

REFEREED PAPER

PRACTICAL APPLICATION OF NEAR-INFRARED SPECTROSCOPY TO PREDICT QUALITY OF SOLID SUGAR

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Abstract

Near-infrared spectroscopy (NIRS) is a fast, efficient and easy method that has enabled Sezela mill to test every consignment of sugar prior to leaving the site during the 2012/13 season. This has ensured customer satisfaction, savings on costs of rejection, and improved supplier credibility, which was encouraging in a season with large variations in weather conditions, cane quality and customer demands.

Conventional analytical methods for pol, moisture, colour, starch and dextran are time consuming and laborious, and make it impractical to test every consignment of sugar. The use of the NIR Fourier Transform Bruker multi-purpose analyser (MPA) therefore becomes attractive as it can read quality parameters of solid sugar in less than one minute.

This paper discusses the practical application of the MPA instrument on very high pol (VHP) solid sugar and presents the comparative analytical data for both conventional and NIRS methods.

Keywords: NIRS, sugar, pol, moisture, colour, starch, dextran

Introduction

Sugar quality parameters are closely monitored by sugar factories. For those factories that have packing plants, it is essential that every pack of sugar meets all of the quality standards before it reaches the consumer. With the use of near-infrared spectroscopy (NIRS) and the high information content in the NIR spectra, a sample of sugar can be measured in less than one minute. This allows for the simultaneous analysis of many different components of that sample with high precision. Such an instrument will enable quality control staff to segregate the product for storage in separate bins before packing based on colour, pol, moisture, starch and dextran.

During the past season Sezela had to supply very high pol (VHP) sugar to two refinery mills and one raw sugar mill, while maintaining the normal supply to the sugar terminal. These mills have very stringent rejection procedures. Sezela mill, which predominantly supplies the sugar terminal, had to ensure that no substandard sugar reached these mills. Sezela produced approximately 6000 one ton bags of sugar during the 2012/13 season, and batches of ten were analysed. The short analysis time and the ability to produce multiple components using a NIR multi-purpose analyser (MPA) has proved most beneficial during the diversion and packing period. Operating the instrument is not very demanding and relatively unskilled staff were recruited for this purpose.

Equipment and software

The following system was loaned to Illovo Sezela by Bruker South Africa:

- Bruker Multi Purpose Analyser – attached to the multi-purpose analyser is an integrating sphere which Sezela made use of for the analysis of VHP sugar. All spectra were obtained in reflectance mode, and the range was between 4000 and 11500/cm (wave numbers). The integrating sphere allows for measuring solids and pastes in diffuse reflection. An optical sample rotator assures high reproducibility for heterogeneous samples.
- The NIRS software used for spectral processing and calibration creation was the Opus Version 7.0. This software was used as an interface, and was relatively easy for staff to learn how to use.

Experimental

Samples

Sezela keeps 150 g of retention sample for every eight hour shift, with wet chemistry results being recorded for each sample. Such samples were used in the calibrations. All analyses including the NIRS predictions were done at a temperature of 20°C, which was maintained by air conditioning. Approximately 50 g of sample was used for the NIRS calibration scans. Sixty-four scans were performed on each sample while the sample was rotating.

Wet chemistry: sugar colour, pol, moisture, starch and dextran

The analytical integrity of the Sezela mill laboratory is high. It has won the Sugar Milling Research Institute's 'Analytical Best Lab' award for the past three seasons and had 88.10% and 100% of samples within tolerance for both sugar and molasses results, respectively. The following equipment was used for wet chemistry routine analysis:

- Polarimeter: Autopol 880
- Refractometer: Rudolf J157
- Crison basic 20 pH meter
- Hotplate
- Vacuum
- Water bath
- Spectrophotometer.

The following are the test methods (Anon, 2005) for wet chemistry that were used for the NIRS comparisons:

- Colour, ICUMSA¹ units SASTA Test Method 7.5
- Pol SASTA Test Method 7.1
- Moisture SASTA Test Method 7.4
- Starch SASTA Test Method 7.8
- Dextran, haze method SASTA Test Method 7.10.

