DIRECT ANALYSIS OF CANE USING A WET DISINTEGRATOR

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INTRODUCTION

For many years a simple and direct method for analysing small samples of cane has been needed. Recent attempts to solve the problem have all centred around the use of some type of disintegrating machine in which cane is disintegrated with water. The liquid obtained after disintegration is analysed for brix and pol and from these figures the brix and pol in cane can be calculated, knowing either the water or fibre content of the cane.

Methods of analysis along these lines have been used for assessing the quality of small lots of cane, e.g. in Hawaii. They were used during 1957 in Queensland to estimate mill input of brix and pol by analysing samples of 1st mill feed taken at regular intervals.

We have developed a method of cane analysis of this type for our own use in assessing cane quality and for checking mill input by other methods, e.g. juice scales. Each step in the analysis has been investigated and an improved method of analysis has resulted.

METHOD OF ANALYSIS

The method of analysis developed depends on the use of a swing hammer mill to shred and intimately mix the cane, and a wet disintegrator for extraction. The hammer mill used is similar to that described by Wadde1, while the wet disintegrator in general use is similar to that developed by Sugar Research Institute, Mackay, Queensland. An experimental high speed (10,000 r.p.m.) wet disintegrator has been built to check completeness of extraction (brix and pol). The wet disintegrators are described in detail later.

Procedure for analysis: A 12 lb. sample of 1st mill feed or chaff-cut cane stalks is charged into the hammer mill and disintegrated for 10 to 15 seconds. In this short period very fine disintegration is obtained.

The per cent water in the cane is determined by drying 200 g samples of hammer-milled cane in a modified type of Spencer oven. In this oven, the cane is dried to constant weight in 2 to 3 hours with negligible decomposition. If a suitable oven for determining water in cane is not available, then fibre in cane is determined by diffusing 100-gm. samples of hammer-milled cane in calico bags according to the official method for Queensland.

To extract the soluble constituents from the cane, 5 lb. of hammer-milled cane and 15 lb. of water are placed in the wet disintegrator. After 45 minutes' treatment, a sample of the slurry is taken and filtered through a 120-mesh screen to remove fibre. The screened liquor is then cooled to 25°C or below, and filtered with Supercel to remove suspended material. This filtration must be rapid, not more than 5 minutes, so that loss of water by evaporation is negligible. The brix of the filtered liquor is de-
terminated by Geissler pycnometer. Pol in the liquor is determined using dry lead defecation and reading in a 400-mm tube.

CALCULATION OF ANALYSIS OF CANE

**Using Per Cent Water in Cane**

\[
\text{Brix or Pol in Cane} \times \text{Weight of cane taken} = [\text{Brix or Pol in disintegrator liquor}] \times [\text{Weight of disintegrator liquor}] \times 0.992
\]

where:

\[
\text{weight of disintegrator liquor} = \frac{\text{Weight of water in cane} + \text{Weight of water added for disintegration}}{1 - \frac{\text{Brix of disintegrator liquor}}{100}}
\]

and 0.992 is the correction factor for hygroscopic or brix-free water adsorbed onto the cane fibre.

For simplicity in calculation, parts weight of cane and water instead of actual weights are used. This gives a factor for converting brix and pol in the disintegrator liquor to brix and pol in cane.

For 5 lb. (1 part) of cane and 15 lb. (3 parts) of water, the factor is as follows:

\[
\text{Factor} = \left[ 3 + \frac{\% \text{ water in cane}}{100} \right] + \left[ 3 + \frac{\% \text{ water in cane}}{100} \times \frac{\text{Brix of disintegrator liquor}}{100} \right] \times 0.992
\]

\[
= \left[ 3 + \frac{\% \text{ water in cane}}{100} \right] \times \frac{\text{Brix of disintegrator liquor}}{100} \times 0.992
\]

**Example of calculation**

Water in hammer-milled cane = 73.00%

Brix in disintegrator liquor = 4.00

Pol " " " " = 3.50

Then

\[
\text{factor} = \left[ 3 + 0.73 \right] \times 0.992
\]

\[
= 3.854
\]

Then

\[
\text{Brix in cane} = 4.00 \times 3.854 = 15.42
\]

\[
\text{Pol " " } = 3.50 \times 3.854 = 13.49
\]

**Using Fibre in Hammer-milled Cane**

\[
\text{Brix or Pol in Cane} \times \text{Weight of cane taken} = [\text{Brix or Pol in disintegrator liquor}] \times \left( \frac{\text{Wt. of added} - \text{Wt. of fibre}}{\text{Wt. of water in cane}} \right) \times 0.992
\]

