

THE CRYSTALLISATION OF MASSECUITES

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During the last few years, a great deal of work has been done on the use of Crystallisers and on Crystallisation in general. In South Africa, we have done practically nothing. In every factory here, there are to be found Herisson water-cooled crystallisers, which are just the plant required for the repetition of the work which has occupied S. J. Saint, in the Barbadoes and the workers at the Experiment Station, Honolulu, Hawaii, for the last few years.

In what follows, the writer has drawn very largely upon the papers which these workers have published, as it seemed the best way of calling general attention, in South Africa, to this work.

A preliminary investigation was made at Doornkop, where there are five Herisson Coolers and six air-cooled crystallisers. The results of this work are given below.

Crystallisers are used to crystallise additional sugar out of the syrup of the massecuite after it has been discharged from the vacuum pan.

In order to effect this purpose, a certain degree of supersaturation must be maintained and then the existing crystals will grow. This degree of supersaturation differs in each grade of massecuite.

High purity massecuite can be submitted to a very rapid cooling without too great a supersaturation taking place and without the formation of secondary grain, but with a massecuite of lower purity it appears that often a period of only $1\frac{1}{2}$ to 2 hours is too rapid, the rate of crystallisation not being great enough to precipitate from solution, the sugar present in the supersaturated state.

According to Classen, the growth of sugar crystals are brought about from those sugar particles

in direct contact with the crystal. As soon as this fine film becomes exhausted no further crystallisation can take place until fresh sugar is introduced into the film from the mother liquor. The adhesion between the film and the crystal is considerable and sugar is introduced into the film chiefly by diffusion. However, the motion of the syrup relative to the crystals can be improved by stirring. A good relative motion will bring supersaturated parts of the syrup nearer to the films round the crystals, and consequently diffusion will be increased.

Savinoff (Nauchnie Lapiski V7 (1929), 416-29) described some experiments in which he determined the velocity of crystallisation (1) where the crystal remains stationary in the supersaturated mother liquor and here the crystals grow through diffusion; (2) where the crystals are rotated in the supersaturated mother liquor thus preventing diffusion. His results show that by rotating the crystals at 100 or more revolutions per minute increases the velocity of crystallisation by as much as 500%. These results were obtained in a solution of pure sucrose of lower supersaturation and hence relatively low viscosity. But under industrial conditions owing to the high supersaturation of the mother liquor, the viscosity is high and it would be more difficult to realise free movement of the crystals relative to its surrounding films of mother liquor, which of course, means that the growth of the crystal in the crystalliser would be mainly by diffusion. In the Herisson there is, however, a fair amount of stirring and if this were increased it would be all to the good. That there is a certain amount of motion of the crystals relative to the mother liquor, is seen in the decrease of the purity in the surface massecuite when the crystals settle out under gravity.

1st MASSECUITE.

Hours in Crystalliser.	MASSECUITE.				MOLASSES.			YIELD OF SUGAR % WT. MASSECUITE.			
	Temperature °C.			Purity.	Purity.						
	Struck.	Cured.	Drop.		Struck.	Cured.	Drop.	Struck.	Cured.	Rise.	
11	61	49	12	90.5	75.4	68.5	6.9	59.3	64.2	4.9	A
13½	60	52	8	91.2	74.9	69.6	5.3	59.0	63.0	4.0	A
12	59	49	10	88.1	67.5	65.1	2.4	57.3	63.3	6.0	A
15¼	57	48	9	90.5	75.0	70.5	4.5	57.8	63.0	5.2	A
6	59	43	16	89.6	71.8	68.1	3.7	59.9	64.9	5.0	W
3½	56	47	9	88.5	70.8	66.7	4.1	58.2	63.3	5.1	W
3½	59	49	10	85.8	69.3	64.8	4.5	57.6	62.4	4.8	W
4	60	49	11	84.4	65.7	62.0	3.7	57.0	61.0	4.0	W

Hours in Crystalliser.	MASSECUITE.		MOLASSES.		Purity Drop.	
	Brix.	Purity.	Purity	Cured.		
3½	89.5	86.2	65.9	20.3	W	
3¾	90.0	86.9	67.3	19.6	W	
5¼	90.5	85.9	66.3	19.6	W	
6¼	90.5	87.1	68.7	18.4	W	
3	90.5	87.4	73.7	13.7	A	
5	89.0	86.6	71.3	15.3	A	
6½	89.5	88.9	73.4	15.5	A	
30	90.5	87.3	65.4	21.9	A	
37¾	90.5	88.1	67.0	21.1	A	

W= Herisson Water-cooled Crystalliser.

A= Herisson Air-cooled Crystalliser, water cut-off.

2nd MASSECUITE.

Hours in Crystalliser.	MASSECUITE.				MOLASSES.			YIELD OF SUGAR % WT. MASSECUITE.			
	Temperature °C.			Purity.	Purity.						
	Struck.	Cured.	Drop.		Struck.	Cured.	Drop.	Struck.	Cured.	Rise.	
25	65	45	20	79.4	55.1	52.3	2.8	57.5	60.8	3.3	A
27¾	64	40	24	75.2	54.0	52.0	2.0	57.1	60.2	3.1	A
10	64	45	19	81.0	49.1	47.0	2.1	56.9	60.3	3.4	W
11	65	44	21	82.0	53.6	50.0	3.6	57.3	60.9	3.6	W

Hours in Crystalliser.	MASSECUITE.		MOLASSES.		Purity Drop.	
	Brix.	Purity.	Purity	Cured.		
9	94.5	73.6	47.7	25.9	A	
8¾	93.0	75.6	57.4	17.6	A	
12	92.0	77.0	58.8	18.2	A	
13	92.5	75.8	54.3	21.3	W	
9¼	93.5	75.4	51.5	23.9	W	
6¼	92.5	77.7	53.1	24.6	W	

W= Herisson Water-cooled Crystalliser.

A= Herisson Air-cooled Crystalliser, water not being circulated.

3rd MASSECUITE.

Hours in Crystalliser.	MASSECUITE: Temperature °C.			MOLASSES: Purity.			YIELD OF SUGAR % WT. MASSECUITE.		
	Struck.	Cured.	Drop.	Struck.	Cured.	Drop.	Struck.	Cured.	Rise.
27	62	27	35	55.5	48.9	6.6	25.8	33.9	8.1
30	62	23	39	53.2	48.1	5.1	20.2	34.6	14.4
20	62	27	35	54.3	48.4	5.9	23.7	32.7	9.0
16	63	23	40	54.6	47.6	7.0	22.7	39.5	16.8
15	63	25	38	55.5	48.3	7.2	22.1	37.6	15.5
21	63	26	37	55.6	49.4	6.2	21.6	32.3	11.7
20	62	23	39	55.0	50.1	4.9	30.1	38.4	8.3
19	62	23	39	55.9	49.1	6.8	30.5	39.1	8.6
21	62	27	35	57.1	49.5	7.6	25.5	35.5	10.0
22½	63	27	36	56.2	50.0	6.2	24.2	35.2	11.0
17	63	28	35	56.6	50.2	6.4	27.9	38.4	10.5
15	63	29	34	56.8	50.4	6.4	31.2	37.0	5.8
24	62	28	34	57.2	49.2	8.0	25.3	35.6	10.3
17	62	30	32	55.7	48.5	7.2	27.6	37.4	9.8
11	62	32	30	56.2	49.7	6.5	29.0	41.4	12.4
(Comparison between Herisson and Air-cooled. Half the Massecuite put into each).									
16W	63	28	35	50.2	46.5	3.7	34.7	43.5	8.8 B on curve
A	63	44	19	50.2	49.1	1.1	34.7	37.5	2.8 A " "
12W	65	28	37	53.0	45.8	6.2	31.5	39.0	7.5 D " "
A	65	48	17	53.0	50.3	2.7	31.5	35.7	4.2 C " "
12W	58	26	32	52.3	47.2	5.1	33.5	41.0	7.5 F " "
A	58	47	11	52.3	51.2	1.1	33.5	37.0	3.5 E " "
16W	65	24	41	54.4	47.9	6.5	32.0	41.7	9.7
A	65	40	25	54.4	50.9	3.5	32.0	37.2	5.2
12W	64	28	36	52.8	47.6	5.2	33.0	43.3	10.3
A	64	40	24	52.8	48.3	4.5	33.0	36.0	3.0

A= Ordinary Air-cooled Crystalliser (no water used).

