Effect of Diet Composition on the Determination of Ash and Moisture Content in Solid Cattle Manure Using Visible and Near-Infrared Spectroscopy

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Visible and near-infrared (Vis-NIR, 350–2500 nm) diffuse reflection spectroscopy (DRS) models built from "as-collected" samples of solid cattle manure accurately predict concentrations of moisture and crude ash. Because different organic molecules emit different spectral signatures, variations in livestock diet composition may affect the predictive accuracy of these models. This study investigates how differences in livestock diet composition affect Vis-NIR DRS prediction of moisture and crude ash. Spectral signatures of solid manure samples ($n = 216$) from eighteen groups of cattle on six different diets were used to calibrate and validate partial least squares (PLS) regression models. Seven groups of PLS models were created and validated. In the first group, two-thirds of all samples were randomly selected as the calibration set and the remaining one-third were used for the validation set. In the remaining six groups, samples were grouped by livestock diet (ration). Each ration in turn was held out of calibrations and then used as a validation set. When predicting crude ash, the fully random calibration model produced a root mean square deviation (RMSD) of 2.5% on a dry basis (db), ratio of standard error of prediction to the root mean squared deviation (RPD) of 3.1, bias of 0.14% (db), and correlation coefficient r^2 of 0.90., When predicting moisture, an RMSD of 1.5% on a wet basis (wb), RPD of 4.3, bias of −0.09% (wb), and r^2 of 0.95 was achieved. Model accuracy and precision were not impaired by exclusion of any single ration from model calibration.

Index Headings: **Diffuse reflection spectroscopy; DRS; Visible and near-infrared spectroscopy; Vis-NIR spectroscopy; Crude ash; Moisture; Manure; Animal feed; Multivariate; Partial least squares; PLS; Pseudo-independence; Particle size.**

INTRODUCTION

Moisture and ash are the primary determinants of manure quality in terms of its use as a fertilizer or fuel. Both are inert constituents that increase the weight of manure, add little or no nutrient value, decrease the higher heating value (HHV), increase gasifier fouling potential, and increase waste-disposal requirements.[1] A commercial bio-energy plant recently built in the Texas Panhandle was designed to operate using manure-fired gasifiers and serves as a contemporary illustration of the rationale for our work. The company's engineering design appears to have been predicated on maximum acceptable moisture content and minimum fuel value benchmarks[1] in order to meet thermal efficiency standards with an engineering factor of safety of 10%.

In previous work[1,2] we confirmed that visible and near-infrared (Vis-NIR, 350–2500 nm) diffuse reflection spectroscopy (DRS) models built from "as-collected" samples of solid cattle manure accurately predict constituents of solid cattle manure such as moisture and crude ash and implicitly predict total solids and organic matter in the lab and in situ.[3,4] However, when analyzing a manure sample from a stock not represented in the calibration set, Vis-NIR model predictions may not be reliable. Moreover, prediction errors stemming from "pseudo-independent validation" can result from randomly selecting from non-independent samples, which can result in an overestimation of predictive accuracy relative to models built from independent samples.[5] For example, a model calibrated with manure samples from several different feedyards may perform very well, but introducing samples from a feedyard not included in the calibration may cause the model to fail. Livestock diets generally differ among feedyards and thus may be an important consideration when building Vis-NIR DRS prediction models.

Ruminant diets contain various feedstocks in different proportions. Each feedstock is characterized by a unique spectral signal. Vis-NIR DRS of manure has been successfully used to detect differences in the dietary intake of domestic ruminant animals such as free-ranging goats,[6] forage-fed sheep,[7] free-ranging cattle,[8] and confined, forage-fed cattle.[9] There has been little research, if any, to determine how differences in rations change the spectral behavior of manure from confined cattle on finishing rations in feedyards.

