

PROGRESS WITH THE IMPLEMENTATION OF A DIODE ARRAY NEAR-INFRARED SPECTROMETER FOR DIRECT AT-LINE ANALYSIS OF SUGARCANE SAMPLES

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Abstract

The feasibility of routine cane testing using Near-Infrared Spectroscopy (NIRS) to replace conventional juice-extraction procedures in the SASEX laboratories was investigated with a Perten DA7000 direct-light, single diode array spectrometer. Validation results were very satisfactory for DM, BRIX and POL ($R^2=0.98, 0.93, \text{ and } 0.94$; $SEP=0.676, 0.768, 1.052\%$, respectively) and somewhat lower for fibre and purity ($R^2=0.89, 0.83$; $SEP=0.912, 9.035\%$). A method was tested for detecting outlier samples during measurement, where predicted moisture+fibre+BRIX which deviates from 100% by more than two standard errors (0.75%) is rejected. Further improvements in temperature control and more regional representation of calibration samples are needed to optimise the performance of this equipment for routine cane testing. Considerable savings in time, labour, chemicals, and hazardous waste disposal can be attained by this rapid NIRS method on shredded cane when compared to conventional juice processing methods.

Keywords: at-line, cane analysis, diode array, NIR, Perten DA7000

Methodology, results and discussion

Routine analysis of whole sugarcane stalk samples is an essential support service to most research programmes at the South African Sugar Association Experiment Station (SASEX). Cane juice extraction, filtering and analysis for POL (apparent sucrose concentration, measured by polarimetry), and BRIX (total dissolved solids concentration, measured by refractometry), and measurement of sample DM (dry mass; gravimetrically by 16-h drying) is a slow and expensive process. Besides, the juice filtering step requires lead acetate to flocculate solids, and disposal of the resulting lead-contaminated waste is increasingly difficult and expensive.

Near-Infrared Spectroscopy (NIRS) is an attractive remote-sensing technology which can substitute all the conventional processes with a single rapid at-line measurement. The initial capital cost of NIR spectrometers is high, but can be justified by cumulative savings of time, materials, waste disposal, and labour. The SASEX sugarcane breeding programme is being expanded in 2000, such that the additional research trials will require a peak laboratory capacity of up to 400 samples /d. To cope with these increased samples in the most cost-effective and environmentally friendly manner, NIRS was adopted as the primary method for future cane analysis. The objective of this communication is to describe progress made with the implementation of a diode array near-infrared spectrometer for direct at-line analysis of sugarcane samples in the SASEX cane testing laboratory.

A Perten DA7000 Fleximode single diode array (950 to 1700 nm) NIR spectrometer was chosen for the task, because of its "direct light" mode, and rapid measurement speed (one averaged scan in three seconds). Direct light or post-dispersive configurations differ from pre-dispersive ones in that the sample is placed between the light source and the monochromator (diffraction grating), rather than between the detector and the monochromator. Obvious advantages of post-dispersive instruments like the DA7000 are that a more powerful polychromatic light source (typically from a halogen lamp) is directed at the sample, which therefore reduces interference from ambient light, targets and penetrates a wide sample segment, and the distance from instrument to sample can be increased to about 14 cm.

The DA7000 NIRS was mounted above a sliding stainless steel sample tray (L 1250 mm, W 210 mm, D ~80 mm) guided by a stainless steel slot. During operation, the tray containing the shredded sample from 12 cane sticks, was pulled slowly by hand under the light beam of the scanning DA7000, while ten continuous average scans were collected. Earlier subsampling investigations using a small circular (13.5 cm diameter) stationary tray had demonstrated the need for at least five averaged scans per sample, so that the calibration and prediction multiple correlation coefficients (R^2) were maximised, and the standard errors (SE) were minimised (Figure 1). Total duration for the NIRS meas-

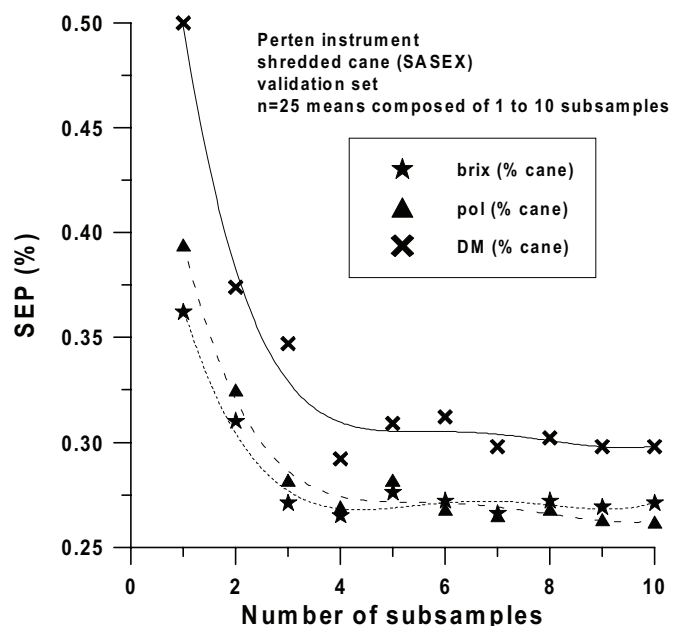


Figure 1. Reduction in standard errors of prediction (SEP) using NIRS, by increasing the number of scans per cane sample.

