

C-MASSECUITE PROCESSING IN THE LABORATORY: SOME PRELIMINARY RESULTS

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Abstract

It is widely accepted that simulating full scale boiling of C-massecuite on a small experimental scale is particularly difficult if not impossible due to the very high viscosities encountered. Recent work using the new pilot scale pan in the THS laboratories has demonstrated that it is possible to produce high brix C-massecuite on a small scale. To extend this test facility, a set of six batch laboratory crystallisers has been refurbished and fitted with the necessary controls to undertake cooling crystallisation of massecuite produced by the pan. These crystallisers can be programmed to operate with any desired cooling profile. Each crystalliser doubles as a Nutsch filter, allowing the progress of crystallisation to be monitored by sequential Nutsching of the individual crystallisers at intervals over the total crystallisation period. Preliminary results indicate that target purity can be achieved with this combination of boiling and cooling crystallisation in the laboratory.

Keywords: C-massecuite, pan boiling, crystallisation, exhaustion

Introduction

During the 1970s, Tongaat-Hulett Sugar Ltd (THS) undertook a major investigation into the exhaustion of low grade massecuities. Influenced by the difficulties of simulating pan boiling on a pilot scale, the experiments were designed to use a pilot scale crystalliser installed at the Darnall factory so that it could process massecuities produced in the full-scale factory pans. The work was structured to investigate the factors which affect exhaustion using the approach of factorially designed experiments (Lionnet, 1978). The extensive body of data from this experimental program were supplemented with results from boiling down tests, following the procedures developed at the Sugar Milling Research Institute (SMRI) by Bruijn (1977). The entire body of data was evaluated in detail by Lionnet and Rein (1980) who fitted a mathematical model of the cooling crystallisation process to the experimental data, providing fundamental information on sucrose solubility and crystallisation rate. The results demonstrated the importance of high non-sucrose to water ratios in achieving high levels of exhaustion, and recommended the use of 'high brix, high purity' C-massecuite as the best route to low final molasses purity. It is also clear from the results that, for low grade massecuities, pan boiling and cooling crystallisation need to be considered in combination, rather than as individually optimisable processes, if the maximum overall exhaustion is to be achieved. The sucrose solubility data estimated from fitting the model to the experiment measurements correlated well with the target purity estimated using the formula of Matthesius and Mellet (1976), although there was an indication that the target purity was too high by approximately 1.5 to 2 units.

Rein and Smith (1981) subsequently analysed both the boiling down data presented by Lionnet and Rein (1980) and some subsequent test results, producing a new target purity formula. This formula was later superseded by a formula designed to accommodate very low reducing sugar ash levels (Smith, 1995). The currently accepted target purity formula has been confirmed by Sahadeo (1998).

Despite the immense value of this previous work on the exhaustion of low grade massecuites, there is still scope for further work in this area. A facility for performing both pan boiling and cooling crystallisation on a pilot scale could be used to investigate a number of important issues:

- the exhaustability of new process streams, such as those generated by the emerging technologies for direct white sugar production (e.g. Fechter *et al.*, 2001).
- investigating the behaviour of refractory massecuites (e.g. Koster *et al.*, 1992) under closely controlled conditions.
- closer monitoring of the crystallisation process during pan boiling, to identify the relative importance of sucrose solubility and crystallisation rate on molasses exhaustion.

The new design of pilot scale batch pan (Love and Walthew, 2002) recently installed in the laboratories at THS provided an opportunity for developing such a facility.

Equipment and Techniques for Pilot Scale Pan Boiling

In the description of the new THS pilot pan (Love and Walthew, 2002) it was reported that some initial success with boiling C-masseccuite had been achieved. Further tests revealed, however, that the direct drive stirrer that was installed on the pan had insufficient torque to continue rotating during the final stages of boiling if the massecuites were to approach the tightness achieved in the best performing full scale pans. To address this, a gearbox with a 10:1 reduction ratio was installed between the motor and the stirrer shaft. Using the electronic variable speed drive of the motor, the impeller speed could be kept within the original design range while being able to deliver a much higher torque. With this new arrangement, stalling of the stirrer driver is no longer a constraint to producing tight massecuites.

