EXHAUSTION AND COLOUR INVESTIGATIONS IN A-MASSECUITE

By L. M. S. A. JULLIENNE
Sugar Milling Research Institute

Abstract

The effect of the A-massecuite brix at strike on pan exhaustion performance was determined under industrial conditions. The additional crystallisation obtained on cooling in a crystalliser was measured. The change in colour of the A-sugar crystal during its growth in pans and crystallisers and the level of colour transfer in the A-massecuites was measured.

Introduction

Many developments in the last few years in the C-stations of South African mills have led to a marked improvement in the exhaustion of C-massecuites. In contrast, no noticeable improvement in the exhaustion performance of the A-massecuites has been achieved. Jenkins,[1] one of the many authors to discuss the importance of high grade exhaustion, wrote that if improvements are desired in the overall recovery of a factory, it is better to start improving the high-grade work first and contemplate major alterations to the low-grade station, only when satisfied that the maximum practicable recovery is being obtained at the preceding stations. Lamusse[2] is of the opinion that a more positive approach to boiling house efficiency is obtained if, in addition to loss control, insistence is placed on exhaustion of high purity products. It is evident that the lack of emphasis placed by local technologists on high grade exhaustion, may be detrimental to sucrose recovery.

Another aspect which has not received much attention concerns the effect of exhaustion on the sugar crystal quality. These considerations have led to the investigations described in this report, in which colour has been the quality parameter considered.

Experimental Procedure

The topics of the investigation were:

- the effect of the A-massecuite brix at strike on the pan exhaustion performance.
- the exhaustion of A-massecuites on cooling.
- the change in the colour of A-crystal during its growth in pans and crystallisers.
- the level of colour transfer in A-massecuites.

The pan exhaustion tests were carried out at four mills (GH, UC, SZ and UK) which all used conventional batch-type pans supplied with vapour one in the pressure range 30 to 70 kPa g. The boiling cycle was started on a B-magma footing and the A-crystal was grown to its desired size (SGS ± 0,70 mm) in a series of intermediate boilings (2 or 3 depending on the mill) followed by the final strike boiling. The range of brixes obtained during the tests was not deliberate and occurred under normal factory operation. The crystalliser cooling tests were carried out at UK where the last water-cooled crystalliser (45 m^3 capacity) in a series arrangement of six was operated batchwise during the investigation.

The change in crystal colour during growth was investigated at UC, SZ and UK. At UC and UK, crystals at different stages of growth were obtained by sampling the massecuite at "cut overs" between successive boilings. At SZ, one of the pans was fitted with a 2 litre vacuum sampler so that massecuite could be sampled while boiling was in progress. The survey on the crystal colour change during growth in a crystalliser was carried out at UK. The massecuite was immediately centrifuged and washed in a Christ Universal Junior III electric laboratory centrifuge to produce a sugar of about 99° purity. The immediate separation of mother liquor was found to be essential to eliminate nucleation and prevent the exhaustion of the molasses in the sample. The A-crystal was obtained by affination at the Sugar Milling Research Institute. The affination and colour (ICUMSA 420 nm) measurements were carried out according to the procedure described in the Laboratory Manual for South African Factories.

The colour transfer factor which was used to evaluate the level of colour transfer between the mother liquor and the crystal was developed by Chiu and Sloane[3] in Hawaii. The factor is derived from a differential colour and material balance between the crystal and the mother liquor and is expressed as follows:

$$k_p = \frac{\log\left[1 - \frac{JRa_c}{Sa_j}\right]}{\log\left[1 - R\right]}$$

where k_p = colour transfer factor
J = A-massecuite purity
S = A-crystal purity
R = A-massecuite exhaustion
a_j = colour of A-massecuite (colour units on brix)
a_c = colour of A-crystal (colour units on brix)

In the derivation of the above formula, it is assumed that at any instant in the boiling process, the colour intensity of the sucrose deposited on the crystal is a constant fraction (k_p) of the colour intensity of the mother liquor from which crystalisation is taking place, that is:

colour of deposited sucrose = k_p × colour of mother liquor

Results

A-Massecuite characteristics

The characteristics of the A-massecuite at GH, UC, SZ and UK during the period of the tests are shown in Table 1.