¹International Commission for Uniform Methods of Sugar Analysis

NIRS calibrations

The calibrations were loaded onto the instrument using the Opus Quant software. The software is based on a multivariate algorithm called partial least squares (PLS). These regressions were used to draw up calibrations. The software makes it possible to find the best possible calibration automatically. The calibration model was built on previous seasons' data and a method of cross-validation was used to develop the equations.

Results and Discussion

Figures 1 to 5 are scatter plots of sugar despatched during the 2012/13 season, showing the correlation between results obtained in the laboratory and those obtained by NIRS. One hundred random samples were used to predict the quality parameters pol, moisture and colour, while 20 random samples were used to predict starch and dextran.

Pol (°Z)

The correlation between laboratory and NIRS values illustrated in Figure 1 for pol shows a good correlation coefficient of $R^2=0.89$. For process control the trend is adequate and reliable enough to make process decisions.

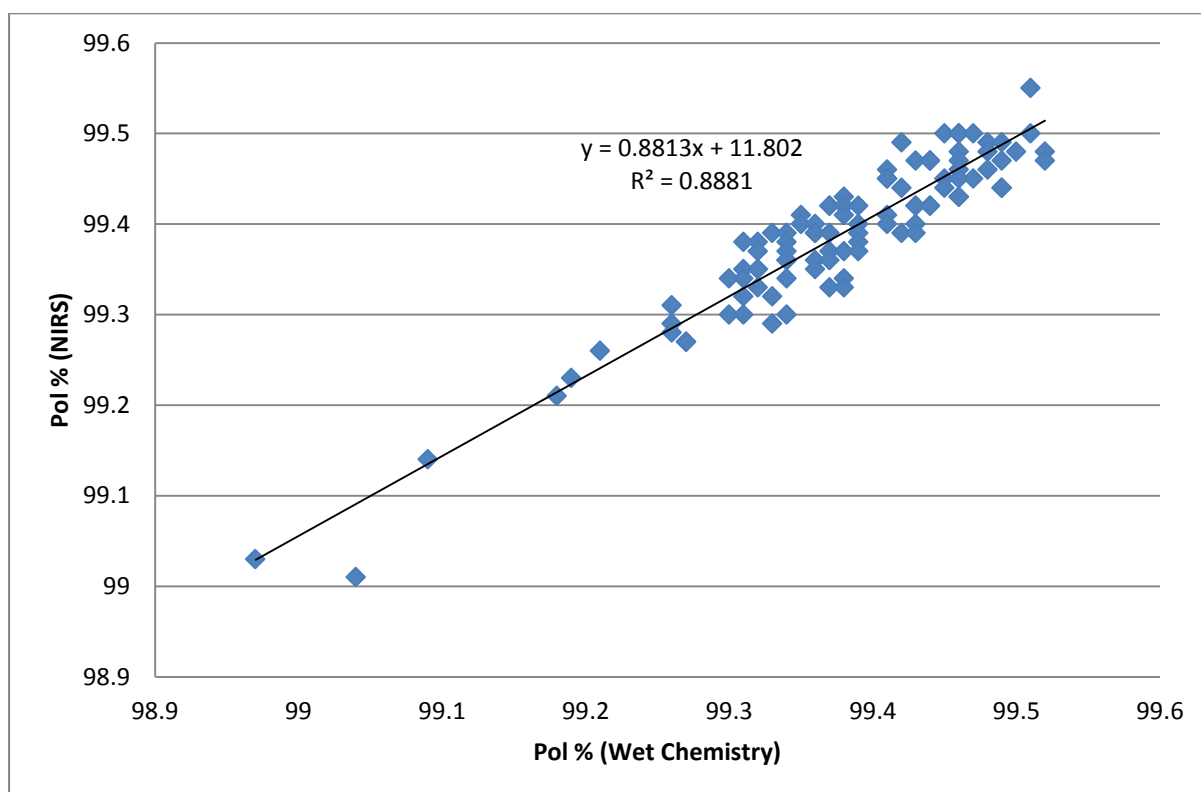


Figure 1. Wet chemistry and near-infrared spectroscopy (NIRS) correlation for very high pol (VHP) sugar (°Z).