The pan technique for boiling low grade massecuites in the pilot pan is still somewhat preliminary and semi-automatic. The basic procedure is as follows:

- 1 Fill the pan with sufficient graining charge so that it will cover the top tube plate when concentrated to the graining point. The material used for this is A-molasses which has been adjusted to 72 purity (to ensure proper grain development) by the addition of dissolved sugar.
- 2 Concentrate the graining charge to the correct graining point. By trial and error this was found to be defined by a boiling point elevation (BPE) of 11°C, corresponding to a brix of approximately 85.
- 3 Hold the pan at the graining point using automatic control of BPE with water feed.
- 4 Seed the pan with 40 ml of standard ball-milled slurry seed in alcohol.
- 5 Wait until sufficient grain is observed, taking samples from the proof-stick and observing them on a microscope slide (approximately 5 to 10 minutes).
- 6 Reduce the BPE set-point to 10°C to allow further grain development without the possibility of secondary nucleation.
- 7 Once it is evident that there is an adequate seed present with no secondary nucleation occurring, switch over to feeding with B-molasses.
- 8 As the feeding with B-molasses continues (using BPE control of feed) increase the BPE set point to ensure sufficient driving force for the crystallisation as the purity of the mother liquor reduces.
- 9 Using the on-line mass balance results displayed by the control computer, monitor the estimated massecuite purity. At a massecuite purity of 53, switch over to water feed and continue to brix up the pan on water feed.
- 10 The final brixing should achieve the highest possible concentration based on the flow characteristics of the massecuites (dry solids values approaching 93 have been achieved).

It must be remembered that it is generally not possible to operate and control batch pans from first principles and that fully automated pans are normally set up to follow the best practices of a competent and experienced pan boiler. Further work is necessary to optimise the boiling techniques for the pilot pan before full automatic boilings can be implemented.

Equipment and Techniques for Pilot Scale Cooling Crystallisation

The approach adopted by Lionnet (1978) in designing a pilot scale crystalliser was to provide a capacity that was sufficiently large, so that samples taken for analysis during the cooling cycle would be relatively small and have no material effect on the experiment.

A different approach was taken in developing the current cooling crystallisation facility, prompted in part by the availability of equipment originally designed and built by the SMRI for boiling down tests Bruijn (1977). This approach uses six identical crystalliser vessels of approximately 500 ml capacity. These vessels are all initially filled with sub-samples of the same massecurite as discharged from the pan and subjected to the same cooling profile. The vessels are constructed of brass and have an integral water jacket, as shown in Figure 1.