W= Herisson Water-cooled Crystalliser.

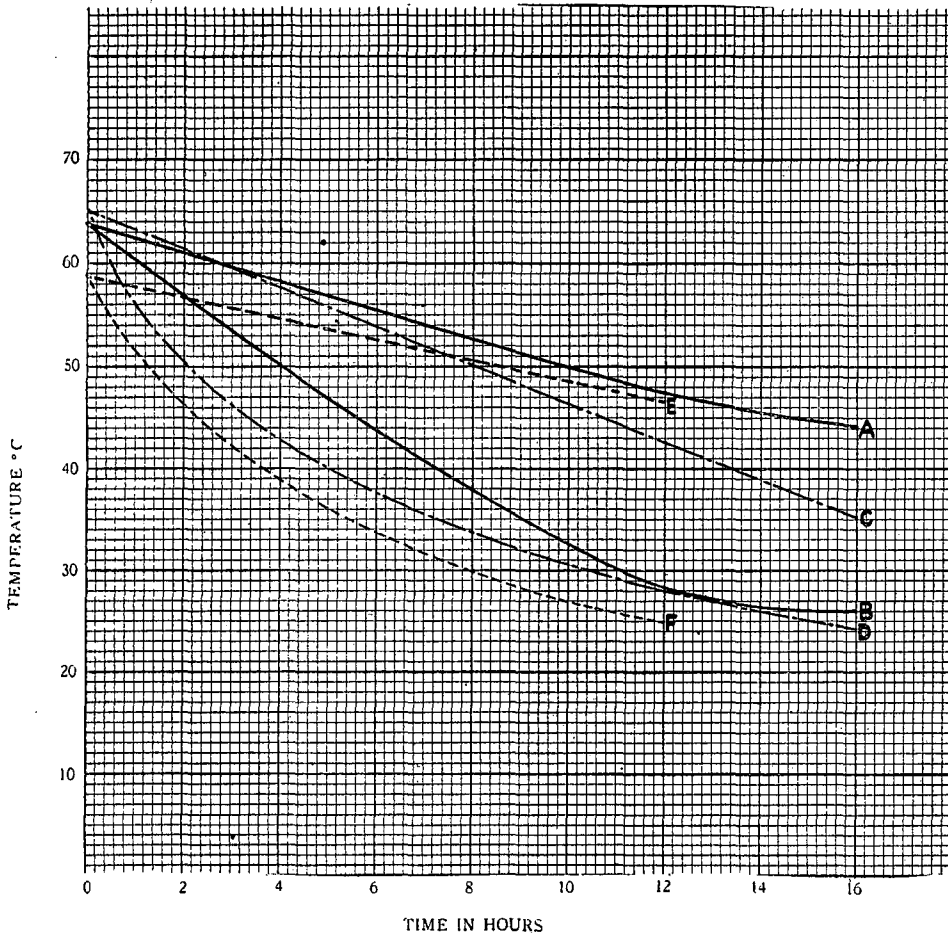


Fig. 1.

The Curves show clearly the value of the water cooling of the third Massecuities.

Three tables, the result of our experimental work, are given above, setting forth the temperature drop during the water and air cooling of 1st, 2nd and low grade Masseccutes. While making these observations, the yield of sugar which resulted from the several treatments was recorded, and it is abundantly clear that the cooling accelerates the yield very considerably in the case of each masseccute. During the work, the temperatures were recorded every hour and the sugar crystallised out was separated from the mother liquor in a laboratory centrifugal. The yield of sugar was recorded and the mother liquor analysed for purity.

This procedure brought to light several points of interest. It was definitely shown that the water inlet end of the crystalliser cooled more rapidly than the water outlet end. Thus at the inlet end, there was found with a first masseccute, a 65.6 per cent. yield, while at the other there was only a 62.8 per cent. yield. In a second masseccute much the same difference was found, i.e. inlet 55.4 per cent. and 52.4 at the outlet end.

Again it is the practice in some factories to steam out the pan after discharging the masseccute and to put the washings on top of the masseccute in the crystalliser. This is a very bad practice as can be seen from the following figures.

A. 1st MASSECCUTE.	Molasses Purity	Per. Cent. Sugar Yield
At discharge	67.5	57.3
After washings have been added to Masseccute	69.3	53.9
2 hrs. later	69.1	53.9
3 " "	68.5	53.6
4 " "	67.9	54.3
B. 2nd MASSECCUTE.		
At discharge	56.3	57.5
After washings have been added to Masseccute	57.6	52.5
3 hrs. later	55.9	53.6
6 " "	54.9	56.6

A is a 1st Masseccute which, when discharged, had a molasses purity of 67.5 and gave a sugar yield of 57.3% on masseccute and B is a 2nd masseccute with a molasses purity of 56.3 when dropped from the pan, and gave a sugar yield of 57.5 per cent.

In both cases it will be seen that it takes several hours before the concentration of the sugar in the masseccute is the same as it was when dropped from the pan. The implications are obvious, all those hours of cooling are lost, for the benefit of the cooler does not begin until the yield of the sugar becomes greater than when the masseccute was first dropped.

The same may be said of "feeding the masseccute with water." It was noticed that the density of the mother liquor rose after water had been sprayed

upon the masseccute to make it possible to turn the shafts and cooling coils. In some South African factories it is the practice to add molasses from the same kind of masseccute to reduce the viscosity.

Good as is the work done by the Herisson, the work done recently in the Barbadoes and in Hawaii has shown that it is not merely a matter of cooling at any haphazard rate. There has been developed a technique in which the Herisson Crystalliser is eminently suited to play a satisfactory part.

This work on low purity masseccutes has had an intensely practical aspect in Hawaii, due to its efficient Experiment Station. The recommendations worked out on theory, have been taken up by the factories, 31 out of 37 have implemented the findings and it is calculated that the improvements in the handling of these masseccutes is worth 900,000 Dollars on a 900,000 ton crop.

A repetition of this work in South Africa would be well worth while, consequently it is reviewed in what follows.

In the crystallisation of sugar, we require to know the solubility of sucrose in cane juices; the coefficients of supersaturation of the solutions, the viscosity of the mother liquor, its concentration and temperature. The velocity of crystallisation is then affected by the purity, supersaturation and the viscosity of the mother liquor. Kucharenco (The Crystallisation of Sucrose, p. 12) has shown that the velocity of crystallisation of sucrose increases rapidly with the supersaturation, and Classen and Horsin-Deon have pointed out that the crystallisation of a sucrose solution can be accelerated considerably by the reduction of the viscosity of the solution. Since the viscosity of a sugar solution rises with the supersaturation, it is clear that the two factors, viscosity and supersaturation, must be mutually antagonistic.

The solubility of the sucrose in cane juices has been worked out by Thiemé (Studies in Sugar Boiling, p. 74) who published Saturation Coefficients from which may be calculated the solubility of sucrose for any given purity.