Colour

The correlation between laboratory and NIRS values illustrated in Figure 2 for colour shows a good correlation coefficient of $R^2=0.89$. For process control the trend is adequate and reliable enough to make process decisions.

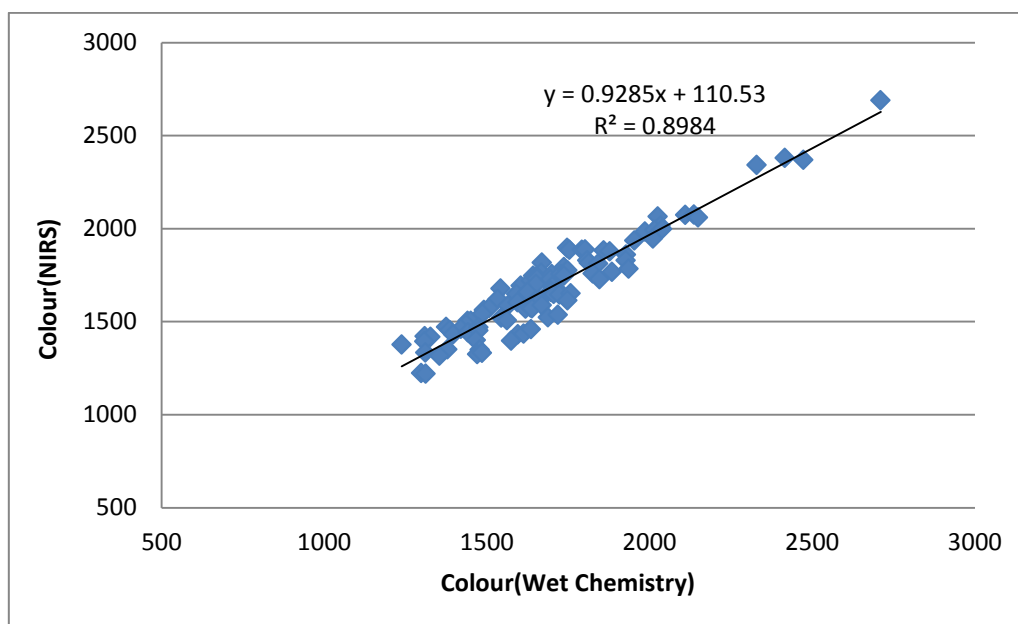


Figure 2. Wet chemistry and near-infrared spectroscopy (NIRS) correlation for very high pol sugar colour.

Moisture

The correlation between laboratory and NIRS values illustrated in Figure 3 for moisture shows a correlation coefficient of $R^2=0.53$. Although the correlation coefficient proved to be satisfactory the results were well within the repeatability tolerance of 0.04% for moisture. For process control the trend is adequate.