New research is revealing that distillers grains in bovine diets may change manure properties in several ways. For example, increasing the fraction of wet distillers grains with solubles (WDGS) decreases the digestibility of the rations and has been observed to increase manure production. This is due to the exchange of corn-derived starch for less-digestible fiber such as arabinose, xylose, and cellulose in the distillation process. A 20% increase of WDGS in feed rations increased dry matter mass in manure by 20% when compared to manure production by animals on steam-flaked corn (SFC) or dry-rolled corn (DRC) diets.[10] In the same experiment, the percent of total nitrogen (N) and phosphorus (P) remained unchanged, but the percent of potassium (K) increased by 5%.

Until recently, finishing diets in Texas High Plains feedyards have been based on whole corn, dry-rolled corn, or steam-flaked corn at inclusion rates approaching 80% of diet dry matter (DM). Recent trends in diet composition, especially the addition of byproduct feeds (e.g., wet corn gluten feed, gluten feed pellets, wet or dry distillers grains), may affect the spectral signature of the resulting manure. In addition, market forces that affect the relative pricing of whole grains and byproducts

Received 26 April 2011; accepted 24 June 2011.
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DOI: 10.1366/11-06333

0003-7028/11/6509-1056$2.00/0

Soil			Fly Ash												Soil		
D45[§]	D60	D30	DRC	D30	D15	D60	D45	SFC	D45	SFC	D30	DRC	D60	D15	SFC	D15	DRC

←West ↑ Prevailing Winds ↑ East→

SCHEME 1. Schematic of the pen layout, surface type, and rations fed to groups of beef cattle. Ration labels are defined in Table I.

may superimpose seasonality on those longer-term trends in diet composition.

RESEARCH OBJECTIVE

This study investigates how differences in livestock diet composition affect Vis-NIR DRS prediction of moisture and crude ash in solid cattle manure.

EXPERIMENTAL METHODS

Sample Collection and Preparation. Manure samples were collected by hand from the pen surfaces of an experimental beef feedyard located in Potter County, Texas. The feedyard contained eighteen pens, the surfaces of which were soil ($n = 6$) and compacted, crushed bottom ash ($n = 12$) from a nearby coal-fired power plant. The soil was a Pullman clay loam, which is a fine, mixed, superactive, thermic, Torrertic Pleustoll and is by far the most common soil type on which feedyards in the Southern High Plains and eastern New Mexico have been built.[11] Finishing cattle received six different diets for 97 to 124 days. Each ration was fed to two ash-surfaced pens and one soil-surfaced pen in a randomized complete block design. Based on Sweeten et al.,[12] we expected pen surfacing to influence the ash and moisture content and therefore noted pen surface types. Scheme 1 presents a schematic diagram of the pens showing surface type and cattle diets.

Samples were collected from an animal performance experiment investigating the inclusion of wet distillers grains with solubles in diets based on steam-flaked corn.[13] The diets differed primarily in the proportion of "concentrates" (ingredients with high metabolic energy density), including steam-flaked corn (SFC), dry-rolled corn (DRC), and wet distillers grains with solubles (WDGS). The SFC and DRC were acquired from different sources. The six diets (Table I) were formulated to provide a minimum of 13.5% crude protein, a minimum of 6.5% dietary fat, and a minimum Ca : P ratio of 1.5 : 1.

Each of the six treatment groups was represented by 36 samples of manure; 12 samples were collected from each of three pens of cattle being fed the same diet. In total, 216 samples were collected in identical plastic bags. One subsample was taken from each manure sample for conventional moisture and crude ash analysis. All samples were stored in a freezer at −12 °C for preservation, and all subsamples were processed immediately.