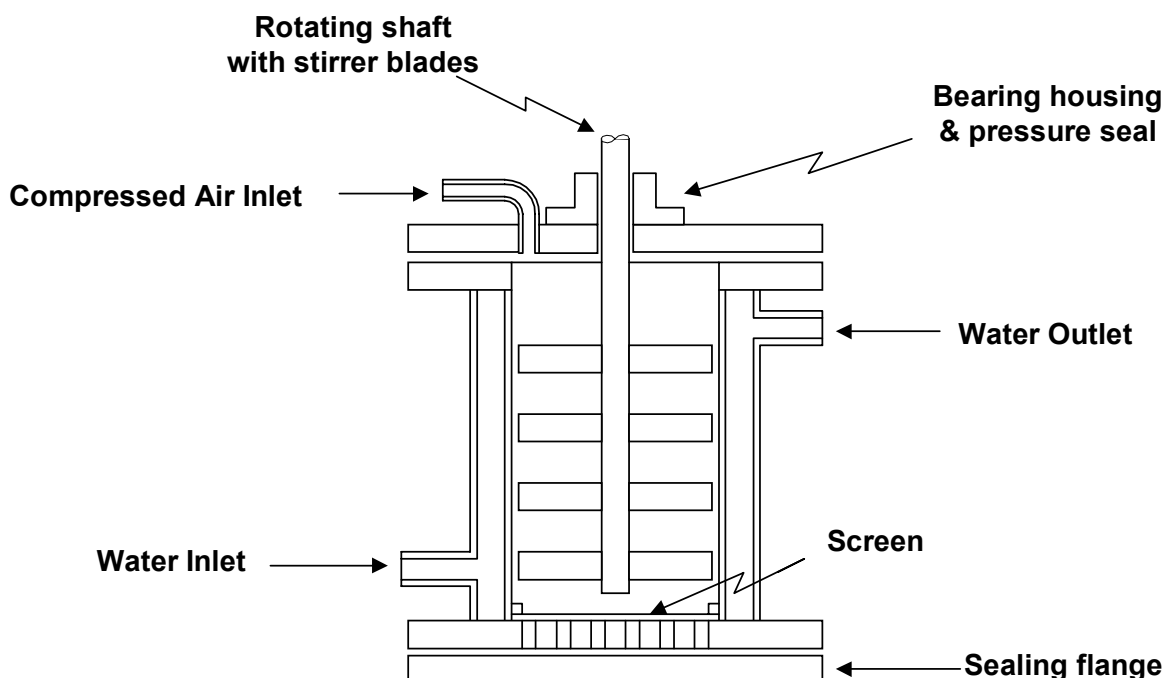


Figure 1. Details of individual crystalliser vessel.

A particular feature of these crystalliser vessels is that they are designed to double as Nutsch filters. Each vessel is bolted to a top flange which is fitted with a compressed air connection to provide the necessary pressure for filtration. The stirrer shaft is fitted with a seal to prevent air leakage during the pressure filtration. The base of each vessel is fitted with a filtration screen, supported by the bottom flange which is provided with a number of drainage holes. To prevent drainage during normal operation, the base is closed with a sealing flange.

To obtain a Nutsch sample, the sealing flange is removed, compressed air is connected to the top of the vessel and the filtered molasses is collected in a beaker placed underneath the crystalliser. It is important to discard the initial runnings as this can be molasses which has seeped through the screen during normal operation and is not representative of conditions within the crystalliser.

By Nutsching the crystallisers sequentially at intervals throughout the cooling cycle, it is possible to monitor the progress of the crystallisation process.

The individual crystalliser vessels are all fitted onto a support frame with a common drive motor and cooling water system, as shown schematically in Figure 2.

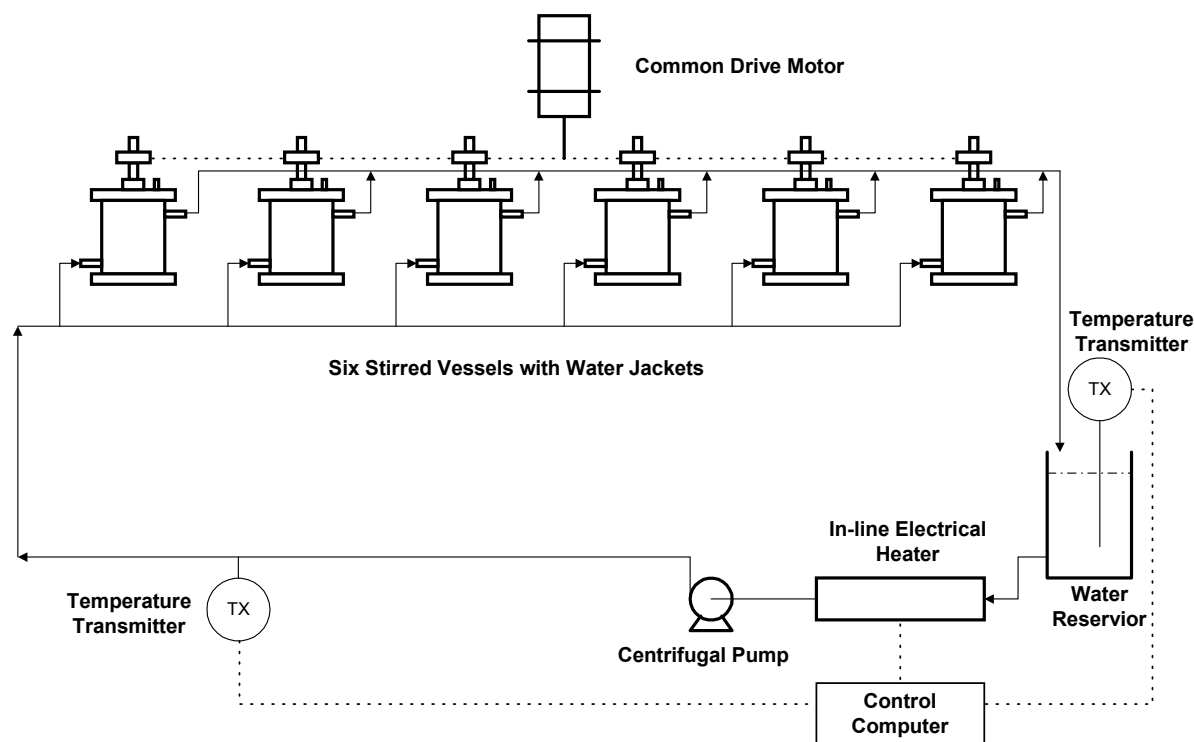


Figure 2. Schematic diagram of cooling crystalliser facility.

