

Remedying Inadequate Crystallizer Capacity

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Received for publication November 27, 1978

Much money is lost each year by beet-sugar manufacturers from excessive loss of sucrose in molasses. There is too much money involved to sell sugar at bargain prices to feed cattle, when it can be sold for a good profit as the re-fined product.

The amount of this loss can be easily calculated by the use of the following formula:

$$\text{\$ per ton of molasses} = (AC - B) \frac{P_1 - P_2}{1 - P_2} \quad (1)$$

where A - the \$ value per ton of sugar and B = the \$ value per ton of molasses; C - the decimal fraction of dry substance in the molasses; P_1 = the purity of the higher purity molasses and P_2 that of the lower purity molasses have the same ds content, the formula gives quite accurate results.

A factory slicing 4,000 tons of beets a day and making 5% molasses on beets, will make 1400 tons of molasses per week. Assuming values of \$300 and \$37 per ton of sugar and molasses, respectively, the \$ per ton of molasses saved by lowering the molasses purity one point from 61% to 60% purity would be \$5.075, or \$7105 per week.

For a company slicing 5 million tons of beets per year, and making 250,000 tons of molasses, the one unit purity drop would involve \$1.28 million. If the molasses purity was reduced to its probable lowest practical or "normal" purity value of 56%, the gain per ton of molasses would be

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\$23.07, or a total of \$5.77 million - a fair annual profit for several sugar companies.

Table 1 shows the molasses performance for North American beet-sugar factories for the 1976-77 campaigns.

Table 1. Average molasses purities (Clerget) for North American beet-sugar factories, 1976-77 campaigns (Courtesy Beet Sugar Institute).

<u>Company</u>	<u>Average</u>	<u>Range</u>
A	(53.0)	(50.0) - 60.0
B	60.8	55.6 - 63.0
C	59.8	55.6 - 70.7
D	58.5	
E	60.5	
F	62.1	59.9 - 63.9
G	62.5	61.0 - 64.0
H	62.0	60.0 - 64.0
I	61.0	58.0 - 63.0
J	58.4	55.2 - 62.5
K	61.8	58.7 - 63.9
L	58.1	
M	59.5	
N	60.3	58.5 - 62.0
averages	60.4	58.1 - 63.7

The low values for the factories of company "A" are omitted from the averages because they are from factories using a special auxiliary process. The purities are Clerget numerators over 1:1 diluted rds denominators. While several manufacturers have made appreciable progress in reduction of molasses purities, as a whole the overall purities are still uneconomically high.

Figure 1 shows a flow plan for a typical raw crystallization station, consisting of a feed-supply tank for the pan, the raw vacuum pan, a receiver (mixer) for the finished massecuite from which the crystallizers are fed (in the case of batch crystallizers these are not needed), a mingler-reheater, and finally centrifugals to separate the molasses from the raw sugar.

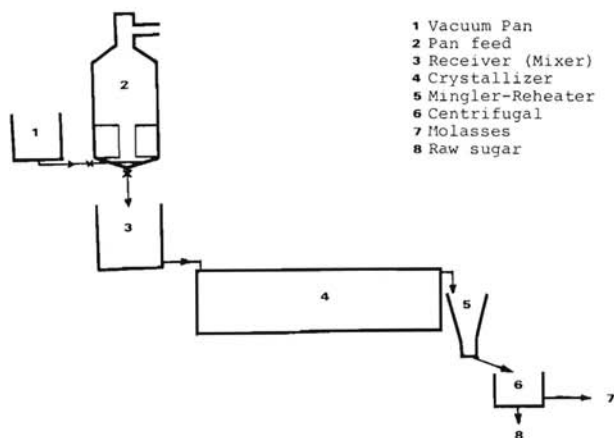


Figure 1. Typical raw crystallization equipment.

DETENTION TIMES IN EQUIPMENT

It is commonly accepted among technologists that in order to complete the crystallization of massecuite which has been properly boiled in the vacuum pans with the usual pan-dropping temperature of about 70°C, at least 30 hours in the crystallizers are required, cooling to a low point temperature of 40°C. This is in addition to the approximate 4 hours spent in the receiver. Table 2 shows detention times in North American factories.

Table 2. Actual detention times in hours in raw crystallizing equipment in North American factories, 1976-77 campaign. (Courtesy Beet Sugar Institute). It should be noted that for Tables 1, 2, and 3, the letter designations do not represent the same companies in each table, to better protect the anonymity.

