

THE DIRECT DETERMINATION OF FIBRE IN CANE

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Abstract

A detailed description is given of a direct method for the determination of fibre in cane. The reproducibility of the method is shown to be within approximately 1% of the mean value.

Introduction

During an investigation² into accurate methods for the direct determination of the constituents of sugar cane, it became apparent that, although fairly accurate indirect methods were available for the determination of fibre, the direct methods left much to be desired. It was decided to attempt the development of a rapid, accurate and direct method for the determination of fibre in cane.

For the purpose of this paper, the definition of fibre complies with that given in "Laboratory Manual for South African Sugar Factories", i.e. "Fibre is the water insoluble matter in cane and bagasse".

The main problem encountered by earlier workers was the difficulty caused by uneven particle size of the prepared cane. Not only did this affect sampling adversely, but it proved impossible to exhaust larger pieces of cane completely. It was apparent that a suitable method of sample preparation was a prerequisite for a satisfactory analytical method.

Method

Cane preparation and sampling

The samples of cane used for the analyses were delivered in bundles of six whole sticks. The sticks were rearranged in a head-to-tail fashion and fed into a chaff cutter. The sticks were cut up into discs of about $\frac{1}{4}$ in. thickness by the chaff cutter, and the six short, uncut ends were discarded. The chopped sample was placed in a pile, well mixed, quartered, and the two opposing quarters placed in an airtight container. The rest of the sample was discarded. The sub-sample was placed in a Waddell shredder and shredded for forty seconds. The shredded sample was carefully removed and placed in an air-tight container for analysis.

The sample prepared in this manner was a homogeneous mixture containing fibre no longer than $\frac{1}{4}$ in.

Apparatus

1. Ultra-Turrax high speed extractors
2. Balance capable of weighing to ± 0.01 g
3. Sintered glass funnel 120 mm diameter porosity 1
4. 1 one litre vacuum flask
5. 2 one litre beakers and stirring rods
6. Dietert Moisture Teller — 200 mesh sieve
7. 500 ml wide mouth flask.

Procedure

200 g (± 0.01 g) of sample was weighed into a one litre beaker. Approximately half the sample was transferred from the beaker into a 500 ml wide mouth flask to which was added ± 300 g clean water. The contents were extracted with an Ultra-Turrax high speed extractor until all the fibres were reduced to small particles. This was usually accomplished within about four minutes. The contents of the flask were poured into a 120 mm, porosity 1, sintered glass funnel which was supported in the neck of a one litre vacuum flask.

While vacuum was applied to drain the funnel the procedure was repeated with the other half of the sample. After addition of the second portion, the runnings (± 600 g) were re-cycled twice through the bed to remove any small suspended particles. The funnel and contents were thoroughly washed with ten one litre aliquots of clean tap water, and the fibre in the funnel was disturbed occasionally during the washing process to reduce channelling. The last washing was carried out using one litre distilled water.

The funnel was allowed to drain well at full vacuum and the dewatering was aided by squeezing the mat with a suitable plunger, e.g. a 250 ml beaker. As much water as possible was removed by this means.

When the runnings ceased the vacuum was released, the funnel removed from the flask and the fibre mat transferred into a 200 mesh sieve by means of blowing down the inverted funnel stem. Any adhering particles were transferred from the funnel to the sieve by means of a clean 1 in. paint brush.

The fibre mat in the sieve was broken up and dried in a moisture teller for one hour at 240°F. The sieve and contents were weighed immediately the moisture teller had stopped to prevent readsorption of moisture.

The fibre weight was expressed as a percentage of the cane weight.

Note on procedure

Samples were taken of the last runnings and brix determinations carried out, using a precision refractometer. Results showed that these values were negligible, ranging between 0.00° and 0.02° brix.

The α -naphthol test for sucrose was also used as a check for exhaustion of the fibre and tests again showed negligible values.

Discussion

Reproducibility tests carried out using the described method gave results with a standard deviation of 0.15 on twenty-five duplicate sets of analyses ranging from 11 to 17% fibre. This represents

approximately 1% of the mean value of 13.16. An independent research team³, using the same basic method obtained results with a standard deviation of 0.18 on twenty duplicate sets of analyses ranging from 12 to 17% fibre, which represents 1.3% of the mean value of 13.85.

Further tests³ showed that the reproducibility of the described direct method is superior to that of the indirect fibre determination proposed for direct cane analysis¹.

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References

1. Buchanan, E. J. An Investigation into the Requirements for Direct Analysis of Sugar Cane. S.M.R.I. Quart. Bull. (28), 1965, 77-86.
2. O'Connor, B. The Determination of Fibre in Sugar Cane. Comm. S.M.R.I. 1953 (17).
3. Sugar Industry Central Board (1969). Unpublished Report.

Discussion

Mr. Smith: Mr. Prince mentions that the described direct method is more reproducible than the indirect fibre determination proposed for direct cane analysis.

Can he give us any comparison of the two methods?

Mr. Loudon: The mean deviation on twenty samples on the direct method was .18 and on the indirect method was 1.00. A comparison of results of the two methods shows the fibre was 14.06% by the direct method and 13.88 by the indirect method.

When comparing the direct and indirect methods one point must be made quite clear—that the indirect method was developed by the S.M.R.I. for a specific purpose, namely, the distribution of sucrose

in cane as an alternative to the Java Ratio system. Therefore any error in the fibre determination has a negligible effect on the sucrose distribution.

If fibre is to be used as a factor in cane quality evaluation then a more accurate figure is required, but I think the indirect method can be improved to give this.

At present only 100 gm of material is being used for moisture determination but if this was increased to say 500 gm a more accurate determination could be made.

Dr. Graham: Is it considered that this method could now be used for routine fibre determinations and is the Ultra-Turrax high speed extractor proving reliable?

When previous comparisons were made between direct and indirect determinations the differences were a lot greater than shown here.

Mr. Prince: This method could be adapted for use in routine fibre determination but not necessarily with the same equipment.

The Ultra-Turrax machine gave trouble at first but is now reliable.

Mr. King: We replaced one set of bearings in the large Ultra-Turrax machine and have since done 800 samples without further trouble. The rotors and stators are showing signs of wear.

The method is at present a bit cumbersome for routine purposes.

Mr. Alexander (in the chair): The S.M.R.I. extractor has proved itself and might be suitable for this type of fibre determination.

Mr. King: I think the main reason for the differences between the methods is that Mr. Prince was drying at 240°F and we were using 267°F.

The higher the temperature used for drying, the lower the weight, as we discovered when we carried out tests at different temperatures on shredded cane.

Mr. du Toit: It is possible that the problem is not one of incomplete drying but of loss of solids by overheating.

Mr. Jennings: It seems necessary to carry out a further series of drying tests on washed fibre at different temperatures.

Mr. Prince: Washed fibre was weighed after being heated first at 240°F and then at 273°F for twenty minutes and there was a negligible difference. I tried again at higher temperatures but the fibre was scorched.