

SOME IDEAS ON A NEW REFINERY BOILING SCHEME

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Abstract

Crystal characteristic dimensions and mass growth rates, in relation to the crystallisation process, have been investigated. Theoretical analyses show that the relation between crystal growth rate and crystallisation time can be approximated by two straight lines; the first shows that about 60% of the growth can take place in 15% of the time, with the second line reflecting the lower growth rate thereafter. These findings are used to propose new concepts about pan boiling in refineries.

Introduction

The crystallisation of sucrose, under conditions relevant to refining, has been studied over the last four years at the Sugar Milling Research Institute (SMRI). The work has dealt with the measurement of the sucrose crystallisation rate. This rate is well documented in the literature (Guimaraes *et al.*, 1995; Grimsey and Herrington, 1994; Maurandi *et al.*, 1984), and a number of definitions have been proposed. The one generally considered the most useful (Bubnik and Kadlec, 1992; Kraus and Nyvlt, 1994) is a mass growth rate expressed as the mass of sucrose deposited per unit time and per unit crystal surface area. A knowledge of the crystal dimension is required, and this has usually been obtained by sieving.

Derivation of parameters

In the present work the approach described by Bubnik and Kadlec (1992) has been followed, but crystal dimensions have been based on an image analysis system which yields two crystal dimensions. A characteristic crystal dimension can then be defined. In addition, three crystal shape factors can be defined, for the conditions relevant to the present work. It is then possible to derive a mass growth rate, G , in kg/s/m^2 . This has been described in detail in a previous paper (Lionnet, 1998b). G is given by Equation 1.

$$G = \frac{3\alpha\rho}{\beta}V \dots\dots\dots (1)$$

where α is a dimensionless volume shape factor, for the sucrose crystal; ρ is the density of sucrose, namely 1587 kg/m^3 ; β is a dimensionless surface area shape factor; and V is a linear crystallisation velocity, in m/s .

The numerical values of α and β , as relevant to the techniques used for the present work, were found to be 0,31 and 2,94, respectively (Lionnet, 1998b).

Finally, V is given by $V = \frac{dD_c}{dt}$

where D_c is a crystal characteristic dimension, in meters, based on the specific image analysis system used here, and t is the crystallisation time in seconds.

Another equation will be needed. Its derivation proceeds as follows:-

$$D_c = (L^2W)^{\frac{1}{3}} \dots\dots\dots (2)$$

by definition, where L and W are the crystal dimensions (m) along the b -axis and c -axis, respectively;

$$\alpha = \frac{m}{\rho L^2 W}$$

by definition, where m is the mass (kg) of one sucrose crystal;

and $N = \frac{M}{m}$

where N is the total number of crystals, M the mass (kg) of all the crystal, and m the mass (kg) of one crystal.

Then, $N = \frac{M}{\alpha\rho L^2 W}$

or $N = \frac{M}{\alpha\rho(D_c)^3}$

D_c can now also be expressed as

$$D_c = \left(\frac{M}{\alpha\rho N} \right)^{\frac{1}{3}} \dots\dots\dots (3)$$

Equipment

The SMRI pilot pan was used to perform a large number of refining boilings, using liquors from local refineries. This pan has been described in detail previously (Lionnet, 1987, 1989; Lionnet and Moodley, 1996) and it has been shown that its behaviour is similar to that of industrial batch pans, particularly refinery pans.

The pan is well instrumented; feed, condensate and massecuite masses are available at any stage of the boiling. These data, in conjunction with the relevant analyses, allow all the necessary material balances, including the mass of crystals, to be determined. The pan also has a sampling facility, which allows a small sample of materials to be removed, at any stage of the boiling, with a minimum of disruption. Massecuite samples can be nutschted immediately, using the sampling device which is a small nutsch filter. The mother-liquor and crystals, at any stage of the boiling, can thus be analysed.

The measurement of D_c

Pilot plant scale

Refinery liquors were concentrated in the pilot pan, seeded with slurry and boiled. Pan contents were sampled throughout the run, the mother liquor was analysed for brix, and the crystals for L and W. Relevant material balances were performed. The crystal characteristic dimension, D_c can now be measured directly by the image analysis of the crystals (equation 2), and calculated using equation 3. A typical example is discussed in detail.

