FACTORY BALANCE SYMPOSIUM

A SOLIDS BALANCE INVESTIGATION

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Abstract

This paper is an abridged version of a thesis which was submitted to the Sugar Milling Research Institute in accordance with the requirements for the Sugar Technology Course.

The objectives of the thesis were to assess the magnitude and significance of errors inherent in the various methods of brix determination, and to investigate a method for calculating a true solids balance in a raw cane sugar factory. The methods of analysis and the facts revealed by the literature survey have been omitted from this paper.

Introduction

Brix is a parameter of major importance to factory control. This investigation was undertaken to assess the magnitude and significance of errors inherent in the various methods of brix determination.

A literature survey which was carried out revealed that a number of investigators throughout the world have looked into the problems associated with brix determinations. The effects of suspended solids, dilution, different techniques of oven drying, etc., have been related to the densimetric and refractometric methods of determining the dissolved solids in factory materials.

It might be justifiable to say that a comparison of methods for determining dissolved solids is unnecessary, since a vast amount of research has already been done in this field. Also, most investigators have been led to the same conclusions, that is, most methods for determining brix are influenced by factors such as suspended solids, non-sugars, etc., and that these factors introduce an error into factory control.

However, the erroneous influence that these factors have on factory calculations and performances has never been examined in any great detail. Examination in detail is therefore the aim and scope of the investigation.

The determination of total solids in factory materials thus enables a solids balance to be made. The procedure which has been adopted for this balance is based on:—

Solids Entering Factory	*Solids Leaving Factory*
Total solids in Mixed Juice	Solids in Filter Cake
solids in Lime added	Solids in Molasses
solids in Phosphate added	Solids in Sugar
solids in Flocculant added	
solids in Bagacillo added	

Due to the relatively small quantity of moisture in the chemicals added, the weights of these materials, as recorded by the factory, have been used.

The investigation was carried out at Darnall where the vacuum flotation clarification process was in use.

The Brix Balance

In other countries, e.g. Java, an attempt was made to introduce a brix balance by assuming no loss of sucrose during clarification, other than in filter cake. Despite the shortcomings of this method as used in Java, it is applied in the Hulett mills and recently there was an occurence at Empangeni which proved its value beyond any doubt.

For a number of years, the "non-Sucrose Ratio" as defined by Perk[19] has found a rather strong following.

$$\text{Non-Sucrose Ratio} = \frac{\text{Tons non-sucrose in final molasses + tons non-sucrose in sugar}}{\text{Tons non-sucrose in mixed juice}}$$

Let:

Tons sucrose in mixed juice	= J
Tons sucrose in sugar	= S
Tons sucrose in molasses	= M
Tons sucrose in combined losses	= L
Tons brix in mixed juice	= j
Tons brix in sugar	= s
Tons brix in molasses	= m
Tons brix in combined losses	= c

Then:

$$\text{Non-Sucrose Ratio} = \frac{(s+m-S-M)}{s+m+c-S-M-L}$$

$$= \frac{(s+m-S-M)}{(s+m-S-M)+c-L}$$

Substituting $W = (s+m-S-M)$

$$\text{Non-Sucrose Ratio} = \frac{W}{W+c-L}$$

The value of W and L are determined fairly accurately and, moreover, are more or less constant quantities. Obviously, variations in the non-sucrose ratio are caused by variations in "c" (tons brix in combined losses).

Assume: S = 75 Normal factory with 2.35% loss in cake+undetermined,
s = 76
M = 8 89% B.H.R.
L = 2 40 purity final molasses
m = 20

For a Non-Sucrose Ratio = 0.9.

$$\frac{W}{W+c-L} = \frac{13}{13-2+c}$$

c = 3.44%

For a Non-Sucrose Ratio = 0.8

$$\frac{W}{W+c-L} = \frac{13}{13-2+c}$$

c = 5.25%

It is therefore submitted that an evaluation of the brix loss will give a better understanding of factory performance than does the present non-sucrose ratio, although it is not suggested that the N-S ratio be discarded.

In the following report, one must differentiate between a *brix* balance and a *solids* balance as the latter includes all suspended (insoluble) solids.

