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# IMPLEMENTATION OF ON-LINE NEAR INFRARED (NIR) TECHNOLOGIES FOR THE ANALYSIS OF CANE, BAGASSE AND RAW SUGAR IN SUGAR FACTORIES TO IMPROVE PERFORMANCE

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#### Abstract

BSES Limited has a long history of developing and commercialising NIR technology for sugar industry applications spanning plant breeding, laboratory and online sugar factory use. This paper traces the development of online NIR systems with FOSS Pacific, for the real-time analysis of cane, bagasse and sugar, describing a range of applications currently applied within sugar factories. The Cane Analysis System (CAS) was developed primarily to measure real-time fibre content of a cane consignment for payment purposes. This enables payment on the overall quality of a consignment rather than making assumptions from average figures. CAS uses a FOSS Direct Light 5000 instrument, with ancillary systems developed for sample presentation, data analysis and integration with mill control systems. Further CAS applications for ash, dry matter, pol and brix in both cane and juice, as well as commercial cane sugar (CCS), were subsequently developed. Data from these calibrations have successfully been used to audit mill laboratories, derive cane quality indices, implement automated control strategies, develop alternative payment systems and provide data on individual farms/blocks, enabling targeted solutions for areas with productivity issues. Based on this platform, online bagasse (BAS) and sugar analysis systems (SAS) were developed. BAS measurements can provide quality-based assessments of bagasse suitable for feed-forward to boiler stations. When linked with a CAS on the same milling train, the effects of mill settings, maceration control and cane quality on pol extraction are determined in real-time, enabling the development of strategies to maximise extraction. SAS calibrations are used to monitor and control critical processes in the production of LoGIcane<sup>TM</sup>, the world's first low glycemic index sugar which was commercialised and released in Australian in 2009. The development of online NIR measurement systems for sugar factories has seen quality, payment and control strategies established which are not possible using normal laboratory data. Almost 30 systems have been installed worldwide, and further uses for online NIR data continue to be developed by existing users in conjunction with BSES and FOSS.

## Introduction

In recent times, near-infrared (NIR) spectroscopic instrumention has been developed for the rapid and reliable analytical determination of parameters of interest within petrochemical (Larrechi and Callao, 2003), beverage (Rodriguez-Saona *et al.*, 2001), food (Dodds and Heath, 2001) and pharmaceutical industries (Carlsson and Janne, 1995). Routine NIR analyses have significant advantages over traditional wet chemistry techniques, particularly in terms of labour and consumable costs, throughput, non-destructive sample preparation and analysis, potential generation of real time on-line results and reduced risk where hazardous materials or procedures are involved. These advantages refer specifically to the analysis itself, whereas further benefits can be obtained from applying the real time data obtained for process control, payment systems and the prediction of multiple constituents from single spectral measurements.

With respect to sugar industry applications, there are well-documented descriptions of at-line NIR spectroscopic techniques used for plant-breeding purposes (Berding *et al.*, 1989; Brotherton and Berding, 1995, 1998). These analyses were primarily performed on shredded or fibrated cane, and were followed by further reports providing data on other substrates derived from sugarcane and various factory streams including molasses and mixed juice (Brotherton and Berding, 1995; Schäffler *et al.*, 1993; Simpson and Oxley, 2008).

The successful plant breeding applications led to an expansion of research activities at BSES Limited, culminating in the development of on-line NIR systems for sugar mill applications. These applications have been developed in conjunction with FOSS as providers of NIR hardware, and have been commercially released as the Cane Analysis System (CAS) and the Sugar Analysis System (SAS). An analogous Bagasse Analysis System (BAS) is currently undergoing development prior to commercial release, although two prototype systems are operational at Mulgrave Central Mill (Cairns, Australia) and the Costa Pinto Mill (Brazil). This paper describes past and recent applications derived from these on-line NIR measurement systems.

## Materials and methods

## **Experimental**

Chemicals : Folin-Ciocalteu reagent and (+)-catechin standard were purchased from Sigma-Aldrich (St Louis, MO) with sodium carbonate obtained from Labserv (Melbourne, Australia). All chemicals used were analytical grade.

