Compositional analysis by near infrared diode array instrumentation on forage harvesters

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Introduction

Near infrared (NIR) spectroscopy, as a non-invasive optical method, offers the opportunity to measure plant constituents very rapidly. As robust NIR diode array spectrometers have been shown to be ideally suited for on-line measurements in several different applications in industry, it is an obvious next step to examine these instruments for agricultural purposes.[1] It has already been shown that, for assessing a multitude of quality parameters in dried forage samples, NIR diode array instruments can perform analytically as satisfactorily as NIR scanning monochromator instruments.[2] Our work is an elaboration of previous attempts to utilise the potential of diode array instrumentation for the on-line compositional analysis on agricultural plot harvesters.[2,3] It first aims to assess the dry matter content in forages without having to handle samples or transport them to a laboratory and dry them.

Materials and methods

This work was performed with the world's first commercially available experimental forage plot harvester equipped with an NIR module for the collection, compression and scanning of forage samples during harvesting (Haldrup NIRS Harvest Line; see Figure 1). The NIR-module includes an InGaAs diode array spectrometer (Zeiss Corona 45 NIR 1.7) and installations for spectrometer referencing, sample preparation and presentation. A representative subsample of each individual plot is automatically chopped and filled into the measuring chamber where it is compressed. Subsequently, the diffuse reflectance of the sample surface is measured by the diode array spectrometer while it moves downwards in front of the measuring chamber. This whole procedure ensures that exactly the same sample that was scanned is available for conventional analysis in order to build up a robust calibration database.

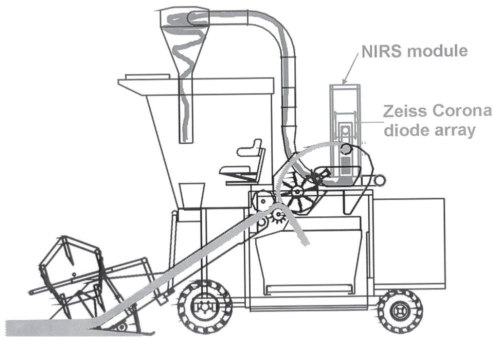

Figure 1. Cross-section of the Haldrup forage plot harvester with NIR module.

In the year 2000, the plot harvester was used for harvesting and analysing typical forage plots (1.4 m × 9 m) consisting of several grass species

Near Infrared Spectroscopy: Proceedings of the 10th International Conference

(*Lolium perenne*, *Lolium multiflorum*, *Festuca pratensis*), a legume species (*Trifolium repens*) and mixtures of both. After NIR measurements were taken in the field, each sample was also measured in the laboratory using a conventional grating spectrometer equipped with both an Si- and a PbS-detector (NIRSystems 6500). Dry matter content (DM) was then determined conventionally by means of oven drying at 105°C for 36 h. Routine chemometric procedures were employed to assess the comparative accuracy and precision of the DM assessment in the spectral range between 960 and 1690 nm by the NIR diode array, as well as by the conventional NIR grating instrument. On-line data acquisition and control of the NIR module was done by means of the Zeiss software CORA. The WINISI II software from Infrasoft International was used to operate the laboratory spectrometer and for chemometric procedures on both types of spectrometers. For calibration, modified partial least squares (MPLS) regression analysis was performed on transformed NIR absorbance values vs DM contents. The accuracy of the DM assessment was estimated through cross-validation by means of the statistical parameters *SECV* (standard error of cross validation) and $1/VR$ (1 minus variance ratio which corresponds to the r^2 value).

Results and discussion

Because of the high absorptivity of water in the near infrared, an NIR method for assessing moisture content (or alternatively dry matter) has become the obvious first target of our on-line forage measurements under field conditions. The NIR spectra obtained during harvesting, using the Zeiss diode array spectrometer, are typified by broad, rolling absorption bands dominated by the strong, first OH stretch at 1450 nm attributable to water in forage. Spectra with minimal and maximal absorbance levels, as well as the average spectrum, are shown to indicate the overall spectral variability of the forages under investigation (Figure 2).