Solids in Mixed Juice

Solids in mixed juice is the starting point of the solids balance and the accuracy of the balance therefore depends to a great extent on the determination of this factor.

Sampling of Mixed Juice

The determination of total solids in mixed juice entails two separate analyses. Firstly, dissolved solids on filtered juice, which can be found by a number of methods and, secondly, suspended solids. The accuracy of the latter determination is more dependent upon the sampling procedure than the actual method of analysis.

The mixed juice sampling device recommended for use in South African factories[1] does not give a representative distribution of sand and other insoluble material in the final sample. Two reasons can be given for this:

(a) If there is not sufficient turbulence in the scale tank before it tips, most of the heavy suspended particles will settle to the bottom of the tank. When the tank empties the juice which reaches the sampling tube first, will contain the greater proportion of sand, yet will represent only an insignificant portion of the total sample.

(b) The tube itself can be compared to a D.S.M. screen, especially if the holes are canted to one side to regulate the quantity of juice obtained per tip. It must be remembered that the recommended tube[1] consists of 15 holes, each 3/32" diameter × 1" pitch. Thus, a large proportion of the sand is deflected away from the tube.

To obtain a representative hourly sample, an entirely new sampling device would have to be employed. However, at the time of the investigation no such device was available. The necessity for an hourly representative sample becomes apparent in the solids balance when one calculates the tons of total solids in mixed juice, i.e. Tons suspended solids+Tons dissolved solids.

The same problem exists when weighing out the juice for the analysis of suspended solids, i.e. the difficulty to ensure an even distribution of insoluble material in the 200 g taken.

At the time of the investigation, there remained no choice but to analyse the sample as received by the available system. More attention will be drawn to this at a later stage in this paper.

Solids in Other Material Added

a. Lime

It is difficult to assess, from one week to the next, the quantity of lime consumed by the process. However a fairly accurate estimation can be made of the weight of lime used over a period of one *month*.

Also, the quantity of solids in lime added is relatively small compared to the total solids in mixed juice. (Solids in Lime % Solids in Mixed Juice = 0.4%).

Thus one can use the monthly estimated weight of lime without introducing any significant error in the solids balance.

b. Mono-Calcium Phosphate

This material is delivered in bags of known weight, and an accurate account of the number of bags used per shift is kept.

Solids in M.C.P. % Solids in Mixed Juice amounts to 0.17%. With an accurate monthly weight of M.C.P. available, the quantity of solids added in this material is easily arrived at.

c. Flocculant

The addition of this material is likewise carefully controlled, and amounts only to 0.004% of the solids in mixed juice.

Again the consumption of flocculant is known to the pound and this facilitates its use in the solids balance.

d. Bagacillo

This material is added to the muds leaving the clarifiers and although it was possible to measure the flow rate of the bagacillo in pounds per minute, a more reliable method of determining the quantity added had to be found.

The method finally decided upon is as follows:

The dry bagacillo content of the mixed juice varies only slightly from hour to hour. By analysing the mixed juice for dry bagacillo per cent a fairly accurate average can be found.

The same applies to the bagacillo content of the filter cake. Assuming that the bagacillo content of clear juice is negligible, the difference between bagacillo in filter cake and bagacillo in mixed juice will

give the amount of bagacillo added to the muds. This method is dependent on a reliable weight of filter cake.

The solids in the filtrate do not have to be taken into account as they are re-cycled continuously.

Solids in Material Leaving the Factory

a. Solids in Filter Cake

The determination of solids in filter cake necessitates both weighing the material and analysing for moisture. Unfortunately, no factories weigh all their filter cake but estimate the total output by weighing an occasional S.A.R. truck and road transport vehicle. The method in use at Darnall is as follows:

An individual truck or lorry is weighed every tenth time it collects filter cake and a record of the number of loads is kept. The average weight of every tenth load is taken as the weight per individual load. Although it is not accurate, this method had to be used for calculating the total solids balance. If this weight is overestimated then the total solids lost in undetermined will, of course, be underestimated, and vice versa.

b. Solids in Sugar

Due to the fact that the sugar is weighed accurately and that moisture determinations are done on samples from each truckload no difficulties are found in calculating tons of solids in sugar leaving the factory.

c. Solids in Molasses

The determination of solids in final molasses is done on a weekly composite sample using the Karl Fischer[8, 9, 10, 11] titrimetric moisture method. The analyses are done weekly by the staff of the Hulett's Central Laboratory.