Moisture was measured according to the procedure recommended by University of Wisconsin Extension.[14] The subsamples were then prepared according to ASTM Standard E1757-01[15] for crude ash analysis by dry oxidation according to ASTM Standard E1755-01[16] in an ashing furnace (Heavy Duty Hi-Temp Muffle Furnace, Model F-A1730, Thermo Scientific, Asheville, NC) with procedural enhancements as described by Preece et al.[1]

The samples were brought uniformly to room temperature for scanning with Vis-NIR DRS. A FieldSpec® 3 Hi-Res spectroradiometer (ASD, Inc., Boulder, CO) fitted with a handheld probe was used to measure the sample reflectance from 350 to 2500 nm with spectral resolutions of 3 nm at 700 nm and 10 nm at 1400 and 2100 nm. Samples were scanned through their plastic bags. The spectrometer was calibrated with a Spectralon® white reference panel placed inside an identical plastic bag to set reflectance to 100% and the calibration was verified after every twenty samples.

Storage and handling caused the manure to settle within the bags, resulting in relatively finer particles on the bottom and coarser particles on the top. Therefore, both the top (Fine) and bottom (Coarse) of the bags were scanned at three separate places. The three scans were averaged, providing two independent spectral datasets ($n = 216$), one for the Fine scan and one for the Coarse scan. A third dataset ($n = 216$) was derived from the mean of all six scans (Mean), and a fourth data set ($n = 432$) contained both the coarse and fine scans (Both).

The raw spectral data were processed in four steps using custom statistical computing code written in R[17] following the procedures of Brown[18] as described in Preece et al.[1] and Sakirkin et al.[2] First, because the spectrometer was equipped with three sensors that detect reflectance over three distinct wavelength ranges, discontinuities created by splicing the reflectance data at the end ranges of the sensors were corrected using the methods of Brown et al.[19] Second, the three raw reflectance scans for each sample were averaged. Third, the

TABLE I. Ration formulations from diets fed to cattle.[a]

Ration ingredient (% of ration as fed)	SFC	D15	D30	D45	D60	DRC
Wet distillers grains with solubles	0.00	15.00	30.00	45.00	60.00	0.00
Steam-flaked corn	76.37	66.68	53.44	39.50	24.45	0.00
Dry-rolled corn	0.00	0.00	0.00	0.00	0.00	76.77
Alfalfa	10.00	10.00	10.00	10.00	10.00	10.00
Cottonseed meal	3.50	0.00	0.00	0.00	0.00	3.50
Yellow grease	3.27	2.16	1.06	0.00	0.00	3.25
Limestone	1.45	1.45	1.45	1.45	1.45	1.45
Molasses	3.00	3.00	3.00	3.00	3.00	3.00
Urea	1.36	0.67	0.00	0.00	0.00	0.98
Custom supplement	1.05	1.05	1.05	1.05	1.05	1.05

[a] SFC = steam-flaked corn; D15 = 15% wet distillers grains with solubles (WDGS); D30 = 30% WDGS; D45 = 45% WDGS; D60 = 60% WDGS; and DRC = dry rolled corn.

TABLE II. General guidelines for reporting the performance of calibrations, based on ratio of standard error of prediction to the root mean square deviation (RPD), for environmental or heterogeneous samples such as manure.[21–24]

RPD < 1.5	Model is not useful
1.5 ≤ RPD < 2.0	Model can possibly distinguish between high and low values
2.0 ≤ RPD < 2.5	Model can be applied to approximate or classify
2.5 ≤ RPD < 3.0	Model is good and can be used for quantitative prediction
3.0 ≤ RPD < 4.0	Model is excellent and can be used for reliable quantitative prediction
4.0 ≤ RPD	Model is reproducible and can be used reliably in commercial applications

average values were smoothed with a weighted cubic spline using 5 nm knots and a smoothing parameter of 0.05. The values for every 10 nm from 350 to 2500 nm for the smoothed raw data and for the first and second derivatives were recorded. Finally, the first and second derivatives of reflectance were calculated in The Unscrambler® 9.7[20] from the 10 nm averages of reflectance.