Under these circumstances, calibration for dry matter content was performed on forages in two separate concentration ranges: (a) the range typically observed in forage trials, i.e. from 12% to 30% DM and (b) in forages with excessive maturity ranging from 30% to 44% DM.

Calibration of the diode array spectrometer, in the typical range of forage maturity, resulted in an *SECV* value of 1.19%, while in forages with excessive maturity the *SECV* amounted to 1.64% DM (Figure 3). The corresponding error levels obtained for the same subsets of samples in post-harvest laboratory measurements on the conventional grating NIR spectrometer were 1.04% DM and 1.42% DM for forages with typical and excessive maturity, respectively (Figure 4). The $1/VR$ values also sup-

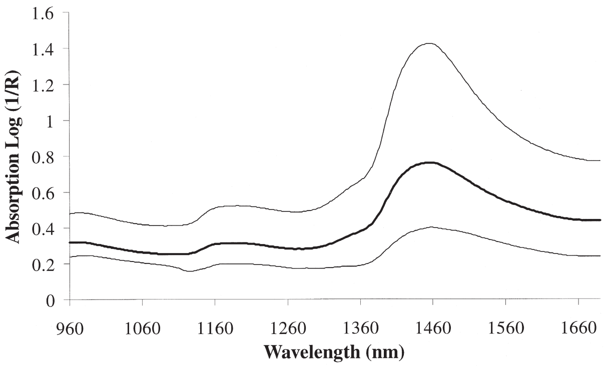

Figure 2. NIR spectra of fresh forage samples measured by Zeiss Corona NIR 1.7 diode array spectrometer during harvesting [maximum, minimum and average spectrum (thick line)].

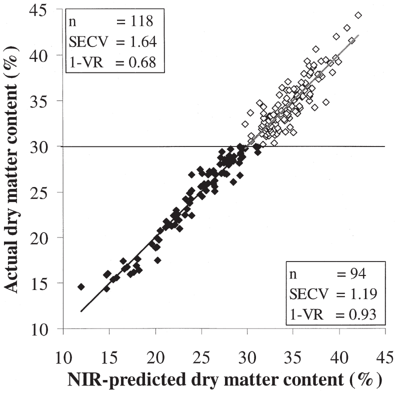

Figure 3. On-line assessment of dry matter content (DM%) in fresh forages by means of the diode array spectrometer Zeiss Corona 45 NIR 1.7 (samples with DM ≤ 30%: black symbols, samples with DM > 30%: white symbols).

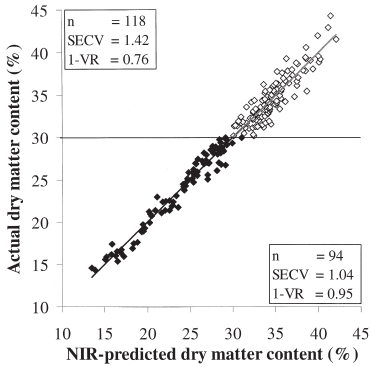

Figure 4. Assessment of dry matter content (DM %) in fresh forages by means of conventional grating spectrometer NIRSystems 6500 in the laboratory (samples with DM ≤ 30 %: black symbols, samples with DM > 30%: white symbols).

port the conclusion that the degree of fit between observed and predicted DM values differs strongly between forages of typical and excessive maturity. It is assumed that—because of increasing morphological differentiation and heterogeneity within plants—the sampling error in forages increases with maturity. Consequently, in forages of excessive maturity, the increase in *SECV* is seen as being caused by the increased sampling error. As a matter of fact, this effect is more noteworthy than the small difference in analytical precision between on-line diode array measurements in the field and post-harvest measurements using the laboratory spectrometer.

The results of this study show that the forage plot harvester with the integrated NIR diode array instrument employed here functions well for the assessment of dry matter even under rugged field conditions. Further refinements appear to be necessary for optimising the process of automated sampling, chopping and filling of the sample compartment to minimise sampling error in highly heterogeneous forage materials. In any case, it is expected that, in future, fundamental savings of energy and labour will be realised by the use of NIR diode array instruments on agricultural plot harvesters, thus replacing the costly and labour-intensive conventional oven drying method for dry matter determination.

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