It has been found that natural cooling from the water circuit is sufficient to achieve the maximum cooling rates desired, and any desired cooling profile can thus be achieved by varying the quantity of heat addition to the water circuit. A personal computer provided with an interface card has been programmed to provide the necessary control functions. The control program allows the user to define the desired cooling profile by selecting a type of mathematical function (linear, exponential or polynomial) and specifying its parameters. The control program uses the specified function to generate a temperature set point and uses this as the input to a PID control loop which adjusts the heat input to the in-line electrical water heater. The control computer also provides data logging so that the full temperature cycle of a test can be recorded and reviewed to check the quality of the temperature control and ensure that no significant temperature excursions have occurred.

Initial tests with this crystallisation apparatus were not successful; no Nutsch samples could be obtained because the massecuite would not filter through the screen. Inspection of the massecuite in the crystalliser vessels showed it to be aerated with tiny bubbles and consequently light cream in colour. It appeared that the stirring was beating air into the massecuite, causing an increase in the effective viscosity of the massecuite and hampering filtration. To address this problem, the existing fixed speed motor and gearbox assembly was replaced with a variable speed motor and gearbox, allowing the stirrer speed to be reduced from 12 to 2 rpm. This eliminated the aeration and filtration problems.

Results of Massecuite Boilings

The objective of the preliminary work reported in this paper was to demonstrate that C-massecuite processing, covering both pan boiling and cooling crystallisation, could be undertaken on a pilot scale in the laboratory and achieve the performance levels of full scale factory equipment. The major focus was on the ability to achieve target purity of the Nutsch molasses at the end of the cooling crystallisation process (with cooling to 40°C). Given this experiment focus, the initial pan boilings concentrated on producing high brix massecuites suitable for feeding the cooling crystallisation process. Detailed monitoring of the progress of crystallisation during boiling by sampling and analysis was not undertaken at this stage.

An example of some of the logged data collected during a pan boiling cycle is shown in Figure 3. The massecuite purity is that estimated by the on-line mass balance. The steps in the boiling point elevation trend show how the set point was adjusted manually to drive the crystallisation. The drop in BPE off the bottom of the graph at around 150 to 170 minutes is as a result of the necessity to stop the pan for a period to empty a vacuum condensate tank.