Company	Crystallizers				
	Vacuum pans	Batch	Lafaeuille	Continuous	Mixers
A	6.3-13.4	26.9-33.4	6.4-8.2	13 - 42	0.3 - 8
B	6 -13			12 - 22	0 - 13
C	5.3- 8.5	11.6-22.2			3 - 9
D	3 - 9	39 -53		45 - 50	3 - 9
E	7 - 9	40 -45	3 -9		3 - 9
F	6 - 8	9 -10.5			3 - 4
G	5.5- 8	30 -36		32 - 40	
H	5.8-12.2			30.4-45.6	
I	12 -15			72 -96	2

Crystallizers - Cont.

<u>Company</u>	<u>Vacuum pans</u>	<u>Batch</u>	<u>Lafaeuille</u>	<u>Continuous</u>	<u>Mixers</u>
J	5 - 7	18 - 20			
K	3 - 6				
Range	3 - 15	9 - 45	3 - 9	12 - 96	0 - 9
Avg.	7.8	27.3	6.7	42	4.5

Three companies using batch crystallizers with Blanchard type agitators are not meeting this requirement; the two companies using Lafaeuille crystallizers are very deficient in detention times, and two of the companies using continuous crystallizers have time shortages.

Thus even though all other variables affecting raw crystallization were in satisfactory condition, which of course is not the case, these crystallizer capacity shortages need rectification.

Addition of more crystallizer units is an unpleasant path. A crystallizer unit today costs about \$300,000 f.o.b., and probably involves another \$100,000 installed. There is likely to be a space problem. Most factories do not have empty spaces for such equipment, and the solution to this might be the installation of the new vertical-type units such as the Toury and the B.M.A., which can be placed on the ground outside the factory building, and connected with magma pumps.

HIGH TEMPERATURES

Since the purpose of this paper is to call attention to this variable, it will be given more attention than other equally important variables. A fundamental principle of sugar crystallization is that as much as possible should be done as far "upstream" as possible. This is, of course, because higher temperatures are normally used with higher purity materials, and crystallization rates are more rapid, and there are more control variables which can be advantageously manipulated in the vacuum pan, such as better agitation, sensitively adjusted supersaturation, syrup

additions, and others. This paper examines primarily the variable of boiling temperature as it affects the crystallization rates.

All the crystallization possible should be done in the white boiling, then all possible of the remainder in the intermediate boiling, and the green syrup from this boiling serves as the feed material for the raw pans. Both white and intermediate mixers should be kept on the full side as much as possible, without causing problems in mixing the grain of different strikes of massecuite. The centrifugals should be operating all the time between pan drops, with no "holes" or idle periods.

In the raw crystallization system, all possible should be accomplished in the pan, and again the raw mixer should be kept on the full side, as considerable crystallization can be done there. The final unit is the mingler-reheater, and if a Stevens type is used, the upper hopper should be kept as full as possible. One factory gains as much as 1 purity point lowering of the molasses, as compared to keeping the level very low.

It is my opinion that most factories are not boiling their raw strikes at temperatures as high as possible without degrading the juice, and are thus wasting valuable crystallization potential.

Table 3 shows the temperatures in raw crystallization units in 1976-77.

Table 3. Temperature data in °C in raw crystallization units for North American factories, 1976-77 campaigns (Courtesy Beet Sugar Institute).

Company	From vacuum pan	Crystallizer low point	To centrifugals
A	72 - 80	39 - 56	46 - 59
B	66 - 77	39 - 48	46 - 51
C	74 - 80	54 - 61	51 - 56
D	60 - 85	36 - 53	46 - 59
E	76 - 82		52 - 59
F	78 - 85	35 - 42	48 - 55

Company	From vacuum pan	Crystallizer low point	To centrifugals
G	64 - 76	48 - 62	48 - 62
H	80	40	54
I	76	36	42 - 46
Range	60 - 85	35 - 62	42 - 62
Average	73	48.5	52

Note that "From vacuum pan" values are the massecuite temperatures after being "Brixd-up," which in a pan without absolute pressure control, often increases the temperature over that of the rest of the boiling. Considering this, it seems probable that only one company has consistently carried a higher than usual boiling pattern.

EFFECT OF TEMPERATURE ON RATES OF CRYSTALLIZATION

It is a well-known "rule of thumb" among chemists that, on an average, an increase of temperature of 10°C will approximately double a crystallization rate. This is a very rough approximation, and deviations from it can be very wide, and even reversed.

Some data from the literature are reproduced to show the temperature effects on sucrose crystallization.

