lagoon effluent peaks occurred around 750 ηm and 800 ηm, and cattle manure peaks occurred at 800 ηm, 1400 ηm, 1900 ηm, and 1975 ηm. The intensities of these peaks differed between manure types.

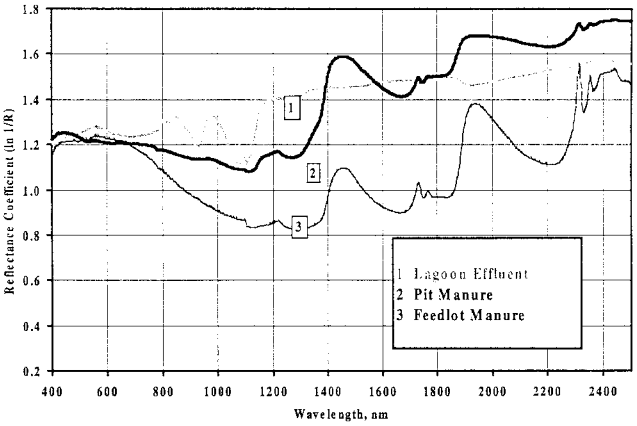

Figure 6–Scans of three different types of manure, lagoon effluent, liquid pit, and solid manure.

Correlation coefficients (r) for the various constituents tested ranged from 0.688 to 0.976 for swine pit manure (table 1), from 0.783 to 0.947 for swine lagoon effluent (table 2), and from 0.764 to 0.955 for solid beef manure (table 3).

Table 1. Liquid swine pit manure—NIRSystems 6500 calibration results for solids, total Kjeldahl nitrogen, ammonia nitrogen, phosphorus, and potassium

Statistic	Solids* (N = 161)†	TKN‡ (N = 152)†	NH_3-N‡ (N = 158)†	P‡ (N = 165)†	K‡ (N = 165)†
No. of PLS factors	6	8	8	11	6
Correl. coeff. (r)	0.920	0.897	0.794	0.688	0.887
SEP§	1.58	2.77	2.76	0.51	2.03
Range:SEP	11.1	9.8	8.0	8.4	8.9
Chemistry	%TS	%TS	%TS	%TS	%TS
Average	6.63	11.1	8.15	3.1	6.22
High	44.98	33.35	26.83	6.32	23.78
Low	0.37	0.78	0.31	0.61	0.57
Outliers removed (%)	7.5	12.6	9.2	5.2	5.2

* % wet basis.
† Values for sample number (N) are based on analysis with outliers removed from sample set.
‡ %of dry solids.
§ Standard Error of Prediction, from one-out cross validation.

Table 2. Liquid swine lagoon effluent—NIRSystems 6500 calibration results for solids, total Kjeldahl nitrogen, ammonia nitrogen, phosphorus, and potassium

Statistic	Solids* (N = 82)†	TKN‡ (N = 95)†	NH_3-N‡ (N = 98)†	P‡ (N = 76)†	K‡ (N = 82)†
No. of PLS factors	11	7	6	11	10
Correl coeff. (r)	0.947	0.829	0.787	0.783	0.84
SEP§	0.08	3.36	3.81	0.44	2.23
Range:SEP	11.1	8.2	7.0	6.8	9.0
Chemistry	%TS	%TS	%TS	%TS	%TS
Average	0.53	17.62	15.07	2.27	20.12
High	2.25	37.52	35.68	5.42	30.45
Low	0.10	5.65	2.78	0.21	3.64
Outliers removed (%)	18.0	5.0	2.0	24.0	18.0

* % wet basis.
† Values for sample number (N) are based on analysis with outliers removed from sample set.
‡ % of dry solids.
§ Standard Error of Prediction, from one-out cross validation.

Table 3. Solid beef feedlot manure—NIRSystems 6500 calibration results for solids, total Kjeldahl nitrogen, ammonia nitrogen, phosphorus, and potassium

Statistic	Solids* (N = 97)†	TKN‡ (N = 96)†	NH_3-N‡ (N = 96)†	P‡ (N = 91)†	K‡ (N = 93)†
No. of PLS factors	9	12	13	16	14
Correl. coeff. (r)	0.955	0.817	0.976	0.764	0.905
SEP§	3.61	0.21	0.02	0.13	0.17
Range:SEP	13.6	12.4	15.9	7.6	12.0
Chemistry	%TS	%TS	%TS	%TS	%TS
Average	66.46	1.42	0.08	0.72	1.42
High	88.43	3.07	0.47	1.53	2.85
Low	39.51	0.52	0.00	0.32	0.63
Outliers removed (%)	6.7	7.7	7.7	12.5	10.6

* % wet basis.
† Values for sample number (N) are based on analysis with outliers removed from sample set.
‡ % of dry solids.
§ Standard Error of Prediction, from one-out cross validation.

