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NIRS AS A TOOL FOR IMPROVED PROCESS MONITORING

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Abstract

The development of NIRS (Near Infrared Spectroscopy) for the rapid routine analysis of factory products has created an opportunity for factories to monitor the sugar making process more effectively. The use of NIRS allows the measurement of inversion loss and target purity difference (TPD) which traditionally was only calculated and reported at the end of the week, to be done at least once per shift. NIRS also allows a mill to monitor inversion loss across the clarifier and evaporator, which previously was done by the Sugar Milling Research Institute in a consulting capacity. NIRS thus enables a rapid identification of potential problems which could impact on undetermined loss. During the 2017 season at Maidstone sugar mill, NIRS was used to make direct estimates of TPD of C-molasses from c-centrifugals and inversion losses over the clarifier and evaporators. These measurements were compared with conventional weekly TPD and UDL figures respectively and good agreement was found.

Keywords: laboratory, NIRS, UDL, TPD, inversion, process control

Introduction

As far back as the 1990s, De Bruijn (1997) demonstrated it was possible for Near Infrared Spectroscopy (NIRS) to be used for process control in the beet industry. Similarly, Schaffler *et al*. (1997) and Schaffler (2001) identified NIRS as an attractive process management tool for the South African cane industry. Despite the substantial advantages offered by NIRS, adoption by the industry has been a slow and steady process.

A significant amount of effort has been spent on determining both accuracy and precision of the method through the development of acceptable prediction equations. In this regard the South African cane industry has been fortunate in having the Sugar Milling Research Institute NPC (SMRI) to develop the equations for the industry, with the support of the Factory Control Advisory Committee (FCAC) (Walford et al (2015), (Simpson et al (2008)). By 2009, the FCAC approved the NIRS method for the analysis of sucrose, pol and brix in final molasses. In 2015, the FCAC recommended the use of NIRS for pol, brix and sucrose in the weekly composite mixed juice, for cane payment purposes (SMRI Method TM401). In addition, in 2015 the FCAC accepted the use of NIRS for factory control and performance reporting. A stipulated requirement is that the factories use the SMRI-NIRS prediction equations. The SMRI provided equations for:

- Mixed juice
- Clear juice
- Syrup
- A, B and C molasses
- A, B and C massecuite

The SMRI continues to maintain the prediction equations for the South African sugarcane industry. Currently the South African factories are in transition from the conventional analysis to adopting NIRS for routine analysis and process control.

In the development work the primary driver was to replace the expensive conventional analytical methods with analysis by NIRS (Simpson and Naidoo 2010). For this reason, the focus has been on pol and brix measurements, which were within the capacity of the factory to perform. TPD was currently only determined by the SMRI for each factory on a weekly basis, using a composite sample. Samples were analysed by the SMRI and results were only reported the following week. There was little detailed information on the individual centrifugals although factories could check purity rise on curing when required (Madho *et al* 2015). Similarly, inversion investigations were undertaken on individual unit operations only when the overall undetermined loss (UDL) was high. UDL was also only reported a week after the fact, based on the overall factory balance.

NIRS presented the factory operations with significantly more opportunity to understand and manage processes. NIRS allowed for the rapid analysis of many analytes from a single sample, including those, that by conventional methods, were prohibitively time consuming and expensive. Fructose, glucose and sucrose (FGS), dry solids and conductivity ash were included in this group. What this means in practice, is that factories could now quite easily calculate measures of performance such as inversion losses and target purity differences (TPD). This enabled factories to rapidly identify any problem areas. These measurements could be made on individual evaporators or centrifugals and/or on a daily or shift basis using NIRS. The Laboratory Information Management system (LIMS) allowed the results to be incorporated into regular and immediate reporting and this could allow timeous interventions when the measured performance was not within an acceptable range.

The work reported by Dairam *et al*. (2016) was a good example of using NIRS to investigate a specific UDL problem, leading to a rapid identification of the problem and a suitable strategy to deal with it.

With the availability of NIRS in all factories the SMRI has also developed a toolbox using NIRS to routinely determine inversion and TPD quickly and easily. This provides an opportunity to measure these performance parameters on a regular basis.

