Comparative analysis of filter papers used in the sugar industry

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Comparative analysis of filter papers used in the sugar industry

The International Commission for Uniform Methods of Sugar Analysis (ICUMSA) is an organization concerned with developing international standards for analytical testing of sugar. These methods provide a foundation for assessing the overall quality of sugar. Filtration is a key sample preparation step in many standard methods that measure the purity of raw and refined sugar. Efficient filtration of juices extracted from cane or beet prior to polarimetry is essential for accurate measurement of sucrose content. Analysis of a filtered sample's color and turbidity enable assessment of contamination levels in refined sugar. Sucrose concentration and refined purity ultimately play a role in determining the price of sugar.

In this study, three commonly used filter paper grades were evaluated for speed and reliability in sample preparation for polarimetry measurements according to ICUMSA GS 1/2/3/9-1. These tests were performed using samples of sugar from Brazil, Zimbabwe, and Guyana. A different set of three commonly used filter papers was evaluated for efficiency in preparing samples for color and turbidity measurements according to ICUMSA GS 1/3-7 and GS 2/3-18, respectively. These tests were performed using samples of Brazilian sugar. All tested papers provided highly repeatable polarization data. However, Whatman™ Grade 91 was shown to provide a much faster filtration time compared with the other papers ($p < 0.001$). Filtration with Whatman Grade 5 paper also resulted in statistically significant ($p < 0.001$) superior turbidity reduction when compared to other grades.

Introduction

Standardized methods for sugar analysis

Founded in 1897, the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) is an international standards body that publishes laboratory procedures for sugar analysis with the aim of standardizing analytical methods across the industry. ICUMSA methods contain instructions for analyzing raw, cane, white, beet, molasses, plantation white, and specialty sugars. Among these are methods for: determination of dry solids content by polarimetry, densitometry, and refractometry; color measurement (extinction coefficient at 420 nm); content of reducing sugars; and presence of metals such as arsenic and copper.

The price paid for product by a sugar mill or refinery depends in large part on sucrose content and purity. In order to determine these parameters, a sample will be taken from a lot of sugar and used for testing. Filtration is a key step in preparing samples for all of these analyses. Polarization analysis on a calibrated polarimeter (or saccharimeter) by measuring the optical rotation of the clarified solution is a prominent method for determining sucrose content. Filtration of extracted juices obtained from cane, beet, and other sources is essential before polarimetry in order to remove clarifying agents such as lead acetate that have been added to a sample. Measuring color and turbidity by spectrophotometry after vacuum filtration allow for determination of purity. Purity determination not only affects the price paid for sugar but also provides refiners insight into the quality of their purification processes. The ability to process these measurements quickly and accurately translates into value for sugar producers and refiners.

Filter paper evaluation

The following study was performed by Salamon and Seaber Ltd., London UK. Analytical chemistry techniques were conducted to evaluate the performance of six filter papers in preparing sugar samples for (1) sucrose content measurement via polarimetry, and (2) purity measurements via spectrophotometry.

Table 1. Study design, n = 10 for all tests

Filter paper	Product code	Lot/batch number	Tests	ICUMSA method	Sugar origin
Whatman Grade 91	1091-110	FC011096	Filtration time Polarization	GS 1/2/3/9-1	Brazil Zimbabwe Guyana
Munktell™ Grade 15	120001	2976			
Sartorius™ Grade 100/N	FT-3-328-090	14-114			
Whatman Grade 5	1005-090	9592085	Color Turbidity	GS 1/3-7 GS 2/3-18	Brazil
Ahlstrom™ Grade 961	9610-0900	104013			
Ahlstrom Grade 962	9620-0900	103890			

Methods

Study design

Three commonly used filter papers were evaluated for their performance in ICUMSA method GS 1/2/3/9-1 for polarization. Additionally, the time to filter 30 ml and 40 ml volumes of raw sugar solution (260 g/liter) prior to polarimetry was recorded, and the repeatability of the polarization results was calculated. Replicates of ten tests were performed on sugar samples from Brazil, Zimbabwe, and Guyana.

