AT-LINE ANALYSIS OF SHREDDED CANE USING A FOSS DIRECT-LIGHT NIR: PRELIMINARY RESULTS PRIOR TO FACTORY TESTS

By
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Abstract
A test rig was built at the Sugar Milling Research Institute to establish the precision and accuracy of the Foss Direct Light NIR for analytes in shredded cane. The rig had the following features: simulation of the ON-LINE application, automation of the cane moving past the scan-head, one metre of cane sample was scanned by the NIR instrument. Over 1200 samples were scanned between August 2000 and December 2000. The calibration and prediction sets were independent of each other. The predictions for pol, brix, moisture and fibre were acceptable. Ash by NIR was only semi-quantitative (e.g. normal or high). The precision, based on duplicate samples, of the NIR estimates rivalled that of the laboratory procedures. The instrument is to be installed at Maidstone sugar mill, adjacent to the Cane Testing Service (CTS) sampling station, for the 2001/2002 season.

Introduction
When analysing constituents in shredded cane, most investigators use a laboratory-based instrument that produces monochromatic light prior to interaction with the sample. These instruments can be fitted with the:
- transport module and the coarse sample cell (Edye and Clarke, 1996; Schaffler, 1996; Peterson and da Silva, 1998);
- remote reflectance probe (Berding and Brotherton, 1999).

The Foss post-dispersive or Direct-Light NIR, a process instrument, allows for the on-line analysis of analytes on conveyor belts and other factory applications. The system has been successfully used in the Australian sugar industry at several sugar mills, mainly for fibre analyses (Staunton et al., 1999).

In South Africa, individual consignments of cane are analysed by an extraction technique known as ‘the direct analysis of cane’ (DAC) as performed by the South African Cane Testing Service (CTS). Although DAC is probably the best sampling procedure available, it is expensive. A preliminary feasibility study was undertaken by the Sugar Milling Research Institute (SMRI) into the precision and accuracy of predictions for pol, brix, moisture and fibre using the Direct-Light NIR with a test-rig.

Experimental
Description of the test rig
A schematic of the SMRI test rig is shown in Figure 1. A photo of the rig with the NIR instrument is included in Figure 2.

Fig. 1—Schematic diagram of the SMRI Test-Rig: (A) = Side view, (B) = Top view.
1 = Scanning table, 2 = glass scan plate, 3 = NIR scan-head, 4 = bottomless scanning drawer holding shredded cane, 5 = guide rails, 6 = fibre optic bundle to NIR spectrophotometer, 7 = DC motor, 8 = pulley, 9 = drive belt, 10 = bearing, 11 = M12 threaded bar, 12 = 12 mm nut, support attached to drawer and threaded bar, 13 = micro switch, 14 = control box (stop/start | forward/reverse).

KEYWORDS: Shredded Cane, Direct Cane Analysis, NIR, Cane Testing, Analysis.
Shredded cane, from the CTS full-width sampling-hatch, was loosely packed into the bottomless drawer (1000 mm long × 250 mm wide and 80 mm deep). A lid was used to improve sample presentation and to reduce evaporation. A DC motor drives a threaded bar which in turn moves the sampling drawer past the glass scanning window (speed is variable from 0.5 to 1.2 metres per minute). The NIR scan head is mounted on height adjustable supports below the glass window. When scanning a cane sample, the NIR recorded 10 reference scans, followed by 105 sample scans followed by 10 reference scans.

Results

Prediction results for pol and other quality parameters

Samples scanned from August to October 2000 were used for calibration. The accuracy of these calibrations was tested by predicting analytes in shredded cane scanned during November and December 2000. The results are presented in Table 1.

The results in Table 1 imply that:

- pol and brix estimates were excellent with little bias and exceptionally good statistics;
- a SEP of 1.0 unit for moisture is satisfactory as this represents an error of less than 2%;
- for the DAC procedure, fibre % cane is derived from moisture % cane and brix % cane. A NIR error of 1.1 in 16 is therefore reasonable. In practice when using NIR, fibre will be calculated from the NIR estimates of brix and moisture;
- the ash estimates were poor (Figure 3). Reasons for this could be:
  - ash is inorganic with little absorbance in the NIR;
  - total ash levels in shredded cane are low (0.5 to 1.5). NIR is not a sensitive technique;
  - the precision test (see below) showed that the laboratory method for ash in cane produced errors of 20%, thus NIR predictions could also show scatter;
  - the NIR estimate for ash is probably more qualitative than quantitative. In fact for quality control purposes, an ash estimate expressed as ‘NORMAL’ or ‘HIGH’ is probably adequate.

Table 1—Prediction results, Calibration set from Aug to Oct, Prediction set from Nov to Dec 2000.