TABLE 1

Characteristics of A-Massecuite at GH, UC, SZ and UK

	Apparent purity	Strike temperature	G + F* / ash
GH	88,5	60	0,5
UC	87,5	54	0,6
SZ	83,5	65	0,8
UK	87,5	64	0,8

* G + F/ash is the ratio of glucose % + fructose % to ash % in final molasses.

The effect of massecuite brix at strike on pan exhaustion performance

Three different parameters are commonly used to evaluate exhaustion performance, namely the purity difference between massecuite and mother liquor, the crystal content and the exhaustion.

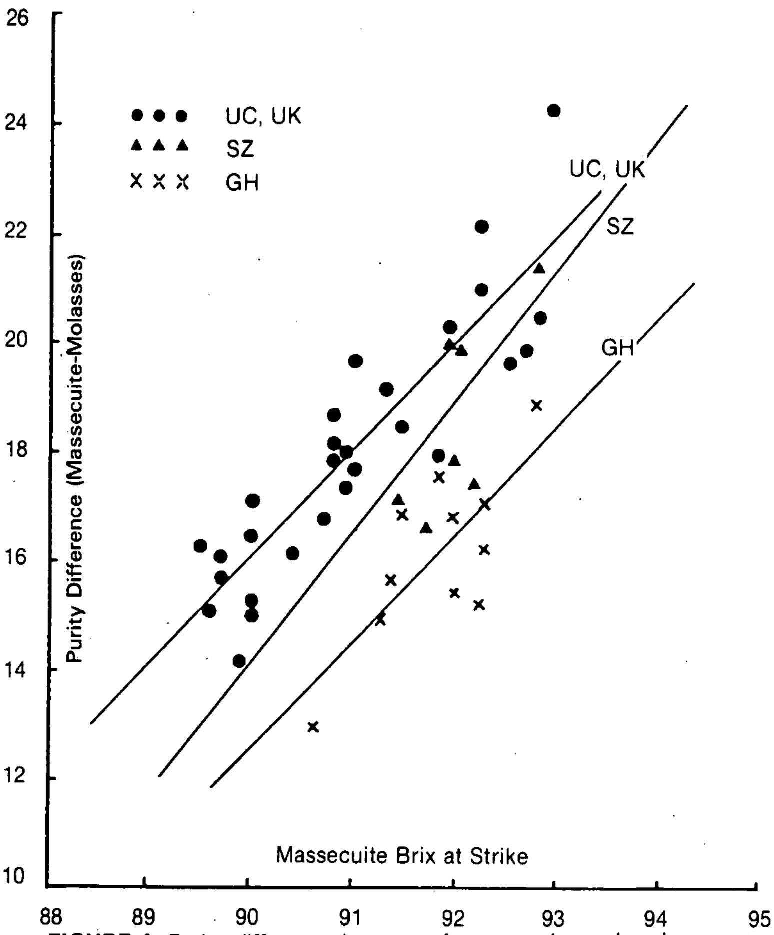

FIGURE 1 Purity difference between A-massecuite and molasses at strike

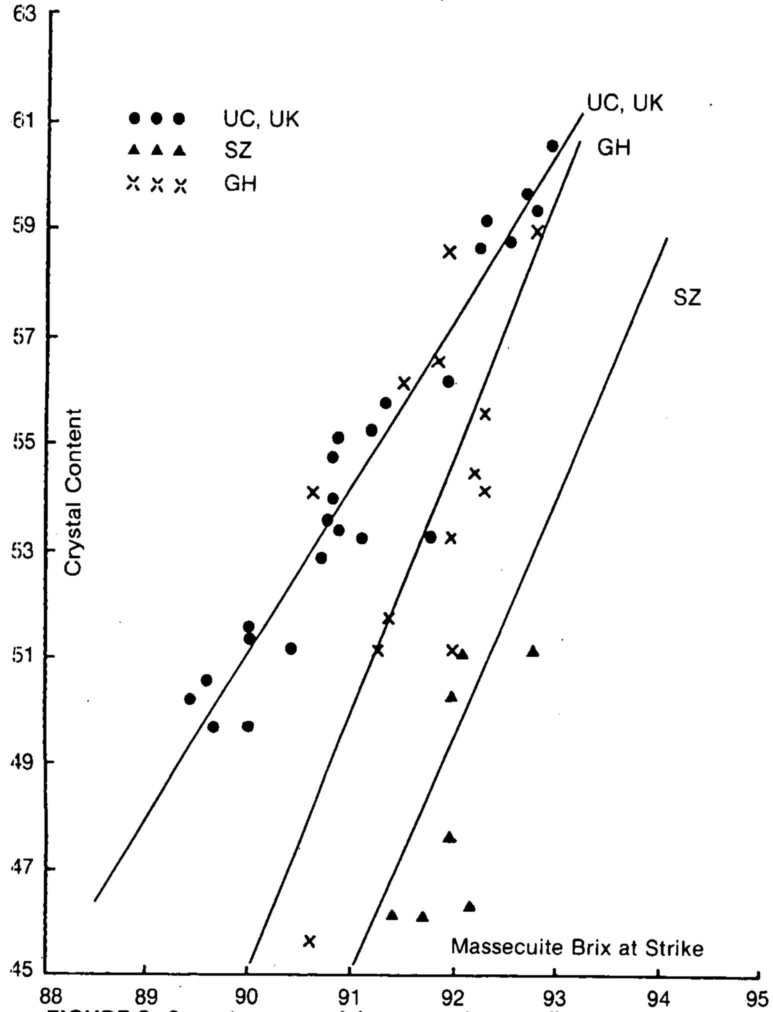

FIGURE 2 Crystal content of A-massecuite at strike

