

THE DISTRIBUTION OF IMPURITIES DURING CRYSTALLISATION

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Abstract

The influence of certain changes in process techniques on the inclusion of gums, starch and ash in the crystal from Hulett's Mills is discussed.

Introduction

The distribution of sucrose through a sugar factory is the prime concern of the operating technologist. The percentage of the sucrose present in syrup which is crystallised into sugar forms the basis for formulae used to gauge boiling efficiency.

While sucrose remains the major interest from an economic standpoint, the distribution of certain impurities between sugar and final molasses becomes increasingly important as more and more emphasis is placed on sugar quality.

Ratios relating the weights of impurities in crystal and final molasses have been calculated for four factories, from data accumulated at Hulett's Central Laboratory during the past two seasons. Ratios for sulphated ash have been determined for both the 1966/67 and 1967/68 seasons, but data for gums and starch were available for the 1967/68 season only.

The Factories

The following are brief descriptions of the pertinent manufacturing techniques used by the factories considered:

1966/67 SEASON

- Empangeni* Milling followed by simple defecation. Full remelt boiling system.
- Felixton* Milling, simple defecation, and partial remelt system (all C-sugar and part B-sugar remelted).
- Amatikulu* Milling, simple defecation and full remelt.
- Darnall* Milling followed by simple defecation until November when the experimental vacuum clarification system was introduced. Full remelt until November, then single magma system.

1967/68 SEASON

- Empangeni* Commenced the season with milling and simple defecation. The B.M.A. diffuser was brought into operation in mid-August while vacuum clarification of a portion of the juice has been operating intermittently since July. Single magma boiling for the whole season.
- Felixton* Milling, simple defecation and partial remelt.
- Amatikulu* Milling plus simple defecation until mid November, when vacuum clarification of part of the mixed juice was

introduced. Full remelt system until the end of December, followed by partial remelt.

Darnall Milling plus vacuum clarification of all mixed juice. Single magma boiling system.

Method of Calculation

The method used for calculating the inclusion ratios can best be illustrated by a worked sample.

Tons sugar produced, T	5572	
sugar pol, s	97.23°	
sugar moisture, m	0.72%	
Tons molasses produced, M	1210	
molasses solids	82.9 %	
molasses purity, p	44.4	
molasses ash	15.93%	
molasses starch	0.78%	
molasses gums	3.28%	
Analysis of affinated sugar (crystal):			
crystal ash	400	ppm
crystal starch	340	ppm
crystal gums	730	ppm

Tons sucrose in sugar		
Ts	5417.7
Tons solids in sugar		
Tb=T(100-m)	5531.9
Tons non sucrose solids in sugar		
Tb-Ts	114.2

Assuming that the purity of the film is the purity of final molasses, then

Tons sucrose in molasses film		
$(Tb - Ts) \times \frac{p}{100 - p}$	91.2

Tons crystal		
Ts-91.2	5326.5

Tons film		
T-5326.5	245.5

Tons final molasses		
M+245.5	1455.5

	Tons		
	Ash	Starch	Gums
5326.5 tons crystal contains (A)	2.13	1.81	3.89
1455.5 tons molasses contains	231.86	11.35	47.74

Total available for inclusion.. (B)	233.99	13.16	51.63
Inclusion ratios $\frac{100 A}{B}$..	0.91	13.75	7.53

The inclusion rate for starch free gums is

$$\frac{3.89 - 1.81}{51.63 - 13.16} \times 100 = 5.41$$

Assumptions Made

1. Laboratory affinated sugar is assumed to be crystal. This assumption is incorrect since crystal is, by definition, 100% sucrose, whereas the impurities in affinated sugar form the basis for this paper. However, as the assumption is used only for calculating crystal tonnages, the errors involved have little effect on the final ratios.

In the worked example, non sucrose % crystal
 = 0.04 + 0.07 %
 = 0.11 %

Therefore, actual sucrose per cent crystal
 = 99.89 %

Now

100 tons sugar
 (97.23 sucrose, 0.72 water, 2.05 non-sucrose)

X tons crystal	(100 - X) tons film
.9989 X sucrose	97.23 - 0.9989 X sucrose
.011 X non-sucrose	2.05 - 0.0011 X non-sucrose

Since the ratio of sucrose to non-sucrose in the film is 44.4
 100 - 44.4

It can be calculated that X = 95.78

	Crystal	Film	Total Molasses
Actual tons	5,336.9	235.1	1445.1
Assumed tons	5,326.5	245.5	1455.5
Error	0.19 %		0.72 %

2. By definition raw sugar is composed of crystal surrounded by a film of the same purity as final molasses. Strictly speaking the moisture content of the film should be taken into account when calculating the weight of potential final molasses which surrounds the crystal.

