Analytical use of NIR diode array spectrometers on forage harvesters

Christian Paul and Christian Pfitzner

Institute of Crop and Grassland Science, Federal Agricultural Research Centre (FAL), Bundesallee 50, 38116 Braunschweig, Germany, christian.paul@fal.de

Introduction

Compared to the time when "wet chemical" methods were the usual means of analysing forage composition, the availability of adequately calibrated NIR spectrometers has contributed to lowering the costs and expanding the volume of forage analyses in routine agricultural laboratories. The costs of sampling, drying and grinding forages prior to analysis now often exceed those of the actual NIR measurements. Here, the advent of robust NIR diode array (DA) spectrometers has provided an opportunity to reduce costs even further by analysing forages at their point of production/distribution, i.e. directly in forage trials, farmers' fields or at farmyard storage points. Following the first reported use of a NIR diode array spectrometer on an agricultural harvester[1] an analogous commercial experimental plot harvester for grass and clover was presented by us[2]. The present paper contains information on its further development and calibration.

Materials and methods

NIRS Harvest Line

The forage plot harvester by J. HALDRUP a/s is designed for yield and quality testing in forage plots of 1.4m width. Part of the harvested forage is bypassed through a chopper (Figure 1) and fed into a module for automatic sample presentation. The sample passes on a conveyor belt underneath an integrated InGaAs DA spectrometer CORONA (CARL ZEISS), which measures NIR reflectance. White referencing and dark current measurement is carried out automatically by tilting of the spectrometer in the direction of a dark chamber and a ceramic reflectance standard. Data collection is initiated by the driver of the harvester via touch-screen PC by means of the software HARVEST MANAGER (HALDRUP) which interacts with the NIR data collection software CORA (CARL ZEISS). False spectra were eliminated from the NIR data files with filters developed using the CORA add-on software MASK FACTORY (CARL ZEISS) – see below. Calibration was performed at the computing office using WINISI II software (Infrasoft International). Regression analysis between NIR spectral data and conventional dry matter data was carried out using the modified partial least square (MPLS) analysis. Also, an outlier elimination routine served for the elimination of samples with atypically high residuals between actual and predicted DM values and/or atypical spectra identified by extreme global H values. Statistical performance of the calibration in cross validation was characterised by the standard error of cross validation (SECV) and the proportion of explained variation in cross validation (1-VR).

On line sample temperature measurement

Forage temperature was monitored by means of a non-contact infrared temperature sensor (CI by Raytek Corporation) during passage of each sample on the conveyor belt inside the NIR module.

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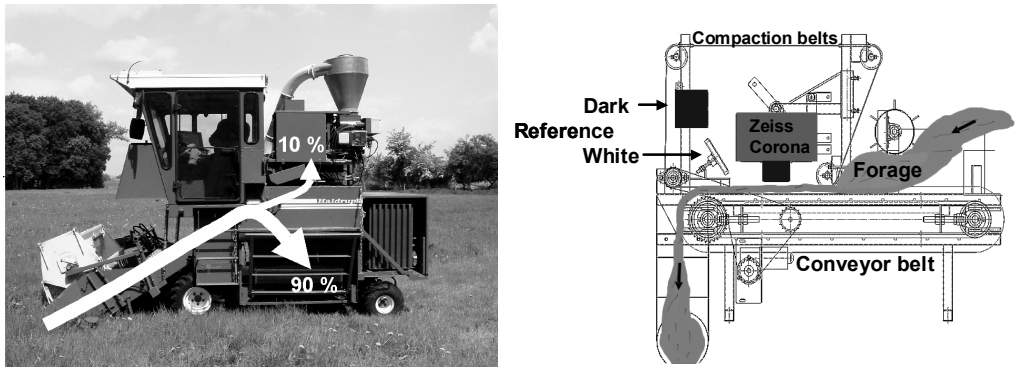

Figure 1. View of Haldrup forage plot harvester (left) with bypass scheme for introducing chopped forage into NIR module and close up diagrammatic view of NIR module (right).

Dry matter assessment

Dry matter content (DM) of the forages in the laboratory was assessed by conventional oven method at 105°C over 36 hours duration.

Laboratory NIRS

For comparative purposes NIR measurements on forages were also performed in large rectangular sample cells on a scanning monochromator NIRSystems 6500 equipped with a sample transport module. The resulting spectra were subjected to regression analysis in an analogous way to those obtained on the DA spectrometer () .

