ONLINE MONITORING OF BATCH COOLING CRYSTALLISATION

L. Rózsa, J. Rózsa

ZUTORA Ltd., Hungary

Abstract

Batch cooling crystallisation is a widely practiced production process in several industries. In the sugar industry it is mainly used as the first step to produce massecuite (footing magma) to seed batch or continuous evaporating crystallisers and to increase the final exhaustion of sugar from the basic raw material (beet or cane). In the pharmaceutical industry it is used to produce the final product directly. Cooling crystallisation is carried out in different types of equipment, the typical ones having cooling coils or jacketed vessels with built-in stirrers.
During *batch evaporative crystallisation*, widely used in the sugar industry advanced instruments (process refractometer, microwave and SeedMaster device etc.) are able to provide quite many online data (including supersaturation) on the most important parameters of the massecuite. Contrary to this practice, instrumentation in cooling crystallisers used in the sugar industry is rather poor: monitoring and control of the process mostly relies only on the measurement of temperature.
This paper reports on a series of tests carried out in a sugar mill in Central Europe to monitor online the most important parameters (supersaturation, crystal content and mean crystal size, product (crystal) yield, mother liquor purity etc.) by using a built-in process refractometer and a SeedMaster 3 (SM-3) device communicating with the plant DCS.
The test results clearly testify: the present control practice relying on a pre-set temperature profile (*control of the process is not the subject of this presentation*) could and should be considerably improved by using supersaturation, the most important parameter of crystallisation for control. It would also help to maintain consistent final product crystal content. In addition, data on the other parameters, for example: mean crystal size (MA) can also be used to improve local seeding practice.

Introduction

When visiting sugar mills and refineries in different countries of the world it is common to find a series of batch evaporative crystallisers equipped with a fairly large set of instruments. The times are over when the operation of the vacuum pans relied exclusively on the expertise of the pan-men and on the exclusive use of the vacuum gauge and the mercury thermometer. Even the more sophisticated devices measuring boiling point elevation, density or some electrical property of the massecuite (simple conductivity or radio frequency (RF) probes) have lost ground on the pan floors [1], [2], [3], [4], [5], [6]. Their roles have been taken over by process refractometers and microwave instruments, which are widely used to measure liquid (syrup, mother liquor) and total solids concentration of the massecuite, respectively.
It is well known that the most important parameter of crystallisation is its “driving force”: supersaturation. It is certainly true not only for the case of sugar crystallisation, but also for the crystallisation of other products, like pharmaceuticals as well.
Attempts to develop an instrument for the online measurement of supersaturation in batch vacuum pans, which are widely used in the sugar industry, had limited success. The reason: supersaturation is a function of several parameters of the technical sugar solution, like concentration, temperature, non-sugar content and syrup quality parameters, all of which have considerable influence on it. However, without reliable information on the “driving force” of crystallisation, its control still was and is quite often even today reminiscent of the TAE (Trial And Error) practice of the old times.
The situation with batch cooling crystallisation in the sugar industry is somewhat different. When filled to full volume, there is no further syrup feed in the crystalliser. There is no evaporation and the amount of the different components (sugar, non-sugars and water) remains constant all over the time. However, the role of supersaturation during batch cooling crystallisation remains the same: it is the most important parameter of the process and its monitoring and control is of primary importance. During the cooling crystallisation of sugar the only control parameter is temperature, so most of the papers published on the subject are devoted to the discussion of the development of an optimal time dependent temperature trajectory. There are several ways to develop this trajectory, namely:

- By the familiar and traditional trial and error (TAE) method, which is sensitive to process disturbances (for example: changes of the feed syrup parameters) and can be a never-ending process lagging by one batch time behind real time.
- By using a model-based batch-to-batch control strategy [7]. It is a step-by-step method hopefully leading to a good temperature reference profile.
- By implementing supersaturation-based control of the process temperature [8].