Polyphenol Analysis : 40 g of raw sugar sample was accurately weighed into a 100 mL volumetric flask. Approximately 40 mL of distilled water was added and the flask agitated until the sugar was fully dissolved after which the solution was made up to final volume with distilled water. The polyphenol analysis was based on the Folin-Ciocalteu method (Singleton and Rossi, 1965) using a protocol adapted from the work of Kim *et al.* (2003). In brief, a 50  $\mu$ L aliquot of appropriately diluted raw sugar solution was added to a test tube followed by 650  $\mu$ L of distilled water. A 50  $\mu$ L aliquot of Folin-Ciocalteu reagent was added to the mixture and shaken. After 5 minutes, 500  $\mu$ L of 7% Na<sub>2</sub>CO<sub>3</sub> solution was added with mixing. The absorbance at 750 nm was recorded after 90 minutes at room temperature. A standard curve was constructed using standard solutions of catechin (0-250 mg/L). Sample results were expressed as milligrams of catechin equivalent (CE) per 100 g raw sugar.

Analytical results for pol, brix, ash, fibre, moisture were generated using standard methods within the Australian sugar industry (BSES, 1991). Bagasse samples were analysed for lignin content according to the methods published by the US National Renewable Energy Laboratory (2008).

## **System Description**

On-line NIR systems were developed based on the NIRSystems 5000 scanning monochromators with direct light attachment and the Infrasoft International (ISI) chemometrics package. The attached scanning head is sealed against the mill chute for both CAS and BAS installations while, for the SAS configuration, it is mounted above the conveyor belt which transports raw sugar from the dryer. For all configurations, the substrate is scanned through a heat-treated toughened glass window mounted within a stainless steel housing. Vibration dampening systems are installed within both the scanning head unit and the NIR instrument cabinet, which is also fitted with an airconditioning system and an uninterruptible power supply. The system is integrated with the mill payment and control computers, and software was developed to process relevant mill signals, control scanning equipment and to distribute data to the appropriate mill systems. This software also interfaces with mill tracking systems to ensure that individual consignments of cane are recognised, and is capable of registering various errors, alarms and internal checks to provide result integrity.

An important feature of these systems is their setup within a secure networked environment. In this way, online diagnostics, system checks, uploading of new and/or upgraded calibration equations and data storage can be handled by remote operators either over a local/wide area network or via the internet. Methods developed for sampling, analysis, NIR scanning, equation development, equation validation and the evaluation of system performance have been described previously (Staunton *et al.*, 1999).

# Cane Analysis System (CAS)

The CAS system has been well described previously and many applications of CAS data have been reported. It was originally envisaged primarily as a means of determining the fibre content of individual consignments of cane supplied to the mill, thereby providing a way of moving away from class fibre systems or payment methods in which cane fibre values were averaged or given assumed values. Upon the development of the system, it became rapidly apparent that the ability to provide real time data provided strong incentives to examine additional options, develop useful calibrations and apply them to gain direct benefit in both the mill and the field.

In particular, CAS data have been used to develop online cane analysis systems for fibre analyses (Staunton *et al.*, 1999), payment purposes (Staunton *et al.*, 2004), canequality schemes (Pope *et al.*, 2004), and for process control purposes using fibre rate control (Jones *et al.*, 2002). There are currently 23 CAS installations worldwide supporting these applications within sugar mills.

## Sugar Analysis System (SAS)

An earlier report outlined the initial development of the SAS system (Bevin *et al.*, 2002), where an instrument configuration similar to that developed for CAS was used to measure various sugar parameters within sugar mills and at bulk sugar terminals. This report also outlined control charting techniques and comparisons of NIR versus laboratory analyses. For mill applications, the SAS sampling head is mounted over a sugar conveyor belt (Figure 1). The plough shown on the right hand side of the setup has been designed to maintain a particular depth of sugar on the belt as it passes under the scanner from right to left, thereby minimising data scatter and error. A description follows of the development of specific SAS equations for process control and quality monitoring of a low glycemic index (GI) sugar product produced at Mossman Central Mill during the 2008 crushing season.