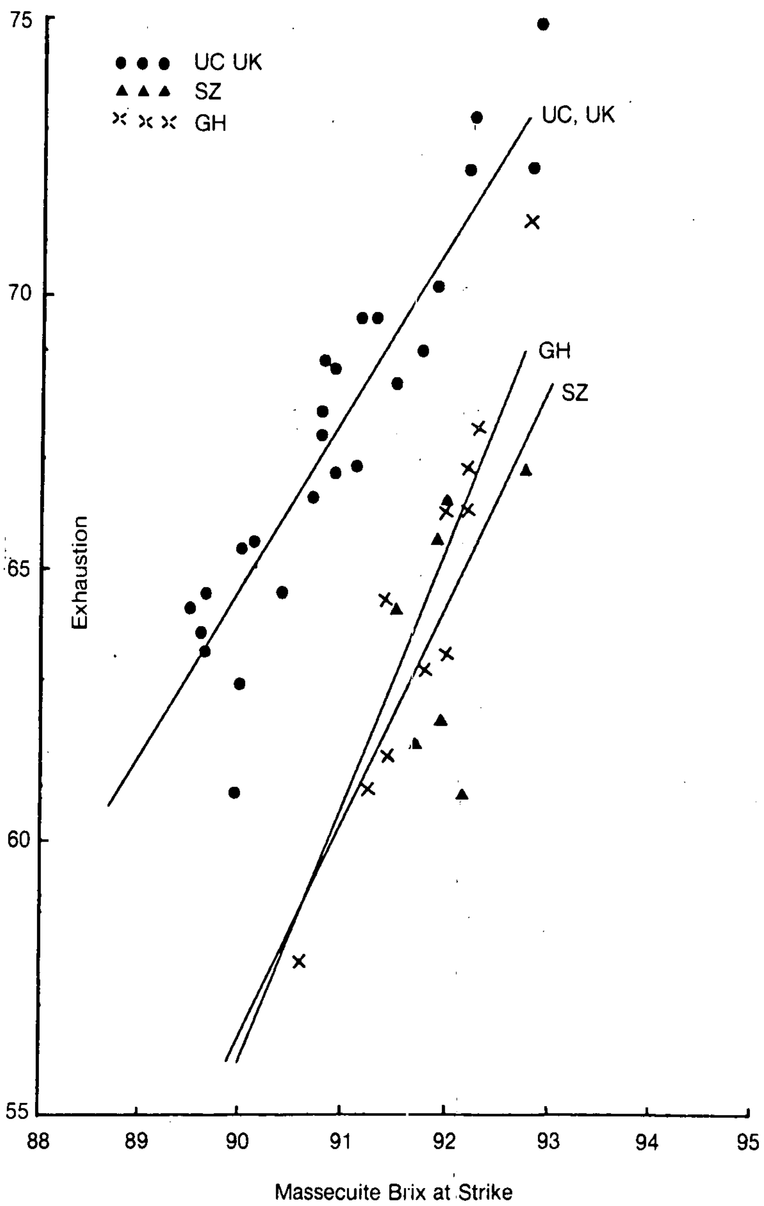

FIGURE 3 Exhaustion of A-massecuite on strike

The positive effect of the brix on these parameters is shown in Figures 1, 2 and 3. In the range of brixes obtained during the tests, the relationships between the massecuite brix and the performance parameters were found to be linear and the equations are given in Table 2.

TABLE 2

Regression equations between A-Massecuite Brix (B_a) at strike and exhaustion performance

	Purity difference	Crystal content	Exhaustion
GH	$= 2{,}00\ B_a - 167{,}7$	$= 4{,}89\ B_a - 395{,}1$	$= 4{,}71\ B_a - 366{,}9$
UC,UK	$= 1{,}97\ B_a - 161{,}5$	$= 3{,}11\ B_a - 229{,}0$	$= 3{,}13\ B_a - 216{,}9$