Forages

In the autumn of 2002 at three different sites in North Germany three identically equipped forage plot harvesters were employed for harvesting and *on line* NIR measurement of 1695 plots of forage grass (mainly different cultivars of perennial ryegrass, *Lolium perenne L.*).

12 forage mixtures with extreme variation in dry matter content were harvested by hand and reduced to a short and long chop length of < 0.5cm and > 5cm, respectively to serve for comparative conventional dry matter assessment by oven and laboratory NIR measurements.

Results and discussion

The NIRS Harvest Line concept in forage trials

The rationalisation of modern field trial operations calls for savings of time, labour, chemicals and energy while maintaining high experimental precision in both yield and quality assessments. In the laboratory, the precision of dry matter content measurements of freshly harvested forages is usually higher with conventional oven drying than with NIR diffuse reflectance measurements (Figure 2). In this case, the standard deviation of dry matter determination was estimated on the basis of six replicates of 200g fresh forage in the oven drying method and six replicates measured in specially designed cells for heterogenous, high moisture sample (holding 50 to 100g of fresh forage) in a scanning monochromator NIR instrument (NIRSystems 6500). Figure 2 shows that the standard deviation increases with increasing dry matter content. This applies both to the oven and NIR method. The increase is due to the increased morphological heterogeneity of forages with high dry matter content, which results in increased variability between samples. The higher standard

deviation for long chop compared with short chop can be also be attributed to increased variability between samples.. The steeper rise in error for NIR vs. oven DM assessments is linked to the same phenomenon. NIR measurements require much larger sample surfaces for the minimisation of sampling error in forage analysis to compensate for sampling error than are foreseen in the presently available laboratory NIR instruments.

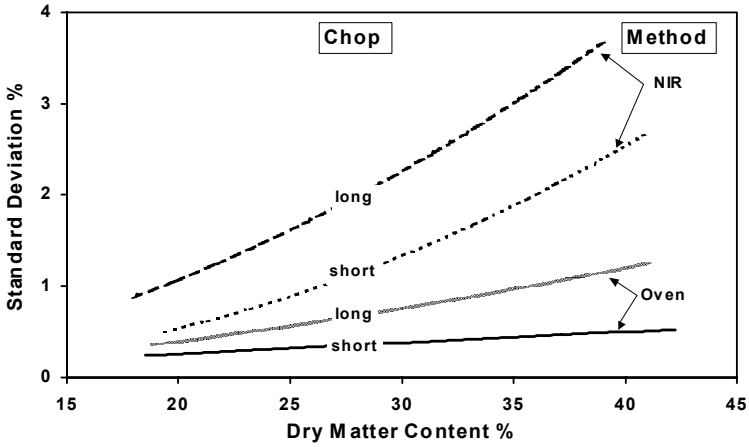

Figure 2. Standard deviation of dry matter assessment using conventional oven drying method and lab NIR assessment in forages of short and long chop length.

Based on such considerations and other empirical knowledge we have formulated a concept for introducing an *on line* dry matter assay into routine forage yield testing in plot trials. It centers on NIR diffuse reflectance measurements and follows several basic rules:

- Shorten the process (minimize the delay between harvesting and analysis)
- Avoid manual sampling (minimize sampling errors through automatic sampling)
- Minimize physical sample preparation (only chopping; avoid drying and grinding)
- Maximize spectrometric sampling (fast continuous scanning of large sample surfaces)
- Use short wavelength NIR (maximise effective path length at low absorptivity)

Continuous sample presentation and spectral filtering

The sample presentation system as part of the NIR module of our harvester was designed to ensure the continuous collection of information of forage characteristics. Yet, practical experience with a diversity of forages demonstrated that more or less unavoidable discontinuities in the forage stream take place. This is regularly the case when at the beginning and end of a plot the transport of forage on the belt starts later and ends earlier than the collection of spectral data. This lack of synchrony is exemplified by the data gained from a single forage plot. A multifile of the raw spectra collected within the plot is shown in Figure 3 (left). It contains typical forage spectra as well as atypical "false" spectra resulting from the absence of forage on the belt. Based on a suitable calibration, NIR predicted DM% and distance measures (global H statistic) were derived from the spectra of this multifile and analysed as a time sequence. For the first 66 spectrometric subsamples the results showed a relatively stable prediction of DM% (Figure 4) and acceptable global H values (<5.0). However, the later spectra gathered within this plot gave rise to atypically low or high DM% and H-values rising to levels above 100. This was because, after the 66th spectrum, the flow of chopped forage particles on the conveyor belt within the NIR module had come to an end.

