Determination of the quality of fresh silages by near infrared reflectance spectroscopy

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Introduction

Grass silages may constitute up to 80% of the dry matter in the winter and summer diets of dairy cows. Although near infrared (NIR) spectroscopic analysis of dried and ground forages is very fast, considerable time and effort may be required to prepare the samples. While the use of dry matter is necessary for practical reasons, it would be useful to analyse fresh silage as it is without any preparation and in the same form that it will be fed to livestock. In addition, wet sample analysis would be beneficial because the drying of the silages before NIR spectroscopic analysis can result in a loss of volatiles.

This study was designed to evaluate if is better to determine quality and nutritive values with NIR spectroscopic analysis of undried or dried grass silages.

Material and methods

Sample collection and preparation for NIR measurement

In the winter of 1994–1995, 65 grass silage samples were collected from different farms. The samples were frozen on the day of collection and stored until analysed.

First, the pH was determined. A representative sub-sample was pressed to obtain the juice. The pH was measured with an automatic titrator. Each sample was then cut into sections of 2–5 cm. After cutting, 250 g of forage was used to fill four high fat/moisture cells, thus ensuring that the analysis is fully representative of the original material provided. Samples were measured in quadruplicate, using a NIRSystems 6500 in reflectance mode with a Sample Transport Module operating in the range 1100–2500 nm. Immediately after being scanned, the contents of the four cells were dried[1] in a convection oven at 60ºC for 24 hours. All samples were ground with a Pulverisette-15 (Fricht) mill to pass through a 0.75 mm screen and then scanned in the same range using a NIRSystems 5000 spectrophotometer.

Chemical analysis

Dry matter (DM) and ash were determined according to Van der Meer[2] in a thermogravimetric determinator MAC 500 (Leco Instruments). Crude protein (CP) was calculated by multiplying the Kjeldahl N by 6.25. Neutral detergent fibre (NDF) was determined according to Van Soest *et al.*[3] The method of Riveros and Argamentería[4] was used for enzymatic digestibility organic matter (EDOM). All samples were analysed twice in the laboratory.

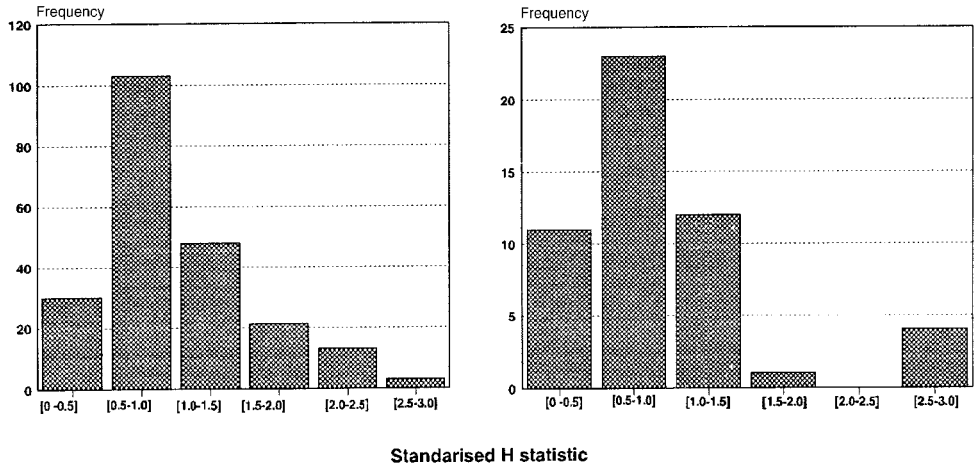

Figure 1. Histogram of distances from the mean of wet and dry silages.

Calibration and validation

For routine NIR spectroscopic analyses, the sample set of 65 samples was divided randomly into two sets; four-fifths of the silage samples in a given lot were used for a calibration set and one-fifth for a validation set.

To determine if oven drying errors would influence the selection of calibration equations for NIR spectroscopic analysis, the NIR spectroscopic analyses were compared using calibrations developed on undried silages and dried silages.

Table 1. Chemical composition of fresh silage.

Constituent	Mean	Range	*SD*
pH (of juice)	4.546	3.87–5.45	0.343
DM	28.837	13.47–62.02	10.368
Ash	2.764	0.89–6.76	1.063
CP	3.216	1.70–6.80	1.083
NDF	17.234	8.61–38.27	6.612
EDOM	55.928	38.67–80.43	8.405

DM: % Dry matter.
Ash: % Ash.
CP: % Crude protein.
NDF: % Neutral detergent fibre. } % fresh
EDOM: % Enzymatic digestibility organic matter.

Table 2. Chemical composition of dry silages.