*References p. 198.*
where:

\[ \text{weight of fibre in cane} = \frac{\text{weight of cane} \times \% \text{ fibre in cane}}{100} \]

Using parts weight of cane and water the following factor is obtained:

\[ \text{Factor} = \left( 3 + \frac{100 - \% \text{ fibre}}{100} \right) \times 0.992 \]

**Example of calculation**

- Fibre in hammer-milled cane = 11.50\%
- Brix in disintegrator liquor = 4.00
- Pol " " " = 3.50

\[ \text{Factor} = \left( 3 + \frac{100 - 11.50}{100} \right) \times 0.992 \]
\[ = 3.854 \]

Then

- Brix in cane = 4.00 \times 3.854 = 15.42\%
- Pol " " " = 3.50 \times 3.854 = 13.49\%

The brix of the water with which the cane is disintegrated is taken as zero in the above examples. If the brix of the water used is greater than 0.01\%, then a correction must be made in the calculations.

**FACTORS INVESTIGATED**

The following factors have been investigated during the development of the above method of cane analysis:

**Loss of Water from Cane during Hammer-milling**

- It has been found that the loss of water from cane during hammer-milling can be considerable (1 to 2\% on cane) if the time of hammer-milling is prolonged, e.g. 30 seconds or more.
- Overcharging of the hammer mill also causes rapid heating of the cane and longer times are required for disintegration. This results in evaporation losses.
- The optimum sized charge is 10-12 lb. when maximum disintegration occurs in 10 to 15 seconds. The evaporation loss is then negligible.

**Fibre Determination in Cane**

It has been found that direct methods of fibre determination, using some means of cane disintegration followed by diffusion in single calico bags, give an under-estimation of fibre. In a series of experiments the loss from clean, fibrated cane was found to be about 0.2\% in a determined fibre figure of 13\%. This is due to some fine material escaping through the weave of the bag. The loss can be greater if the cane contains fine clay and mud which can easily pass through the bag. It has also been found that the fibre determination is dependent on the degree of fineness of the cane preparation, the finer the preparation the greater the loss.
Losses through the bag can be reduced by taking suitable precautions, e.g. use of double bags and centrifuge washing. However, the determination of water in cane is a simpler procedure.

**Determination of Water in Cane**

The determination of water in hammer-milled cane was investigated by two methods: vacuum drying at 70°C, and hot-air drying at atmospheric pressure. It was found that identical results were obtained by both methods when the hot-air drying was carried out with a rapid flow of air at a temperature not exceeding 110°C at the air inlet to the oven.

A new type of Spencer drying oven was built for this purpose giving a controlled air temperature of 110°C ± 1°C at this point (Fig. 1). It was found important to position the thermometer immediately above the air inlet. If it were placed even slightly away from this position, lower air temperatures were indicated and excessively hot air could be introduced in attempting to reach 110°C. In an oven of this type, cane was dried to constant weight in 2 to 3 hours as against 5 to 6 hours in the older type of Spencer drying oven.

**Determination of Brix in Disintegrator Liquor**

It is desirable that the measurement of brix (gravity solids) in the disintegrator liquor should be accurate to 0.01°. This requirement cannot be met by the use of a...
brix spindle or the normal Abbé type of refractometer, but is obtainable with a pycnometer. A Geissler pycnometer was used because of its convenience with the inbuilt thermometer for rapid determination.

The disintegrator liquor, after being strained through a 120-mesh screen and cooled to $25^\circ C$ or below, is filtered with Supercel filter aid to remove fine suspended material before determining brix and pol. This procedure was adopted as serious overestimation of brix was found to occur with liquors containing fine suspended solids.

Rapid vacuum filtration (<5 minutes) of the liquor with Supercel (2% on liquor) causes negligible brix error due to evaporation or adsorption of soluble solids on the Supercel.

**Determination of Pol in Disintegrator Liquor**

The determination of pol presents no difficulty. The minimum amount of lead defecating agent should be used. To obtain greater accuracy a 400 mm. tube is used.

**Specifications of Wet Disintegrators**

*a. Standard Wet Disintegrator*

This is the machine in general use (Fig. 2). It is similar in design to the wet disintegrator developed by The Sugar Research Institute, Mackay, Queensland3.
The shaft with cutting blades rotates at 5,720 r.p.m. and is driven by a 2 hp. 1,430 r.p.m. motor. Three blades, made of spring steel, 6 in. overall length, 1\(\frac{1}{2}\) in. wide and \(\frac{1}{8}\) in. thick, are positioned at the bottom of the shaft.