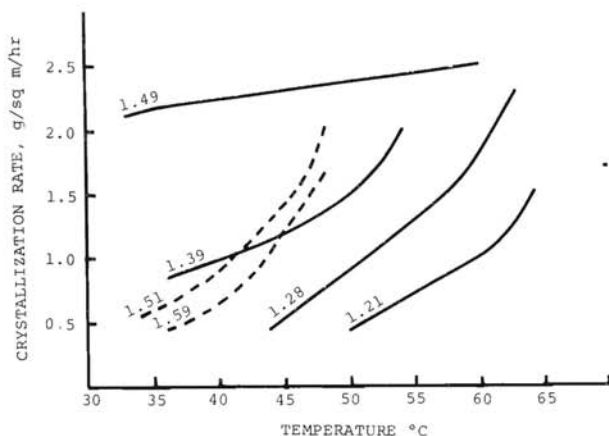


Figure 2. Crystallization rates in typical crystallizer raw massecuite (4). Claassen supersaturations. (Massecuite; 35% crystals, 92.7 ds, 76.7 apc Syrup, 62.5 apc, 83.5 rds).

Table 4 shows data from Kukharenko, on pure sugar-water solutions.

Table 4. Velocity of crystallization in pure sucrose solutions, derived from data of Kukharenko (3).

Kukharenko Supersaturation	% rate of crystallization increase 10 °C intervals		
	40° - 50°	50° - 60°	60° - 70°
1.005	65	42	150
1.010	72	47	136
1.015	63	67	91
1.020	78	84	81

Figure 2 shows crystallization rates in crystallizer massecuite, and the data in Table 5 are taken from these curves.

Table 5. Rates of crystallization in crystallizer massecuite, calculated from data of McGinnis, Moore and Alston (4).

Claassen Supersaturation	10°C temperature intervals	% increase in rate
1.22	54 ° - 64 °	130
1.28	52 ° - 62 °	107
1.39	44 ° - 54 °	77
1.49	50 ° - 60 °	5

Unfortunately no data are readily available in the 80°-85°C region. Susic (5) conducted factory scale tests at 80°C, with obviously very rapid rates of crystallization. Attempts at measuring such rates in the laboratory have indicated they are so rapid that precise control is difficult on a small scale.

Thus, if the boiling temperature is raised as high as can be done without degradation of sucrose or nonsucroses, the rates of crystallization will be quite rapid. As the concentration of the sucrose in the mother liquor is lowered in the latter part of the boiling, the massecuite temperature will have to be lowered gradually to maintain the supersaturation, and after tightening, the pan will end at about 70°C, with a maximum amount of crystallization performed. There will be much less for the crystallizers to

do, and if supersaturations are kept optimum in them, the total crystallization achieved will be much more than if the pan had accomplished less. The percent gross crystallization (percent on sugar in the pan feed) should be close to 60%, and with 6 to 8% added by the crystallizers, better molasses exhaustion should result.

The percentage of crystallization in a pan boiled by this "hot" technique, if the massecuite is properly tightened and dropped at 70°C will be the same as in a boiling at a lower temperature, but much less boiling time will have been required, and certain slow-boiling massecuites (because of the nature of their nonsucroses) will crystallize much more rapidly. There will be much less for the crystallizers to do, and if the factory is short of crystallizer capacity, probably the best possible results with the existing equipment will be attained.

It should be kept in mind, however, that this "hot" boil-

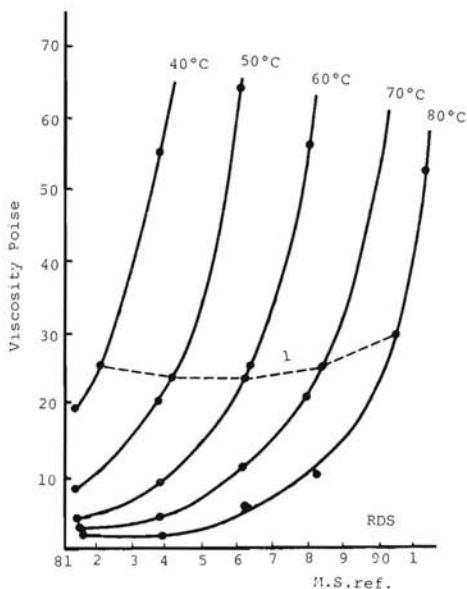


Figure 3. Viscosity of typical beet molasses at various rds values and temperatures. The curve, 1, is at 1.0 saturation (5).

ing technique will be of much less, or no interest at all, if the factory has ample crystallizer capacity, - enough to give crystallizer detention times or 30 - 40 hours or more.