The supersaturation of the mother liquor of a masseccute is affected by two main factors, i.e. concentration and temperature. At a given temperature, the supersaturation of the mother liquor increases with the concentration of sucrose in solution, and at each definite concentration of the mother liquor the supersaturation increases as the temperature decreases.

During the investigations referred to above, the effect of cooling on the exhaustion of the mother liquor in the crystalliser was examined by the separation and analysis of samples of the mother liquor at definite time intervals. These analyses made possible the calculation of the coefficients of supersaturation of the mother liquor and the percentage of crystal sugar in the masseccute during the time that the masseccute remained in the crystalliser, as follows:—

TABLE 1.
ORDINARY AIR-COOLED CRYSTALLISER, BARBADOES.

TIME (Hours)	MOTHER LIQUOR.			MASSECUITE.	
	Dry Matter.	True Purity.	Co-efficient of Supersaturation	Temperature ° C.	Per Cent. Crystal Sugar.
At discharge	86.51	50.26	1.270	68	24.70
2 hours later	86.33	48.94	1.221	66	26.34
12 " "	85.60	46.40	1.223	60	29.34
24 " "	85.11	44.47	1.236	52	31.47
36 " "	84.37	43.34	1.227	46	32.63
48 " "	84.20	42.30	1.225	42	36.69
60 " "	84.27	41.53	1.258	38	34.42
72 " "	84.26	40.72	1.262	36	35.16
84 " "	83.94	40.01	1.246	35	35.84
118 " "	83.90	39.81	1.253	32	36.02

The small variation of the coefficient of supersaturation during the entire period of cooling in the crystalliser is one of the striking facts brought out by these figures. The crystalliser was of the ordinary type air-cooled type. This co-efficient lay between 1.27 and 1.22 and the crystal sugar increased during this period from 24.7 per cent. to 36.02 per cent. which increase is entirely due to the fall in solubility of the sucrose in the cooling mother liquor and not to any reduction in the supersaturation.

Another point brought out was that since the coefficient of supersaturation remained unchanged, the only other factor which affected the growing viscosity of the mother liquor was the temperature, and it was shown that, with the initial temperature at 64° C., when 35° C. was reached the viscosity of the mother liquor is so pronounced that for all practical purposes crystallisation has ceased. Nevertheless, the coefficient of supersaturation remains at 1.22, and the examination of the molasses by Kalshoven's method (Archief 27 (1919), p. 1560) disclosed that 6.6 per cent. of the sucrose of the final molasses was present as false grain.

(At the Experiment Station, Mount Edgecombe, we had eight or ten final molasses from various factories examined and it was found that in every case there was a considerable part of the sugar present (up to 9 per cent.) as false grain).

The consideration of the facts set forth in the table, which records the results from an air-cooled crystalliser, led to the following conclusions.

The greatest crystallisation of sucrose takes place in the pan so it is desirable to discharge the massecuite at as high a concentration as possible, but this will result in a relatively high supersaturation of sucrose in the mother liquor, which in turn will produce a high viscosity which tends to retard the velocity of crystallisation. Kucharenco's (loc. cit.) results show that if the temperature of the mother liquor is high increasing supersaturation has relatively little effect.

It has been found also that the velocity of

crystallisation is greatest at 60° C. and falls considerably as the temperature drops, it would appear logical to suggest that the temperature of the massecuite be maintained at the striking temperature until there is a considerable reduction in the supersaturation of the mother liquor. This crystallisation of sucrose will lower the viscosity, and the effect of the lower viscosity on the velocity of crystallisation will produce a lowering of the supersaturation. It may therefore be found that when the supersaturation has decreased to say 1.1, the velocity of crystallisation can be increased by cooling and there is little doubt that with the lower supersaturation of the mother liquor, this cooling could be carried out rapidly.

Further, as the table discloses that the mother liquor of the cooled massecuite is considerably supersaturated, and although this sucrose is theoretically recoverable, it is lost under practical factory conditions because there is insufficient time available to allow crystallisation to go to completion. Hence it suggests itself that the massecuite **could be cured** by raising the temperature from say 32° to 50° C. There is no fear of dissolving the grain, i.e. without undersaturating the mother liquor. This method would appear much more advantageous than the more usual method of adding water to the crystalliser, since in the former case, the viscosity of the massecuite is lowered not only by a reduction in the supersaturation of the mother liquor but also by the increase in temperature.

With these ideas in mind, three low purity massecuites were allowed to cool in the crystalliser, under the following conditions:—

1. Air-cooled for 40 hours.
2. Maintain strike temperature for 24 hours, and then cool rapidly for another 24 hours.
3. Maintain strike temperature for 10 hours, and follow with rapid cooling for 30 hours.

The three strikes used in these experiments, had practically the same initial analyses, and the results are shown in the tables and curves.

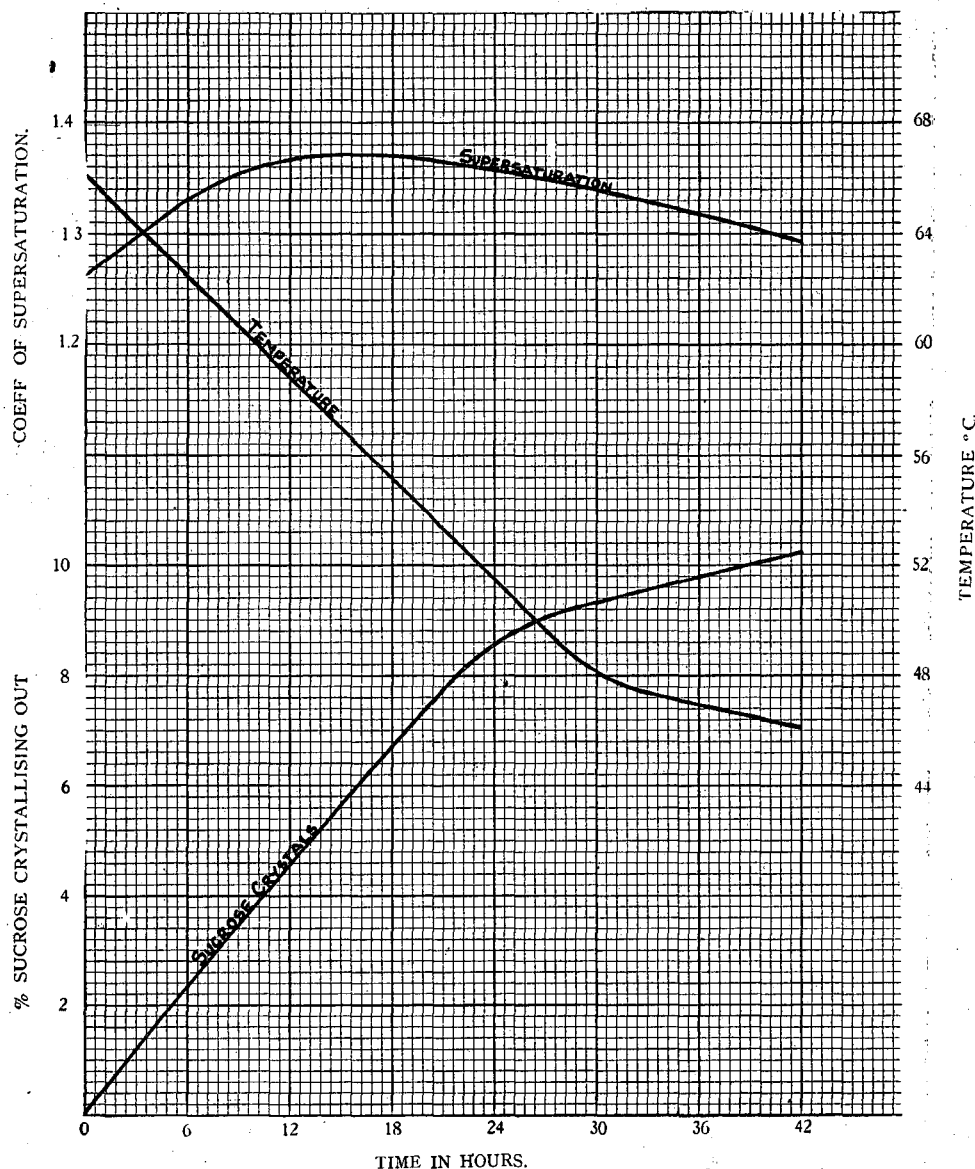


Fig. 2.