SZ	$= 2{,}45\ B_a - 206{,}4$	$= 4{,}24\ B_a - 340{,}6$	$= 4{,}01\ B_a - 304{,}5$

All purities are apparent and crystal content and exhaustion are calculated as follows:

$$\text{Crystal content} = \frac{(\text{pty mcte} - \text{pty mol})}{(100 - \text{pty mol})} \times \text{bx mcte}$$

$$\text{Exhaustion} = \frac{10\,000\ (\text{pty mcte} - \text{pty mol})}{\text{pty mcte}\ (100 - \text{pty mol})}$$

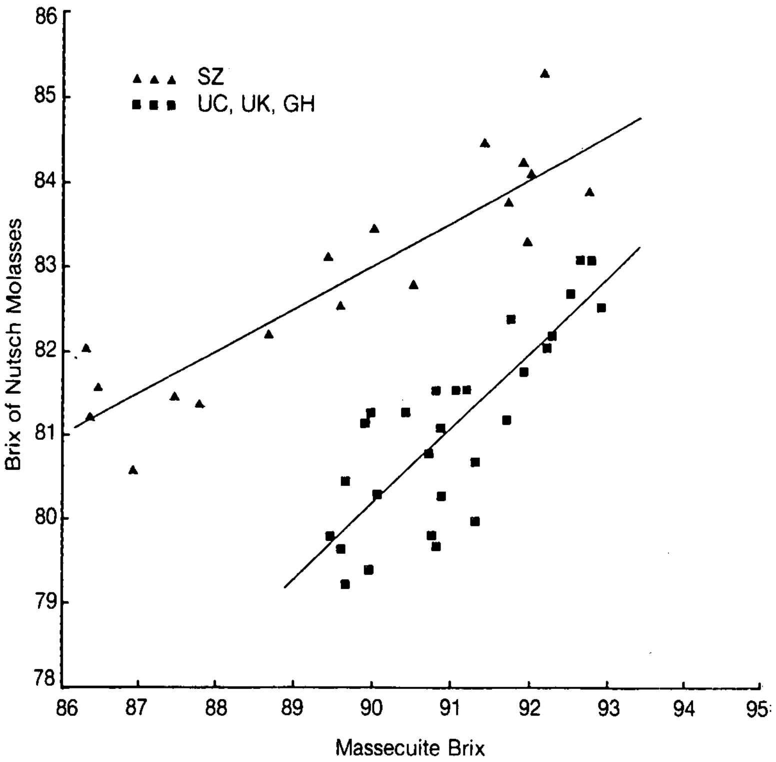

FIGURE 4 Brix of nutsch molasses at different A-massecuite brixes

It was found that a change in massecuite brix resulted in a corresponding change in the brix of the mother liquor (see Figure 4). The linear relationship between the two brixes is as follows:

For GH, UC and UK,
molasses brix = (0,91 × massecuite brix) − 1,54
(n = 26, r = 0,83)

For SZ,
molasses brix = (0,52 × massecuite brix) + 36,19
(n = 18, r = 0,90)

Exhaustion performance of A-massecuite on cooling

The results of the cooling tests carried out in a 45 m^3 capacity water-cooled crystalliser at UK are given in Table 3. The rate of cooling of the massecuite was about 1°C h^{-1} in all the tests.

TABLE 3

Results of A-Massecuite cooling tests at UK

Month of test	Massecuite				Molasses purity	
	Brix	Pty	Temp. °C	Temp. °C	In	Out
July	92,4	86,4	62	56	64,9	63,2
August	92,2	87,9	66	51	68,4	64,9
August	91,7	88,1	60	53	68,0	66,6
September	92,6	87,7	67	50	68,3	63,9
September	91,7	87,8	66	50	67,0	64,1
September	91,8	86,5	66	52	66,5	63,6
December	89,5	86,7	62	44	72,9	68,1
December	88,5	85,3	61	42	73,5	68,4

The improvement in exhaustion as expressed by the three performance parameters already mentioned above, is given in Table 4.

TABLE 4

Exhaustion performance of A-Massecuite on Cooling (8 tests)

Molasses purity drop per °C	Crystal content			Exhaustion		
	In	Out	Increase per °C	In	Out	Increase per °C
0,28	57,9	59,5	0,27	72,1	74,1	0,32
0,24	56,9	60,4	0,23	70,2	74,5	0,29
0,20	55,2	59,0	0,20	71,3	73,1	0,26
0,26	56,7	61,0	0,25	69,8	75,2	0,32
0,19	57,8	60,5	0,17	71,8	75,2	0,21
0,21	54,8	57,8	0,21	69,0	72,7	0,26
Average 0,23	56,5	59,7	0,22	70,7	74,1	0,28
0,27	45,6	52,2	0,37	58,7	67,3	0,48
0,27	39,4	47,3	0,42	52,2	62,7	0,55
Average 0,27	42,5	49,8	0,49	55,5	65,0	0,52

Colour changes during crystal growth

The colour (ICUMSA 420 nm units) of the crystal at different stages of growth during pan boiling is shown in Table 5 for UC and UK where there were three and four boilings respectively in a full growth cycle.

TABLE 5

A-crystal colour during pan boiling at UC and UK

	No. of tests at UC				No. of tests at UK			
	1	2	3	4	1	2	3	4
B-magma	850	812	895	1056	1074	980	–	–
First "cut over"	422	–	454	801	396	290	–	–
Second "cut over"	371	367	369	614	335	280	–	–
Third "cut over"	–	–	–	–	354	255	1 318	750
Final A-mcte	528	540	333	863	556	510	1 987	987
% colour increase in last boiling	+42	+47	−10	+41	+57	+100	+51	+32

At SZ, the colour of the A-crystal was measured at different stages of the final boiling only. The results are given in Table 6.

TABLE 6

Colour of crystal during final boiling at SZ

Boiling stage	Test 1	Test 2	Test 3
Start	935	835	1 170
Before brixing	1 210	805	1 055
At strike	1 350	1 305	1 070
% colour increase	+44	+56	−9

Three tests carried out at UK to determine the change in colour of the A-crystal during exhaustion in a water-cooled crystalliser gave the results shown in Table 7.