Crude Ash Model Creation and Validation. Partial least squares (PLS) regression models were built on mean-centered data using a segmented, cross-validation PLS method in The Unscrambler software and validated with a test-set holdout. The segments for cross-validation of the calibration were chosen randomly and represented 4% of the calibration dataset. The Unscrambler used a standard nonlinear iterative partial least squares (NIPALS) algorithm and chose the number of factors to include in each PLS model by minimizing the residual variance of the calibration cross-validation.

The first derivative of the raw reflectance with respect to wavelength ($\partial R/\partial\lambda$) was used in the construction of all models because we have found it to be consistently the most predictive in characterizing manure[1,2] as compared to the raw spectra and the second derivative of the raw spectra. This behavior was confirmed in several models for different constituents in the current study, but we report here only the results from models built on the first derivative of the raw reflectance.

Samples from both types of pen surfaces were included in the calibration models to avoid errors arising from dependency related to pen surface type. The number of PLS factors was restricted to a maximum of four to ensure comparability between models and prevent over-fitting. A limit of four PLS factors was prescribed because over-fitting manifested after four factors in some models in preliminary analysis and also in previous work.[2] The root mean square deviation (RMSD), ratio of standard error of prediction to the root mean square deviation, (RPD), coefficient of determination (r^2), bias, and number of PLS factors were considered in the evaluation and comparison of model performance. Table II shows the general guidelines suggested in the literature for reporting the reliability and stability of calibrations for environmental or heterogeneous samples (such as manure).

To determine the effect of particle size on Vis-NIR DRS model performance, four models were created using results from all samples with one-third of the samples withheld from calibration for use as a validation set. One model was created for each of the Fine, Coarse, Mean, and Both spectral data sets previously described.

To investigate the spectral difference between treatments, seven different groups of Vis-NIR DRS models were created. In the first group, two-thirds of all samples were selected as a calibration set and the remaining one-third was used as a validation set. In the remaining six groups, samples were grouped by treatment (ration). Each treatment in turn was held out of calibrations and then used as a validation set.

RESULTS AND DISCUSSION

Ash and moisture results are presented in Table III. The moisture content ranged from 4.3 to 28.1 with a mean of 11.5% on a wet basis (wb). The ash content ranged from 18.1 to 44.5 with a mean of 25.7% on a dry basis (db). These results are similar to those of samples characterized in previous studies[1,2] and are typical of feedyards in the region. Additional samples collected from the same pens at a different time were also analyzed by a commercial laboratory. The ash content as reported by the commercial laboratory ranged from 17.1 to 44.0, with a mean of 25.9% (db), which agrees closely with our results.

Pen surfacing influenced the ash and moisture content of the manure as expected. Manure collected from the soil-surfaced pens contained 11.4 to 20.4% more ash (wb) than manure collected from pens with a fly-ash surface. This difference agrees with previous studies, which also reported reductions in ash content with fly-ash surfaces.[12,25] Samples from both types of pen surfaces were included in model calibration.

The Vis-NIR DRS models built from the Coarse spectral data set strongly outperformed the models built from Fine, Mean, and Both spectral data sets. This result is consistent with earlier findings[2] that milling manure samples to reduce particle sizes impaired prediction of ash. Using scans of unprocessed manure taken from the top of the sample bags yielded successful models with improved performance relative to models developed in previous studies.[1,2] The Coarse model based on all treatments with a random one-third of the samples

TABLE III. Mean, minimum, and maximum moisture percent (wb) and crude ash percent (db) for each treatment group of manure samples.

Ration treatment[a]	Mean moisture (% wb)	Minimum moisture (% wb)	Maximum moisture (% wb)	Mean ash (% db)	Minimum ash (% db)	Maximum ash (% db)
SFC	7.7	4.3	14.2	26.7	21.2	40.4
D15	9.4	5.3	15.2	24.6	20.2	34.2
D30	10.2	6.5	21.1	24.5	18.1	36.2
D45	13.7	5.1	28.1	27.9	19.7	44.5
D60	16.2	8.0	26.8	26.7	20.3	41.5
DRC	11.7	5.9	25.1	24.1	18.3	34.2

[a] SFC = steam-flaked corn; D15 = 15% wet distillers grains with solubles (WDGS); D30 = 30% WDGS; D45 = 45% WDGS; D60 = 60% WDGS,; and DRC = dry rolled corn.