Solids in Stock

The impossibility of determining accurately the quantity of solids in stock, necessitates the use of "Made and Estimated" figures for sugar and molasses.

Errors are frequently made in estimating the weekly stock and these will therefore give erroneous figures for solids in sugar and molasses made and estimated. However, by doing a solids balance on the monthly or to-date figures one can minimise the effect of these stock errors. Moreover they are, eventually, self-correcting.

Results of Investigation

The methods used in this investigation for calculating the total solids balance relies on the application of factors to the monthly or to-date figures supplied by the factory.

These factors are as follows:

1. The *average* difference between factory recorded mixed juice brix (spindle) and dry substance per cent filtered mixed juice. This enables one to arrive at an estimated dry substance per cent filtered mixed juice from the factory figures.
2. The average suspended solids per cent mixed juice is also applied to the factory recorded weight of mixed juice. (This analysis has since become part of the daily routine carried out at all Hulett factories.)
3. The average dry bagacillo per cent filter cake is used to estimate the quantity of bagacillo added to the muds from the factory recorded weight of filter cake.
4. An average figure for dry bagacillo per cent mixed juice enables one to apply this figure to the factory recorded weight of mixed juice.

Analysis of Mixed Juice

No. of Analyses	Spindle Brix		Dry Subs % Filtered M.J.	Suspended Solids % M.J.	Dry Bagacillo % M.J.	Spindle Brix Recorded by Factory
	Filtered	Unfiltered				
60	12.80	12.96	12.79	0.37	0.06	13.08

These figures are the arithmetic means obtained during the period of investigation. The last column shows the weighted mean brix recorded by the factory during the same period.

The disparity between the factory figure (13.08) and that recorded by the author (12.96) is due to two factors:

1. The hydrometer used by Darnall was checked against an S.M.R.I. calibrated spindle and found to be 0.1° out.
2. The technique used by the testers was found to be incorrect. They immersed the spindle into the jar as soon as the vessel was filled. This procedure leads to air bubbles adhering to the hydrometer.

This difference of 0.12 was consistent with each *hourly* sample and is therefore not due to the fact that the one figure is an arithmetic mean, while the other is a weighted mean.

Analysis of Filter Cake

Bagacillo		Filter Cake	No. of
Moisture %	Rate of Addition	Dry Bagacillo %	Tests
56.0	65 lb/min.	8.66	25

Filter cake was analysed for moisture in parallel with the laboratory and no difference was found. A check was made on the amount of evaporation occurring from the filter cake between the sampling point and the weighbridge. This was found to be negligible and only occurred from the top layer of cake in the truck. Thus, the mill figures for moisture per cent cake are used in the solids balance.

It was stated previously that in the solids balance, the quantity of dry bagacillo added to the muds is found by difference, i.e.:

Tons dry bagacillo added = Tons dry bagacillo in filter cake - Tons dry bagacillo in mixed juice.

To check the accuracy of this assumption, one can compare the quantity of bagacillo found by measuring the rate of addition (65 lb. per minute), with that found by analysing the filter cake for dry bagacillo per cent (8.66%). The latter figure should be greater than the former because of the bagacillo already contained in the mixed juice.

In fact, the difference between the two figures should equal the quantity of bagacillo in mixed juice (0.06% on juice).

The following figures will illustrate the above check on the accuracy:

Darnall—August 1968:

Tons mixed juice=184 776
Tons filter cake= 7 866

By Analysis

Tons dry bagacillo in filter=

$$\frac{8.66}{100} \times 7\,866 \doteq 681 \text{ tons}$$

Tons dry bagacillo in mixed juice=

$$\frac{0.06}{100} \times 184\,776 = 111 \text{ tons}$$

By difference tons bagacillo added ... = 570 tons

By Measuring the Rate of Addition

65 lb. per minute at 56.0% moisture is equivalent to ... ... 590 tons
Difference ... ... 20 tons

A similar calculation for September gives a difference of 16 tons.