This paper reports on the use of NIRS to:

1. Determine inversion measurements across the evaporator and clarifier and as verification to compare to the trend of undetermined loss.
2. Determine TPD for the c-centrifugals at the Tongaat Hulett Maidstone mill during the 2017 season and compared to the SMRI results for TPD over the same period.

This was carried out with a view to improving process control by incorporating such measurements in the LIMS across all the Tongaat Hulett mills

Experimental

Maidstone factory was selected to test the feasibility of incorporating Bruker MPA NIRS for routine reporting of TPD and inversion during the 2017 season. The trial was run over an eight week period between weeks 21 and 28 of the season. The trials were carried out by laboratory staff with the assistance of students on experiential training. The staff involved were given training on the operation of the NIRS and the necessary sampling procedures. A Bruker NIRS was used for the trial which had been uploaded with the SMRI prediction equations.

Inversion: It is generally accepted that the loss of sucrose due to inversion across the clarification and evaporator station is well correlated with UDL (Dairam *et al.* 2016; van Hengel (1962). This excludes obvious physical losses due to spills or leaks which would not

need sophisticated analysis to identify. This measurement should allow a timely warning of possible UDL problems.

Samples of mixed juice, clear juice and syrup were taken several times daily and analysed by NIRS using the standard procedures (SMRI Toolkit Standard operating procedures 2015). From the fructose, glucose and sucrose results, the inversion was calculated, according to the following equation (Schaffler 2001)

$$\%\text{sucrose lost} = \frac{\left(\frac{\text{glucose}}{\text{Brix}}\right)_{out} - \left(\frac{\text{glucose}}{\text{Brix}}\right)_{in}}{\left(\frac{\text{sucrose}}{\text{Brix}}\right)_{in}} \times \frac{\text{Sucrose molecular weight}}{\text{Glucose molecular weight}} \times 100$$

This Formula assumes that there was no destruction of invert sugar during the process.

Target Purity Difference: Maidstone factory has five 'C' centrifugals. The standard operating procedure prescribed by the SMRI Toolkit was used for the trials. Samples were analysed for pol, brix, sucrose, conductivity ash and dry solids using NIRS.

The TPD was calculated using the method proposed by Smith (1995).

Where TPD = True purity – Target purity

and Target Purity = $43.1 - 17.5(1 - e^{(\frac{F+G}{A})})$

Where: F-Fructose % final molasses
G-Glucose % final molasses
A-Conductivity ash % final molasses.

and True purity = $\frac{Sucrose}{Dry\ solids}\ x\ 100.$

Results

Inversion

The daily results for the inversion calculations were averaged for the week using the (simple average) technique. The average weekly inversion loss for the clarifier and evaporator station is shown in Figure 1. This showed the bulk of the inversion took place in the evaporators as compared to the clarifier.

The sum of the weekly average results for the clarifier and evaporator losses allowed the results to be compared with the weekly undetermined loss reported by the SMRI. This is illustrated in Figure 2. It should be noted that two data points have been excluded. The first for week 21, when the UDL was exceptionally high due to other events, not of relevance to this study, and for week 23 when the UDL was not reported due to technical problems with the LIMS reporting system. Considering the very different ways in which the final weekly figures were calculated, the graph shows a reasonably good correlation. Thus, the data demonstrates that routine reporting of the inversion losses across the clarifier and evaporators, using NIRS, can give a rapid indication of potential undetermined loss problems.