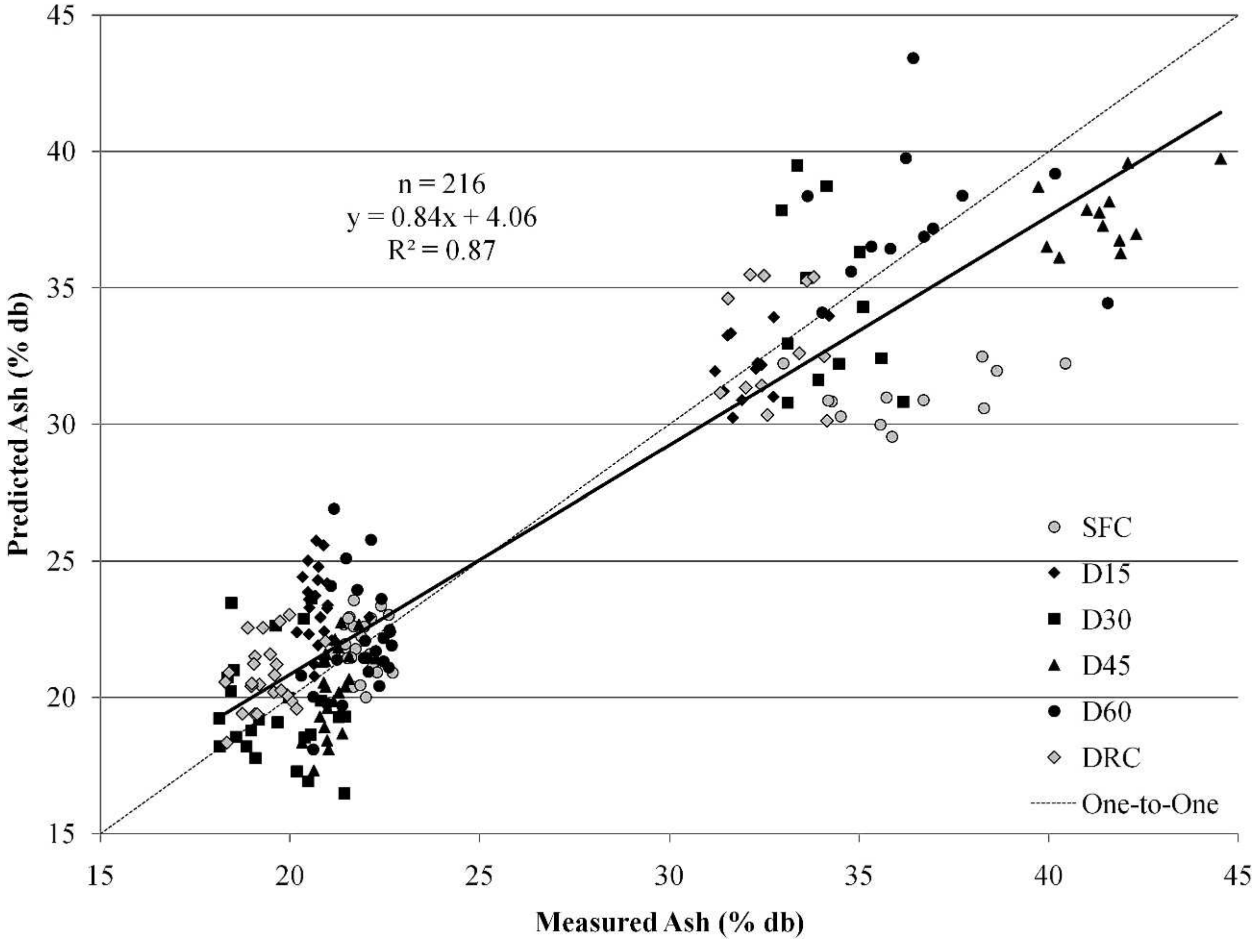

FIG. 1. Predicted ash (% db) versus measured ash (% db) from six models calibrated by holding out each treatment group (ration) in turn. A linear global trend line is shown in bold, along with a dashed one-to-one line for reference.