Fig. 1 – Typical mounting system for the SAS scanning head over a sugar conveyor belt

LoGiCane<sup>TM</sup> is the world's first all natural low GI cane sugar product and was developed in Australia by Horizon Science. It was produced on a large scale for the first time during the 2008 season at Mossman Central Mill and was officially launched in March 2009 (Burke, 2009). LoGiCane<sup>TM</sup> has been independently tested and certified by the Glycemic Index Foundation in Australia with a low GI of 50, whereas white sugar has a medium to high GI of 65. Low GI foods provide a health benefit by producing gradual increases in blood glucose and insulin levels following consumption, thereby having implications for weight control and diet choice for people with type 1 and type 2 diabetes. The GI of LoGiCane<sup>TM</sup> is reduced due to the presence of various natural components of sugarcane, particularly polyphenols and minerals, which have been added back during the production process.

Assays for these components are vital to characterise polyphenol and mineral levels across the production process and within the final product. In conjunction with Horizon Science and Mossman Central Mill, BSES developed an online process control solution for LoGiCane<sup>TM</sup> production using SAS technology. The SAS instrument installed not only measures traditional sugar parameters such as pol, moisture, ash, colour and reducing sugars, but also provides data on the concentrations of polyphenols, minerals and a measure of antioxidant capacity. These critical measurements provide instant process feedback, enabling the development of process control solutions based on the SAS data, as well as systems to ensure product consistency.

Calibration and validation plots for the prediction of total phenolics in sugar are shown in Figures 2 and 3. The calibration results were very good and included spectral results from two separate laboratory instruments. When applied to the SAS instrument, the validation results contained some bias, scatter and skew; however, the results proved to be adequate for process control and would improve with further development. Importantly, the calibration developed contains no data from the online SAS instrument. The validation results are demonstrating effective calibration transfer even at this early stage of development. As this development process continues (viz. inclusion of on-line SAS data and instrument variation in the global calibration), significant improvements in equation performance are expected.



Fig. 2 – Calibration plot for total phenolics in sugar at Mossman Central Mill. Phenolic units are mg catechin equivalents per 100 g sugar



Fig. 3 – Validation plot for total phenolics in sugar at Mossman Central Mill. Phenolic units are mg catechin equivalents per 100 g sugar

## **Bagasse Analysis System (BAS)**

The development of BAS instrumentation has been described previously (Staunton and Wardrop, 2006) and the system will be available commercially in the near future. The previous report described in some detail the system development, sampling, analysis and preliminary factory applications for calorific value estimation of bagasse to aid boiler operation as well as the determination of online pol extraction across the

milling train where both CAS and BAS installations are present. Here data are presented for online pol extraction from the 2008 crushing season obtained from the two prototype BAS installations at Mulgrave Central Mill (Australia) and Costa Pinto Mill (Brazil), where both milling trains also have CAS installations at the front end. Early developmental work on an online lignin determination on bagasse is also presented.

## **Online Pol Extraction**

The Mulgrave Central Mill BAS installation is currently in its fifth year of continuous operation, having been installed during the 2005 season. In order to develop a measure for online pol extraction, appropriate calibration equations and software were developed to calculate pol extraction using real time data from the mill tracking systems and the CAS and BAS units and applying the following formula which assumes no loss in fibre (Staunton and Wardrop, 2006).

$$Polextraction = 100 * \left(1 - \frac{Pol_b * Fibre_c}{Fibre_b * Pol_c}\right)$$
  
where: c = cane, b = bagasse

The 2008 crushing season laboratory validation statistics for a selection of CAS and BAS calibrations are shown in Table 1. The low levels of bias observed and the fact that standard errors of prediction (SEP) are lower than the equation error control limits (ECL) show that the CAS calibrations performed close to or within expectations in terms of analysis accuracy and precision. The BAS validation statistics show relatively poor slope and coefficients of determination due to the relatively high SEPs, combined with restricted ranges in parameter values. This is especially true for pol%bagasse and is due, in part, to the difficulties involved in collecting representative samples of bagasse that span a sufficient range of pol%bagasse values.