TABLE 7

A-crystal colour during exhaustion in a crystalliser

	Test 1		Test 2		Test 3	
	In	Out	In	Out	In	Out
Retention time (h)	10		19		20	
Mcte. temp. (°C)	58	41	65	44	61	42
A-mol purity	66,7	61,7	75,6	68,1	73,5	68,4
A-crystal colour	512	514	721	778	679	622

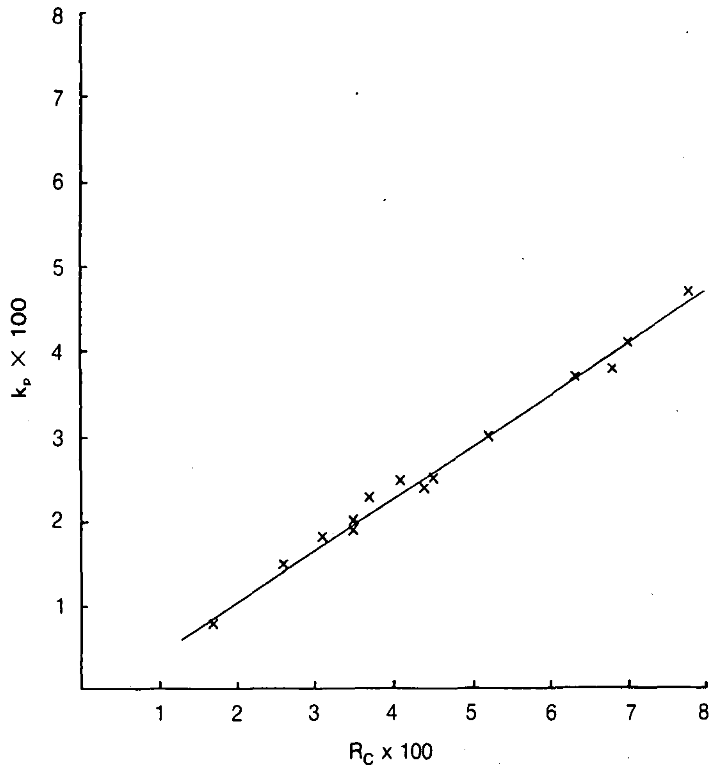

FIGURE 5 Correlation curve between colour ratio (R_c) and colour transfer factor (k_2)

Level of colour transfer in A-massecuites

The two parameters used to quantify the level of colour transfer during pan boiling are the colour transfer factor (k_p) which has been described and the ratio (R_c) of colour in crystal to colour in massecuite (both colour values expressed in ICUMSA 420 nm units on brix). Both coefficients are highly correlated as shown in Figure 5, the relationship being

$$k_p = 0{,}61\ R_c - 1{,}4 \times 10^{-4}$$

Low values of both coefficients correspond to a low level of colour transfer from mother liquor to crystal and vice versa. In other words, an efficient colour separation during crystallisation would yield low values for k_p and R_c. The results of colour measurements and corresponding values of k_p and R_c for a few mills during October and November 1983 are given in Table 8.

TABLE 8

Colour of A-massecuite, A-molasses and A-crystal and the corresponding colour transfer factor (k_p) and Ratio (R_c)

Date	Colour			$k_p \times 100$	$R_c \times 100$
	Massecuite	Molasses	Crystal		
MS 21/11	37 230	—	1 300	1,9	3,5
UC 13/10	19 410	41 590	860	2,5	4,5
SZ 23/11	40 500	54 460	1 240	1,8	3,1
23/11	28 890	76 670	1 000	1,9	3,5
24/11	34 390	65 020	910	1,5	2,6
25/11	34 570	60 230	2 680	4,7	7,8
25/11	34 700	67 700	1 790	3,0	5,2
UK 14/11	28 960	55 430	1 820	3,7	6,3
14/11	30 920	57 030	2 160	4,1	7;0
15/11	26 130	45 990	1 060	2,5	4,1
15/11	27 350	55 710	1 870	3,8	6,8
17/11	23 780	42 770	890	2,3	3,7
17/11	24 930	49 940	1 090	2,4	4,4
MH 21/10	22 020	55 190	370	0,8	1,7