TABLE 2.

MASSECUITE No. 1

TIME (Hours)	MOTHER LIQUOR.			MASSECUITE.	
	Dry Matter.	True Purity.	Co-efficient of Supersaturation.	Temperature °C.	Per Cent. Crystal Sugar.
At discharge	87.60	45.67	1.256	66.7	23.52
2—6	87.87	43.15	1.329	64.2	26.52
8—12	87.56	41.61	1.365	59.8	28.21
14—18	86.32	39.63	1.391	55.6	30.27
20—24	87.04	37.50	1.351	50.9	32.3
26—30	86.88	36.95	1.347	47.4	32.86
32—36	86.58	36.47	1.307	47.0	33.30
38—40	86.62	36.00	1.320	45.8	33.71
Total Increase in per cent. Crystal Sugar					10.19

In No. 1, which was air-cooled, it will be seen that as the temperature fell the coefficient of supersaturation rose from 1.26 to 1.36, showing that the velocity of crystallisation had not been able to keep pace with the lowering of the solubility of the

sucrose in the mother liquor as the temperature fell. At 50° the velocity of crystallisation fell, indicating that to maintain the same velocity of crystallisation, the supersaturation must be higher than 1.34.

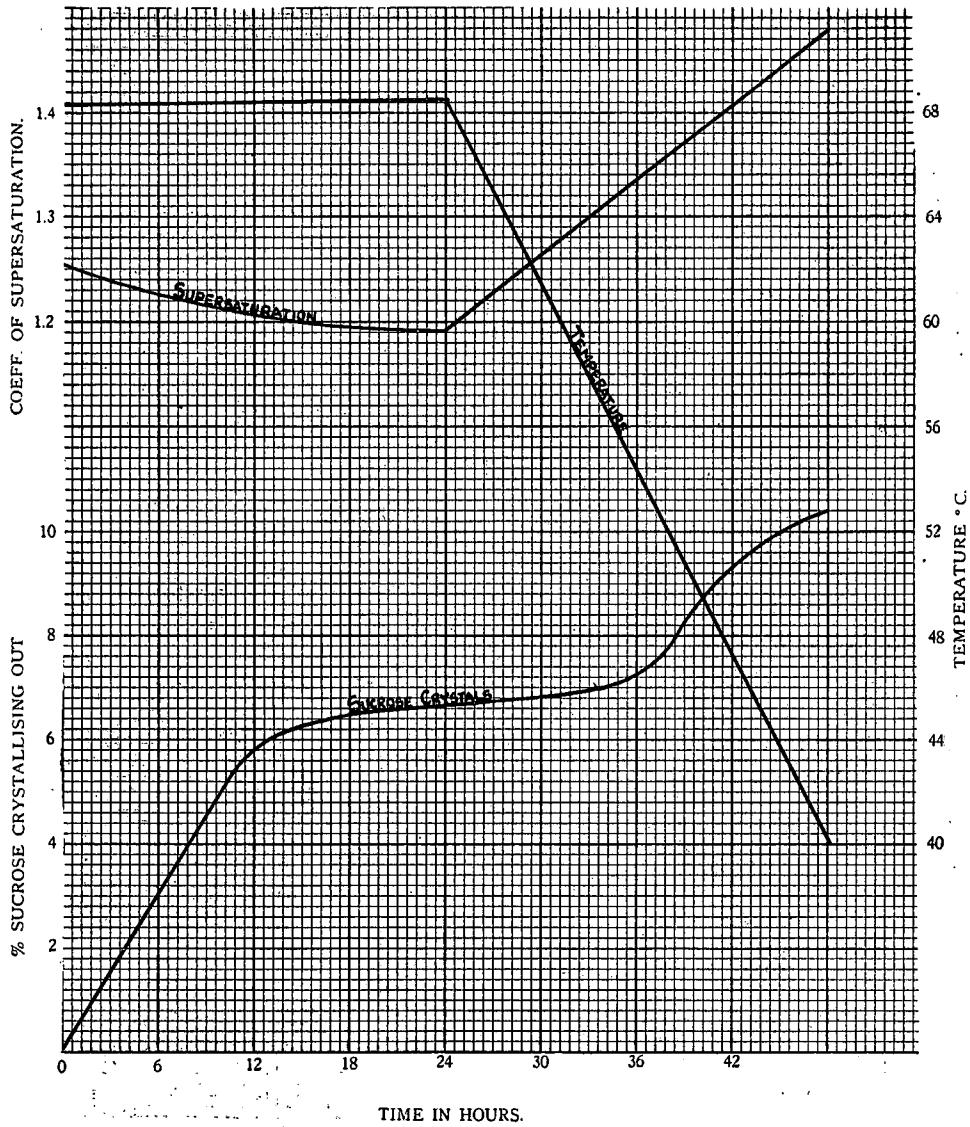


Fig. 3.

TABLE 3.

MASSECUITE No. 2

TIME (Hours)	MOTHER LIQUOR.			MASSECUITE.	
	Dry Matter.	True Purity.	Co-efficient of Supersaturation.	Temperature °C.	Per Cent. Crystal Sugar.
At discharge	86.00	51.42	1.257	68.3	22.08
2—6	86.14	49.13	1.235	68.5	25.13
8—12	86.23	46.72	1.204	68.2	28.07
14—18	86.15	46.40	1.197	68.4	28.44
20—24	85.81	47.62	1.191	68.5	—
26—30	86.05	45.95	1.272	61.2	28.95
32—36	85.81	45.72	1.331	54.1	29.21
38—42	86.02	43.58	1.373	48.9	31.52
44—48	86.32	42.65	1.485	39.5	32.47
Total Increase in per cent. Crystal Sugar					10.39

In No. 2, the striking temperature was maintained by circulating hot water in the coils for 24 hours. The massecuite began to show signs of froth fermentation after being heated for 10 hours.

On the curves it will be seen that crystallisation took place readily for the first 10 hours until the coefficient of supersaturation dropped to 1.2 and for the next 14 hours practically ceased. The

massecuite was then cooled and the velocity of crystallisation remained slow until at 54°C. the coefficient of supersaturation had risen to 1.33 when the higher velocity of crystallisation was resumed. The cooling was carried out rapidly by circulation

of water and as the increase in supersaturation shows, the sucrose crystallising out could not keep pace with the drop in solubility, at 40° C. the supersaturation was 1.4 and the velocity of crystallisation had only slightly decreased.