CAS Parameter	SEP	Error Control Limit	Slope	Bias	R <sup>2</sup>	Range	Ν
Pol%Juice	0.32	0.38	0.94	0.017	0.97	11.9-23.9	11 312
Brix%Juice	0.30	0.38	0.93	0.009	0.97	15.2-26.2	11 312
Direct CCS	0.32	0.32	0.95	0.018	0.95	8.0-18.0	11 312
Fibre%Cane	0.69	0.66	0.73	-0.12	0.80	11.4-22.4	588
BAS Parameter	SEP	Error Control Limit	Slope	Bias	R <sup>2</sup>	Range	Ν
Dry Matter%Bagasse	1.04	1.07	0.64	0.04	0.64	47.5-56.2	160
Moisture%Bagasse	1.04	1.07	0.64	-0.04	0.64	43.8-52.5	160

 Table 1 – Calibration equations and validation statistics for Mulgrave Central Mill CAS and BAS installations

Fibre%Bagasse	1.19	1.13	0.6	-0.11	0.61	43.2-54.1	160
Ash%Bagasse	1.09	1.48	1.00	-0.04	0.65	0.8-9.0	160
Pol%Bagasse	0.24	0.22	0.28	-0.02	0.31	0.7-2.0	160

The pol extraction validation results are shown in Figure 4. Both the slope and the correlation coefficient for the linear regression were considered to have utility, especially when considering the relatively small population size, the narrow range of values for the extraction data and the relatively high standard errors of prediction reported for BAS constituents. Even with these limitations, online pol extraction by NIR represents a significant advance in the monitoring of milling train performance and has the potential to advance the understanding of the key parameters affecting extraction, leading to better process control and more consistent extraction outcomes.



Fig. 4 – Validation plot for online pol extraction at Mulgrave Central Mill

A typical 24 hour trend for calculated online pol extraction is shown in Figure 5. This trend shows that, across this period, relatively constant pol extraction of around 97% was achieved. However, the graph does show particular examples where extraction has dropped by up to 2-3% before returning to the average extraction value. These variations are attributed to a step decrease in cane quality (with no or little change in pol%bagasse) and the return of the extraction value back to the average reflects a return to better quality cane in the following consignment.



Fig. 5 – Typical online pol extraction trend for a continuous 24 hour period at Mulgrave Central Mill

Currently, Mulgrave Central Mill uses NIR determined prepared cane pol to fibre ratio to determine milling train maceration levels, where the maceration%cane fibre is varied within a defined operating range depending on the measured quality of incoming cane. This is an example of a feed forward control system, whereas the application of real time online pol extraction data provides a potential feed backward maceration control system.

In an analogous fashion, online pol extraction predictions were developed and assessed at the Costa Pinto Mill during the 2008 season. Calibration statistics for the CAS and BAS equations are shown in Table 2. There were a number of significant challenges to overcome in order to replicate the system that had been developed in Australia at Mulgrave Central Mill. In particular, the equations used on the CAS and BAS systems were initially developed and constructed using Australian methods and data. As expected, Costa Pinto Mill uses slightly different analytical procedures and sampling methods, which by definition will lead to slightly poorer agreement with the NIR data (i.e. higher SEP values). The ECLs have been calculated from Australian data which include processes such as automated juice sampling and fully audited laboratory methods. The corresponding Costa Pinto data were generated by manual sampling and, as such, an increased sampling error and therefore increased ECL was expected. The magnitude of acceptable ECL errors has not yet been determined and will require more data and larger populations of cane scanned in order to better represent them.