Constituent (% as is)	Mean	Range	*SD*
pH	4.546	3.87–5.45	0.343
DMr	94.976	85.34–97.21	1.991
Ash (% as is)	9.393	5.19–18.65	2.428
CP (% as is)	10.88	7.44–17.05	2.039
NDF (% as is)	56.782	42.44–66.79	5.659
EDOM	51.567	38.67–80.43	8.207

DMr: % Dry matter, residual
CP: % Crude protein.
NDF: % Neutral detergent fibre.
EDOM: % Enzymatic digestibility organic matter.

Table 3. NIR reflectance spectroscopy results in analysing undried silages for dry matter, ash, crude protein, neutral detergent fibre, enzymatic digestibility organic matter and pH.

	Calibration (4/5)				Validation (1/5)
Constituent (% fresh)	PLS terms	*SEC*	R^2	*SECV*	r^2
DM	8	1.815	0.969	2.087	0.94
Ash (% fresh)	9	0.363	0.883	0.403	0.66
CP (% fresh)	11	0.259	0.943	0.337	0.87
NDF (% fresh)	8	1.174	0.968	1.323	0.93
EDOM	10	3.024	0.868	3.285	0.85
pH	11	0.123	0.870	0.147	0.59

SEC: Standard error of calibration.
R^2: Validation r^2.
SECV: Standard error cross-validation.
r^2: Validation r^2.

Modified PLS calibrations were performed for all constituents with math treatments (1, 5, 5), (1, 10, 10), (1, 20, 20), (2, 5, 5) and (2, 10, 10). The maximum number of PLS terms was 12. The math treatment and number of terms giving the lowest standard error of cross-validation *(SECV)* for each constituent were used to develop the final equations (see Figure 1).[5]

Table 4. Near infrared reflectance spectroscopy results in analysing dried silages for ash, crude protein, neutral detergent fibre, enzymatic digestibility organic matter.

	Calibration (4/5)				Validation (1/5)
Constituent (% as is)	PLS terms	*SEC*	R^2	*SECV*	r^2
Ash	5	0.994	0.832	1.407	0.92
CP	12	0.163	0.994	0.499	0.90
NDF	6	1.342	0.944	1.837	0.92
EDOM	9	1.428	0.970	2.941	0.75

SEC: Standard error of calibration.
R^2: Validation r^2.
SECV: Standard error cross validation.
r^2: Validation r^2.

Results and discussion

Chemical composition and nutritive value

The mean *(±SD)* values and range for the different chemical analyses are shown in Table 1 for fresh silages and Table 2 for dry silages.

The results for the different constituents had low minimum and high maximum values. This indicates that NIR spectroscopic calibrations were performed on samples having fermentation and nutritive characteristics that were representative of those which may be encountered in practice.

Quality estimation of undried and dried silages by NIR spectroscopy

Table 3 shows the results for the analysis of DM, ash, CP, NDF, EDOM and pH by NIR spectroscopy on wet silages.

R^2 values for all components were much higher than 0.85 and the standard errors of calibration *(SEC)* were all within acceptable limits. However, small R^2 values for ash and pH were obtained. In the case of pH, this would be because this parameter is a vegetable matter property linked to several chemical components.[6] For ash it could be explained because it is very difficult to relate the mineral linkages of organic matter with wavelengths.

The NIR spectroscopic calibration relationships with respect to dried silage nutritive values are show in Table 4. The second derivative was the best math treatment for all constituents, both undried and dried silages.

When the dried silages used in this study were analysed by NIR spectroscopy, the calibration set R^2 for CP and EDOM were higher than with the undried silages (Table 3). Also, to compare *SEC* values for both states, small *SEC* values were found (in %) for dried silages in all parameters. In both cases, standard error values were higher for NIR prediction than for NIR calibration. This is in agreement with results of Shenk *et al*.[7] and Marten *et al*.[8]

In Tables 3 and 4, only validation r^2 values are given, because for the validation of these equations, unfortunately we had too small a number of samples for a reliable prediction. In future work we will need to increase the number of samples.

While results for grass silages in their natural state are less accurate than with dried grass silages, they show that the former can be analysed rapidly, even with moisture levels of over 85%. In addition, wet sample analysis would be beneficial because drying silages before NIR spectroscopic analysis may cause loss of volatiles.

Conclusions

The results of this study indicates that: (i) although the accuracy of NIR spectroscopy with wet silages is not as good as it is for dry silages, we can obtain acceptable results for the former; (ii) analysis of grass silages in this fresh state is more convenient and would allow rapid nutritive determination on site and (iii) the processes of drying and grinding are time-consuming and expensive. In addition, the composition changes during the drying process.

References

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