withheld from calibration for validation produced an RMSD of 2.5% (db), RPD of 3.1, bias of 0.14% (db), and r^2 of 0.90 when predicting crude ash. The number of factors that minimized the residual variance of the calibration cross-validation was three, with the first factor explaining nearly 60% of the variance in the data. When predicting moisture the model produced an RMSD of 1.5% (wb), RPD of 4.3, bias of −0.09% (wb), and r^2 of 0.95. The number of factors minimizing the residual variance of the calibration cross-validation was two, with the first factor explaining nearly 40% of the variance in the data. The RPD values indicate that both models are stable and reliable. Previously developed models based on as-collected (field moist, unprocessed) manure had an RMSD of 3.2 and an RPD of 2.1 based on random validation.[2]

The number of significant wavelengths (p-value < 0.05) for the crude ash calibration model was 168, while it was 194 for moisture over a total of 214 possible wavelengths. (Spectral endpoints 350 and 2500 nm were not included in models based on $\partial R/\partial \lambda$.) This number is much higher than the 23 significant wavelengths identified by Preece et al.[1] in determining crude ash in oven-dried and milled solid cattle manure adulterated with soil and is higher than the 58 significant wavelengths found by Sakirkin et al.[2] when predicting crude ash in air-dried manure samples. The increase in the number of significant wavelengths may perhaps be explained by the differences in sample moisture content between the studies. In this study the samples were not dried and contained up to 30% more moisture than those from the other two studies. Covalent and hydrogen bonds associated with water molecules are spectrally active and with increased moisture content the intensity of these bands will be greater across the Vis-NIR DRS spectrum.[26] The fact that the number of significant wavelengths increases with moisture content of the manure samples across the three studies supports this explanation. In further support is the report by Sakirkin et al.[2] of improved accuracy in models that included water bands (1390–1410 and 1890–1910 nm) compared to those in which they were excluded. However, prediction accuracy was unaffected when moisture was included along with spectra as a prediction variable.

In general, Vis-NIR DRS models calibrated by holding out samples by ration were as reliable as the model calibrated by using samples from all treatment groups while holding out random samples. Plots of the predicted versus measured ash from the six models calibrated by holding out each ration in turn are presented in Fig. 1. This figure shows an evident grouping of samples into higher and lower ash content due to the two pen surface types. Table IV contains summary statistics of ash content model validations. Ash content of SFC and D45 were under-predicted, as quantified by larger biases (Table IV). These rations may have produced manure with different ash and moisture characteristics than the other rations (Table III), but a plot of observed moisture and ash values versus their

TABLE IV. Validation statistics of Vis-NIR DRS models predicting crude ash for each ration treatment with that treatment left out of the calibration model, and the model calibrated by holding out random samples regardless of treatment. Root mean square error (RMSD) and ratio of the standard deviation over the RMSD (RPD), coefficient of determination (r^2), bias, and number of partial least squares (PLS) regression factors are shown.

Treatment[a]	SFC	D15	D30	D45	D60	DRC	ALL
Validation	Ration	Ration	Ration	Ration	Ration	Ration	Random
RMSD (% crude ash db)	3.3	3.8	2.8	2.7	2.6	2.0	2.5
RPD	2.1	1.4	2.6	3.7	2.8	3.3	3.1
Bias (% crude ash db)	−1.7	1.7	0.0	−1.9	0.5	1.0	0.1
PLS factors (count)	4	4	3	3	3	4	3

[a] SFC = steam-flaked corn; D15 = 15% wet distillers grains with solubles (WDGS); D30 = 30% WDGS; D45 = 45% WDGS; D60 = 60% WDGS; DRC = dry rolled corn; and ALL = all treatments.

respective prediction residuals revealed no trend (data not shown). Figure 2 presents a plot of the predicted versus measured moisture from the six models calibrated by holding out each ration in turn. Summary statistics of moisture content model validations are presented in Table V.