 Table 2 – Calibration equations and performance statistics for Costa Pinto Mill CAS and BAS installations

CAS Parameter	SEP	Error Control Limit	Slope	Bias	R <sup>2</sup>	Range	Ν
Pol%Juice	0.45	0.38	0.89	-0.15	0.78	12.3-20.5	330
Pol%Cane	0.41	0.32	0.86	-0.09	0.75	10.6-17.0	330
Brix%Juice	0.43	0.38	0.84	-0.12	0.78	15.8-23.0	330
Brix%Cane	0.39	0.32	0.81	-0.07	0.75	13.5-18.9	330
BAS Parameter	SEP	Error Control Limit	Slope	Bias	R <sup>2</sup>	Range	N
Moisture%Bagasse	0.75	1.07	0.80	0.01	0.74	44.8-52.0	145
Pol%Bagasse	0.22	0.22	0.79	0.02	0.65	1.5-3.8	145
Fibre%Bagasse	0.83	1.13	0.65	0.04	0.76	44.3-52.4	145

Further difficulties in drawing direct conclusions between the two installations are due to the differences in tracking systems employed. The tracking system is a critical element of online pol extraction determination since any measure of extraction requires the analysis of cane and bagasse from the same input cane. This cannot occur simultaneously due to the time lag involved along the milling train, but this time lag can be determined approximately by monitoring the progress of dye through the milling train. The measured time delay is used to synchronise CAS and BAS response for the same cane parcel. At Mulgrave Central Mill, cane is delivered to the mill in individual consignments which can be traced back to individual farms and blocks. As such, there is little difference in cane quality across several measurements within single consignments. Conversely, at Costa Pinto, the cane being crushed may have been sourced from several farms/blocks and mixed prior to milling, thereby increasing the variability in the pol extraction results from consecutive measurements.

In order to validate the online pol extraction measurement, a set of 24 samples was collected for complete cane and bagasse analysis (Figure 6). These samples were identified using a BSES tracking system (NCS Tracking) from CAS to BAS. The results were very good with 95% of NIR data appearing within 1 unit of the lab extraction value. This level of accuracy is more than adequate for application across process monitoring and control functions. Further, the validation results provide confidence that the variation shown within a typical 24 hour trend for NIR calculated pol extraction is real (Figure 7).



Costa Pinto Mill, 2008: NIR Calculated Milling Train Pol Extraction Validation Plot

Fig. 6 - Validation plot for online pol extraction at Costa Pinto Mill



Costa Pinto Mill, 2009 Online 24 Hour Milling Train Pol Extraction Trend (20 second scans)

Fig. 7 – Typical online pol extraction trend for a continuous 24 hour period at Costa Pinto Mill

Despite the fact that these results have not been generated across large numbers of samples, there are important trends that are apparent. The results for NIR calculated online pol extraction are very similar at both sites, demonstrating that the technique is applicable across any milling train and is a robust measurement despite existing differences between the mills and their milling trains. Based on the similarities observed, we conclude that the measurement itself does not appear to be adversely affected by variations in cane variety, harvesting methods or the mill preparation of shredded cane. Further, the authors expect that similar measurements should be applicable to any milling or diffusion extraction process, providing the ability to generate real time pol extraction. Access to this data will allow a mill to better understand pol extraction trends and to determine various optimised mill settings and maceration strategies which importantly can be altered in real time, in response to variations in the incoming cane supply and the observed extraction trends.

## **Online Lignin Determination**

Since BAS installations must be located at the end of the milling train, they are ideally located for the real time analysis of bagasse which could be potentially employed for value adding processes other than as a boiler fuel. Bagasse has potential feedstock applications for many downstream applications including lignocellulosic applications, furfural production, lignin applications (Doherty *et al.*, 2008) and others (Banerjee and Panday (2002). With this in mind, it was decided to explore the potential of BAS installations to serve as fibre or biomass analysers for parameters of interest to other processes. The starting point for this examination was the determination of lignin content, as it was envisaged that these data could have applications within lignocellulosic processing as well as impacting on the quality of bagasse as a boiler fuel for energy and cogeneration processes.