The calibration data are reported as a percentage of each constituent on a dry matter basis. In general, NIR worked best for the solid manure based on the higher range:SEP ratios. Tables 1, 2, and 3 summarize the results for the three manure types.

Outliers were identified by viewing plots of "scores" for each constituent and by viewing a leverage plot for each constituent in The Unscrambler software (The Unscrambler 6.11a, 1997). The "scores" plot shows similarities and differences among the samples allowing investigation of patterns and outliers. Leverages are used for the detection of samples that are far from the center within the principal component space described by the model. A sample with a large leverage may be different from the rest, indicating the possibility of being an outlier. Large leverage indicates a high influence on the model (The Unscrambler 6.11a, 1997). The percent of outliers removed during analysis of each constituent is displayed in tables 1, 2, and 3.

INDIVIDUAL CONSTITUENT ANALYSIS

Total Solids. TS results are shown in column 2 in tables 1 through 3. Analysis of total solids for all three manure types resulted in r values of 0.920, 0.947, and 0.955, and ratios of range:SEP of 11.1, 11.1, and 13.6 for swine pit manure, swine lagoon effluent, and beef feedlot manure, respectively. These ratios ($8 <$ range:SEP ≤ 12) indicate that swine pit manure and lagoon effluent will be predictable with further work and that beef feedlot manure has good predictability (range:SEP >12).

Total Kjeldahl Nitrogen. TKN results are shown in column 3 in tables 1 through 3. Range:SEP values for these manures were 9.8 for pit manure, 8.2 for lagoon effluent and 12.4 for dry cattle manure. Pit manure and lagoon effluent will require further work ($8 <$ range:SEP ≤ 12), but the beef feedlot manure shows good predictability (range:SEP >12). Correlation's were 0.897 for swine pit manure, 0.829 for swine lagoon effluent, and 0.817 for beef feedlot manure.

Ammonia Nitrogen. NH_3-N results are shown in column 4 in tables 1 through 3. Range:SEP for NH_3-N were 8.0 for the swine pit manure, 7.0 for lagoon effluent. NH_3-N in pit manure or lagoon effluent is not predictable at this time (range:SEP ≤ 8). Range:SEP of 15.9 for dry beef feedlot manure indicates good predictability of NH_3-N in beef feedlot manure (range:SEP > 12). For NH_3-N, r values were 0.794, 0.787, and 0.976 for swine pit manure, swine lagoon effluent, and beef feedlot manure, respectively.

Phosphorus. P results are shown in column 5 in tables 1 through 3. Low ratios of range:SEP (6.8 and 7.6) and low correlation values (0.688 to 0.783) indicate that NIR did not perform well in predicting P levels in the lagoon effluent and beef feedlot manure samples (range:SEP ≤ 8). The range:SEP of 8.4 for swine pit is only slightly better. Additional work will be required before manure P can be predicted accurately by NIR.

Potassium. K results are shown in column 6 in tables 1 through 3. Range:SEP ratios were 8.9, 9.0, and 11.8, which indicates that K may be predictable with further work ($8 <$ range:SEP ≤ 12) in all three manures. Correlation coefficients were 0.887, 0.840, and 0.905 for pit manure, lagoon effluent, and solid beef manure, respectively. Potassium peaks occur in the same area where plastic bag interference occurred, which contributes to the problem of predicting potassium accurately.

Outliers. As tables 1, 2, and 3 show, a significant of number of samples (up to 24% for swine lagoon P) were identified as outliers from some constituent analyses. This represents a problem that must be addressed in further work, because a producer would not know if a sample he submitted for analysis were an outlier or not in a field situation.

CONCLUSIONS

This study showed that NIR is a potentially usable method to determine manure nutrient and solids concentrations in liquid and solid forms of manure. Although further work will be necessary to refine procedures, results are encouraging based on the ratios of SEP to data ranges for manure from swine pits, lagoons, and beef feedlots. The technique did the poorest job of prediction on P in swine lagoon effluent. It worked the best on solid beef feedlot manure. Results of this initial research were positive enough to justify further investigation on the applicability of the NIR procedure to all forms of manure, regardless of moisture contents.

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