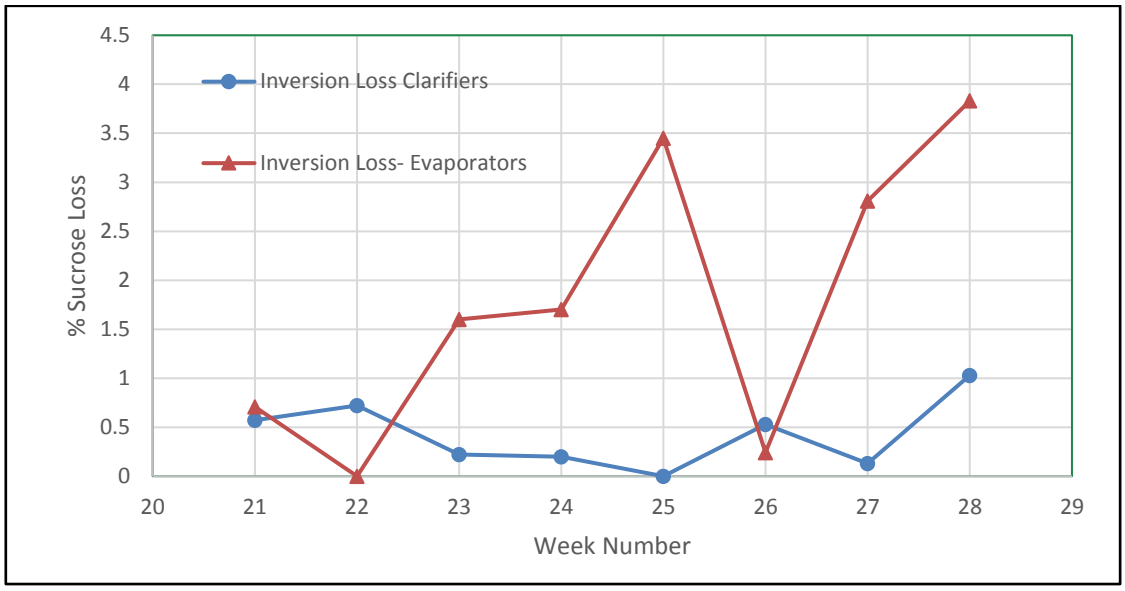

Figure 1. Average weekly inversion loss across the clarifier and evaporator.

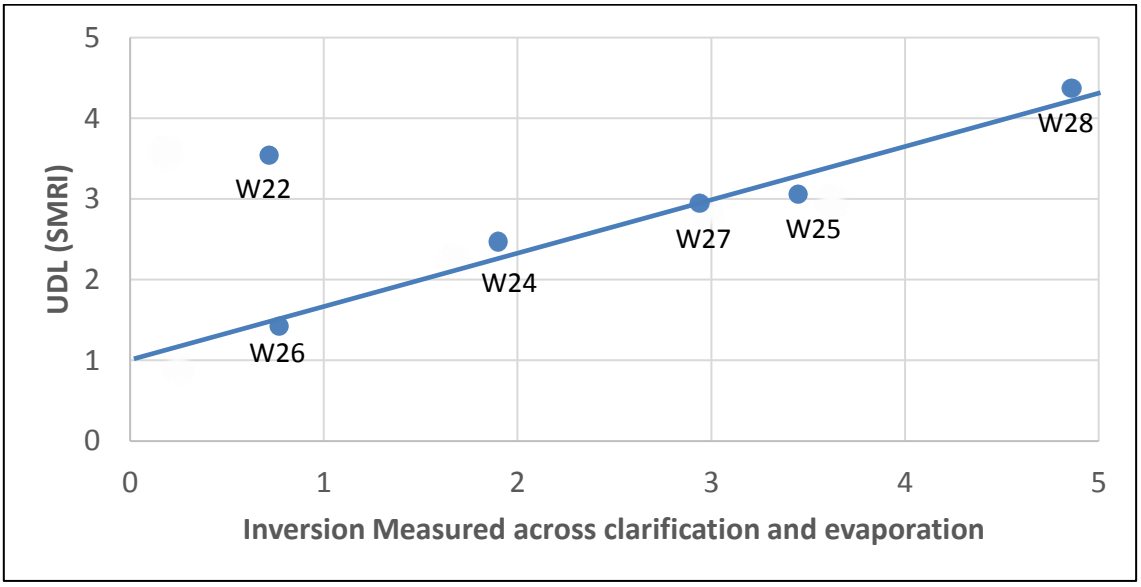

Figure 2. Undetermined loss reported by the Sugar Milling Research Institute compared with the sum of the weekly average inversion loss measured across clarification and evaporation.