It is assumed that the number of crystals in the massecuite is constant. Then, N can be calculated, using the strike (final) massecuite data, using the equation:

$$N = \frac{M}{\alpha \rho L^2 W}$$

For the selected example:-

$$M_{\text{final}} = 6,68 \text{ kg}$$

$$L_{\text{final}} = 268 \times 10^{-6} \text{ m}$$

$$W_{\text{final}} = 188 \times 10^{-6} \text{ m}$$

$$\alpha = 0,34$$

$$\rho = 1587 \text{ kg/m}^3$$

Then $N = 9,2 \times 10^8$

At any time t, during the boiling, N is constant and equal to $9,2 \times 10^8$; α and ρ are constant and the mass of crystals in the massecuite, M_t , can be calculated through material balances. Equation 3 can thus be used to calculate the value of D_c at any time t.

Values of D_c are also available by the actual measurement of L and W, for the time t. It is thus possible to compare the two values as has been done in Table 1 and Figure 1.

Data are available for other runs; in all cases the results are similar to those discussed above.

Three comments may be made from the results:

- The calculated and measured values of D_c agree reasonably well. This confirms the validity of the approach.

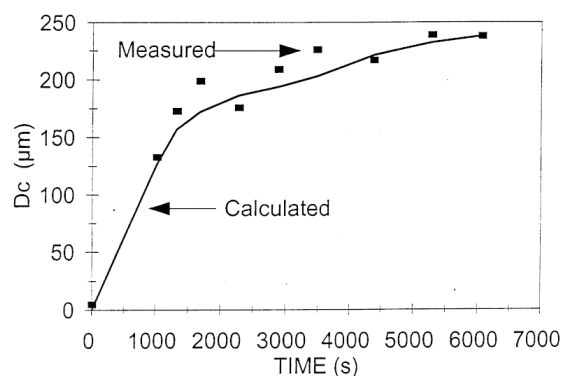


Figure 1. The measured and calculated values of D_c plotted against crystallisation time.

Table 1. Measured and calculated values for the crystal characteristic dimension D_c

Crystallisation time (s)	L (μm)	W (μm)	D_c measured (μm) ($L^2 W$) ^{1/3}	Crystals (kg)	D_c calculated (μm) ($M / \alpha \rho N$) ^{1/3}
0	0	0	0	0	0
1020	149	105	133	1,01	127
1320	198	131	172	1,92	157
1680	228	152	199	2,53	172
2280	199	137	176	3,21	186
2880	239	159	209	3,61	194
3480	259	171	225	4,13	203
4380	250	164	217	5,36	221
5280	273	184	239	6,20	232
6060	268	188	238	6,68	238

- The form of the curve is interesting. There is a rapid initial increase in the value of D_c followed by a much slower rate of increase. White *et al.* (1998), using an analysis based on growth rate dispersion modelling, arrive at a similar finding. This again confirms the validity of these results.
- If these trends are clearly evident under industrial conditions, then optimisation of the crystallisation process in refining may be possible.

Industrial scale

Samples of massecuite were taken, at specific times, throughout the boiling period, from pans in two local refineries. The proof stick was used, and the samples were kept in sucrose saturated glycerol. L and W were measured by the usual SMRI image analysis system. The sampling techniques, sample preservation and time delay between sampling and the image analysis, were not ideal when compared to the situation with the pilot pan at the SMRI. Nevertheless it is clearly evident that the form of the relationship between D_c and crystallisation time is similar. Typical results, from industrial batch pans are shown in Figure 2.

If the trends shown in Figure 2 are approximated by using two straight lines, then about 60% of the final value of D_c is reached in about 15% of the crystallisation time. The slope of the straight line yields an approximation for a linear crystallisation velocity. This velocity is about $0,25 \mu\text{m/s}$ for the first straight line, but only about $0,03 \mu\text{m/s}$ for the second; the initial rate is thus about 8 times faster.

Industrial Implications

Reference to Figures 1 and 2 shows that refined sugar crystals with a characteristic dimension (D_c) of 150 to 200 μm are pro-

duced about 30 minutes after nucleation; a further 40 to 60 minutes are then needed for D_c to reach 250 to 300 μm . The linear crystallisation velocity is about eight times faster during the first 30 minutes.

The question that should now be asked is whether the above findings can be used to optimise the industrial crystallisation of white massecuites in terms of crystallisation time, pan capacity and pan design.

The following points need thorough investigations.

- Effects on yield. Stopping the boiling after the first 30 minutes, without having reached the required yield, is obviously unacceptable. The concept of backblending needs to be considered, for example:
 - (i) Boil the massecuite until crystals with D_c values of 150 to 200 μm are produced.
 - (ii) Strike.
 - (iii) Cure the massecuite to produce refined sugar. A portion of the jet is recirculated to the pan feed while the rest is stored, to be boiled separately on its own.

The factors to be investigated here would be the final brix and crystal content of the massecuite in (i) above, the handling of this low brix white massecuite, the split of the jet in (iii), and the impact of backblending on sugar quality, particularly in terms of colour and of ash.