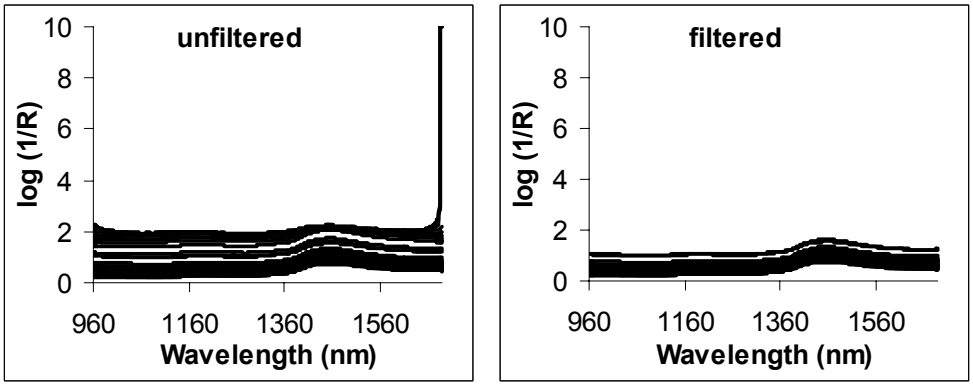

Figure 3. Unfiltered multifile containing raw spectra collected within one single forage plot (left); filtered multifile containing only typical forage spectra of the same plot (right).

A further source of "false" spectra were gaps in the sample flow which occurred irregularly. To discriminate between such "false" and true forage spectra we developed a filter using the MASK FACTORY software. It served to help in the evaluation of our data and to support future routine work. Using this filter, the true forage spectra could be extracted and averaged to form the database characteristic for the particular forage plot (Figure 3 right).

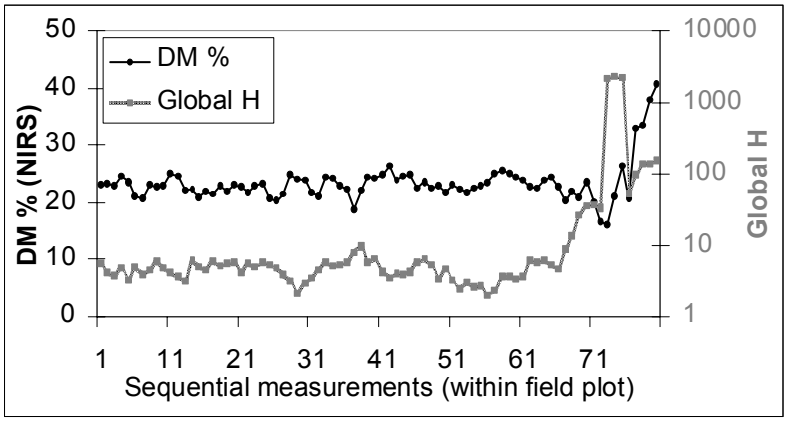

Figure 4. Time sequence of NIR predicted DM% and distance measures derived from the unfiltered spectra shown in Figure 3 of one single forage plot.

Wide scale testing

In the autumn of 2002 the robustness of the NIRS Harvest Line concept was tested with three separate harvesters in forage cultivar trials at three different sites in Northern Germany which provided data from 1695 field plots. After the exclusion of spectral data resulting from a malfunctioning of the sampling and sample presentation system, 1489 acceptable spectral multifiles were recovered, filtered and averaged. In the corresponding data base the individual samples were first ranked according to DM% and split by allocating the odd and even numbered samples to a

calibration and a validation set respectively. In addition to the validation set containing filtered spectra, an further validation set was formed containing the equivalent unfiltered spectra.