Target purity difference

Samples of final molasses were taken from each of the centrifugals every two hours a day over the 8-week period from Tuesdays to Fridays. The TPD was calculated for each sample taken. All the data points collected are shown in Figure 3. While this shows general trends, it was difficult to distinguish between the different centrifugals. To make more sense of the data, the weekly average TPD was calculated from the daily data (Simple average). The

results are shown in Figure 4. This shows differences between the centrifugals and allows any poorly performing centrifugal to receive attention.

Finally the weekly average was calculated for all the centrifugals and compared with the SMRI reported TPD weekly figures for the weeks of interest. The result is shown in Figure 5 and shows good agreement. Currently the SMRI calculates the weekly TPD for a factory based on a composite molasses sample using NIRS so the good agreement should not be that surprising.

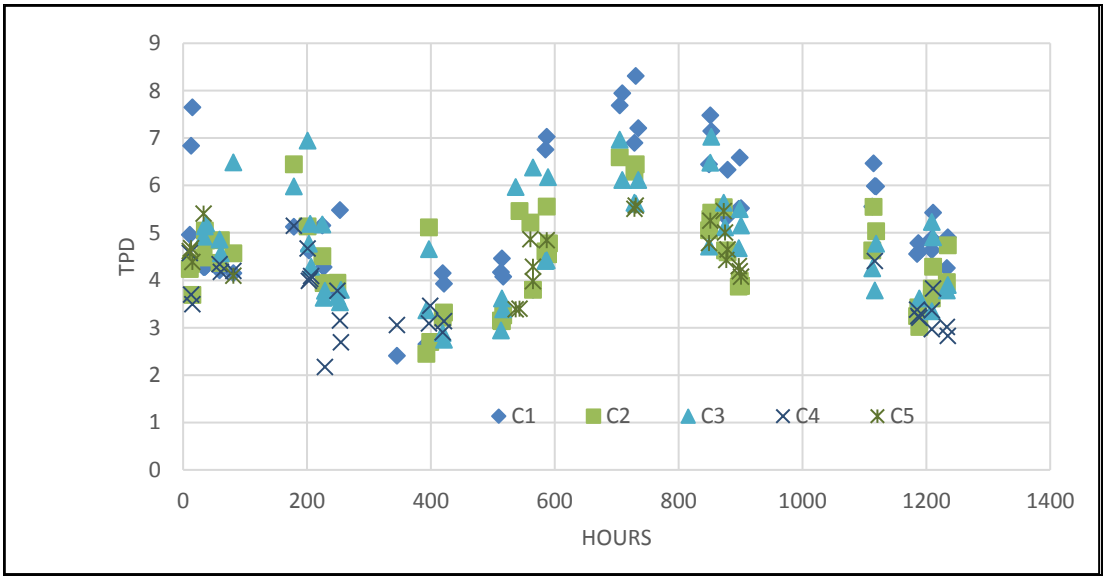

Figure 3. Target Purity Difference of all the C-centrifugals over the trial period.

