Environmentally friendly methods for raw sugar analysis.

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Abstract

The International Commission for the Unified Methods of Sugar Analysis (ICUMSA) has included in its latest manual a clarifier-free method for pol that employs NIR polarimetry and also a method for ash determination that uses conductivity measurements. These two methods are now used routinely by the Sugar Industry Research Institute in the analysis of raw sugars obtained from the seven sugar factories operating in Jamaica.

This paper outlines comparative results obtained using the ICUMSA methods GS 1/2/3-1 employing wet lead and GS 1/2/3-2 using NIR polarimetry. The results for % ash obtained using the gravimetric method compared to those derived from conductivity measurements are also presented.

Introduction

In the sugar industry, lead subacetate has been used as the clarifying agent in pol analysis of cane juice, raw sugar and other factory products for over 160 years (Schoones, 2004). With growing environmental awareness it became necessary to find methods of determining pol that were environmentally friendly. Two approaches were followed: one was to identify non-toxic substitutes for lead sub-acetate (Clarke, 1989; Clarke, 1990; Dunsmore, 1993; Mustafa, 1995; Peart and Byfield, 1991) and the other to determine if polarisation without clarification was possible. With the advent of NIR technology, the latter approach became possible since at 880 nm solutions of high colour could be read (Altenburg and Chou 1991 and 1993; Homer, 1998; Wilson, 1996). Subsequently, several sugar industries have made the change to NIR polarimetry and for the 2003/4 crop, South African sugar industry started using this method (Schoones, 2004).

Ash of sugarcane juice and its products comprises the soluble inorganic salts present in the juice (Chen, 1993). It is estimated by various procedures involving burning off the carbon in samples at temperatures in the range 500 – 800°C (gravimetric) or by conductometric methods as approved by ICUMSA. The conductivity ash gives a measure of the concentration of ionised soluble salt present and the gravimetric/sulphated ash gives the sum of water-soluble and water-insoluble ash. Studies have shown that conductivity ash of sugar and sugar products is highly correlated with ash determined by gravimetric methods (Mullins, 1986). The analysis of
sugar products for ash by conductometric methods is based on the fact that deionised water is an exceedingly poor conductor of electricity and any soluble mineral impurities found in sugar dissociate in water into electrically charged ions which conduct electricity thereby increasing the conductivity of the aqueous solution.

**Methods**

The Amstar Contract Form 2021-91G, issued by Domino Sugars, USA, is the guide that is used by the Jamaica sugar industry and states the specifications for raw sugars and the methods to be used in the analyses. In recent times, the two methods discussed in this paper have been made official. Sugar exported from Jamaica is expected to meet or exceed the minimum specifications set out in the Amstar contract. The first amendment, dated March 5, 1993 speaks to pol analysis and outlines a method employing NIR technology. The second, dated May 22, 2003 outlines ash determinations by conductivity measurements.

These two methods are approved by ICUMSA, the body that oversees methods of sugar analysis. The laboratory at the Sugar Industry Research Institute, acquired an NIR polarimeter in 1997 and a conductivity meter suitable for ash determination in 2000.

**Results**

A total of 842 raw sugar samples were analysed during the 2001 crop using ICUMSA lead and NIR methods. The analyses were done concurrently. The mean pol for the leaded and lead free solutions was similar. The average difference obtained was 0.32 with NIR values higher than leaded values. A correlation of 0.78 was observed (Fig 1).

![Fig 1 Plot of Lead vs NIR pol for sugar 2001 crop](image1)
For the 2004 crop, test solutions were treated with ‘filter cel’ to reduce turbidity and this resulted in much closer correlation, 0.99 (Fig 2), between the two methods. Turbidity free filtrates were obtained for all samples prepared for NIR pol analyses.

![Fig 2 Plot of Lead vs NIR pol for sugar 2004 crop](image)

\[
y = 1.0324x - 3.1836 \\
R^2 = 0.9876
\]

Results obtained using 100 raw sugar samples to assess the conductivity method and to compare values obtained with those from the gravimetric method showed that conductivity values were generally 0.02 units higher than gravimetric values. A correlation of 0.92 was obtained (Fig 3).

![Fig 3 Plot of Gravimetric Ash vs Conductivity Ash](image)

\[
y = 0.9327x + 0.0251 \\
R^2 = 0.922
\]
Discussion

The two new methods reported are environmentally friendly and efficient. New environmental regulations seek to discontinue the use of lead subacetate in Jamaica and so alternative methods available to the sugar industry are needed. The earlier studies conducted in 2001 used celite 505 as the filter aid. Many of the filtrates obtained then were turbid and sometimes too cloudy to be read on the NIR polarimeter. The results indicated differences in polarization by the two methods for the same sugar sample as great as 0.5 units.

The change to NIR for pol analyses of sugars and conductivity measurements for ash determinations by the Sugar Industry Research Institute allows for more easy comparison of results to those received on out turn reports. The out turn results for pol and ash of the last two shipment of sugar from Jamaica when compared to the results obtained at the Sugar Industry Research Institute were excellent; average difference for pol was 0.06 and for ash 0.03. The change also allowed the Laboratory to meet one of its mandates to reducing the use of toxic chemicals in the Jamaica sugar industry.

Generally, values obtained by NIR are higher. Although dark samples are measurable by NIR, Homer experienced some difficulty in reading a number of dark samples. The results indicated that non-sucrose constituents impact more on the polarization values obtained by NIR. This author experienced similar difficulty in reading several dark samples and noted that in those cases the filtrates were cloudy. Evidently, samples must be free from turbidity (Trott, 1994). Altenburg investigated the impact of filter aid and filter paper on the results and general performance of NIR and found that the filter cel grade of filter aid was ideal (Altenburg 1993). The use of filter cel significantly reduced turbidity levels in resulting in better correlation between the two sets of data (Fig 2).

Studies showed that an increase in dextran as determined by the Haze method resulted in an increase in pol by NIR over that from ICUMSA lead method (Wilson 1996). Homer observed a higher correlation between 589 nm and NIR pol measurements for low colour samples compared to high colour samples (Homer, 1998). This could explain why low correlation between the two methods was sometimes observed for sugars that were relatively low in dextran but high in colour. Differences as great as 0.5 unit and higher were noted in samples high in colour and dextran. Additional work needs to be done to fully assess the exact impact of various constituents on the results obtained by NIR (Wilson 1996).

Just as there are positives from this method using NIR, there are negatives as well. The increase in pol of raw sugar has a direct and dramatic financial impact and with the climate in which we now operate with so many restrictions and stipulations and the extremely competitive marketplace for sugar, there is little room on the bottom line for unrecoverable costs. Since the reporting of raw sugar pol has now increased without a corresponding increase in the sucrose content of the final product, there is a built in loss factor that cannot be reduced.
In most instances, the conductivity and gravimetric ash values are exactly the same or extremely close. However it has been noticed that on several occasions in the case of one factory and occasionally one or two other factories, the conductivity ash has been as much as 0.1 unit higher. Usually these samples are high in colour. Further studies are necessary to assess the impact of polysaccharides and colourants on ash by conductometric measurements. In terms of premium/penalty the value for ash is much less than that for pol, but penalties however small can still impact on the bottom line.

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