Process monitoring in sugar mill by near infrared spectroscopy

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Introduction

Evaluation of the sugar content of juice and syrup samples is an important aspect of routine sugar milling processes, such as concentration and crystallisation. However, the values obtained from the samples are not used to control the processes because the chemical method currently used by the technicians is too time-consuming. It is necessary to develop a simple monitoring system, based on a rapid, nondestructive and low-cost measurement protocol. To achieve this, near infrared spectroscopy (NIRS) was investigated. NIR spectroscopy affords a rapid method for the determination of the Pol and SSC (Soluble solids content) of sugarcane juice or shredded samples.[1–3] In Okinawa, a compact NIR system is used for cane quality measurement as part of the payment system in place in all 10 sugar mills in operation. In this study, the NIR spectra of milled juice and syrup samples were measured to ascertain the potential for a sugar quality measurement system. Calibrations for SSC and Pol value of juice and syrup samples were developed, and the influence of syrup temperature on the calibrations was evaluated.

Materials and methods

Samples and NIR measurements

Juice from the first milling and syrup samples were collected from one of the 10 sugar mills operating in Okinawa (Daito Togyo Co. Ltd., Japan) during the 2007/2008 harvest season. Measurements were performed using a compact NIR system (InfraXact, Foss Co. Ltd.) equipped with a slurry cup, and a 0.5 mm thick gold reflector was used for NIR data acquisition from liquid samples. The NIR reflectance spectra were measured from 1100 to 1848 nm in 2 nm increments, in order to determine the target properties.

Chemical analysis

To provide reference data for the development of a set of calibrations, SSC and polarimeter readings were measured using a refractometer (RX-500FB, ATAGO Co., Ltd, Japan) and polarimeter (AP-300, ATAGO Co., Ltd, Japan), respectively. One-hundred mL of milled juice were placed in a flask and 1 g of lead acetate was added. The contents of the flask were shaken and filtered. The filtrate was transferred to a polarimeter tube (200 mm) and the Pol reading was measured. The Pol value was calculated from the Pol reading and SSC value. The Pol and SSC value of the syrup sample were then again measured after four-fold dilution.

Data analysis

Sixty juice and syrup samples were randomly selected and used for the calibration model, with the remaining samples used for evaluating the model prediction error. All NIR calculations were performed using Unscrambler software (ver.9.7, CAMO). A partial least squares regression (PLSR) analysis was applied to the original absorbance spectra for predicting the SSC and Pol value. Calibration equations were then tested against the validation set.

Results and discussion

NIR calibration

Figure 1 shows NIR reflectance spectra of first mill juice and syrup samples.

Strong absorption bands, due to water, were observed at 1450 nm.[4] Clear differences in absorbance can be seen at 1450 nm in these samples, reflecting the lower water content of the syrup samples. The average Pol of the syrup and milled juice are 15.2 and 52.3%, respectively.

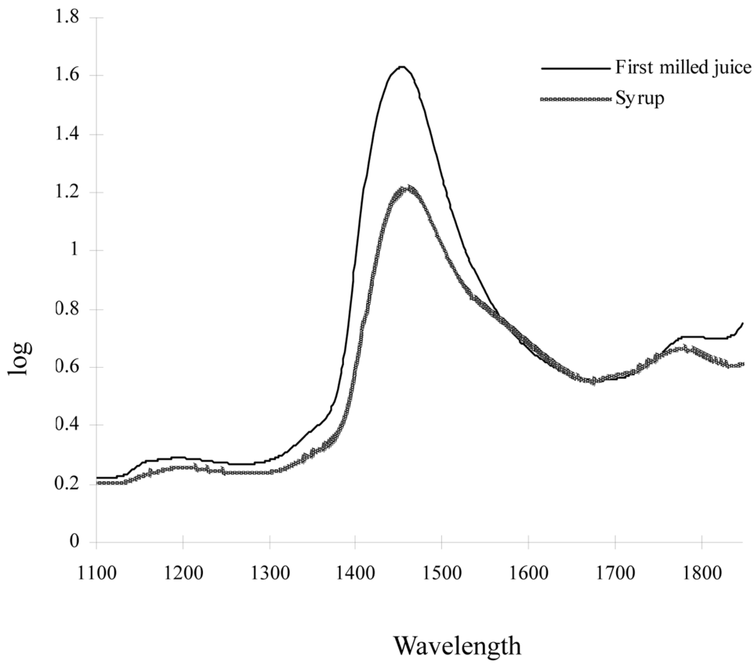

Figure 1. Original reflectance spectra of first mill juice and syrup sample.

Table 1. Calibration and prediction results for SSC and Pol values of first mill juice samples.

Property	Treatment	Calibration set			Prediction set	
		nF	*R*	*RMSEC*	*SEP*	*Bias*
SSC (°Brix)	Centrifugation	7	0.934	0.16	0.17	0.02
	No-centrifugation	6	0.944	0.15	0.12	−0.01
Pol (%)	Centrifugation	11	0.950	0.14	0.15	0.04
	No-centrifugation	6	0.899	0.20	0.19	−0.03

nF: Number of factors used in the MPLSR calibration; *R*: Correlation coefficients;
RMSEC: Root mean square error of calibration;
Bias: The average of difference between the NIR and actual values;
SEP: Bias-corrected standard error of prediction.