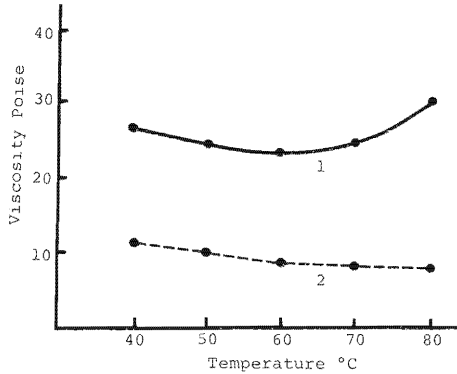


Figure 4. Viscosity of typical beet molasses, 1, and pure sucrose water solution, 2, at various temperatures. Both are at 1.0 saturation (5).

VISCOSITY

When boiling at high temperatures it is necessary to keep the rds of the massecuite high, not only because of the more rapidly-increasing mass of crystals, but also to maintain high enough supersaturation.

Fortunately viscosities of the syrup phases will not increase appreciably. The recently published work of Karadzic and Terek (2) shows that in the ranges of rds and temperature which would be involved in high-temperature boiling, the syrup viscosity changes only by a few poises at constant saturation, and we may reasonably assume, at constant supersaturation. This is shown clearly in Figures 3 and 4.

FACTORS IMPORTANT IN HIGH-TEMPERATURE BOILING

1. The molasses produced must have a pH value higher than 6.9. If lower, milk of lime or magnesia should be added to any of the following: intermediate boiling, raw pan

feed, or the raw pan. Do not use caustic soda or soda ash, as these are very melassigenic.

2. Temperatures should be raised by degrees when first trying high temperature boiling. Watch for evidences of degradation, such as inversion, or color increase, not only in the massecuite as discharged, but also in proof-stick samples taken at intervals during boiling. If such evidences are found, boiling temperatures should be lowered several degrees. Generally a suitable boiling temperature will be in the area, 80°-82°C.

3. With proper seeding with milled seed, temperatures should be held in the high range, and the massecuite rds held sufficiently high, and also the N/W, of course, so that the supersaturation will be in the correct range. The rds will surely be over 93 before the massecuite is dropped, and the N/W over 3.0.

4. During boiling, the massecuite should be carefully kept in the correct range; i.e., below the saturation at which false grain would form.

5. The mass of crystals will increase more rapidly than at lower temperatures, and after a certain point is reached, the supersaturation cannot be held by increasing the rds because of the high viscosity of the massecuite, and the temperature must then be gradually lowered. Do not fail to do this, as otherwise the supersaturations will become too low for maximum crystal growth, once the most suitable boiling consistency has been reached and is being maintained.

6. When the pan is filled and Brixing-up is started, watch for lowering temperatures, as if there is not absolute pressure control this will happen, and false grain will form if the labile zone is entered.

7. The massecuite must circulate sufficiently during boiling. If there is no mechanical stirrer, there must be vapors of sufficient temperature in the calandrias. This may mean an alteration of the factory's steam and vapor distribution system, and the cost of the additional energy could contra-indicate high-temperature boiling.

8. The crystallizer temperature pattern may well be about the same as with lower-temperature boiling, with the same low-point temperature.

9. If the factory has continuous raw centrifugals, the high rds of the massecuite fed to them may cause difficulties with certain models.

Figure 5 shows the appearance of a typical pan temperature-vacuum chart for an 80°C boiling. Note that the temperature referred to is that of the majority of the boiling, and not just that at which the massecuite is dropped.

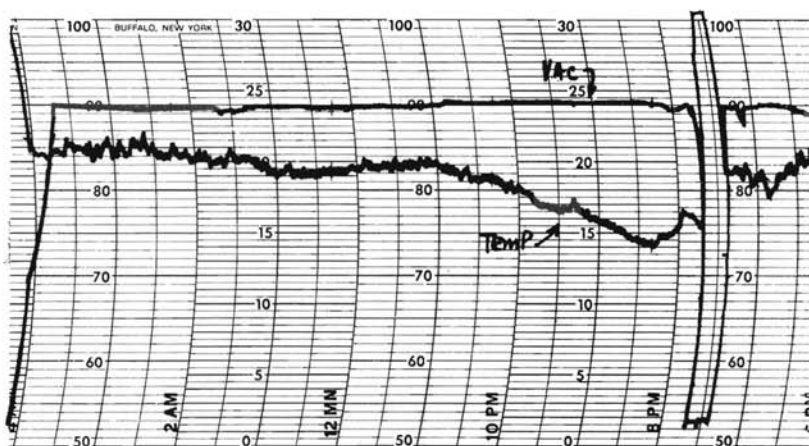


Figure 5. Appearance of pan chart of 80°-85° boiling. (simulated because of inability to secure permission to reproduce original.)