<table>
<thead>
<tr>
<th>Analyte</th>
<th>SEP</th>
<th>Bias</th>
<th>Slope</th>
<th>RSQ</th>
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</thead>
<tbody>
<tr>
<td>Pol</td>
<td>0.4</td>
<td>0.1</td>
<td>0.94</td>
<td>0.92</td>
</tr>
<tr>
<td>Brix</td>
<td>0.4</td>
<td>0.1</td>
<td>0.90</td>
<td>0.84</td>
</tr>
<tr>
<td>Moisture</td>
<td>1.0</td>
<td>-0.2</td>
<td>0.94</td>
<td>0.76</td>
</tr>
<tr>
<td>Fibre</td>
<td>1.1</td>
<td>0.3</td>
<td>0.91</td>
<td>0.69</td>
</tr>
</tbody>
</table>

SEP = standard error of prediction, RSQ = correlation coefficient squared

Fig. 3—Comparison of ash in shredded cane, N = 55 samples, Laboratory versus NIR estimates.
Evaluation of the precision of laboratory and NIR methods

NIR predictions are based on the laboratory data used for calibration. The quality of the CTS DAC data was evaluated by analysing duplicate shredded cane samples for a limited period. Duplicate subsamples were also packed into the NIR sampling drawer for scanning. The results of these repeatability tests are presented in Tables 2 and 3.

The results from Tables 2 and 3 indicate that:
- the precision of the NIR estimates for pol, brix and moisture are virtually identical to the repeatability of the laboratory results;
- NIR precision for both fibre and total ash was superior to the laboratory methods; this could be due to:
  - fibre is a derived analyte (errors from both moisture and brix % cane);
  - ash is a difficult determination and error-prone;
  - the NIR calibration averages out laboratory errors (partial least squares);
  - the fact that NIR scanning is simple and precise.

### Table 2—Precision of the DAC procedure, duplicate samples, N = 55.

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Grand Mean (% cane)</th>
<th>Mean SD of duplicates</th>
<th>RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pol</td>
<td>11.3</td>
<td>0.2</td>
<td>2.0</td>
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<tr>
<td>Brix</td>
<td>13.7</td>
<td>0.4</td>
<td>2.6</td>
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<tr>
<td>Moisture</td>
<td>69.8</td>
<td>0.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Fibre</td>
<td>16.4</td>
<td>0.6</td>
<td>3.3</td>
</tr>
<tr>
<td>Ash</td>
<td>1.2</td>
<td>0.2</td>
<td>20.5</td>
</tr>
</tbody>
</table>

### Table 3—Precision of NIR estimates, samples scanned in duplicate, N = 55.

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Grand Mean (% cane)</th>
<th>Mean SD of duplicates</th>
<th>RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pol</td>
<td>11.3</td>
<td>0.2</td>
<td>2.0</td>
</tr>
<tr>
<td>Brix</td>
<td>13.8</td>
<td>0.3</td>
<td>2.5</td>
</tr>
<tr>
<td>Moisture</td>
<td>69.9</td>
<td>0.5</td>
<td>0.8</td>
</tr>
<tr>
<td>Fibre</td>
<td>15.9</td>
<td>0.2</td>
<td>1.4</td>
</tr>
<tr>
<td>Ash</td>
<td>1.6</td>
<td>0.1</td>
<td>6.0</td>
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</table>

### References


ANALYSE A ECHELLE DE LABORATOIRE DE CANNES DEFIREEES PAR LA SPECTROSCOPIE PROCHE INFRA ROUGE DE FOSS UTILISANT L'ILLUMINATION DIRECTE: RESULTATS PRELIMINAIRES AVANT LES TESTS EN USINE

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Résumé
Une plate-forme expérimentale fut installée au Sugar Milling Research Institute afin d'évaluer la précision de la spectroscopie proche infra rouge (PIR) en illumination directe fabriqué par Foss pour la détermination des composantes dans la canne défibréée. La plate-forme avait les caractéristiques suivantes:

• simulation de l’application en milieu réel
• automatisation de l’échantillon de canne passant devant la tête de lecture
• examen d’un mètre d’échantillon de cannes par la PIR


L’appareil sera installé à l’usine de Maidstone, tout près de la station d’échantillonnage du Cane Testing Service (CTS), pour la saison 2001–2002.

Mots clés: cannes défibréées, analyse directe des cannes, PIR

ANALISIS EN LÍNEA DE CAÑA DESFIBRADA UTILIZANDO UN NIR FOSS DE LUZ DIRECTA: RESULTADOS PRELIMINARES ANTERIORES A LAS PRUEBAS EN FÁBRICA

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Resumen
En el Sugar Milling Research Institute, se construyó una infraestructura de prueba con el propósito de establecer la precisión y exactitud del NIR para analitos en caña desfibrada.

La infraestructura tenía las siguientes características:

• Simulación de la aplicación en línea.
• Automatización del paso de la caña ante el sensor.
• El NIR efectuó un barrido a muestras de cañas de 1 metro de profundidad.

Entre Agosto y Diciembre de 2000, más de 1200 muestras fueron sometidas al barrido del NIR. Las corridas de calibración y predicción fueron independientes entre sí. Las predicciones para pol, brix, humedad y fibra fueron aceptables. La determinación de cenizas fue sólo semi-quantitativa (v.gr. normal o alta). La precisión resultante, para los estimados por NIR, basada en muestras duplicadas, cumplió en forma muy estrecha con la obtenida en los procedimientos seguidos en el laboratorio.

El instrumento será instalado en el molino de Maidstone, cercano al servicio de pruebas de caña (Cane Testing Service—CTS) de la estación de muestreo, para la zafra 2001–2002.

Palabras claves: Caña desfibrada, análisis directo de caña, NIR, prueba de caña, análisis.