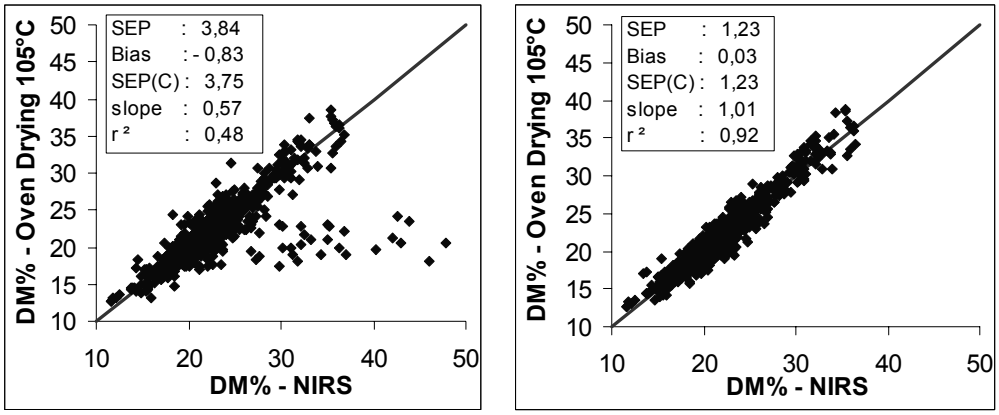

Figure 6. Validation of DM% prediction in unfiltered (left) vs. filtered (right) spectral data originating from three harvesters operating in late 2002 at three North German trial sites.

Throughout the set-up of the calibration/validation sets no account was taken of possible systematic effects due to either NIR instrument or reference lab.

DM% prediction in the unfiltered 739 validation spectra resulted in a poor fit between actual and predicted data (Figure 6 left). Less than half the variation observed in DM% was accounted for by the predicted data. A swarm of outliers with DM% contents of around 20% but much higher predicted values appeared to be the main cause of this poor fit. Filtering of the spectral data remedied the outliers and dramatically improved the goodness of fit (Figure 6 right). Filtering can thus be regarded as a necessary means of quality assurance in the continuous collection of forage spectra on our harvester. The standard error of prediction of 1.23% reached under these circumstances should be set against the level of error of 1.1% DM which has been found in an inter-laboratory comparison of conventional forage DM determination by oven drying[3] in the three testing stations being part of our NIRS Harvest Line test. In due consideration of these findings the precision of our *on line* operation on the forage harvester seems satisfactory.

Consideration of sample temperature

The introduction of a continuous quality control system for field conditions requires a proper understanding of the fundamental processes and effects of varying sample temperature on NIR absorption. In a previous laboratory study[4] we used frozen forages to inspect the OH- first overtone shifts during warm-up and their implications for NIR measurements under large variations in ambient temperature. Under North-West European conditions during harvesting the sample temperature in the field may fluctuate between 5°C and 30°C and higher. This leads to OH-band shifts – which in comparison to customary laboratory calibration experiments – are enormous. Upon analysis of the 1489 spectra obtained in the autumn of 2002 also considerable temperature effects were found. A calibration/validation experiment formed from selected samples collected at defined temperatures on the three plot harvesters of this study showed that a hypothetical forage sample of 18% DM at a given sample temperature of either 15°C, 10°C or 5°C would be predicted with a bias of 1%, 2.3% and 4% respectively if based on calibration samples of 20°C. In the present study such

effects were avoided by representation of all sample temperatures in the calibration set. Consequently, in NIRS Harvest Line calibrations utmost attention must be paid to ensuring robustness towards variations in sample temperature.

Conclusions

Considerable progress has been achieved in setting up a pilot system for the *on line* assessment of forage quality in plot trials. The feasibility of predicting dry matter content of chopped forage particles while being transported on a conveyor belt underneath an InGaAs diode array spectrometer on a commercial plot harvester has been proven. Malfunctioning of the sampling and sample presentation system (mainly due to occasional blockages of chopped forage material at various points in the bypass) lead to more than 10% of the total plots being lost from the exploitable data base. The causes of this were identified and noted for later constructional improvements. However, the lack of synchronisation of presenting the actual sample and collecting spectra from it can be compensated by filtering of spectral data to retain typical forage spectra for the final evaluation. Calibration sample sets formed from such field data must implicitly include sample temperature as an important safeguard against the observed temperature induced shifts of absorbance bands.

In future - as a logical extension of the work presented – we will need to go beyond dry matter content as the lead parameter for developing NIRS calibrations. Additional quality parameters will be water soluble carbohydrate, protein and energy content of forages. Furthermore, we want to form a collaborative network to support non-NIR-specialist managers of field trials who need to use this technology for saving labour and energy costs.

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