- Continuous boiling - The modifications discussed above could suit continuous boiling, for the massecuite in (i) above. Encrustation and scaling (Rein, 1990) can be severe problems in continuous pans, particularly at high purities. The low massecuite brix and crystal content proposed here would possibly counteract the effect of purity.

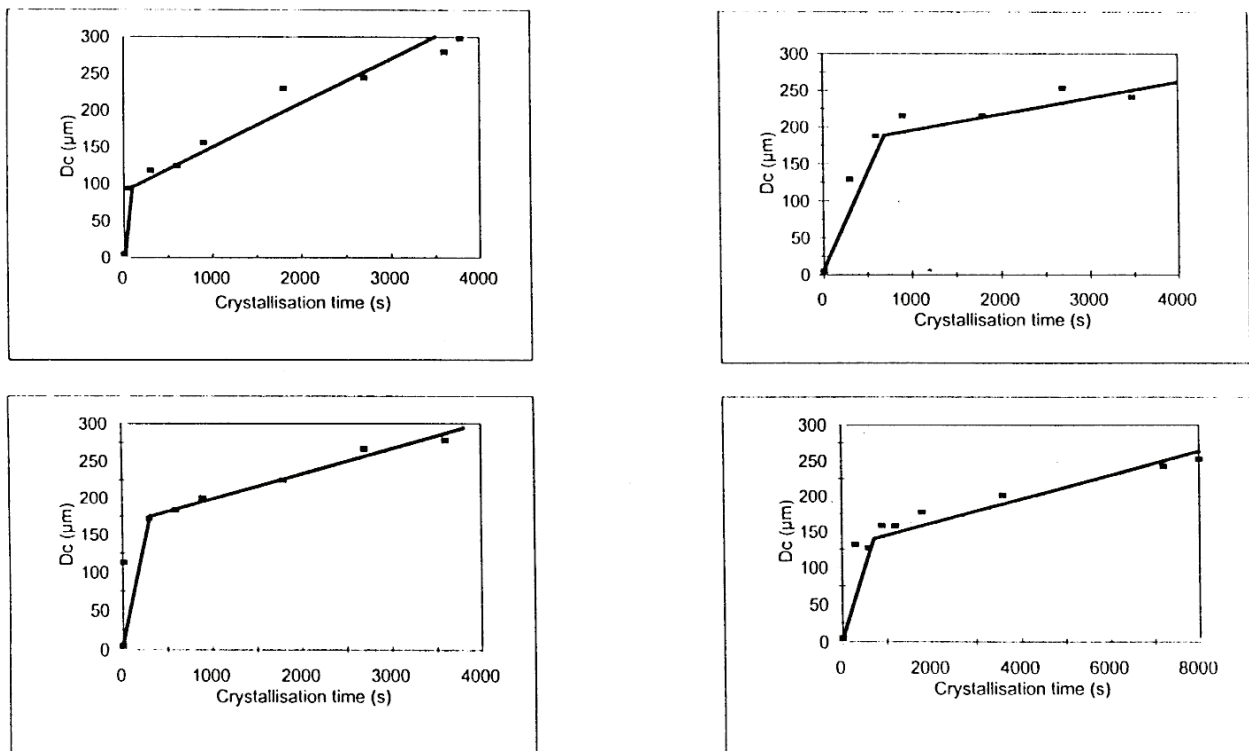


Figure 2. Crystal characteristic size plotted against crystallisation time, for industrial pans.

The mother-liquor is still supersaturated and encrustation problems on the pan walls would need attention. Temperature control and the spraying of liquid on pan walls have been suggested to control these problems.

- Technical and commercial implications of smaller refined sugar crystals - Process modifications to maximise the use of the first crystallisation period will probably result in smaller crystals. The concept of backblending and the boiling procedures described above were tested as a batch process, in the SMRI pilot pan. The refined sugar produced had MA (mean aperture) values of 0,53 to 0,56 mm, and specific grain sizes of 0,53 to 0,43 mm. This does not seem to be out of line but would need to be confirmed industrially. Sugar quality, in terms of colour and of conductivity ash, for the back blended boilings also appeared normal; affinated sugar colours ranged from 12 to 15 ICUMSA units, while conductivity ashes were close to 0,001%.

During the batch test the final massecuite brix ranged from 84 to 85, with crystal contents of about 45% (m/m). Normal white massecuites from the SMRI pan usually have brixes of about 90 and crystal contents of about 54%.

Conclusion

Much more work is required to test the ideas presented here. The basic concept of different crystallisation velocities, is however, well established.

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