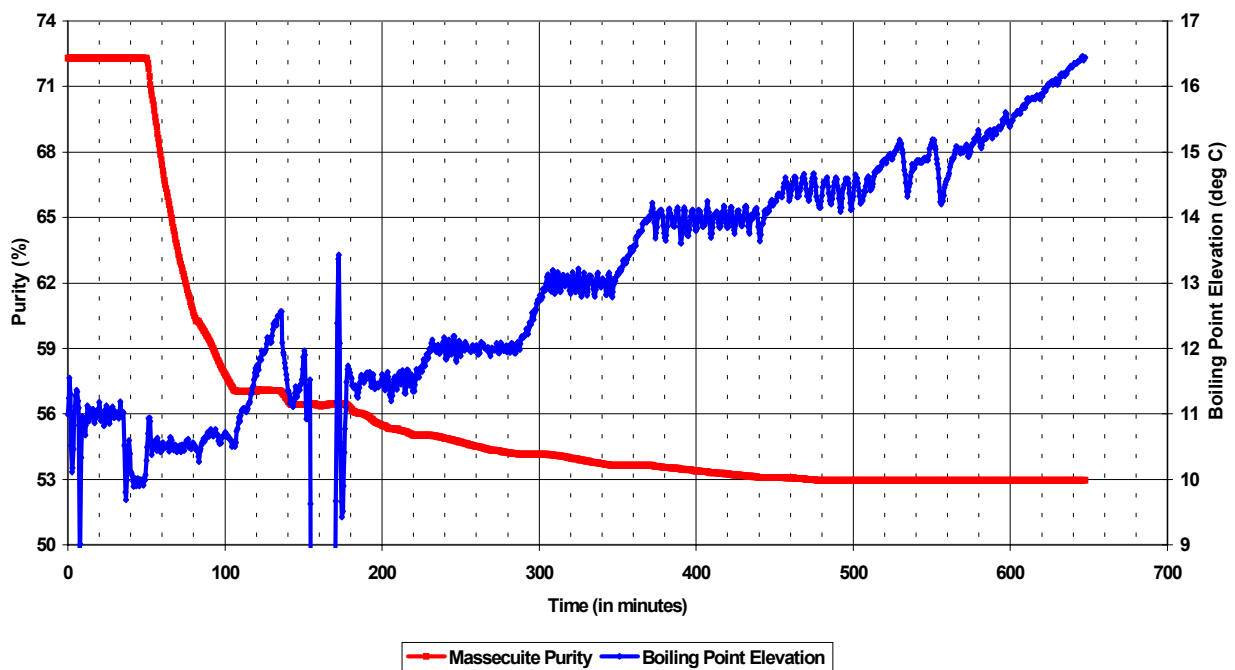


Figure 3. Trends of logged data for a pan boiling cycle.

Proof-stick samples were taken from the pan during the boiling to monitor the crystal growth. Digital images of these samples under a microscope were captured to computer disk and subsequently analysed to provide estimates of the average crystal size, giving an indication of crystal growth during the boiling, as shown in Figure 4.

A polynomial curve is fitted to the data points to provide a smoothed trend line for the change in crystal size with time. The derivative of this curve provides an estimate of the crystal growth rate throughout the boiling cycle.

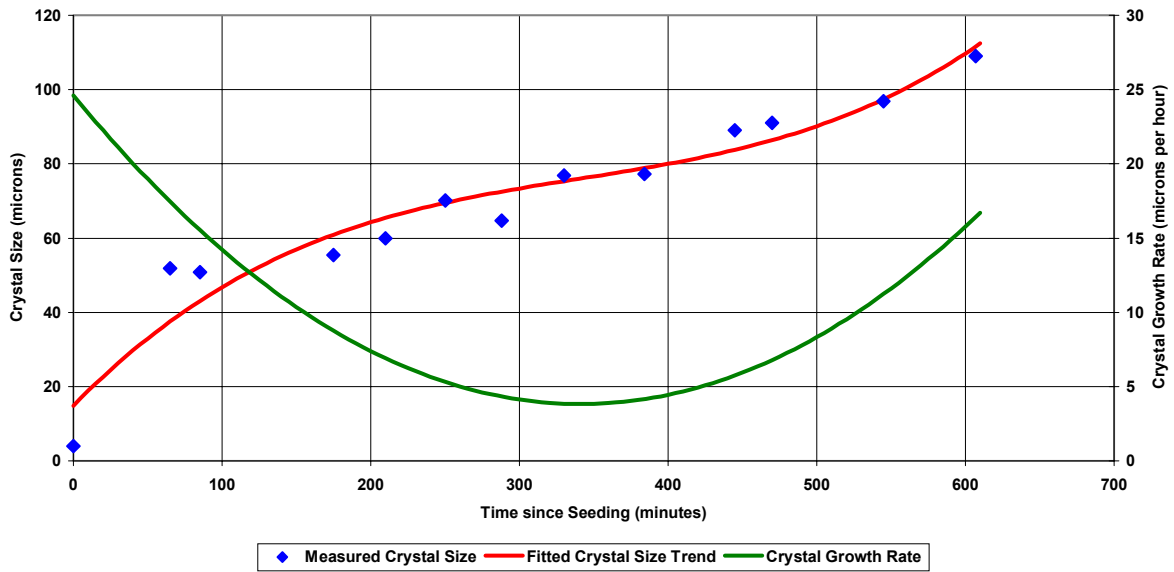


Figure 4. Growth of crystals during a pan boiling cycle.

The pan boiling was done at a fixed absolute pressure of 15 kPa, yielding a massecuite strike temperature of approximately 70°C. This is at the upper limit of acceptable strike temperatures to avoid the Maillard reaction, and future work will investigate the use of lower temperatures.

There is clearly scope for substantial further work to optimise the procedure for C-massecuite boiling in the pilot pan. The pan is also ideal for detailed investigations into the crystallisation process, with the potential for using both logged data and sample analyses as inputs to a mathematical model to yield information on the fundamentals of the process.