We can therefore accept the analysis figures to be sufficiently accurate for the purposes of a monthly solids balance.

Calculation of the Solids Balance

The complete method of calculation is given in Annexure I. The results of this typical example are as follows:

Solids In		*Solids Out*	
Mixed Juice:			
Suspended Solids	684	Filter Cake .	1 862
Dry Substance	23 343	Molasses ...	4 209
Chemicals:		Sugar ...	17 883
Lime	95		
Mono-calcium Phosphate	42		
Separan ...	1		
Bagacillo	570		
Total (Tons)	24 735		23 954

Undetermined Solids Lost: 781 tons.

Undetermined solids lost per cent solids in clarified juice=3.41%.

The factory reported the following losses for the same period:

	Tons	*Per cent in Clarified Juice*
Brix	848	3.65
Sucrose	398	2.01

Discussion of Results

At the outset of this investigation it was realised that it would be difficult to draw concrete conclusions from the results obtained. However, it was felt that an investigation would at least reveal the shortcomings in more detail and thus provide sufficient information to enable us to orientate our way of thinking towards establishing a more reliable system for tracing losses in the factory.

The results of the balances are difficult to evaluate and lead one to a choice of two conclusions:

(a) The solids balance is as inaccurate as the simple brix balance as applied in all the Hulett mills.

(b) The solids balance, despite being more accurate analytically (because it is based on dry substance), has nevertheless given answers which are, for all practical purposes, the same as the brix balance. This, therefore, seems to indicate that both methods have the same degree of reliability.

Due to the following reasons, the former conclusion seems the more likely one:

1. It was mentioned earlier that the determination of suspended solids per cent mixed juice was known to be inaccurate due to the sampling procedure employed.
2. The weight of filter cake is more of an estimate than a true weight and can influence the final results considerably.
3. The application of "factors" to the Mill Report Sheets can only lead to reliable results if they are determined over a long period. That is, they should result from *routine* analyses so as to obtain weighted averages for the period under consideration.
4. The brix balance as applied in the Hulett mills assumes no loss of sucrose during clarification, other than in filter cake:
   Then:
   Tons sucrose in clear juice=tons sucrose in mixed juice - tons sucrose in filter cake.
   Tons brix in clear juice=

$$\frac{\text{tons sucrose in clear juice}}{\text{purity of clear juice}} \times 100$$

   These assumptions clearly give rise to the following inaccuracies:
   (a) Losses of sucrose do occur during clarification and the magnitude of these losses can differ widely from factory to factory.

(b) It is known that the presence of large quantities of insoluble solids cause an error in the determination of sucrose in mixed juice, and this error is carried forward into the brix balance.

(c) An apparent purity of clear juice (i.e. pol/brix ratio) is used to calculate the tons of brix in clear juice.

It would appear that our measuring tools have become too crude for this investigation and that the increase in factory efficiency has gone beyond the point of reliable information.

Conclusions

Although a step in the right direction has been taken by using dry substance instead of specific gravities and refractive indices, the solids balance, as presented in this report, requires refinement.

Empangeni Mill now lends itself to a more reliable investigation due to the following:

1. A sampling device has been designed and installed at Empangeni to give a representative sample of mixed juice for the determination of suspended solids.
2. A riffler apparatus has been designed and tested to enable sub-sampling of the mixed juice sample.
3. Empangeni has installed a Servo-Balans to weigh syrup.

A point to bear in mind is the possible introduction of the new cane payment system in South Africa. This system would make the water scales available for syrup weighing and once the syrup sampling problems have been solved, one would know what quantity of material is entering the boiling house.

If any further work is envisaged on this project, the following recommendations are tentatively put forward:

1. All analyses must be done on a routine basis so as to obtain weighted averages.
2. Filter cake must be weighed.
3. Correct sampling procedures must be adopted.
4. Syrup scales must be installed.
5. At present, it is doubtful whether any existing laboratory staff could handle the extra work load entailed and one would therefore require extra staff.