If

- Tb = tons solids in sugar
- Ts = tons sucrose in sugar
- p = purity of final molasses
- m = solids % final molasses

Tons potential final molasses in film

$$= \frac{10,000 (Tb - Ts)}{m (100 - p)}$$

Using this formula in the worked example:

Tons potential final molasses in film .. 247.8

Therefore

True tons final molasses	1457.8
Assumed tons final molasses	1455.5
Error	0.16 %

The true inclusion ratio can be calculated if neither assumption is made:

	Ash	Starch	Gums
True ratio	0.92	13.84	7.60
Assumed ratio	0.91	13.75	7.53

The magnitude of errors introduced is small compared to the time saved during calculation.

3. The third assumption is that the total quantity of an impurity which is available for inclusion in the crystal is the sum of the quantities of that impurity in sugar and final molasses. This assumption, basically that solids in sugar plus solids in molasses equals solids in syrup, makes no allowance for undetermined losses (or gains) during the boiling process.

It is difficult to suggest a practical way of overcoming the problem as factories do not weigh syrup, and no regular analyses of the impurities in the syrup have been made. As the size of the errors involved cannot be established they have to be ignored and the assumption has been taken as a definition for the purposes of this investigation.

Discussion

The factors which may influence the inclusion of impurities in crystal can be broadly divided into two groups; those which cannot be controlled by the factory and those related to the techniques of manufacture.

The variety, age and quality of cane crushed, seasonal and climatic conditions and geographical environment, are factors which may combine to produce impurities with a greater or lesser affinity for syncrystallisation. The influence may be direct, by a change in the physical properties of the impurities, or indirect, if the amount of impurity included in the crystal is proportional to the quantity available for inclusion. While these influences are usually beyond the Miller's control, they must be taken into account when comparing results from different factories.

The second group of factors include those related to the techniques of extraction, clarification and crystallisation. The extraction process determines the quantity and may influence the physical characteristics of the impurities of mixed juice. By removing varying amounts of individual impurities from mixed juice, the clarification station determines the quantities available for inclusion during crystallisation. In the final stage the inclusion level is influenced both by the boiling system and by the equipment available.

TABLE I
Inclusion Ratios for Ash 1966/67 and 1967/68 Seasons

	Empangeni		Felixton		Amatikulu		Darnall	
	ppm in crystal	ratio	ppm in crystal	ratio	ppm in crystal	ratio	ppm in crystal	ratio
1966/67								
May	660	1.05	760	1.45	470	0.78	700	1.09
June	570	1.11	650	1.42	470	1.14	490	1.15
July	800	1.57	570	1.25	320	0.73	420	0.84
August	740	1.36	730	1.45	460	1.04	550	1.11
September	810	1.57	850	1.89	500	1.13	570	1.33
October	850	1.70	990	2.06	530	1.17	620	1.43
November	700	1.39	750	1.56	480	1.08	1,110	2.49
December	910	1.69	960	1.71	480	1.04	1,070	2.42
January	1,010	1.68	1,040	1.90	680	1.35	950	1.90
February	1,620	2.45	1,170	2.04	600	1.19	960	1.68
Mean \bar{x}		1.557		1.673		1.065		1.544
s		0.370		0.272		0.176		0.539
100s		37.0		27.2		17.6		53.9
\bar{x}		23.76		16.26		16.53		34.91
1967/68								
May			810	1.37	600	1.26	790	1.74
June	1,680	3.16	760	1.49	460	1.01	810	1.67
July	1,490	2.84	820	1.66	420	0.94	890	2.08
August	1,680	3.08	950	1.94	410	0.91	920	1.74
September	1,680	2.78	810	1.55	450	0.96	1,000	2.01
October	1,440	3.13	820	1.70	400	0.83	1,140	2.20
November	1,570	2.39	660	1.47	370	0.79	920	1.95
December	1,360	2.36	650	1.36	410	0.85	1,030	2.09
January	1,350	1.89	600	1.15	420	0.86	1,070	2.08
February	1,500	2.06	980	1.73	470	1.00	1,060	1.95
Mean \bar{x}		2.632		1.542		0.941		1.951
s		0.447		0.214		0.126		0.167
100s		44.7		21.4		12.6		16.7
\bar{x}		16.98		13.88		13.39		8.56
1966/67 and 1967/68								
Mean \bar{x}		2.07		1.61		1.00		1.75
s		0.674		0.252		0.164		0.448
100s		67.4		25.2		16.4		44.8
\bar{x}		32.62		15.67		16.35		25.63

The number of variables is too large to allow significant conclusions to be drawn from simple statistical analysis of the results of one or two seasons. If the accumulation of data can be continued, it may be possible to determine seasonal trends for each mill. Anomalies outside the trends could then be investigated.