Results of Cooling Crystallisation Tests

Once the equipment problems mentioned earlier had been eliminated, three successful tests were conducted on the cooling crystallisation apparatus. Each test used a different cooling profile.

The results of the test using a linear cooling profile are summarised in Table 1 and Figure 5.

Table 1. Cooling crystallisation using a linear cooling profile.

Time (hours)	Sample type	Fructose	Glucose	Sucrose	Dry solids	Cond. ash	True purity	(F+G)/A	Target purity	TPD
0.0	M'cuite	5.59	4.64	52.08	92.43		56.35%			
0.0	Nutsch	6.82	5.68	41.27	89.92	14.74	45.90%	0.85	34.94%	11.0%
5.0	Nutsch	7.24	5.89	36.95	89.44	15.88	41.31%	0.83	35.09%	6.2%
15.5	Nutsch	7.51	5.92	34.13	88.00	16.40	38.78%	0.82	35.15%	3.6%
21.0	Nutsch	7.70	6.10	33.71	88.16	16.72	38.24%	0.83	35.10%	3.1%
26.0	Nutsch	7.59	6.04	33.14	88.56	16.81	37.42%	0.81	35.20%	2.2%
44.0	Nutsch	7.89	6.17	31.08	87.84	17.03	35.38%	0.83	35.10%	0.3%

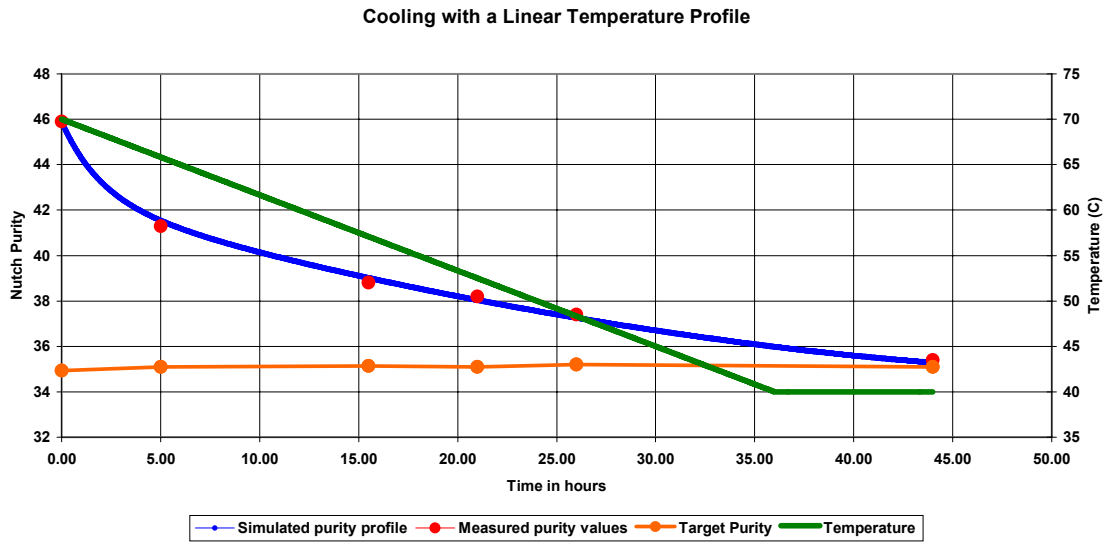


Figure 5. Trends during cooling crystallisation with a linear temperature profile.

The results of the test using an exponential cooling profile are summarised in Table 2 and Figure 6.

Table 2. Cooling crystallisation using an exponential cooling profile.

Time (hours)	Sample type	Fructose	Glucose	Sucrose	Dry solids	Cond. ash	True purity	(F+G)/A	Target purity	TPD
0.0	M'cuite	5.57	4.61	50.18	90.40		55.51%			
0.0	Nutsch	6.68	5.53	40.38	89.14	14.46	45.30%	0.84	34.97%	10.3%
4.6	Nutsch	7.38	5.99	36.28	88.39	15.58	41.05%	0.86	34.87%	6.2%
17.3	Nutsch	7.73	6.31	31.59	87.57	16.91	36.07%	0.83	35.07%	1.0%
40.5	Nutsch	8.01	6.41	29.11	85.93	17.32	33.88%	0.83	35.05%	1.2%
44.5	Nutsch	8.02	6.43	28.71	86.23	16.92	33.29%	0.85	34.90%	1.6%