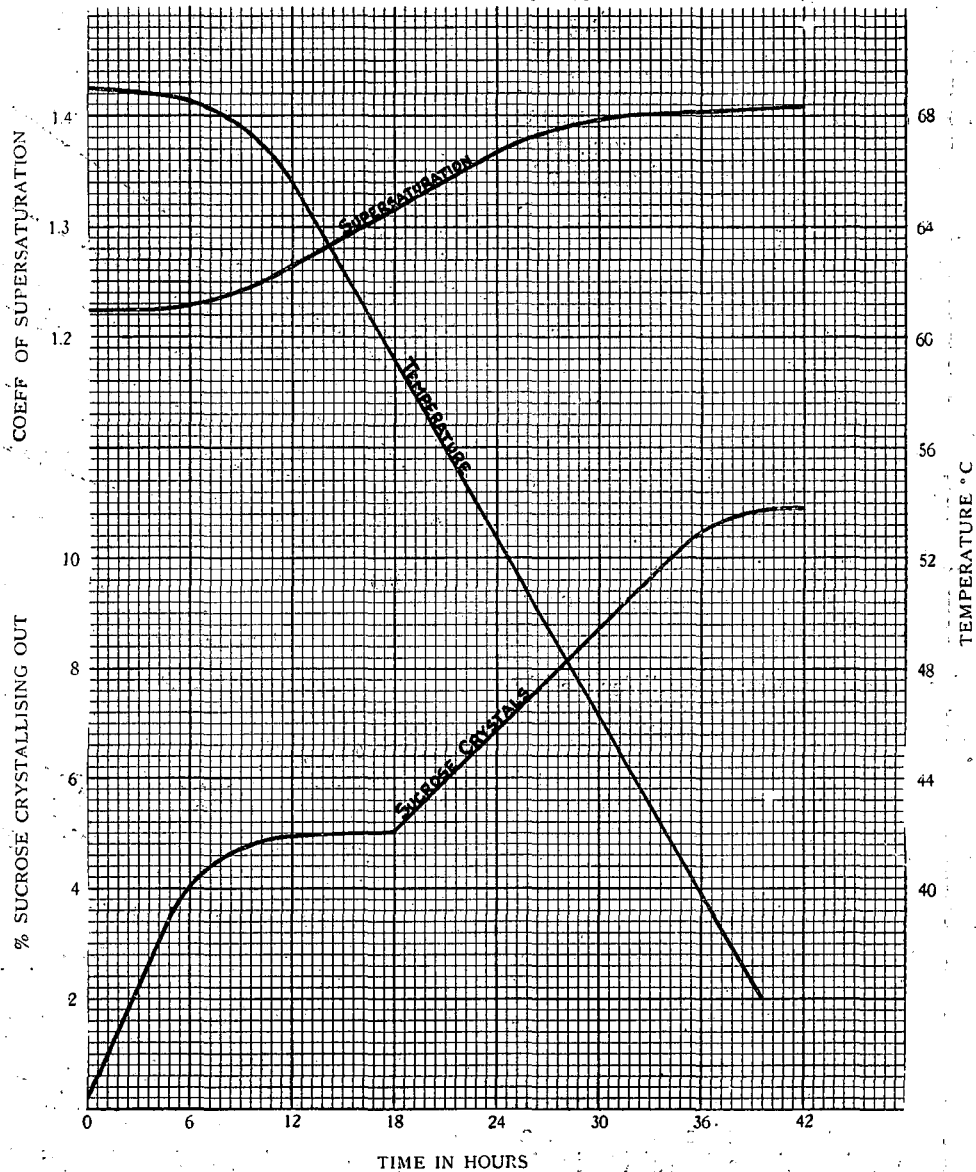


Fig. 4.

TABLE 4.

MASSECUITE No. 3.

TIME (Hours)	MOTHER LIQUOR.			MASSECUITE.	
	Dry Matter.	True Purity.	Co-efficient of Supersaturation.	Temperature °C.	Per Cent. Crystal Sugar.
At discharge	86.60	47.31	1.225	68.9	24.04
2—6	87.03	44.01	1.229	68.5	27.97
8—12	87.21	43.13	1.263	66.3	28.95
14—18	86.91	23.05	1.326	59.2	29.03
20—24	86.54	41.33	1.363	51.1	30.84
26—30	86.92	39.25	1.393	45.7	32.90
32—36	86.54	37.57	1.394	40.7	34.45
38—40	86.61	37.10	1.412	39.2	34.88
Total Increase in per cent. Crystal Sugar					10.84

No. 3 massecuite also showed signs of froth fermentation and after 10 hours the temperature was lowered by circulating cooling water in the coils, and, just as was the case with No. 2, cooling did not increase the velocity of crystallisation until the coefficient of supersaturation of the mother liquor had risen to over 1.3 at 60° C. During the following 18 hours, the temperature fell from 60° to 40° C. and the supersaturation coefficient of the mother liquor rose from 1.32 to 1.4. This confirms the results shown by No. 2 massecuite, viz., that even at 40° C. the velocity of crystallisation will remain high, provided that the coefficient of supersaturation is maintained at 1.4.

It is clear from these and such results that the controlling factors influencing the velocity of crystallisation in low purity massecuites are the temperature and coefficient of supersaturation of the mother liquor.

When the supersaturation falls below 1.2 at 68° C., the velocity of crystallisation becomes very slow. As the temperature decreases and the viscosity of the mother liquor increases, a higher supersaturation is necessary, if the velocity of crystallisation is to be maintained.

It will be noticed that there is little difference in the total amount of sucrose crystallising out under the different methods of cooling adopted for these experiments. It is therefore clear that there is no great advantage to be gained by maintaining the striking temperature of the massecuite since, although crystallisation of sucrose takes place for a time, the coefficient of supersaturation is so reduced thereby that a considerable cooling has to take place before the coefficient of supersaturation can be raised sufficiently for a normal velocity of crystallisation to proceed at the reduced temperature.

Another very useful and important point emerges from this study. At the time of curing, a relatively high supersaturation of sucrose exists in the mother liquor which cannot be recovered by the factory. Owing to this high degree of supersaturation the temperature of the massecuite could be raised to over 70° C. without any sign of dissolution of the sugar crystals in the massecuite. This would result in a most important reduction in the viscosity of the mother liquor and a great saving of time at the centrifugals.

This work has been extended in Hawaii and for the last three or four years they have been devoting a great deal of experimental work to the subject of crystallisers, crystallisation and centrifugals.

It is the practice in South Africa, in some factories, to add gradually, water to their low grade massecuites as the massecuite tightens. The accom-

panying chart shows how the gravity purity of the molasses is enriched at the expense of the sugar crystals and shows how dangerous it is to warm such diluted massecuite before centrifuging.

CURVE SHOWING THE INCREASE IN PURITY OF THE MOTHER LIQUOR ON DILUTION WITH HEATING.

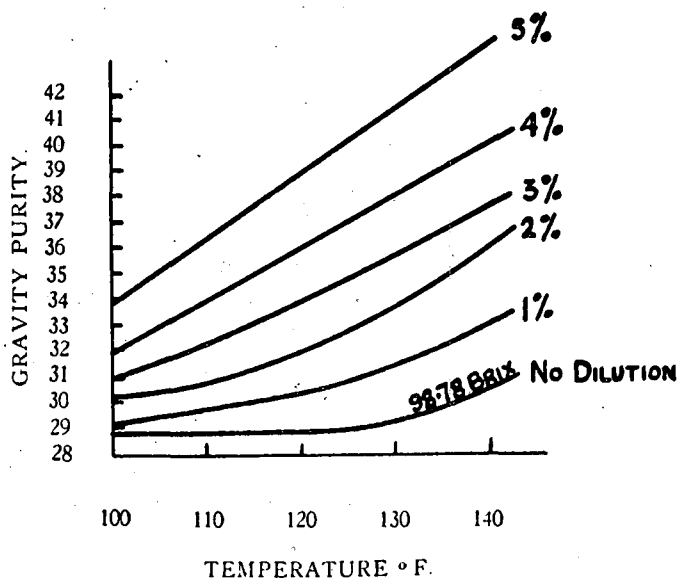


Fig. 5.