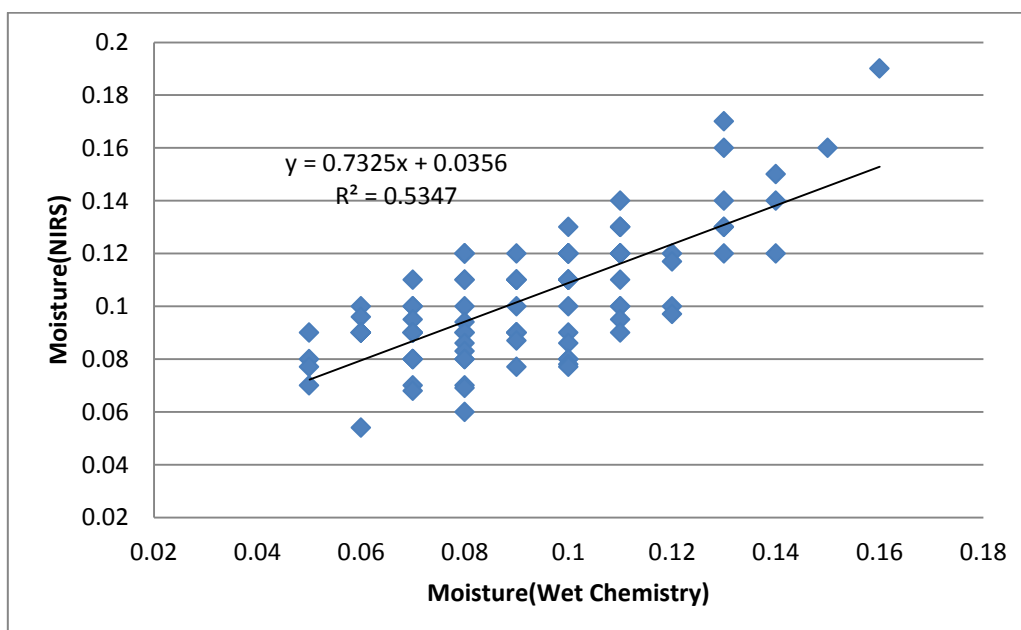


Figure 3. Wet chemistry and near-infrared spectroscopy (NIRS) correlation for very high pol sugar moisture.

Dextran

The correlation between laboratory and NIRS values illustrated in Figure 4 for dextran shows a correlation coefficient of $R^2=0.64$. This correlation coefficient is deemed satisfactory.

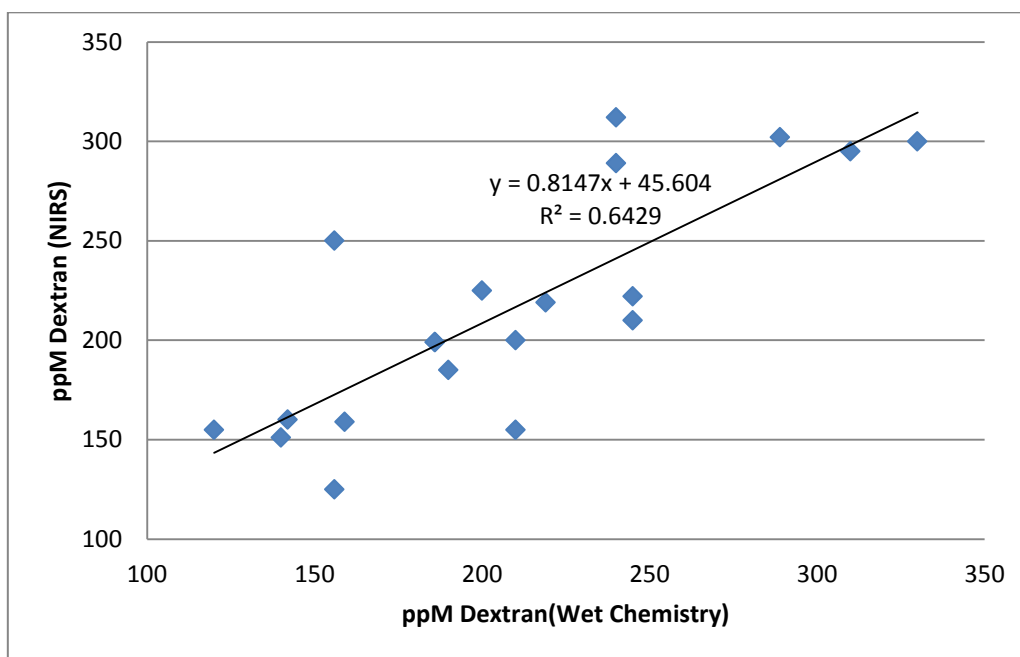


Figure 4. Wet chemistry and near-infrared spectroscopy (NIRS) correlation for very high pol sugar dextran.

Starch

The correlation between laboratory and NIRS values illustrated in Figure 5 for starch shows a correlation coefficient of $R^2=0.72$. This correlation coefficient is acceptable.