Discussion

Pan exhaustion

At all four factories the brix of the massecuite at strike was found to have a strong positive influence on the pan exhaustion performance. In the range of brixes tested (89 to 93°) a one point increase in brix improved the exhaustion and crystal content by 3 to 5 % and yielded a 2 point drop in A-molasses purity (see Figures 1, 2 and 3 and Table 1). Because of different massecuite characteristics (purity, and glucose plus fructose-to-ash ratio) and strike temperatures, the four mills did not achieve the same exhaustion performance or have similar handling characteristics (fluidity) at the same brix. By and large the pan exhaustion performance measured during the tests was high. For example, at a brix of 92,3 which is the average reported by the industry for the 1983/84 season, the exhaustion was 73% at UK and UC, and 68 and 67 % at GH and SZ respectively. The importance of the massecuite fluidity lies in the fact that it determines the pan striking time which, with the existing equipment, appeared to be the factor limiting higher brixes and better performance. Consequently to achieve optimum performance under a given set of conditions, the massecuite must be boiled to a fluidity level which results in the maximum tolerable striking time irrespective of the brix. In other words, the target given to the pan boiler ought to be the striking time and not the massecuite brix. In this regard it is worth noting that the striking times recorded during the investigation were found to be very erratic, varying from less than 2 minutes to about 30 minutes, and are certainly an aspect of process operation which deserves better control.

Exhaustion performance on cooling

The cooling of the high brix massecuites (± 92° brix) yielded on average, a drop of one point in molasses purity for each 4°C of cooling which corresponded to an increase of 1% in exhaustion and crystal content (see first 6 tests in Table 4). In spite of the fact that the improvement in the exhaustion of the low brix (± 89°) massecuites of Tests No. 7 and 8 were better on cooling, their overall performance (including pan exhaustion) was found to be markedly lower, with a 10 per cent lower exhaustion and crystal content. During the cooling of the high brix massecuites of Tests No. 1 - 6 it was found necessary, because of the high crystal content obtained to "lubricate" the massecuites with 5 per cent by volume of 78° brix A-molasses. This dilution was not found to have any marked detrimental effect on the exhaustion performance.

Crystal colour

The colour of the A-crystal, which was grown from a relatively high-colour B-sugar nucleus, was found to improve considerably during its growth in the intermediate boiling stages. However, in the final (strike) boiling there was, in most cases a 40 to 50 % increase in colour (see Tables 5 and 6). A few preliminary tests to determine at which stage of the boiling this jump in colour took place, were not conclusive (see Table 6). There are, however, good reasons to suspect that the colour is included in the crystal during the brixing-up period. Data collected during this investigation have shown that up to 40% of the total crystal mass is deposited during this period, which is normally of about 30 minute duration. The rate of crystallisation is then almost three times faster than average and there is a marked increase in the colour concentration of the mother-liquor. A fast rate of crystallisation has been found to increase inclusion.[4] Exhaustion in a water-cooled crystalliser did not result in any increase in the crystal colour (see Table 7). This is an indication that from the colour aspect, a crystalliser could be better equipment for sucrose crystallisation than the vacuum pan.

In spite of the relatively few results available, the level of colour transfer during A-massecuite crystallisation was found to vary widely both within and between mills (see Table 8). The highest level recorded was about five times the lowest figure.

At this early stage the operational factors and/or composition of colour bodies which may be responsible for the variation in colour separation efficiency during crystallisation have not been investigated.

Conclusions

The brix of the massecuite at strike has a strong positive effect on the pan exhaustion performance which is at a maximum level when the massecuite is boiled to a "tightness" resulting in the maximum tolerable striking time. Generally, an increase of one point in the massecuite brix at strike yields the same crystallisation as 8° C of cooling in a crystalliser. There is a substantial increase in the colour of the A-crystal during the last boiling of a full pan cycle and there are wide variations in the level of colour transfer in A-massecuites.

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