Differences in model performances between rations were very small; the RMSD was less than 1.5% db for ash and less than 0.7% for moisture content. The largest RMSD (which corresponds approximately to a 95% confidence interval) was less than 4.0% when predicting ash and less than 2.6% when predicting moisture. These accuracies for both water content and ash are considerably lower than the 10% engineering factor of safety in gasifier design.

CONCLUSION

We predicted crude ash and moisture with an accuracy of ±4% (db) and ±3% (wb) by weight, respectively, in as-collected (unprocessed) samples of solid cattle manure by scanning the samples through plastic bags with a hand-held Vis-NIR DRS probe. Withholding samples grouped by OC source from model calibration did not impair prediction; the differences in livestock diet composition had little effect on the accuracy or reliability of Vis-NIR DRS models. Therefore, based on this experiment we can reliably predict both ash and moisture content (by mass) in solid manure, in as-collected

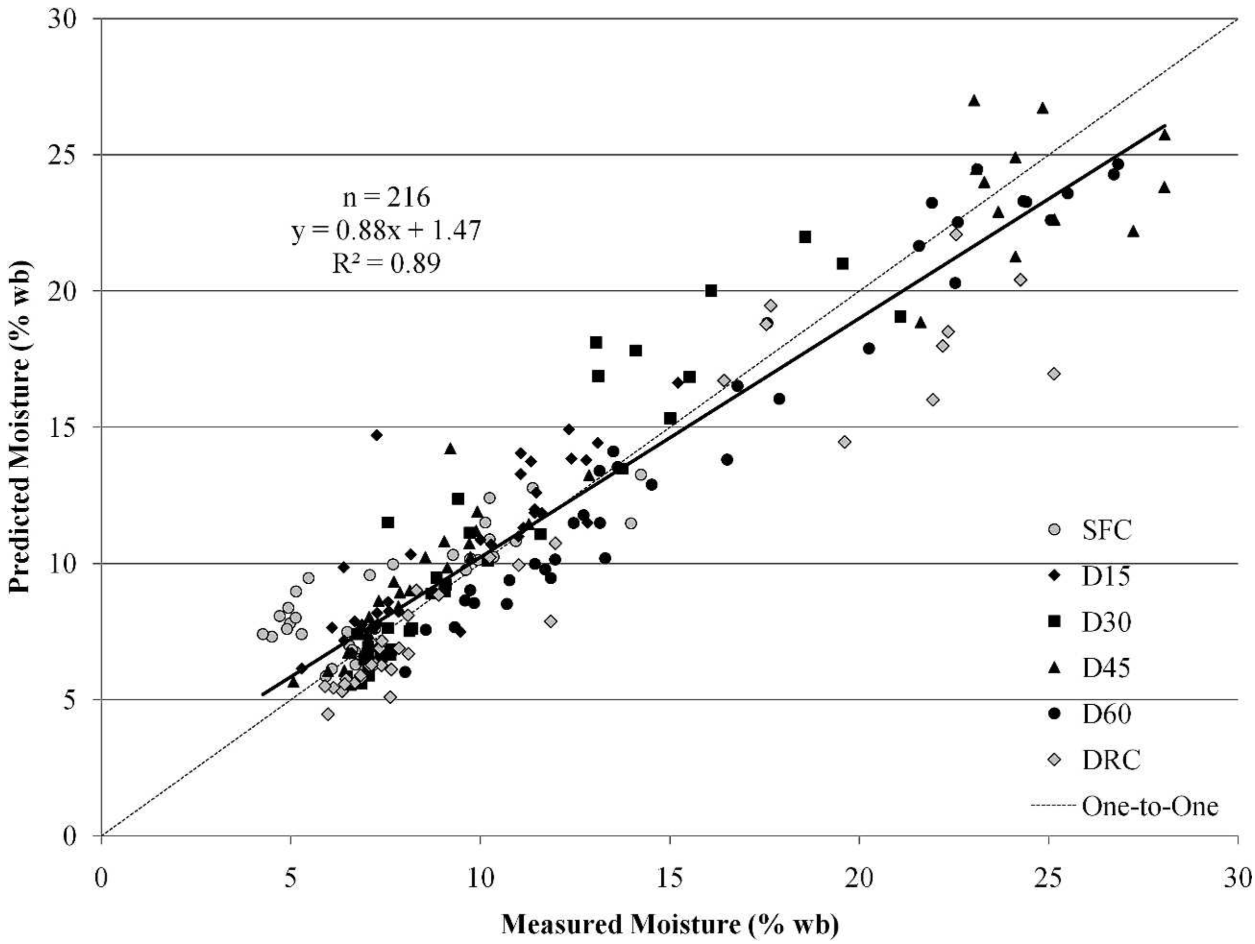

FIG. 2. Predicted moisture (% wb) versus measured moisture (% wb) from six models calibrated by holding out each treatment group (ration) in turn. A linear global trend line is shown in bold, along with a dashed one-to-one line for reference.

TABLE V. Validation statistics of Vis-NIR DRS models predicting moisture for each ration treatment with that treatment left out of the calibration model, and the model calibrated by holding out random samples regardless of treatment. Root mean square error (RMSD) and ratio of the standard deviation over the RMSD (RPD), coefficient of determination (r^2), bias, and number of partial least squares (PLS) regression factors are shown.

Treatment[a]	SFC	D15	D30	D45	D60	DRC	ALL
Validation	Ration	Ration	Ration	Ration	Ration	Ration	Random
RMSD (% moisture wb)	1.9	1.9	1.9	2.0	1.72	2.5	1.5
RPD	1.4	1.4	2.2	4.1	3.6	2.5	4.3