Calibration for first mill juice after centrifugation

The calibration equations for the Pol and SSC of the first mill juice were developed using partial least squares regression (PLSR). The first mill juice was contaminated by soil or "mud" so that centrifugation was necessary as a pre-treatment step. In this study, we investigated the effect of sample centrifugation on the calibration performance (Table 1).

The standard error of calibration (*SEP*) was 0.19% for the Pol calibration of first mill juice with no centrifugation. PLSR analysis using centrifuged samples showed an *SEP* of 0.15% (R=0.950, $RMSEC$=0.14%). The *SEP* of centrifuged and uncentrifuged samples obtained from SSC calibration was 0.17% and 0.12%, respectively. These results show that centrifugation had no significant effect on calibration accuracy for first mill samples.

Calibration for syrup samples

The calibration equations for the Pol of syrup samples were also developed using PLSR. Results of the calibrations for samples at 20°C gave *RMSEC* and *RMSEP* values of 0.34% and 0.35%, respectively. In the sugar milling process, syrup is heated to approximately 50 to 60°C to encourage condensation. To investigate the influence of temperature, calibration equations were developed for samples at 20, 30, 40, 50 and 60°C. The results are shown in Table 2.

Table 2. Calibration results for the Pol values of syrup samples at different sample temperatures.

Temp	20°C	30°C	40°C	50°C	60°C	20–60°C
R	0.95	0.93	0.93	0.90	0.94	0.85
RMSEC	0.34%	0.37%	0.40%	0.46%	0.42%	0.58%
RMSEP	0.35%	0.40%	0.41%	0.46%	0.42%	0.58%

R: Correlation coefficients; *RMSEC*: Root mean square error of calibration;
RMSEP: Root mean square error of prediction.

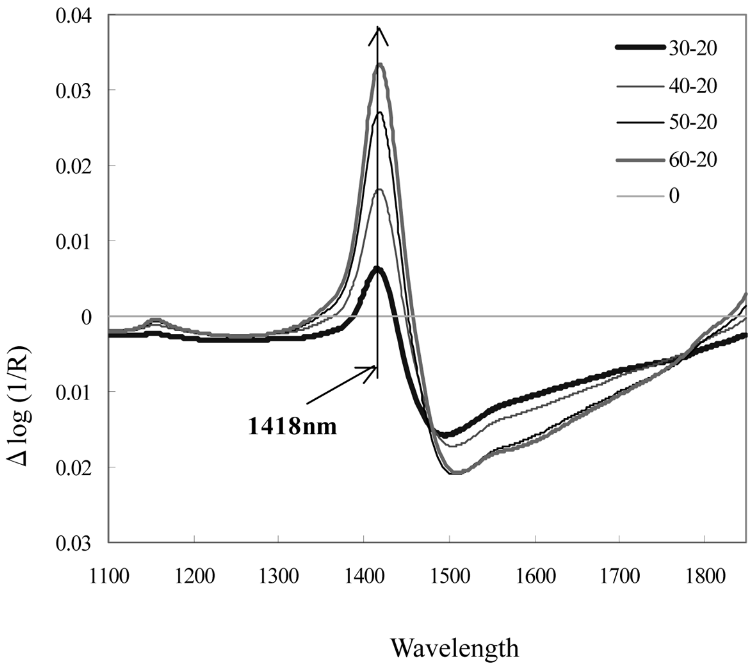

Figure 2. Difference spectra of syrup samples (30–20, 40–20, 50–20, 60–20°C).

The calibration developed for the Pol value at 20°C showed a high degree of accuracy for unknown samples. Figure 2 shows the difference spectra for samples of different temperatures.

A positive peak was observed at 1418 nm in the difference spectra. Sample temperature was found to affect the NIR spectra markedly, mainly the water absorption band. As a next step, to cover the temperature range expected during the sugar milling process, a robust calibration was developed using samples at different temperatures. The *RMSEP* of the calibration for the temperature range from 20 °C to 60 °C was lower than that obtained from the calibrations models for each specific temperature, indicating that Pol values for syrup samples at different temperatures can readily be obtained.

We conclude that NIS is a useful tool for the measurement of milled juice and syrup quality during the sugar milling process.

Conclusions

This study has established NIR calibrations using PLSR for estimating the quality (SSC and Pol values) of juice and syrup samples during the sugar milling process.

We found that there were no significant differences in the accuracy of calibration models for centrifuged and uncentrifuged juice samples, indicating that the NIR technique will be useful for the analysis of first mill juice contaminated with soil and mud. Further, we found that the NIR spectra of syrup were affected by sample temperature, with particularly large changes observed in the water absorption band. Although the results of the robust calibration model were less accurate than those of temperature-specific models, it was nonetheless sufficient for effective application to the measurement of Pol of syrup samples of different temperatures.

References

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