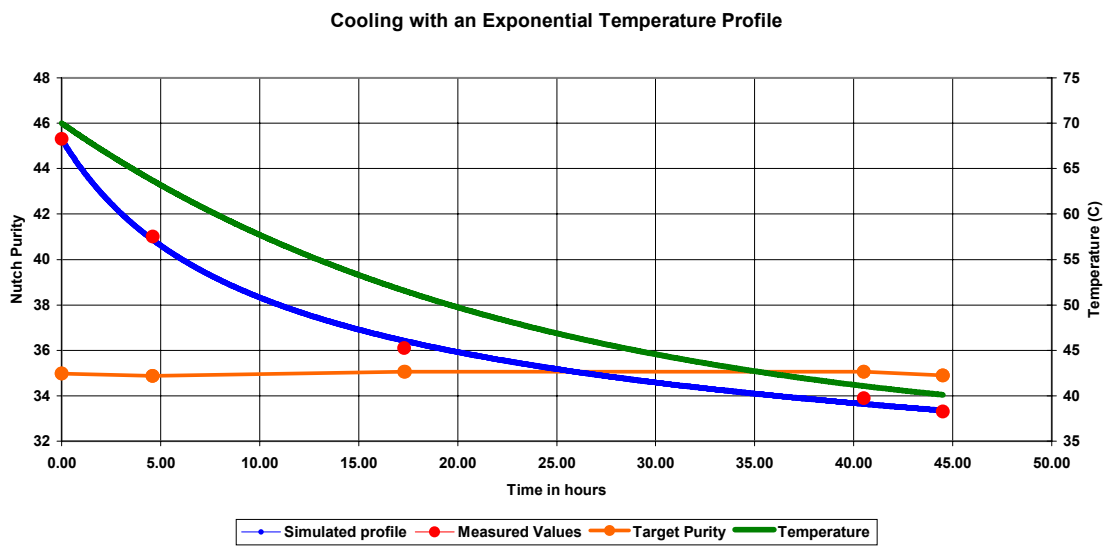


Figure 6. Trends during cooling crystallisation with an exponential temperature profile.

The results of the test using a polynomial cooling profile are summarised in Table 3 and Figure 7.

Table 3. Cooling crystallisation using a polynomial cooling profile.

Time (hours)	Sample type	Fructose	Glucose	Sucrose	Dry solids	Cond. ash	True purity	(F+G)/A	Target purity	TPD
0.0	M'cuite	5.86	5.88	51.39	92.96		55.28%			
0.0	Nutsch	7.12	7.17	40.16	89.63	14.34	44.81%	1.00	33.97%	10.8%
5.8	Nutsch	7.05	7.70	36.65	88.71	15.50	41.31%	0.95	34.25%	7.1%
10.8	Nutsch	7.92	7.90	33.98	89.14	15.80	38.12%	1.00	33.94%	4.2%
17.2	Nutsch	7.30	6.88	34.80	87.91	16.18	39.59%	0.88	34.75%	4.8%
20.8	Nutsch	7.44	6.85	33.77	87.75	16.60	38.48%	0.86	34.86%	3.6%
40.8	Nutsch	7.73	7.08	31.14	86.74	16.65	35.90%	0.89	34.66%	1.2%
47.0	Nutsch	7.81	7.29	29.57	85.32	22.29	34.66%	0.68	36.20%	1.5%