Bias (% moisture wb)	1.1	1.1	1.0	0.3	−1.2	−1.5	−0.1
PLS factors (count)	2	2	2	2	2	2	2

[a] SFC = steam-flaked corn; D15 = 15% wet distillers grains with solubles; D30 = 30% WDGS; D45 = 45% WDGS; D60 = 60% WDGS; DRC = dry rolled corn; and ALL = all treatments.

form, from Pullman clay loam or fly-ash surfaced pens, at feedyards feeding DRC, SFC, or WGDS-based rations.

The fact that models built from scans of coarse manure from the upper surface of manure in bags were most reliable and most successful has important consequences for the adaptation of Vis-NIR DRS in commercial applications. We have shown that Vis-NIR DRS may be convenient in situations where immediate in situ determination of ash and/or moisture in as-is manure is required, such as in industries using large volumes of manure as a fuel. In these industries manure may be transported in trucks or held in stockpiles and be subject to settling similar to that in the manure samples in this study. The ability to scan the surface of a load or pile and obtain a reliable result without having to process the manure in any way is paramount to the usefulness of the method.

In practical terms, the vast majority of feedyards in the Texas, Oklahoma, and New Mexico region use SFC, DRC, and/or WDGS in their rations and are located on Pullman clay soil. The model reported herein is likely to be useful throughout this region, which represents well over 30% of the cattle on feed in the United States.[27,28] Because the model is unaffected by ration differences among the dominant feed stocks, recalibration seems unnecessary when a feedyard changes the composition of its diets. However, to expand the geographic applicability of the model beyond the southern High Plains and eastern New Mexico, it would be necessary to add samples of any atypical or regionally exotic feedstocks and/or soils to our existing calibration set.

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