A different set of three commonly used filters was evaluated in preparation for spectrophotometry to measure color and turbidity according to ICUMSA methods GS 1/3-7 and GS 2/3-18 respectively. For these tests, only Brazilian sugar was used, because it is known to have the highest natural level of purity of the three varietals, which minimizes the amount of additional sample preparation required that might otherwise contribute to variance in study results. Again, tests were repeated ten times each. Table 1 provides a summary of the study design.

Statistical analysis

One-way analysis of variance was used to analyze the results. All data were normally distributed.

Results and discussion

Polarization measurements and filtration time

Polarization results

ICUMSA method GS 1/2/3/9-1 was followed to prepare samples for testing and to obtain polarization data and filtration time. Raw sugar samples from Brazil, Zimbabwe, and Guyana were filtered using Whatman Grade 91, Munktell Grade 15, or Sartorius Grade 100/N filter papers. All filtrations were performed using gravity. The polarization results are shown in Figure 1. The data obtained were not significantly different when results were derived using the three different filter paper grades ($p > 0.05$).

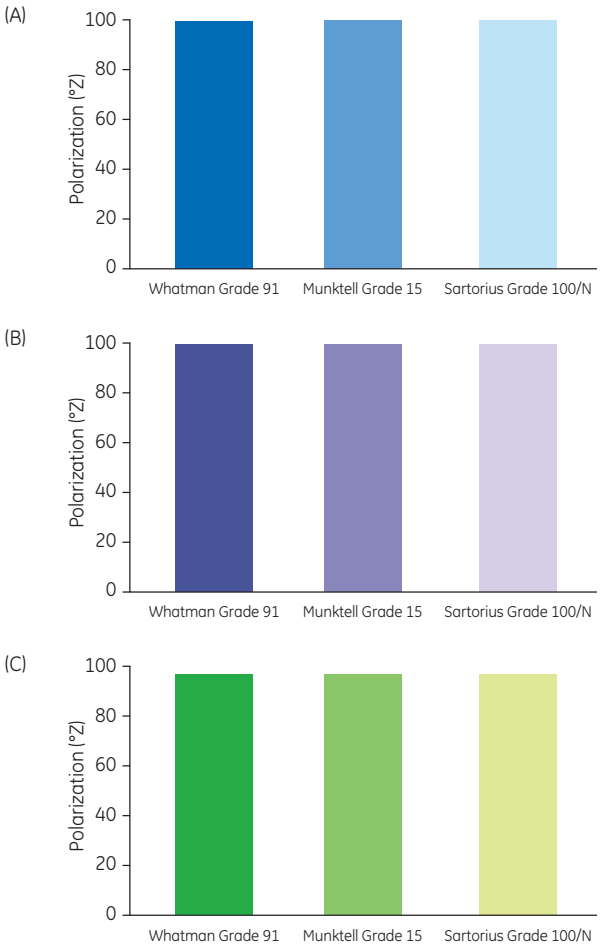

Fig 1. Polarization data from raw sugar solutions obtained from A) Brazil; B) Zimbabwe; C) Guyana. Data, in ICUMSA units (°Z), are shown as mean polarization (n=10). Standard deviations (SD; see Table 2) were too small to be graphed.

Table 2 summarizes the repeatability of the polarization results obtained after filtering samples of the different raw sugars using the three papers. Highly repeatable results of less than 0.10°Z were obtained for all filters and across all geographies from which the sugar samples were sourced.

Table 2. Repeatability of polarization results (n=10).