EXAMPLES OF RESULTS OF HIGH-TEMPERATURE BOILING

It is difficult to obtain statistically-significant data, unless all other variables are correctly set, and I have been unable to show these figures. However, use of high-temperature boiling has resulted in some of the lowest purity molasses in the history of certain factories.

EFFECTS OF OTHER VARIABLES

It is obvious that raising the temperature of raw boiling is only one of numerous ways in which the molasses exhaustion can be made more complete, and also that it is not applicable under all circumstances.

Following are listed some of the other factors which frequently are found responsible for poor performance.

1. The raw massecuite purity should be as low as possible, and still produce a satisfactory raw sugar, which will not recycle too much color and floc. This is the result of the application of the principle of doing more crystallization upstream in the intermediate pan. This purity cannot be more than 20 purity points higher than the molasses purity desired, unless precentrifuging is used.
2. The raw pan feed syrup must be free of fine crystals, which can be made certain by heating the raw pan feed syrup to a temperature equal to or above that carried in the pan. Hotter feed, flashing as it enters the pan, can enhance massecuite circulation if properly distributed well outside the calandria center well.
3. The pans should be seeded with milled seed slurried in isopropanol, with the correct number of seed to yield ample crystal surface area for crystallization, and yet not so fine that purging will be hindered.
4. False grain must not be permitted to enter at any point. Low purity molasses and good quality raw sugar

cannot be obtained with badly mixed grain.

5. The viscosity of the molasses as centrifuged should be set the same as for molasses from lower temperature boilings.

6. The syrup phase should be kept in the proper supersaturation range in all of the raw crystallization units. Using the VanHook supersaturation formulation (same N/W in numerator and denominator) this means 1.3 - 1.45 in the pan, receiver, and crystallizers, and a reheating to a little over 1.0 for centrifuging.

7. Water must not be added after the massecuite has left the pan. Condensates from pan steamout should not be routed to the raw pan receiver. Water is by far the most melassigenic nonsucrose substance. If the massecuite consistency must be reduced, and it cannot be accomplished by raising the temperature curve, use final molasses which has been freed of fine grain and air bubbles. It mixes in more quickly than water, and does no damage other than slightly reducing the syrup phase supersaturation.

8. The vacuum pan should be properly equipped, preferably with a mechanical circulator; with a condenser system which permits operation by any desired pressure; with vacuum or absolute pressure control; a meter of some type to permit estimation of supersaturation and perhaps consistency; a suitable pan thermometer capable of rapid rate of response should be located at a point of good vapor velocity in the entrainment separator, so that it measures the vapor temperature as it leaves the massecuite surface. Measurements taken much below the top massecuite level are subject to considerable error, and vary with the circulation pattern.

9. Saturation determinations should be made by the laboratory at appropriate intervals, either with a Saturascope or with saturation runs, so that supersaturations can be

estimated with validity.

10. If the pan does not have a mechanical circulator, there should be sufficient vapor pressure in the calandrias to maintain good circulation. Circulation can be accomplished by bleeding steam or water into the boiling massecuite, but this is very costly, energy-wise.

11. The pan receiver should be properly-sized and insulated, so that rapid crystallization can continue while the massecuite is in residence there.

12. The crystallizers should have effective heat transfer equipment and agitation. Cooling water should be controlled so that the temperature difference between the water and the massecuite will not result in plating out crystals on the massecuite side.

13. The interiors of the cooling elements should be cleaned at long intervals by "boilout" or other means, to remove microbial growths.

14. All raw crystallization equipment should be kept full and in use at all times, growing crystals as fast as possible, and this includes pans. If the pans get ahead of the syrup supply, reduce the amount of seed to increase grain size.

15. Temporary high massecuite viscosities in the crystallizers should be relieved with raised temperatures and not with water.

16. The massecuite that flows through the crystallizers should be uniform.

17. All control and measuring equipment should be kept accurately calibrated and in use at all times.

If a Stevens mingler is used, keep the upper hopper full to obtain more crystallization before the massecuite reaches the heating coils.

I hope I have gotten across the points that increased boiling temperatures is only one of many ways to increase molasses exhaustion, and may not be indicated for certain factories because of shortage of steam, or centrifugal requirements, but if not prevented by these factors, and for those very short of crystallizer capacity, the technique can and has proved profitable.

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