In view of these last-mentioned factors, an analogy to Lost Absolute Juice % Fibre is being sought for application in the boiling house. That is, one must attempt to express the sucrose losses in terms of a substance which remains a constant quantity (other than that which is actually lost) such as fibre in the milling tandem. At present, an investigation is being conducted by Hulett's Research and Development Department into the feasibility of a chloride ion balance.

If this proves successful, it will be possible to express sucrose as a ratio to chloride in all factory materials thus enabling one to pinpoint any losses.

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Although all these references are not used in this abridged report, they are given for the convenience of those wishing to make use of them.

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ANNEXURE I

CALCULATION OF THE SOLIDS BALANCE

The following factors are applied in the calculation :

1. The difference between factory recorded spindle brix and dry substance % filtered mixed juice = 13.08 — 12.79 = 0.29
2. Suspended Solids % Mixed Juice 0.37
3. Dry Bagacillo % Filter Cake 8.66
4. Dry Bagacillo % Mixed Juice 0.06

Although many balances were calculated, the following is given as a typical example.

BALANCE FOR DARNALL — AUGUST 1968

The complete method of calculation is given in this example:

(*a*) *Factory figures from Monthly Report Sheet*

Tons Mixed Juice weighed	184 776
Tons filter cake	7 866
Tons lime	95
Tons Mono-Calcium Phosphate	42
Tons Separan	1
Tons Sugar made and estimated	17 959
Tons Molasses made and estimated	5 231
Mixed juice spindle brix	12.97
Moisture % sugar	0.423
Karl Fischer solids % Molasses	80.47
Moisture % filter cake	76.33

(*b*) *Estimated Figures*

Dry substance % filtered mixed juice = 12.97 – 0.29 .. .. 12.68

(*c*) *Calculation of Balance*

(1) Tons suspended solids in mixed juice = $\frac{0.37}{100}$ x 184 776 .. .. = 684

(2) Tons actual mixed juice = tons mixed juice weighed – tons of suspended solids = 184 776 – 684 .. .. = 184 092

(3) Tons dry substance = estimated dry substance % mixed juice x (2) = $\frac{12.68}{100}$ x 184 092 .. .. = 23 343

(4) Tons total solids in mixed juice = tons dry substance + tons suspended solids = (3) + (1) = 23 343 + 684 = 24 027

(5) Tons dry bagacillo in filter cake = dry bagacillo % filter cake x tons filter cake = $\frac{8.66}{100}$ x 7 866 .. .. .. = 681

(6) Tons dry bagacillo in mixed juice = dry bagacillo % mixed juice x tons mixed juice weighed = $\frac{0.06}{100}$ x 184 776 = 111

(7) Tons dry bagacillo added to the mud = (5) – (6) = 681 – 111 .. .. = 570

(8) Tons total solids into clarifiers = total solids in mixed juice + tons lime + tons mono-calcium phosphate + tons Separan = 24 027 + 95 + 42 + 1 .. .. = 24 165

(9) Tons solids in filter cake = tons cake – tons moisture in cake = 7 866 – $\frac{76.33}{100}$ x 7 866 .. .. = 1 862

(10) Tons solids in mud leaving the clarifiers = Tons solids in filter cake – tons dry bagacillo added to mud = 1 862 – 570 = 1 292

(11) Tons solids in clear juice = tons solids into clarifiers (8) – tons solids in mud (10) = 24 165 – 1 292 .. .. = 22 873

(12) Tons solids in Molasses made and estimated = $\frac{80.47}{100}$ x 5 231 .. .. = 4 209

(13) Tons solids in sugar made and estimated = 17 959 – ($\frac{0.423}{100}$ x 17 959) .. .. = 17 883

(14)

Solids In			*Solids Out*	
Mixed Juice :	Suspended solids	684	Filter Cake	1 862
	Dry Substance	23 343	Molasses	4 209
Chemicals :	Lime	95	Sugar	17 883
	M.C.P.	42		
	Separan	1		
Bagacillo		570		
Total		24 735		23 954

Undetermined Solids lost = 781 tons.

The factory reported the following losses for this period

	Tons	*% in Clear Juice*
Brix	848	3.65
Sucrose	398	2.01

For discussion on this Paper, see page 50.