A NEW FILTRATION SYSTEM FOR GUM ANALYSIS IN RAW SUGAR AND MOLASSES

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Abstract

'Gums' are defined as a heterogeneous group of compounds that form a precipitate on addition of acidified alcohol. Starch and natural gums occur in undamaged sugarcane. Natural gums include hemicelluloses, pectins and dextrins. Bacteria also produce gums such as dextrans and levans, during and after the milling process (Jennings, 1964). These molecules are all soluble in water but insoluble in alcohol, providing an easy method of separation.

Increased health and environmental concerns have prompted an investigation into finding an alternative filtration system to asbestos in the gums method. In the Fibroxcel range of filter aids (AEB Africa), Fibroxcel 10, with a glass fibre pre-filter, displayed potential in reproducing results comparable with the asbestos filtration system, with the added benefit of being environmentally friendly and improving filtration time.

A ring test was conducted between four laboratories in the South African sugar industry and results confirmed reproducibility of the new method in raw sugar and molasses. A tolerance for gums in molasses has never existed and an intra-laboratory comparison conducted at the SMRI established a tolerance of ± 2000 ppm. The Factory Control Advisory Committee approved the revised methods for determination of gums in molasses and raw sugars on 10 March 2004.

Keywords: sugar, gums, filtration

Introduction

In a cane sugar factory, gums are only partially removed by clarification so that some of the soluble fraction is carried through to the raw sugar. In fact, the amount of gums present in raw sugar is a fairly good indicator of the deterioration in sugarcane and will affect the filterability of a solution of that sugar. This may cause severe problems in refining and other subsequent processes (Meade and Chen, 1977).

In the filtration procedure of gum analysis, asbestos fibres form the filter pad in the Gooch crucible (Ruff and Withrow, 1922). The health and environmental concerns associated with asbestos have prompted the investigation of alternative filtration systems. Over the past six years, the SMRI has evaluated various types of sintered glass frits (in terms of porosity) with commercial filter aids. However, none of these gave results that were repeatable and within tolerance of results obtained using the asbestos filtration system (unpublished data[1]).

[1] Internal reports 1998-2001, Sugar Milling Research Institute, University of KwaZulu-Natal, Durban, South Africa.

The Fibroxcel range of filter aids (AEB Africa) was initially developed to replace asbestos fibres in filtration applications. The Fibroxcel filter aids are easily discarded and environmentally friendly (AEB Africa, 2002). When the Fibroxcel filter aids were used inside sintered glass frits for the analysis of gums in raw sugar, one in particular (Fibroxcel 10) displayed potential in terms of reproducing results comparable with the use of the asbestos filtration system (de Jager, 2002). However, it was soon clear that the sintered glass frits could not withstand repeatable ignitions at 650°C. The use of Gooch crucibles, which are made of porcelain, was initially hampered due to the filter aid particles being small enough to flow through the holes in the crucibles. Closing of the holes with filter paper would not have been viable at these temperatures. However, use of a glass fibre pre-filter, which can withstand temperatures of up to 650°C, proved successful in keeping the filter aid intact. In addition, filtration times were much shorter when using the Fibroxcel 10 filter aid with the glass fibre pre-filter, than when using the asbestos filter pad.

Method for raw sugar and molasses (SMRI, 2003)

Sample preparation for raw sugar
A 50E Brix sugar solution was filtered through a screen into a centrifuge tube. The solution was centrifuged and the supernatant liquid weighed into a beaker. The refractometer Brix of the sugar solution was measured. Acidified alcohol (1:1) was added to the supernatant and the sample was allowed to stand for 16 hours.

Sample preparation for molasses
A 10E Brix molasses sample was prepared and centrifuged. The supernatant liquid was weighed into a beaker and acidified alcohol (1:1) added to the sample. The molasses sample was allowed to stand for 16 hours.

Gooch preparation
A slurry was prepared with Fibroxcel 10 filter aid and water. The slurry was poured into a Gooch crucible with a glass fibre pre-filter to form a smooth filter pad. The Gooch crucibles were ignited in the muffle furnace at 650E and cooled.