Sugar origin	Filter paper	Mean polarization (°Z)	SD	Repeatability (°Z)[1]
Brazil	Whatman Grade 91	99.590	0.057	0.064
	Munktell Grade 15	99.644	0.064	0.048
	Sartorius Grade 100/N	99.640	0.069	0.053
Zimbabwe	Whatman Grade 91	99.362	0.070	0.062
	Munktell Grade 15	99.374	0.039	0.030
	Sartorius Grade 100/N	99.348	0.063	0.042
Guyana	Whatman Grade 91	96.634	0.039	0.037
	Munktell Grade 15	96.670	0.042	0.037
	Sartorius Grade 100/N	96.662	0.043	0.029

[1] Repeatability is equivalent to mean difference.

Filtration time

Data were collected on the time required to filter the samples of sugar solution in preparation for polarimetry according to ICUMSA method GS 1/2/3/9-1. Samples (30 ml) from the three sugar sources were filtered with the three filter papers used in the polarization measurements. The mean filtration times and SD (n=10) are provided in Figure 2.

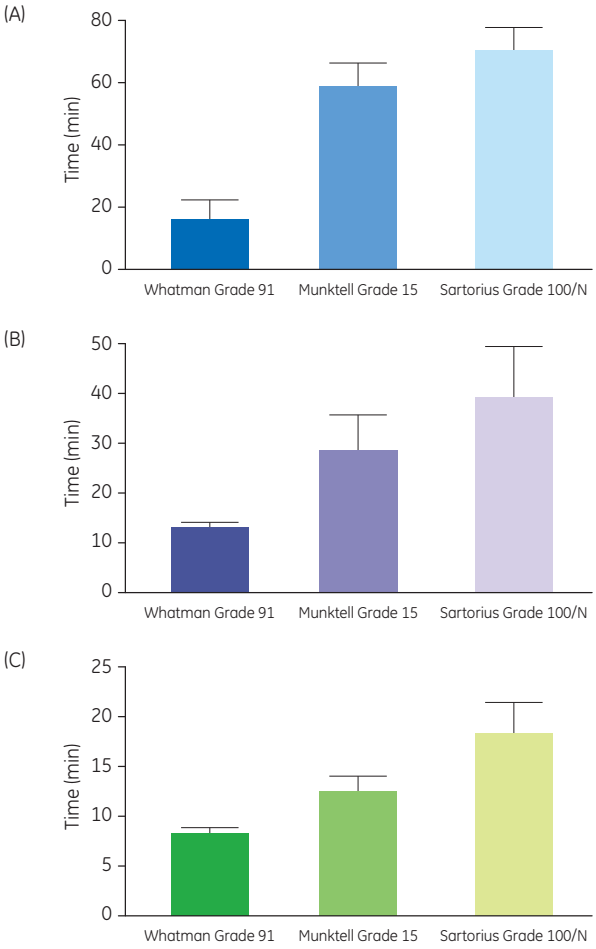

Fig 2. Filtration times using three grades of filter papers and 30 ml of raw sugar solution from A) Brazil; B) Zimbabwe; or C) Guyana. Bar heights represent the mean of 10 replicates. Error bars represent the SD.

The filtration times required using Whatman Grade 91 filter paper were less than those obtained using the other two filter paper grades at a highly significant ($p < 0.001$) level. The Brazilian sugar showed the greatest difference; filtration times were more than 3-fold higher for the Munktell and Sartorius papers compared with Whatman Grade 91. The substantially longer time required to filter the Brazilian sugar using any of the filters is consistent with visual observations of its high viscosity compared with the other sugar samples. It was observed in a subsequent test of 40 ml samples prepared according to the same method that the Whatman filter was the only one in the test group capable of filtering the entire volume. As the full volumes were not fully filtered by the Munktell and Sartorius filters, no further analysis was performed.

Color and turbidity

Color

A sample solution of Brazilian sugar was prepared for spectrophotometry using ICUMSA method GS 1/3-7. The color of the filtrate obtained from each of the three filter papers is provided in Figure 3. All filtrations were performed using a vacuum. The filtrate color (in ICUMSA units - IU) was lower with Whatman Grade 5 than with the other papers at a highly significant ($p < 0.001$) level.