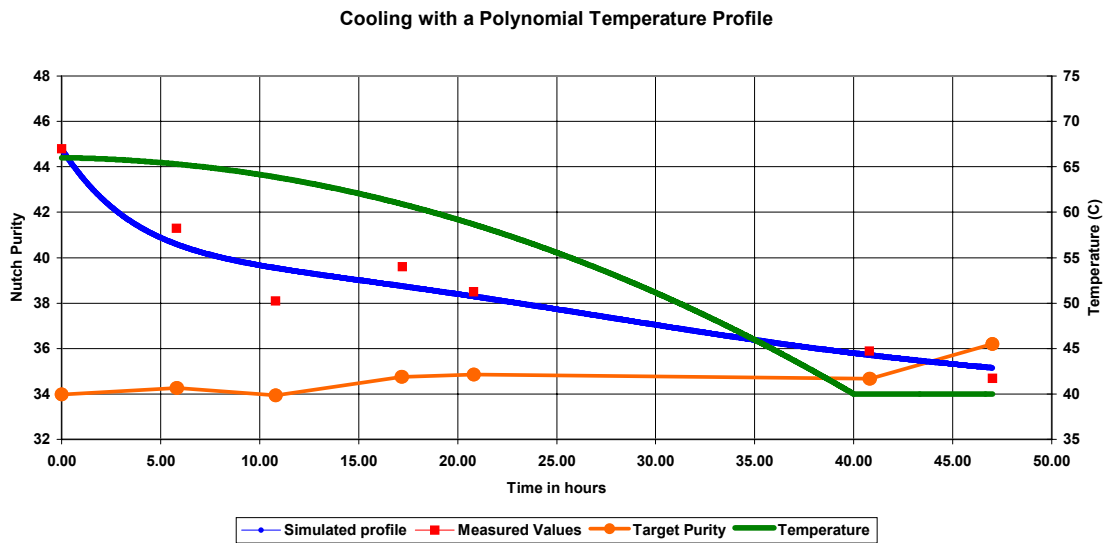


Figure 7. Trends during cooling crystallisation with a polynomial temperature profile.

In each of these tests the data have been fitted to a simple mathematical model of the crystallisation process, as described by Lionnet and Rein (1980). This was done within a standard spreadsheet program using the simple Euler method for the solution of the differential equation and using the built-in solver function to determine the parameters which provide the best (least squares) fit. The parameters determined in this way are summarised in Table 4.

Table 4. Parameters determined by fitting the crystallisation model to experiment data.

Parameter	Linear profile	Exponential profile	Polynomial profile
SC	1.18	0.77	1.25
k_1	4.70E+09	4.70E+09	4.70E+09
E_A	65026	66905	66145

Where:

SC = solubility coefficient.

k_1 = parameter combining both mass transfer coefficient and interfacial area for mass transfer.

E_A = activation energy, defining the temperature dependence of the mass transfer process.

Unfortunately, in this model (as commented on by Lionnet and Rein, 1980) the values of k_1 and E_A are highly correlated and the same quality of fit can be achieved for a range of assumed values of k_1 by altering the fitted value of E_A to compensate. The values of k_1 in Table 4 are the initial assumed values (based on the results of Lionnet and Rein, 1980) and were unaltered during the fitting process.

While it is necessary to exercise restraint in drawing conclusions from this limited body of preliminary data, there are a number of observations which can be made:

- It has been possible to reach, and even drop below, target purity when simulating the processing of C-massecuite in the laboratory on pilot scale equipment. (The full analysis of each Nutsch sample provides multiple estimates of the target purity for each test, and, since the target purity should not change, gives an indication of possible errors in analysis.)
- The fitting of a crystallisation model to the results of the cooling crystallisation tests has yielded parameters in a ranges similar to those estimated by Lionnet and Rein (1980) from tests processing massecuite boiled in full scale factory pans.
- The best results were achieved with a high initial cooling rate (exponential cooling profile), agreeing with the predictions of Lionnet and Rein (1980).

Conclusions

A new facility has been developed to allow the processing of C-massecuite on a pilot scale in the THS laboratories. To achieve this, the stirrer on the existing pilot pan has been modified to accommodate high brix massecuities and a set of small crystalliser vessels have been refurbished and modified to provide a cooling crystalliser. Preliminary tests have shown that C-massecuite can be processed in this facility to achieve target purity of the Nutsch molasses at the end of the cooling cycle.

The cooling crystallisation apparatus is designed to provide samples during the cooling cycle. By analysing these samples and fitting the results to a mathematical model of the crystallisation process, it is possible to obtain valuable information on the fundamentals of the crystallisation process.

Although boiling procedures still need to be optimised and further automated, the new facility has demonstrated the ability to simulate full-scale operation in the laboratory under closely controlled conditions. The facility has enormous potential as a research tool for investigating the exhaustion of low grade massecuities.

Acknowledgements

The long boiling times for C-massecuities and the need to Nutsch crystallisers at regular intervals over an extended (48 hour) cooling period made for inconvenient working hours for a laboratory that is not staffed for shift work. Rakesh Sevlall provided valuable assistance in achieving the success of this project in these conditions.

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