From the point of view of technique, Felixton has remained the most constant of the four factories reviewed over the past two seasons. This factory has the most simple manufacturing process of the four; milling, simple defecation and partial remelt. If differences in "uncontrollable factors" are not considered, useful speculations may still be made by comparing the ratios from other mills with those of Felixton.

Ash

Over two seasons the inclusion ratios for ash at Felixton ranged from 1.15 to 2.06 with the mean 1.61 and standard deviation of 0.252, 15.67% of the mean.

Amatikulu, where process conditions remained stable until the middle of the second season, recorded ash inclusion ratio figures ranging from 0.73 to 1.35, with the mean 1.00 and standard deviation of 0.164, 16.35% of the mean.

The standard deviation for ash ratios over two seasons at Empangeni was 0.674, 32.62% of the mean; while the equivalent figures for Darnall were 0.448 and 25.63%. Clearly the process changes at these mills influenced the inclusion of ash in the crystal significantly.

In the case of Darnall the changes following the adoption of vacuum clarification and, more important, the dropping of the remelt system, can be seen from Table 1. The ratio for October, 1966, 1.43, increased to 2.49 during November, the first month of vacuum clarification and no remelt.

At Empangeni the changes appeared with the new season. A mean inclusion ratio of 1.56 for the 1966/67 season increased to 2.63 for the 1967/68 following the dropping of remelt. While the inclusion

ratios for ash at Empangeni dropped in the latter half of this season the drop cannot be ascribed with any certainty to the changes in extraction and clarification techniques as there is a parallel trend at neighbouring Felixton. Conclusions concerning the influence of diffusion on the ash content of crystal would be premature.

The salient feature of the ash inclusion ratios is the benefit of remelting. Even the partial remelt system used by Felixton results in considerably less ash being included in the final sugar, while the full remelt system can yield sugar with a very acceptable crystal ash content. The Table below summarises the average crystal ash contents of the sugars for the past two seasons.

		Ash in Crystal (ppm)			
		EM	FX	AK	DL
1966/67	850*	840†	480*	520*	'till Oct.
1967/68	1570	790†	430*	970	

*Full remelt for greater part of season.

†Partial remelt.

Gums

There are pronounced seasonal trends in the gum inclusion ratios for both Felixton and Amatikulu, with maximum values in the period September to December. The trend is also indicated by ratios for the inclusion of starch-free gums (Table 4) but the decrease in these ratios at the end of the season is more noticeable in the case of Amatikulu, and may be connected with the introduction of vacuum clarification.

The gum inclusion ratios for Empangeni show no pronounced seasonal trend apart from a drop in February. Darnall ratios exhibited a similar drop in February following a slight increase through the season.

The influence of various process changes on the inclusion of gum in crystal is difficult to assess from the figures available. The slight evidence, already mentioned, that vacuum clarification may have helped to reduce the ratios at Amatikulu, is not substantiated by results at Empangeni. Inclusion ratios at Darnall and Amatikulu were of the same order, both being somewhat lower than the ratios for Felixton.

Whether the lower degree of inclusion is due to remelt, at Amatikulu, and vacuum clarification at Darnall, or to "uncontrollable factors" cannot be determined. There is, however, some evidence to

show that the remelt process reduces the inclusion ratios for starch-free gums. The mean values for Amatikulu (full remelt) and Felixton (partial remelt) are lower than the means for the two factories using the magma process.

Starch

Apart from a drop in February, the starch inclusion ratios for Felixton showed minor variations, the standard deviation being 2.38, about the mean of 20.6. Amatikulu starch ratios decreased through the season, a more rapid drop in the last four months again indicating the possible influence of vacuum clarification.

It is in the case of Empangeni that changes are most marked. Following the introduction of diffusion and vacuum clarification the starch content of the crystal dropped from 600 to 400 ppm, while the inclusion ratio decreased from 26 to under 20. Since the vacuum clarification was used intermittently, to treat only a portion of the total juice, it appears likely that the major cause of the reduction of the starch content of Empangeni crystal has been the adoption of diffusion.

Darnall has been the only Hulett Mill to employ the vacuum starch removal process with real success. There is little to learn from the inclusion ratios at Darnall, but the efficiency of the process can be judged from the improvement in the starch content of the crystal from 375 ppm for the period May to October, 1966, to 145 ppm for the 1967/68 season.

The influence of remelt on starch inclusion ratios is not clear. While Amatikulu recorded the lowest ratios, with a mean value of 15.3, Felixton recorded the highest, if the June and July values for Empangeni are omitted.

Conclusions

Inclusion ratios, used in conjunction with the actual impurity content of crystal, can be a useful guide to the operating efficiency of a new technique. If controls could be kept constant, it should be possible to use measurement of impurity inclusion to test the comparative efficiency (from a standpoint of sugar quality) of vacuum pans or pan boiling systems.