The can for holding the cane and water is an ordinary 4-gal. oil drum with a wooden lid, a peripheral rubber gasket being used to prevent loss of fluid.

b. **High-speed Wet Disintegrator** (Fig. 3)

This was built to check the completeness of extraction obtained in the standard machine. The shaft with cutting blades rotates at 10,000 r.p.m. and is driven by a 3 hp. 2,880 r.p.m. motor through a flat nylon belt. Cutting blades used are similar to the standard machine.

The can for holding the slurry is heavy-gauge metal. Three small baffles, fitted in the can near the bottom, are necessary to prevent small pieces of cane escaping disintegration. The can is cooled with water to dissipate the heat generated and minimize evaporation from the slurry.

**Completeness of Brix and Pol Extraction in the Wet Disintegrators**

Much attention has been devoted to ensuring that maximum extraction is obtained in the wet disintegrators and the following factors have been examined:

a. The clearance of the 3 cutting blades above the bottom of the can.

A position as near the bottom as possible was found to give the fastest rate of extraction and the finest preparation. The 3 blades are equally spaced around the...
shaft with \( \frac{1}{2} \)-in. between blades and the bottom blade is about 1 in. from the bottom of the can.

b. Thickness and length of cutting blades.
It was found that maximum extraction was obtained with the thin blade, \( \frac{1}{4} \)-in. The thin blade has the additional advantage of not needing to be sharpened. Extraction was found to be slower with blades shorter than 6 in. in length.

c. Baffling
This gave no positive advantage in the standard machine. Baffling was, however,

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\[ \text{Fig. 4. Fibres after disintegration in standard machine.} \]

necessary in the high-speed machine to ensure that no small pieces of cane escaped disintegration.

d. Time and temperature of wet disintegration.
A time of 45 minutes was selected for the standard machine and 30 minutes for the high-speed machine. Longer times did not give any increase in extraction, but shorter times occasionally resulted in incomplete extraction. This was found mainly to be a function of variety.

The temperature of wet disintegration was examined and it was found that hot conditions, i.e., 80°-90°C, did not raise the extraction above that obtained at room temperature.

e. The effect of the cutting blade speed in both the standard machine (5720 r.p.m.) and the high-speed machine (10,000 r.p.m.).
It was found that the cane was reduced to a considerably finer state of preparation in the high-speed disintegrator, as illustrated in Figs. 4 and 5.
The higher speed generally extracted a fraction more brix from the cane than the slower speed but, generally, there was no difference in pol extraction at the higher blade speed. It was found, however, that some cane varieties were more resistant to extraction than others and gave slightly higher extractions of both brix and pol at the higher blade speed.

In Table I, Vesta, Trojan and CP 34/120 are examples of "extraction-resistant" canes.

Work is being continued on the effect of blade speed on extraction. However, the results do not indicate that the slower-speed standard wet disintegrator is giving serious underestimation due to incomplete extraction.

Preservation and Deterioration

The preservation of hammer-milled cane prior to analysis was examined in order to be able to sample 1st mill feed over a long period, to mix all samples and then to get a representative sub-sample for analysis.

It was found that hammer-milled cane could be stored satisfactorily for up to 12 hours at 0° to 5° C in polythene bags with 1 ml. of toluene added per pound of cane. If longer storage is required, the cane must be held at temperatures below 0° C.

Deterioration during wet disintegration can result from inversion of sucrose or by microbiological destruction of sucrose. This effect was examined by disintegrating with and without additions of sodium carbonate to raise the pH to counteract acid inversion and mercuric chloride to prevent microbiological destruction. Results

References p. 198.
TABLE I
THE EFFECT OF BLADE SPEED ON WET DISINTEGRATOR EXTRACTION

<table>
<thead>
<tr>
<th>Cane variety</th>
<th>No. of comparisons</th>
<th>Concentration in disintegrator liquor</th>
<th>Brix</th>
<th>Pol</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Standard speed</td>
<td>High speed</td>
<td>Standard</td>
</tr>
<tr>
<td>Vesta</td>
<td>9</td>
<td>4.10</td>
<td>4.11</td>
<td>3.53</td>
</tr>
<tr>
<td>Trojan</td>
<td>12</td>
<td>4.86</td>
<td>4.88</td>
<td>4.10</td>
</tr>
<tr>
<td>CP34/120</td>
<td>3</td>
<td>4.32</td>
<td>4.34</td>
<td>3.68</td>
</tr>
<tr>
<td>Pindar</td>
<td>12</td>
<td>5.31</td>
<td>5.32</td>
<td>4.75</td>
</tr>
<tr>
<td>Q5o</td>
<td>11</td>
<td>5.60</td>
<td>5.61</td>
<td>4.73</td>
</tr>
<tr>
<td>Erros</td>
<td>6</td>
<td>5.52</td>
<td>5.53</td>
<td>4.49</td>
</tr>
<tr>
<td>Q57</td>
<td>3</td>
<td>4.87</td>
<td>4.88</td>
<td>3.00</td>
</tr>
<tr>
<td>Vidar</td>
<td>3</td>
<td>4.90</td>
<td>4.90</td>
<td>4.41</td>
</tr>
</tbody>
</table>