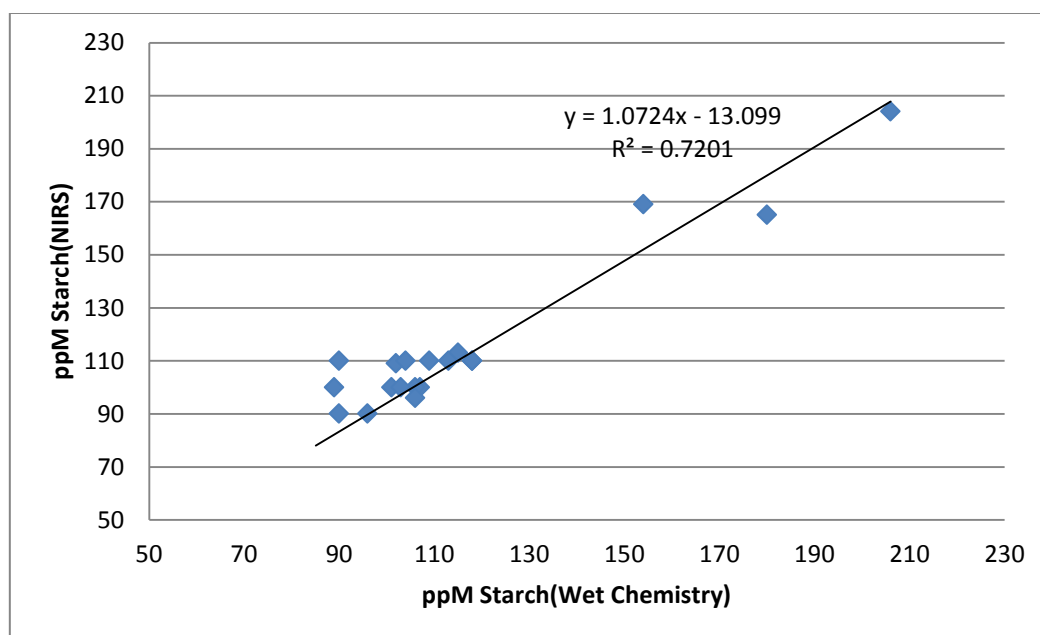


Figure 5. Wet chemistry and near-infrared spectroscopy (NIRS) correlation for very high pol sugar starch.

Table 1 is a brief summary of the overall results obtained.

Table 1. Summary of wet chemistry and near-infrared spectroscopy prediction results for very high pol (VHP) sugar.

VHP sugar	Pol	Colour	Moisture	Dextran	Starch
Sample count	100	100	100	20	20
Slope	1.00	0.97	0.73	0.79	0.67
R ²	0.89	0.89	0.53	0.64	0.72

Practical experience

Approximately midway into the 2012/13 season, marketing demands dictated that Sezela supply three sugar mills with good quality VHP sugar. At short notice temporary staff had to be sourced to complete this task. Severe rainfall events and a transport strike which impacted negatively on sugar quality complicated the distribution task.

The distribution method was set up as follows:

- Every vehicle to be sampled during loading.
- This sample to be analysed using the NIR MPA instrument.
- A certificate of analysis to be generated and handed to the driver, who in turn hands the certificate over to the customer.
- A sample to be retained for verification purposes, should this be necessary.

No customer complaints were received, except for one which was withdrawn after citing a problem in their laboratory. Customers were able to segregate sugar into different storage bins for varying quality requirements. In situations where the quality was border-line, staff were able to optimise distribution by consulting with the customer before diversion.

It must be emphasised that the simplicity of the instrument allowed relatively unskilled staff with minimal training to be employed. In addition, the rapid output of results allowed for ease of distribution. A high level of confidence was placed in the system.

Sezela was able to meet its distribution objectives successfully and with relatively few problems.

Conclusion

The NIRS MPA instrument enabled staff at the Sezela mill to despatch sugar to four different destinations with ease and confidence. The short analysis time and the ability to read multiple components proved most beneficial. There were no customer rejections. The comparative analytical data achieved thus far indicated a high level of reproducibility, and it is recommended that the system be further tested for online quality monitoring.

Acknowledgement

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REFERENCES

Anon (2005). SASTA test methods. SASTA Laboratory Manual, including the Official Methods, 4th Edition. Published by the South African Sugar Technologists' Association, Mount Edgecombe, South Africa.