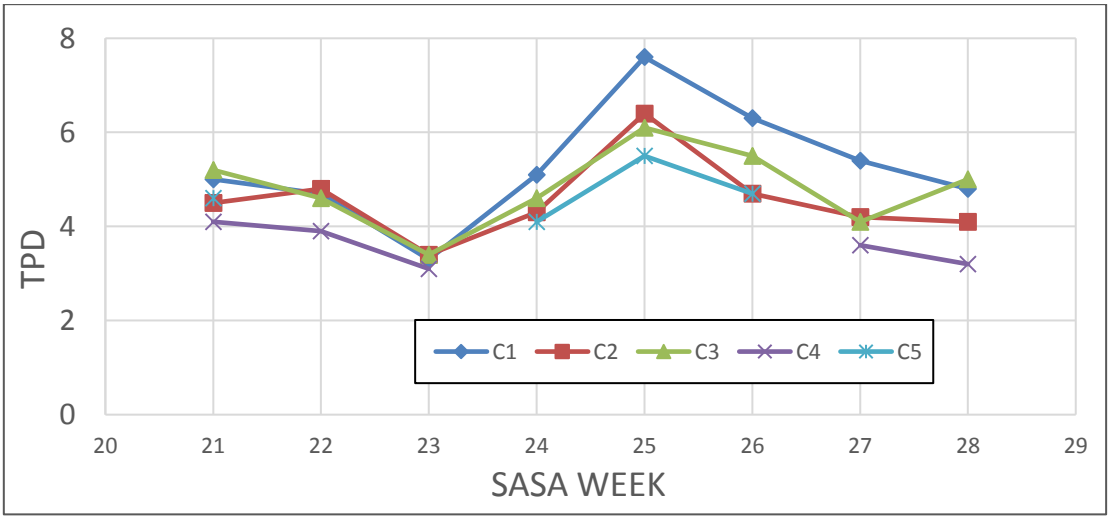

Figure 4. Weekly averaged Target Purity Difference figures for each centrifugal.

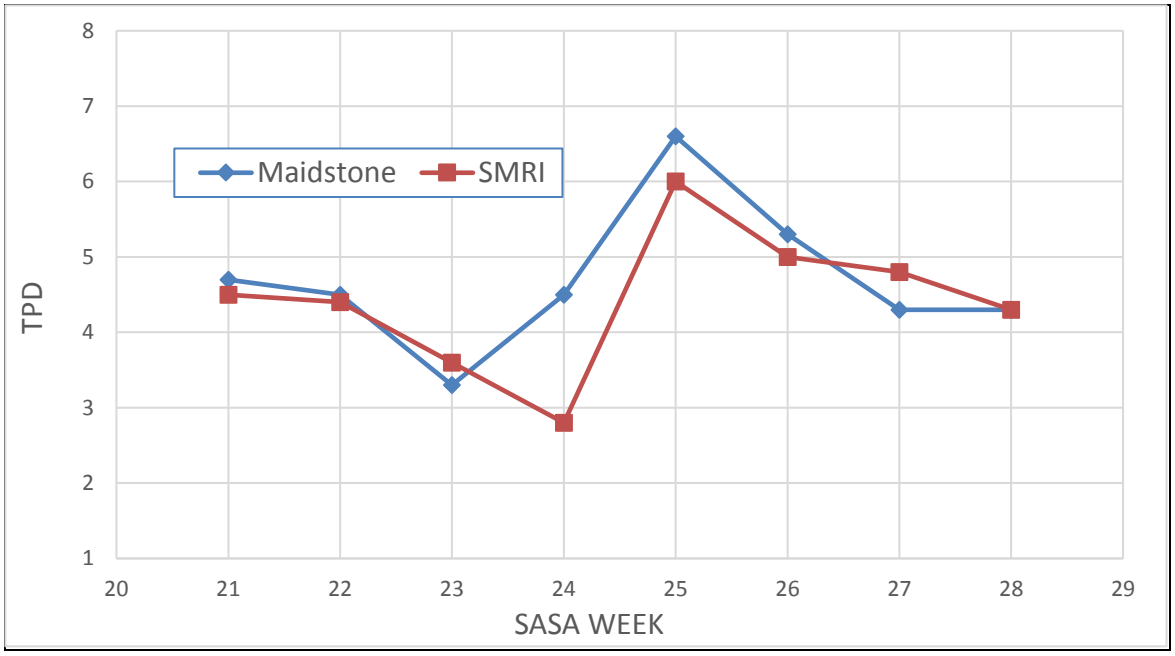

Figure 5. Target Purity Difference (TPD) measured by the Sugar Milling Research Institute compared with a weekly average TPD measured at Maidstone by near infrared spectroscopy.

Conclusions

NIRS not only simplified and reduced the cost of existing measurements required for process control but offered greater opportunity for process management with minimal added effort. NIRS allowed additional analysis to be performed by the factory laboratory staff in many cases without any extra sampling required (e.g. while measuring pol and brix in a factory sample, NIRS would also report fructose, glucose and sucrose).

This work has demonstrated that the NIRS technique, combined with the SMRI support system developed, makes it possible for factories to determine performance measures such as TPD and inversion losses on a routine basis. The trial results showed good agreement with the weekly SMRI reported figures. The results generated were easily incorporated into the LIMS which makes reporting and trending straightforward and rapid. Therefore the NIRS system would assist factory management in identifying potential problems earlier in the week and enable them to monitor and manage those situations more effectively thus allowing for improved process monitoring.

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