indicated that deterioration is negligible in the 45-minute disintegration time with sound cane.

10 ml 10% sodium carbonate and 5 ml saturated mercuric chloride solution are added as a precaution when deteriorated cane is being analysed.

Accuracy and Reproducibility of Methods of Analysis

1. Error in Analytical Methods

The maximum error to be expected in the analysis of a sample hammer-milled cane has been estimated. The individual errors in each analytical step are as follows:

<table>
<thead>
<tr>
<th>Step</th>
<th>Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weighing cane for wet disintegrator</td>
<td>1 in 200</td>
</tr>
<tr>
<td>water</td>
<td>1 in 600</td>
</tr>
<tr>
<td>Water determination in cane</td>
<td>1 in 500</td>
</tr>
<tr>
<td>Brix determination in liquor</td>
<td>1 in 400</td>
</tr>
<tr>
<td>Pol</td>
<td>1 in 120-200 mm tube</td>
</tr>
<tr>
<td></td>
<td>1 in 240-400 mm tube</td>
</tr>
</tbody>
</table>

When these errors are combined for brix and pol determination in cane, the maximum error is as follows:

<table>
<thead>
<tr>
<th>Determination</th>
<th>Maximum Error</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brix in cane</td>
<td>1.0%</td>
<td>18.00 ± 0.18</td>
</tr>
<tr>
<td>Pol</td>
<td>2.0%~200 mm tube</td>
<td>15.00 ± 0.30</td>
</tr>
<tr>
<td></td>
<td>1.6%~400 mm tube</td>
<td>15.00 ± 0.24</td>
</tr>
</tbody>
</table>

The error normally obtained would be considerably lower than the maximum error where all errors are at their maximum in one direction.

2. Error in Sampling and Analysis of Hammer-milled Cane

The error in quadruplicate wet disintegrator analysis of hammer-milled cane has been determined. The error here includes analytical error.

The standard deviations for brix and pol determinations were as follows:

- Standard deviation of one brix determination = 0.07
- Standard deviation of one pol determination = 0.04
i.e., in 95% of determinations the brix would be for example 15.00 ± 0.14 and the pol 13.20 ± 0.08.

The Effect of Hygroscopic Water on Cane Analysis

Hygroscopic, or brix-free, water exerts its effect in wet disintegrator analysis as water strongly adsorbed onto the cane fibre and unavailable for solution of the soluble constituents. This has the effect of raising the brix and pol in the disintegrator liquor to a higher concentration than would be observed if all the water present were available for solution of these substances.

Work done during 1957 indicated that hygroscopic water was in the order of 30% of the dry weight of the cane fibre under the conditions of wet disintegration. At this level, a correction factor of 0.99, i.e., a 1% reduction, was required for the brix and pol in cane.

We have continued this work and have recently improved our technique for measuring the hygroscopic water. The results which are being obtained now indicate that hygroscopic water is more nearly in the order of 20% to 25% on dry fibre. At this level, a correction factor of 0.993 or 0.992 is indicated.

Our general method of measuring hygroscopic water has been to take the cane fibre after disintegration and wash it free of soluble material, then dry it at 120°C. The dry fibre is mixed with a 4% sucrose solution or liquor from the disintegrator in the proportion of 30 gm. fibre to 500 gm. solution. After equilibrium is reached (in about one hour) the rise in concentration of the solution is observed and the amount of water adsorbed is determined by a material balance.

Pol determination in a 400-mm. tube in a saccharimeter readable to 0.01°C is the best method of observation.

For the purpose of measuring hygroscopic water, brix determination by Geissler pycnometer has been abandoned due to the virtual impossibility of washing the fibre to a state where it gives no brix indication on re-mixing with distilled water. The fibre can, however, be washed to a state where no pol is observed when it is re-mixed with water. As a precaution, a control experiment with distilled water is always carried out to ensure that none of the pol rise has come from the fibre.