The heating of massecuites, as recommended by Saint, instead of dilution, to expedite work at centrifugals has failed previously in the hands of some operators owing to the lack of the fundamental principles.

The first successful application of this principle of re-heating was inaugurated at Waipahu following the installation of crystallisers provided with 30 per cent. of cooling or heating surface in stationary copper coils. It was possible, therefore, to cool the massecuite to a satisfactory temperature in from 54 to 60 hours, then by introducing warm water into the coils to raise the temperature of the massecuite to 105 - 110° F. before discharging to the centrifugal mixer.

Exhaustive experiments had previously convinced them that this practice was sound. It was found, for example, that when the massecuite temperatures were as shown below, that the purity of the sugar was as shown.

Massecuite Temperature.	Apparent Purity of Sugar.
79° F.	63.5
84° F.	67.2
89° F.	70.5
94° F.	73.5
99° F.	75.2
104° F.	76.4
109° F.	77.5

It is clear, therefore, that when the temperature of the centrifuged massecuite is high, a much higher purity sugar is obtained, as a result of the better elimination of the molasses resulting from the reduction of its viscosity.

It has been shown that with proper heating equipment, there should be no increase in molasses purity.

It has been demonstrated also that heating is superior to dilution in an unexpected direction, viz., that at a given viscosity, molasses with a higher saturation temperature has a lower purity than the same molasses at a lower saturation temperature. The accompanying figure illustrates the point by the series of curves at five saturation temperatures.

PURITY CURVES SHOWING GRAVITY PURITY-VISCOSITY IN THE SAME MOLASSES AT SATURATION TEMPERATURES 30-35-40-45-50° C.

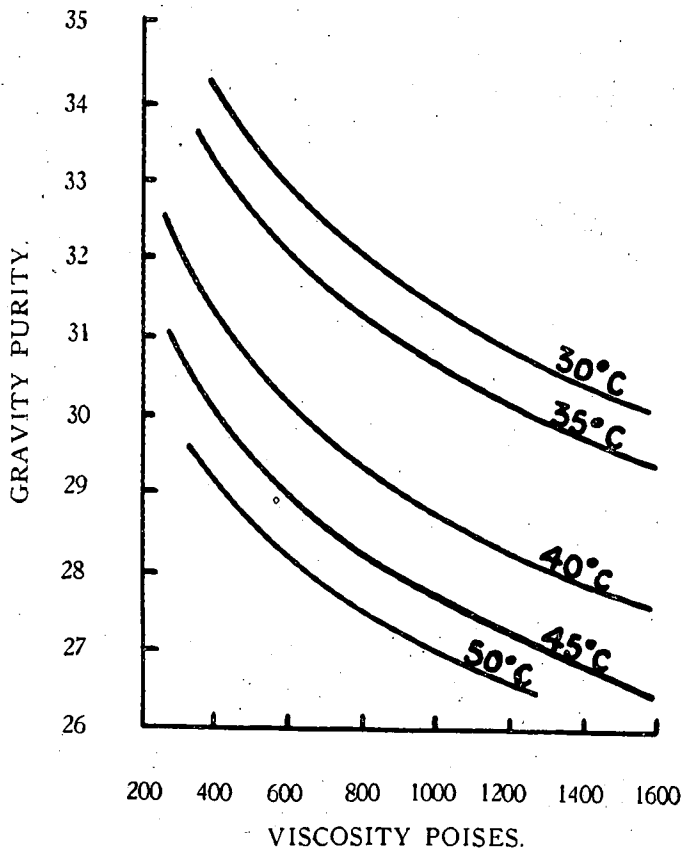


Fig. 6.

Thirty-one out of the thirty-seven factories in Hawaii have taken up this treatment of their low grade molasses (1935) within the short time this treatment has been worked. An enormous amount of work is necessary before applying the principle to the factory, but it has resulted in reductions in the final molasses from 39.18 in 1929 to 33.07 in 1933. This reduction is estimated as worth 900,000 Dollars per year, on a 900,000 tons crop.

The additional plant involved has been warming coils in the centrifugal mixer to maintain a satisfactory temperature in the massecuite going to the centrifugals.

At another factory, sufficient crystalliser capacity to provide time for both cooling and warming was not available and a heater was inserted between the crystallisers and the centrifugal mixer.

A U-shaped trough, 30 inches in diameter and 20 feet long was installed with a heat and stirring element revolving at $5\frac{1}{4}$ R.P.M. and composed of 73 square feet of heating surface in parallel 2 in. O.D. brass tubes operating in multiple, the power needed being 5 H.P. About 75 feet of massecuite per hour pass through this heater, the temperature being thereby raised from 86° to 118° F. with warming water at 180° F. and leaving at 144° F. Massecuite passes from end to end in 80 minutes.

Another similar unit consists of a heating element composed of a hollow shaft and a single helical coil providing 48 square feet of heating surface, or 30 per cent. on volume revolving at 3 R.P.M. heating 110 cubic feet of massecuite per hour from 110° to 118° with water entering at 154° and leaving at 144° F. Massecuite passed through this heater in 20 minutes being first warmed to 100° F. by means of exhaust steam coils in the crystalliser discharge trough.

ACKNOWLEDGMENTS.

In the practical work in this paper, Mr. du Toit, of the Experiment Station, assisted the writer. Thanks are also due to Mr. J. Rault, of Natal Estates, for the figures showing the comparison between the Herisson and Air-cooled Crystalliser in 3rd Masseccuites.

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South African Sugar Association,
Experiment Station,
Mount Edgecombe,
January, 1937.

The PRESIDENT: Dr. Hedley has submitted to us a very interesting paper, and one which, I think, bristles with points for discussion.

Mr. BOOTH: As one who was associated with Dr. Hedley in the first few days of his investigation, I have read this paper with much interest, and I have learned quite a lot. You have asked for comments on the scientific principles laid down. I think that is somewhat difficult. I agree with Dr.

Hedley that the whole question of saturation is somewhat of a new line. He mentions in the early part of his paper, the giving attention to the cooling of massecuites. I think it is only a small portion of the major question of the crystallisation of sugar from syrup.

The appearance of this paper is a confirmation of your own special plea, Mr. President, in your Presidential Address, that there remains a great amount of work to be done in the investigation by way of increasing factory efficiency. To refer to your statement that 35% of the cost of the sugar lies in the manufacture, we realise that we have to reduce our costs further, and the only way to do it is to get a staff of trained technicians to attend to those questions which the factory staff cannot hope to do; they have to cope with their routine duties. Some three years ago, I mentioned in this Congress the superheating of juices and was flattered yesterday to find that you also referred to the same subject. Apparently my words did not fall on deaf ears. Last year, I placed in Dr. Hedley's hands some correspondence regarding the separate control of juices. Vacuum pan design, vacuum pan control, crystallisers, stirring gear and high speed curing, open up new vistas in this interesting subject but, as I said before, the work cannot be done by the ordinary factory superintendent and his staff. Therefore I would suggest that the incoming Committee make capital out of Dr. Hedley's findings and decide as to whether the Experiment Station cannot undertake part of this work.