The remelt system can produce sugars with satisfactory crystal ash contents, while the system may reduce to some extent the amount of gum and starch in the crystal. Vacuum clarification is an efficient technique for removing starch.

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TABLE IV
Inclusion Ratios for Starch Free Gums, 1967/68

	Empangeni		Felixton		Amatikulu		Darnall	
	ppm in crystal	ratio	ppm in crystal	ratio	ppm in crystal	ratio	ppm in crystal	ratio
1967/68								
May			297	3.5	219	3.4	498	9.0
June	825	11.3	480	7.0	483	6.6	509	7.9
July	586	8.9	390	6.6	366	6.0	609	8.4
August	796	11.5	481	8.0	480	8.2	547	7.0
September	843	11.3	584	9.8	599	9.8	729	10.1
October	634	10.4	517	9.5	472	8.4	582	8.1
November	1,137	12.9	673	11.5	634	9.2	828	10.7
December	1,169	13.8	393	8.8	333	7.8	496	9.9
January	992	11.0	869	11.9	535	7.7	1,042	12.4
February	990	8.4	782	7.7	570	6.7	880	7.2
Mean \bar{x}		11.06		8.43		7.38		9.07
s		1.63		2.34		1.74		1.60
100s								
\bar{x}		14.74		27.76		23.58		17.64

Discussion

Mr. J. de St. Antoine (Mauritius): I would like to know a little more about the single magma boiling system as used by Darnall and Empangeni. I would also like to know whether single or double cured C-sugar is used for making magma.

Mr. R. Robinson: At Darnall, a magma of double cured C-sugar is used as a footing for A and B boilings. I would also like to mention that Darnall produces low pol export sugar by cutting down on the wash in the A centrifugals, and not by the usual South African method of coating a good crystal with C molasses. We have found that the quality of the sugar produced by our method is satisfactory.

Mr. Jennings: The boiling system will have a very considerable bearing on impurity inclusion. The use of a magma system based on single or double cured C-sugar will probably result in more inclusion of impurity than a remelt system, regardless of the clarification and extraction process employed.

Mr. Phipson: At Felixton, water is boiled in the evaporators at the start-up each week, to provide condensate for the boilers. As a result, early syrups are often low in brix. I wonder whether the dilute syrups, and also the fact that the syrups early in the week have been concentrated from clarified juice which has been held in the clarifiers for over 24 hours, will affect the inclusion of impurities in the crystal.

Mr. Jennings: I doubt very much whether the dilute syrup will affect inclusion. The point about 24-hour old clarified juice is a good one, I am afraid I have no answers at present!

Mr. de St. Antoine: Is there a relationship between the inclusion of impurity in crystal and grain size. We have found that small grained sugars appear to contain less impurities.

Mr. Jennings: I have no data on this point at this stage. I believe that the Berlin Institute are investigating the subject, while a recent Australian paper on inclusion of mother liquor following erosion of crystal faces would support Mr. de St. Antoine's findings.

Mr. Perk: I am distressed to hear that Mr. Robinson is not over concerned about the quality of low pol export sugar.

Mr. Robinson: I must hasten to assure Mr. Perk that I am most concerned about the quality of low pol export sugar, but with the equipment available at Darnall it is more practical to produce the sugar by the method I described than by coating a high quality crystal.

Mr. Alexander: The answer may lie in treating the final molasses before applying it to the crystal. If the ash and colour could be reduced by some means, an all round good quality sugar could be produced. Hulett's Research are looking into the problem.

Dr. Matic: I must again remark on the accent which is being placed by overseas technologists on the basics of clarification. To my mind an investigation of this kind depends upon the standardisation of basic techniques of clarification and boiling.

Mr. Jennings: It was mentioned in an earlier discussion that Huletts were looking into a project for standardising clarification techniques. What progress has been made with the S.M.R.I. investigation into control of pan boiling?

Mr. Warne: The S.M.R.I. investigations are continuing, but many difficulties have been encountered. I doubt whether the use of inclusion ratios to judge pan boiling efficiency will ever be possible.

Mr. de St. Antoine: In Mauritius we have carried out most of our investigations into pan boiling using a laboratory vacuum pan. The results obtained are usually far more reproducible than is the case with factory pans.

Dr. Matic: We have found that one of the major problems with laboratory pans is the difficulty experienced in trying to produce a "bad sugar", even from low purity syrup the crystal produced is very pure. For the purposes of determining the amount of impurity which would be included in the crystal, a laboratory pan would not be so useful.

Mr. Alexander: The author is to be congratulated on making an attempt to use data which has been accumulated from semi-routine laboratory analyses. Too often data is recorded, filed and never used. I think that the paper has once again shown us how little we know about the path of impurities through our clarification and crystallisation stations.