In 2006, eleven bagasse samples were sampled from Mulgrave Central Mill, analysed for lignin content and matched with existing NIR scans from the BAS instrument. They were further characterised using a FOSS XDS laboratory NIR instrument at BSES Indooroopilly. A further 21 bagasse samples were characterised by laboratory NIR spectra only. The NIR scans from both instruments were pooled to develop an initial calibration. This was an admittedly small data set which was potentially prone to overfitting, but was considered sufficient to generate a calibration which could provide indicative trends but not high quality quantitative data.

**Table 3** – Calibration equation and performance statistics for the lignin equation builtfrom Mulgrave Central Mill bagasse samples

Parameter	SEC	Error Control Limit	Mean	Std Dev	R <sup>2</sup>	Ν	Range
Lignin % Dry Matter	0.09	0.11	16.46	2.56	0.99	43	10.60-18.75

This equation was used without validation and, as such, the precision and accuracy of the predictive equation are unknown. This renders the data generated as qualitative only and capable of indicating trends rather than providing quantitative data. Figure 8 shows the equation applied to the Mulgrave Central Mill BAS installation within a 24 hour period during 2009, and is representative of the variation observed during any such period. The online trend indicates considerable lignin variation within the bagasse, and that there are observable step changes in lignin content occurring between cane consignments. Additionally, there is good clustering of lignin results within individual consignments and good sample definition. These are encouraging results and will be further developed, along with other equations for potential online biomass analysis.



Fig. 8 – Typical online lignin content trend for a continuous 24 hour period at Mulgrave Central Mill

The relative trends in lignin content were examined by determining the unweighted arithmetic mean lignin value for all data observed, and dividing the individual values obtained by this value to produce a range of mean centred results. Using this calculation, a value of 1.0 indicates a result which was the same as the overall mean.

The cane supply at Mulgrave Central Mill is tracked from the field to the factory, thereby allowing the allocation of each bagasse scan to individual farms, blocks and varieties. This permits the analysis of lignin content across multiple varieties, crop classes and environmental situations, as well as permitting cross-correlations with productivity data. When applied to a continuous 2 week operation at Mulgrave Central Mill which covered some 1600 individual cane consignments during the early part of the 2009 season, an indication was gained of the variation in lignin content across cane variety and crop class.

Figure 9 shows the mean centred lignin content data for this period, and also indicates the tonnages of each variety crushed and the relative standard deviation for each of the variety averages. This shows that, across all varieties, the total variation was +/-20% from the mean. The variation within varieties was approximately half of that and was of a similar magnitude for most varieties, irrespective of the tonnages processed. This tends to indicate that there are meaningful differences between lignin content for Australian commercial sugarcane varieties, and that the variation might be enough to warrant consideration of the intended use for the bagasse. Further, such data allow the mill to more accurately value bagasse for different downstream options. One possible scenario might see bagasse of higher lignin content deliberately used as boiler feed material, with lower lignin content material diverted to lignocellulosic digestion or fermentation systems where reduced lignin content may reduce process difficulties. In this way, online NIR data would be used to direct bagasse in a way which would maximise benefits to the sugar mill.



Fig. 9 – Mean centred lignin content data for a continuous 14 day period at Mulgrave Central Mill, showing the variation across commercial varieties scanned

The ability to determine lignin content by NIR methods also has implications for plant breeding processes, especially with increasing interest in the development of energy canes for both cogeneration and for lignocellulosic biofuel/chemical production. Such methods could be applied at earlier stages of selection to identify varieties with desirable cellulose and lignin contents that are suitable for application as energy canes. More work is needed to improve the performance of this NIR calibration, but initial results are promising.

## Conclusion

Online NIR systems have been developed in different configurations for the analysis of cane, sugar and bagasse process streams. They offer tremendous advantages for the continuous, real time provision of analytical data which can be used for quality, process control and payment situations. These data are backed up by laboratory analysis in order to continuously validate and track NIR performance. Recently developed applications of NIR methods have been described for the prediction of sugar and bagasse parameters which can provide significant benefits to the sugar mill.

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