Referring to the paper, there are many points that Dr. Hedley has mentioned. As regards the water-cooled crystalliser, it may be taken for granted that, especially in the second massecuite, they yield better figures and save much time. I have doubts as to whether rapid cooling of 3rd massecuites is to be recommended. At a brix of 95°, the increase in viscosity has to be balanced by applying water, otherwise that massecuite is going to go round in one big blob. If one doesn't watch out, the inside of the crystalliser will be torn out. Some people use molasses to slacken. I don't see much point in that practice. It is certainly a lubricating agent. Why not water the massecuite judiciously to avoid crystalliser grain? Our practice in final massecuites is to seed the second wash of approximately 80 purity, build on 2nd molasses, dropping at the highest possible brix into a Herisson cooled crystalliser with the steamings. Let it consolidate for two hours. Drop the temperature to normal (about 90°F.) raise it to about 110 to 115°F. then spin it in 30 inch machines, at 1,250 revs. per minute. This sugar polarises somewhere about 90. Since all massecuites are examined by microscope before they drop, it is interesting to learn from Dr. Hedley that the samples of molasses he had from us contained false grain. Were those grains spontaneous crystallisation or grains that failed to grow? I

differentiate between the two. We hope to improve upon that, but I have no intention of raising the temperature to 70°C. We are installing a hot water mingler in the final massecuite mixers to raise the temperature from 90°F. to 122°F. to avoid heating in the crystallisers, and if we can get a higher speed in our machines so much the better. The difference between spinning at 1,200 and 1,500 is enormous. We hope that Dr. Hedley, when again working on this subject, will come up to see us at Doornkop, as I am very interested in this saturation business, and by this time next year we may have considerably more information on a very important subject.

Dr. HEDLEY: With regard to the matter of the false grain, I should imagine, in your case, it would not be grain that didn't grow; it might be due to your suddenly cooling it locally. That will do it.

With regard to raising the temperature to 70° and then curing; I think you must have missed the point of this supersaturation curve. The point is you must not raise it above that temperature which will dissolve the sugar which is in the supersaturated condition. If you do, then you will certainly dissolve the saturated sugar, but you can raise it to the temperature which your co-efficient of supersaturation allows, without losing any sugar.

Mr. BOOTH: I don't think you have mentioned any time factor.

Dr. HEDLEY: The heating factor will be governed by the coils of your crystalliser and obviously the heating surface after you leave your crystalliser to your centrifugals. That is a very important point. If heating is going to take too long, obviously you are not getting much by heating.

Mr. BECHARD: I take it that the first part of the paper refers to experiments made by Saint, mostly. I would like Dr. Hedley to tell us whether the experiment referred to at the bottom of page 73 on these three different massecuites refers to South African conditions.

Dr. HEDLEY: Those were experiments which Saint conducted when investigating the subject in Barbadoes.

Mr. BECHARD: Massecuite one, air-cooler crystalliser, showing purity drop of 45.67 to 36 in 40 hours is quite on a parallel with South African experience; also its crystal recovery from 23% to 33%, shows similar results to South African massecuites of that description. It is certainly impossible to do so in air-cooler crystallisers.

Mr. MACBETH: Mr. President and Gentlemen, in rising to add a few remarks on this paper, I do so, not as an expert on the subject, but to give a few details of what I observed during my trip overseas.

Dr. Hedley's paper on the Crystallisation of Masseccutes is certainly a step forward in the right direction and touches upon a subject, which in my opinion, is of very great importance to the Sugar Industry in this country.

Whilst in the Hawaiian Islands, I was fortunate in meeting Mr. Raymond Elliott, who gave me a fairly detailed description of the work carried out on the supersaturation of masseccutes, and as far as I can now recollect they are touched on, besides other details, in Dr. Hedley's paper.

I notice in the paper that no mention is made of two very important instruments that are used in ascertaining the melting point of sugar crystals and the viscosity of the masseccutes due perhaps to not sufficient information being available about them.

I allude to the "Saturoscope" and "Viscometer," both these instruments are the products of the Hawaiian Experimental Station.

Perhaps most of you are aware, the "Saturoscope" is used to observe the melting point of sugar crystals when the masseccutes are subjected to reheating. The instrument is of simple construction, but all the same ingenious and if desired I will endeavour to explain it to you. At this stage Mr. Macbeth described the instrument and the details with reference to procedure of operation, etc.

As far as the "Viscometer" is concerned, unfortunately, this instrument was not on view, as it was still kept closely guarded and was not for inspection during my stay there.

However, I was given to understand that this instrument was to ascertain the viscosity of masseccutes at various stages of cooling and reheating and perhaps further information could be obtained from the Principals of the Hawaiian Experimental Station.

In conclusion, I would like to emphasise that this subject should not be treated lightly, as thousands of pounds sterling per year could be recovered from our final masseccutes and molasses if experimental and research work was carried out in this direction.

I think the time is opportune to appoint a Technologist to be attached to the Experimental Station, to investigate these various important problems which would benefit the Sugar Industry in this country generally.

Mr. RAULT: Mr. President, I would like to thank Dr. Hedley for having brought forward for discussion the very important subject of crystallisation. To speak in terms of crystallisation phraseology his paper should be an "exciter" of discussion on a very vast subject.

When referring to crystallisation we should not narrow our outlook on what happens in the crystallisers only, but should remember that the greatest part of the work is done in the pan itself,

where crystallisation starts. This part of crystallisation is as important as the subsequent one and very often influences in a marked degree the results of the latter operations. Sugar boiling has so far been a craft where the personal element plays a great role, and it is possible that a large measure of improvement could be realised by the help of the recording instruments lately placed on the market and also by the microscope and the camera.

Coming to the crystallisation cooling stage it looks as if the practice of water feeding is now in disfavour. Nevertheless our experience goes to prove that feeding in a judicious way will produce very satisfactory results as will be gathered from the average yearly figures of the past three seasons. Where no remelting seems to take place as evidenced by the increased yields and lowering of molasses purity.

Pan boiling and cooling in motion endeavour to force out of solution a maximum amount of crystals, by bringing the syrups to a very high degree of supersaturation. This high degree of supersaturation is usually the cause of increased viscosity, which handicaps the separation of crystals from the mother liquor in the centrifugal department. The methods used for enforcing maximum crystallisation are in some way antagonistic to maximum efficiency of curing.

The Hawaiian technologists are solving the conflicting problem of maximum crystallisation and maximum centrifugal efficiency by reheating the the last masseccute, and increasing centrifugal speeds. We have so far practiced methodical dilution worked simultaneously with the cooling.

The re-heating appears to be a very promising method, as viscosity is more easily decreased by heating than by dilution and with less risks of remelting.

We are trying the re-heating system, and have found it fairly satisfactory except in so far as the heating after completion of cooling operations consumes a longer time, than the simultaneous cooling and dilution.

It is not correct to state that water feeding of low grade masseccutes will in every case remelt crystals. In our factory our present capacity forces us to masseccute for only 24 to 30 hours in Herrison water cooled crystallisers. During the cooling of this last masseccute we carry out slow feeding and our average results of crystalliser work show a drop of purity molasses from 6 to 7 degrees and an increased yield of $8\frac{1}{2}$ to $11\frac{1}{2}$ per cent crystals on weight of masseccute during this short cooling operation.

There is a wrong impression that the re-heating method increases further crystallisation. If any extra yield is obtained by this method it is purely

as a result of not melting the grain previously formed by cooling.

We have found that in the case of a badly-spinning massecuite, reheating was a very poor palliative which did not cure the trouble.

It should also be noted that the claim of a better purity sugar from reheating a massecuite would indicate that the quality of the sugar turned out of the last boilings of Hawaiian factories is decidedly inferior to anything produced in South African factories. Raising the purity of a sugar from 63 to 77 means nothing to us as our worst sugars are already at a higher purity than 77.

I can assure Mr. Bechard that in my term of office for the coming year, I will do my utmost to revive the boiling house Committee and obtain the necessary apparatus for continuing the good work already done.