urement step is about 40 s/sample, enabling a realistic throughput of about 1 sample /min or 480 samples /d (8 h day).

Calibration of the DA7000 instrument was achieved with both WIN-ISI 1.02a and Grams/32 software, using the modified partial least squares algorithm, and first derivative (3-point gap), standard normal variate (SNV)+detrnd pre-processing of spectra. Grams/32 is packaged with the DA7000 specifically to develop and implement calibration equations for that instrument. Reference data for calibration and validation (training set) were obtained from 307 cane samples analysed by conventional laboratory methods described above. About 66% randomly selected samples (196) were used for calibration, and the remainder (95) for validation. Fourteen sample spectra were discarded because high room temperatures in summer caused unacceptable sensor noise (Figure 2). Specified instrument operating temperature is 10 to 35°C, and in the final placement, a room airconditioner will maintain laboratory air at 23°C and a separate air chiller unit will pre-cool and dry the intake air for the DA7000 to 20°C.

Validation results were very satisfactory for DM, BRIX and POL ($R^2=0.98, 0.93, \text{ and } 0.94$; SEP= 0.676, 0.768, 1.052 %, respectively) and somewhat lower for fibre and purity ($R^2=0.89, 0.83$; SEP=0.912, 9.035 %). Lower precision for fibre and purity may be attributed to the additional cumulative error from conventional measurements in these derived variables. Calibration equations inevitably predict incorrectly on a small fraction of "outlier" samples, especially in BRIX, POL, fibre and purity variables. By monitoring the sum of moisture + fibre + BRIX, which should add up to 100% for each sample, outliers may be trapped during routine analysis with the DA7000, since true laboratory values would then not be known. Preliminary tests of this technique with the same data showed that predicted values, the sum of which deviated from 100% by more than two standard errors (0.75%), were likely to be outliers. In practice, while the test is not infallible, it should assist to filter out the majority of erroneous samples analysed by NIRS, which would

then be diverted to the conventional analytical processes in the laboratory.

Unacceptable regional bias is frequently encountered in NIRS calibrations, and was demonstrated in these data as inaccurate prediction of POL for Durban region samples by a calibration derived from Pongola region samples. The only reliable solution to these bias problems is to enlarge the calibration set with more representative data from all possible regions, over different seasons. Considerably more calibration development is therefore needed before the DA7000 can be used for routine analysis. Final implementation of the spectrometer will proceed after the addition of a computerised air cooling system and reversible motor drive for the sample tray. The proper regulation of temperature and sample transfer speed, followed by thorough re-calibration for all regions, should further enhance the already acceptable performance of the Perten DA7000 instrument in this feasibility study.

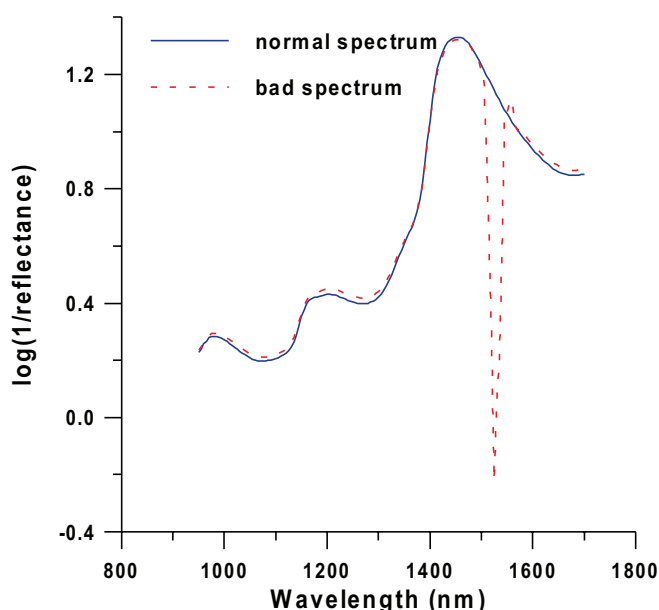


Figure 2. Normal DA7000 spectrum of a shredded cane sample (solid line), and defective spectrum caused by high air temperature (dashed line).