Filtration
The precipitate was transferred quantitatively to the Gooch crucible and washed with acidified alcohol and absolute alcohol. The crucibles were dried in an oven, cooled and weighed. The crucibles were then again ignited in a muffle furnace, cooled and re-weighed.

Results and discussion

Inter-laboratory ring test
Four samples of raw sugar and five samples of molasses were distributed to the Hulett's Refinery laboratory (HR), the TMD laboratory (TMD) and the Sugar Terminal laboratory (SAST), with the SMRI being the fourth laboratory in the ring test. For anonymity, participating laboratories are labelled 'Lab 1' to 'Lab 4'.

Tables 1 and 2 display the average values obtained by each laboratory for the raw sugar and molasses samples respectively, with some additional calculations to aid evaluation.

Table 1. Average raw sugar results for each laboratory.

Sample	A	B	C	D
	Gums (ppm)			
Lab 1	725	800	550	650
Lab 2	725	900	550	525
Lab 3	~	950	600	~
Lab 4	700	825	575	600
Average (ppm)	717	869	569	592
SD RSD (%) 2x SD (ppm)	14 2 40	69 8 138	24 4 48	63 11 126

SD = standard deviation, RSD = relative standard deviation, 2x SD = recommended tolerance

Table 2. Average molasses results for each laboratory.

Sample	A	B	C	D	E
	Gums (ppm)				
Lab 1	16 850	20 150	19 550	18 250	13 400
Lab 2	19 950	23 650	21 950	18 200	14 650
Lab 3	18 500	~	21 800	18 200	15 800
Lab 4	18 050	21 200	21 100	18 800	14 450
Average (ppm)	18 338	21 667	21 100	18 363	14 575
SD RSD (%) 2x SD (ppm)	1281 7 2562	1796 8 3592	1098 5 2195	293 2 585	984 7 1967

SD = standard deviation, RSD = relative standard deviation, 2x SD = recommended tolerance

The accepted tolerance used in the South African industry for the analysis of gums in raw sugar is ±150 ppm (SMRI, 2003). From Table 1 it is clear that the recommended tolerances (2x SD) for the raw sugar samples fall within the existing tolerance of 150 ppm. The maximum relative standard deviation (RSD) for the raw sugar samples was 11%.

From Table 2, the maximum relative standard deviation for the molasses comparisons was 8%, which resulted in a recommended tolerance of 3600 ppm (rounded to the nearest 100 ppm). Since there is no tolerance for the analysis of gums in molasses, this value is difficult to judge. However, it is quite clear that the relative standard deviations for molasses and raw sugar are comparable. This indicates that the method for analysis is reproducible compared with the existing standards (*i.e.* analysis of gums in raw sugar).

Intra-laboratory comparisons for molasses

Three molasses samples were analysed in triplicate by four analysts from the SMRI (Table 3). Results indicated that a repeatability and reproducibility (between analysts) tolerance of ± 2000 ppm gums in molasses would be appropriate once analysts are adequately trained.

Table 3. Intra-laboratory comparison for the analysis of gums in molasses.

Sample	Analyst A		Analyst B		Analyst C		Analyst D		Average	SD
	Average	SD	Average	SD	Average	SD	Average	SD		
1	21 550*	212	23 000*	707	22 667	961	22 550*	212	22 442	624
2	17 133	321	17 067	58	15 733	451	~	~	16 644	790
3	23 850*	919	22 267	153	21 467	493	22 100	819	22 421	1013

*One outlier was removed in each case according to the Grubbs test; outliers were removed for statistical purposes so as not to bias the average.

Conclusions

The use of Fibroxcel 10 filter aid on a glass fibre pre-filter was found to be a suitable substitute for asbestos fibres when analysing for gums in raw sugar and molasses, since results obtained were comparable with those obtained using the original method. Other samples (syrup and juice) and the determination of the amount of bagacillo in sugar will be evaluated using the proposed method in due course.

It is recommended that molasses gum results be rounded to 100 ppm and a tolerance of ± 2000 ppm be used as an industry standard.

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