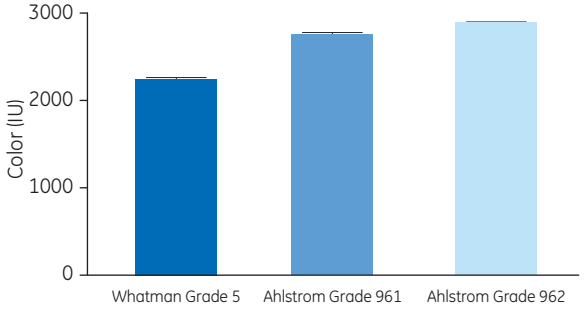

Fig 3. Color of filtrate from Brazilian raw sugar. Data, in ICUMSA Units, are shown as the mean (n=10), with error bars representing the SD.

Turbidity

A sample solution of Brazilian sugar was prepared for spectrophotometry using ICUMSA method GS 2/3-18. The same grades of filter paper tested in color measurement were once again used, and all filtrations were performed using a vacuum. The data as shown in Figure 4 indicate that the turbidity of the filtrate was lower with Whatman Grade 5 than with the two other papers at a highly significant ($p < 0.001$) level. Indeed, the turbidity of the filtrate obtained with Whatman Grade 5 paper was less than half of that obtained using either of the Ahlstrom papers. These data indicate that higher nonsoluble contamination levels are present in the filtrates derived from the Ahlstrom papers compared with Whatman Grade 5. The ability to isolate these contaminants as filtrate allows for simpler downstream analysis and greater insight that refiners can use to optimize their quality processes.

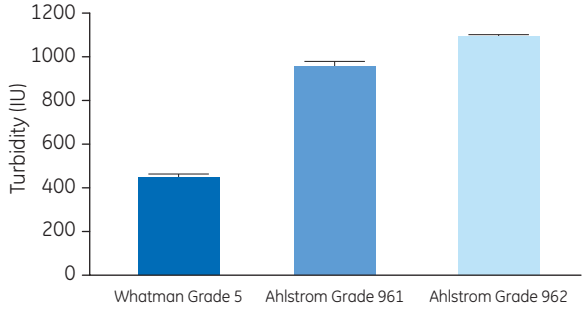

Fig 4. Turbidity of filtrate from Brazilian raw sugar. Data, in ICUMSA Units, are shown as the mean (n=10), with error bars representing the SD.

Conclusions

In this study, polarization measurements using ICUMSA method GS 1/2/3/9-1 were comparable ($p > 0.05$) for all three filter papers that were evaluated. In addition, all three papers provided highly repeatable data across all ten samples. However, filtration time was significantly faster ($p < 0.001$ for 30 ml sample volume) using Whatman Grade 91 filter paper compared with the Munktell Grade 15 and Sartorius Grade 100/N papers. Notably, the Whatman filter was the only one found in this study to adequately complete filtration of 40 ml of the Brazilian raw sugar. Laboratories that conduct a large number of polarization tests could realize substantial savings in both time and labor by using a filter paper that provides a rapid flow rate without compromising the quality of polarization data.

Using ICUMSA methods GS 1/3-7 and GS 2/3-18, the results for color (filtrate clarity) and turbidity were significantly better ($p < 0.001$ for both assays) with the Whatman Grade 5 filter paper compared with the two Ahlstrom papers that were tested. The turbidity values for Whatman Grade 5 were less than 50% of those obtained with either of the other papers.

The data generated in this study support the use of Whatman Grade 91 in ICUMSA method GS1/2/3/9-1 (1) and of Whatman Grade 5 to effectively remove contaminants from sugar samples using ICUMSA methods GS 1/3-7 (2) and GS 2/3-18 (3).

References

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3. International Commission for Uniform Methods of Sugar Analysis. ICUMSA Method GS2/3-18. *The determination of the turbidity of white sugar solutions – official* (2013).

The data used to support this study can be made available upon request to TechSupportUK@ge.com.

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