We have improved the duplication of our determinations by using vacuum de-aeration of the fibre while it is being mixed with the solution. This presumably results in better wetting.

The estimation of hygroscopic water still remains, however, relatively inaccurate, but we doubt whether the present accuracy of measurement results in significant differences between fibres from different cane varieties.

We have also checked the effect (on the hygroscopic water) of drying the fibre at 120°C compared with fibre that has not been dried at any stage. No significant difference has been found, i.e., drying does not alter the amount of water adsorbed.

This has been done by taking washed fibre and pressing it to about 50% water content. Water is determined in the fibre by drying at 120°C, and some of the wet fibre is mixed with a solution of known concentration as in the dry fibre determination. A material balance gives the hygroscopic water.

Work is continuing on hygroscopic water with the aim of improving the accuracy of its measurement. The main source of error appears to lie in non-uniform water adsorption by the fibre.

References p. 198.
FACTORY

SUMMARY

Various factors involved in the direct method of cane analysis using a wet disintegrator have been investigated with a view to laying down a procedure for quality determination of small lots of cane.

The factors which have been investigated include:

a. preservation of shredded cane samples prior to analysis;
b. loss of water from cane during hammer-milling;
c. completeness of extraction of brix and pol and deterioration during wet disintegration;
d. water and fibre determination in cane;
e. brix determination in disintegrator liquor;
f. effect of hygroscopic water on cane analysis;
g. accuracy and reproducibility of methods of analysis.

The method favoured consists of wet-disintegrating 1 part of hammer-milled cane with 3 parts of water for 45 minutes using 3 blades, 6" overall length x 4/32" thick, on the bottom of a 1" shaft rotating at 5700 r.p.m. in a 4-gallon can. The resulting slurry is first strained through a 120-mesh screen and cooled to 25°C or below, and then filtered with Supercel filter aid giving a filtrate for determination of brix (by Geissler pycnometer) and pol (in a 400 mm. tube). A water determination on hammer-milled cane is done in a Spencer-type oven; fibre is also determined.

The analysis of cane is then calculated using either the water or the fibre figures and applying a correction factor for hygroscopic water of 0.992 (equivalent to 25% water adsorption on dry fibre).

ACKNOWLEDGMENT

The authors wish to express their appreciation to the Management of The Colonial Sugar Refining Company, Ltd., for permission to publish this paper. Also gratefully acknowledged are the contributions from the work of members of the Company's staff.

REFERENCES


DISCUSSIONS

D. H. Foster (Australia): The higher extraction of the very high speed machine might be due to the fact that by very fine subdivision of cane more sugar is extracted from the cells of the true fibre. These are distinct from the sugar storage cells. We have found 1 to 2% brix in some of the fibre cells. This may vary according to the speed of sugar manufacture in the plant.

M. A. Mascaro (Cuba): In the process developed by Vasquez, the cane was finely ground on carborundum stones. This gave very fine disintegration. The process was actually intended for the manufacture of paper and building boards, but the important point was that it did involve very fine grinding of the bagasse on carborundum wheels.

L. J. Rhodes (Hawaii): Relative to hygroscopic water, is this phenomenon also an influencing factor in crushing plant calculations?

C. W. Davis (Australia): I don't think it is necessary to think in that way. I believe all you need say is that the cane contains whatever fibre be determined plus some water which has to be allowed for - say 0.3%. Then Juice = 100 - 0.3 = fibre. I agree that this would disturb the existing crushing plant calculations.

Mr. Rhodes: If we disregard hygroscopic water in crushing plant calculations, then I think
that if we are using the wet disintegrator method for factory control we might disregard hygroscopic water in our determinations using the disintegrator too.

Mr. Davis: I do not suggest you disregard it. You can’t disregard the facts of life, and it is a fact that everything we know is consistent with the fact that for the purposes of determining pol % cane there is fibre and there is this 0.3% water and the rest is juice. If you don’t think along those lines you’re bound to be walking into error.

Mr. Rhodes: This machine may be adapted to secure analytical data for milling tests. How much time would be required for one man to make 5 determinations?

Mr. Davis: In the actual program, hammermilling would take 12 seconds and disintegration about 3/4 hour. Half an hour later you have the answer. If you want % water as well, the process will be longer unless you can use some quick water method. I would say that you can have results within a couple of hours, and in that couple of hours one could have an entire set of figures on which one could in fact run a mill.

Mr. Rhodes: In regard to “hot” vs “cold” extraction, it would appear to me that when extraction is complete in the “cold” it would surely be complete in the “hot”.

Mr. Davis: When you say that extraction is complete—that’s like seeking the ultimate truth you know.