I am sorry not to see amongst us here to-day Mr. Jelley, Chief of Fabrication at New Guelderland, where excellent boiling house results have been obtained. I do not think that with the limited staff at his disposal, it has been possible for Mr. Jelley to have many opportunities for extensive scientific control, but he has nevertheless produced the goods. Exhaustion of molasses and in consequence, boiling house recovery have in the factory reached a point, never known in South African factories.

I have occasionally visited this factory and been impressed with the extent of its capacity in boiling, cooling and curing. If exhaustion of molasses and boiling house recovery are merely a matter of capacity, the question of crystallisation would be easily solved; but I am sure that other factors are also contributory to the excellent results of New Guelderland. It should be the purpose of the boiling house Committee to raise the level of the boiling house efficiency to that of the small North Coast factory.

Mr. du TOIT: I am under the impression that the question by Mr. Bechard has not been answered yet, in connection with figure 6. I think Mr. Bechard is under the impression that Dr. Hedley said that by simply raising temperature you could get lower gravity purity. Now, Mr. Chairman, I think that is certainly not the case. These graphs only show that a lower purity is obtained when you have a higher saturation temperature, which is something quite different. Then I should also like to point out, in connection with viscosities, that the viscosity, as Dr. Hedley has pointed out, depends on the supersaturation, the temperature, etc., but I think it also depends to a large extent on organic non-sugars, for if we take the different factories in Natal and work them at the same concentration and the same temperature, we will find quite a difference in the viscosity of the different molasses. That, I think, can only be due to the organic non-sugars in the molasses. In so far the

actual viscosity of the molasses might be reduced perhaps by certain methods of clarification.

In connection with the dilution vs. heating, I should like to say this, that at the stage where viscosity really becomes a major problem, I suppose it is during the last stages, and at that stage the supersaturation temperature is fairly high, with the result that the temperature may be increased quite a lot without dissolving any grains. At the same time if it is super-saturated, vertically, of course, it may be diluted by quite an amount, and in that way the viscosity can be reduced, but I doubt it very much whether you will get the same reduction of viscosity by simply diluting to saturation, as you would get by heating up to saturation. I think you will probably get more by reheating. And then, apart from that, I can hardly see how you can add any appreciable quantity of water to massecuite without dissolving sugar, for the moment your water comes into contact with certain parts of the crystal, a certain amount of re-solution must take place. On the other hand, the amount of heat required can be regulated much better than the amount of water, for, as Dr. Hedley pointed out, you can work, say, at that particular temperature where super-saturation occurs. In these conditions, no re-solution can take place. In practice I believe they work about five degrees higher than that. Even under these conditions, I believe the chances are extremely small that any of the sugar will be dissolved.

Mr. BECHARD: Two things have got to be kept in mind when we are dealing with this question, one is the theoretical position, and one is the practically possible, and the same thing applies to the problem raised, dilution vs. heating. Working with the saturation instrument you come to the conclusion very quickly that you can reach a fairly high temperature before dissolution takes place. At the same time it is not practical. The main reason is that you get caramelisation of the sugar. Also that a certain amount of drying takes place round the coils, and transmission becomes very difficult. There is one thing also that one must bear in mind. It has not been emphasised sufficiently, and that is the energy of mixing in the crystalliser. We fear that the strength of the stirring gear in the crystalliser is not sufficiently great to deal with high viscosities and high densities, also the power used at the crystalliser is insufficient. If the revolutions were to be raised to anything like the revolutions necessary to give a good mixing a good many of these problems would disappear.

Mr. HAYES: I should like to join with Mr. Rault in the remarks he made, particularly in connection with the opening of this paper, in which Dr. Hedley says: "During the last few years, a great deal of work has been done on the use of Crystallisers and on Crystallisation in general. In South Africa we have done practically nothing." I think that statement should be qualified. In fact some work has

been done in this country. Mr. Rault mentions one instance where valuable work has been done, and I think it should be the function of any Committee that is formed to help in classifying other data.

Regarding the paper, it is rather difficult to sort out the local practical work from that done in other countries, and I should like to ask Dr. Hedley what the actual experimental methods were in determining viscosity for instance.

Dr. HEDLEY: No, we have no means of determining the viscosity of molasses. As Mr. Macbeth told you, when he was in Hawaii, the viscosimeter was a secret. The viscosimeter which they use in Hawaii has been published this year in their journal.

Mr. HAYES: A great difficulty with the work of this kind lies in taking of samples. Some figures shown here demonstrate the effect of running steamings on a massecuite. I should rather like more information on this, because we have found that it is virtually impossible to take a sample representing the whole bulk of a massecuite. Most people admit that it is not advisable to run any amount of steamings on to the top of a crystalliser, but the difficulty is what are you to do with it? When it has been run on the crystalliser, it is very hard to estimate the damage done, in that it is virtually impossible to take a sample representing the whole bulk of that massecuite. As Mr. Rault pointed out, the work of the crystalliser can really be divided into two stages; the first is the attempt to get out as much sugar as possible by crystallisation, but the second stage is the antagonist, in that it is desired to reduce viscosity to such an extent that the sugar will cure easily. At the Refinery, we work for as complete a separation of molasses from the crystal as possible. We have done numerous experiments on the re-heating of massecuites, and also the feeding with water and have found it amazing how much water one can add to a massecuite if the feeding is done very slowly, and this without any apparent damage to crystals, but in fact with an increased yield.

To mention one instance, we set aside a crystalliser containing a very low-grade massecuite. We expected some difficulty curing it. But by judicious feeding of water, a steady drop in purity was obtained, and a fairly steady increase in yield. But on the whole, we have found that re-heating a massecuite seems the more correct line on which to work for reducing viscosity. Nevertheless

when judicious water feeding is employed to increase crystallisation, good results can be obtained.

Mr. W. A. CAMBELL: Mr. President, I have listened to the debate from various members here. May I suggest to the Director of Experiment Station that Dr. Hedley follows this matter up this coming season with various factories. I can assure you—I am sorry that the owners of other factories are not here to assure you also—that we will give Dr. Hedley every encouragement, and put our staffs at his disposal to bring fruition to the questions arising out of his paper. It is a most important matter to the factory. Mr. Hayes brought some very interesting data, and I do ask you, sir, if you can allow Dr. Hedley, with his technical knowledge to go into the various factories and follow this matter up this coming season, as he has so successfully followed up the question of boilers and furnaces.

Mr. DODDS: Mr. Chairman, this subject of the crystallisation of massecuites is of the greatest importance to South Africa in its influence on the losses of sucrose in molasses, which we know is very high indeed in this country. If I might refer again to the table which we have already considered this morning, we find that in this country we have a purity of final molasses of 45 for the past season and 46 for the season before. In Hawaii, where the matter has been gone into systematically, as we have just heard the purity of final molasses is 34. The result is that the total losses of sucrose in molasses in the country is about 9 or 10% of the total loss per cent. sucrose in cane: in Hawaii it is only between 6 and 7. This difference means a difference of three units in the boiler house recovery, and this involves an amount of 15,000 tons of sugar, annually in our crop. The saving of 15,000 tons of sugar or even a considerable part of it, is, of course, a very important matter, and would justify a considerable amount of expense in experimental work on the subject. In answer to Mr. Cambell's suggestion, I have already strongly recommended to the Sugar Association that some work on these lines be continued during the coming season.

The PRESIDENT: I am sure we are all very much indebted to Dr. Hedley for bringing this very interesting subject before the Conference. I think the biggest compliment which can be paid to this paper is the interesting discussion which has taken place. I would ask you to join me in a vote of thanks to Dr. Hedley for his most interesting paper. (Applause.)