To sum it up: for good control of sugar crystallisation in cooling crystallisers, availability of reliable online data on supersaturation is a must. However, besides supersaturation there are quite a few other important massecuite parameters which can be used to improve local control practice.

Background

Realising the need to acquire online data on supersaturation to control sugar crystallisation in batch evaporative crystallisers, K-PATENTS Oy., Finland, pioneer of the digital process refractometers and PROFICON Ltd., Hungary, a control engineering

company with experience in mill-wide automation of sugar manufacturing have joined forces in the late 1990-s to develop the first SeedMaster Instrument. A few years ago PROFICON's role had been taken over by ZUTORA Ltd., operating also in Hungary. The result of the development is the SeedMaster Instruments family. Its youngest member is the third generation SM-3, manufactured by K-PATENTS. The different members of the family can be found in operation in quite many sugar mills and refineries of the world [9], [10], [11].

A typical instrument configuration to be used for sugar crystallisation monitoring in batch vacuum pans consists of

- a K-PATENTS process refractometer measuring liquid (syrup, mother liquor) concentration and temperature,
- one per pan (typically microwave) instrument to measure the total solids content of the massecuite,
- one per pan level transmitter and
- a single SM-3 Instrument capable to serve 2 vacuum pans simultaneously.

Fig. 1 K-PATENTS process refractometer

Fig. 2 SM-3 configuration showing the optional analogue and digital input/output unit (left) and the stand-alone main unit (right)

Based on the online data listed above the SeedMaster Instruments provide online data to the plant DCS or PLC via standard current (4-20 mA) output, or via fully digital communication (Modbus, Profibus or Ethernet/IP). The only (optional) control action provided by the instruments is automatic seeding by operating the seeding valve when a pre-set value of supersaturation is reached.

The online data provided by the SeedMaster instruments are listed below:

1. Supersaturation (--)
2. Massecuite solids content (%)
3. Crystal content (% vol.)
4. Crystal size (mm)
5. Mother liquor purity (%)
6. Mother liquor concentration (%)
7. Temperature (°C or °F)
8. Massecuite level (%)
9. Massecuite density (kg/m3)

Parallel to the development of the SeedMaster Instruments Family, development of a new crystallisation control strategy for batch vacuum pans had utmost priority. Accordingly, advanced control of crystallisation based on online data provided by the SeedMaster instruments has been commissioned in different countries of the world [11], [12], [13], [14], [15].

Batch cooling crystallisation of sugar

One can find a large number of papers and presentations on the subject of batch cooling crystallisation. Most of them are trying to develop a kind of control which results in optimal process parameters. These parameter requirements are usually:

- Product crystal size and size distribution meeting target requirements
- Crystal content and product yield
- Time of crystallisation

The methods of control range from simple temperature or concentration control using prescribed trajectories (set point values) to different, more elaborate model based iterative (batch to batch) learning control solutions. Development of temperature or concentration profiles for control however is a time-consuming process and similarly to model based control, they are prone to changes of the different parameters (initial concentration, temperature, purity etc.) of the highly nonlinear process of crystallisation. Supersaturation based control of the process is still relatively rare. It is mainly due to the unfamiliarity with the SeedMaster instruments.

The different methods of control used for cooling crystallisation of sugar in the industry still rely on a very restricted use of instruments. Online data on the process in most cases is reduced to temperature, with the addition of occasionally taking samples for laboratory analysis (to determine the time of correct seeding, for example). The result is a lack of online information on important parameters, something similar to the list of those available when using K-PATENTS process refractometers and SeedMaster instruments.

Sensing the need to provide more online information on the process, first of all on supersaturation for its advanced control, it was decided to develop a version of the SM-3 device, capable to fill this need. This presentation is devoted to publish the results of a series of tests carried out in a sugar mill in Central Europe to monitor the process of cooling crystallisation. *It was not intended to deal with or to modify the actual control of the process as practiced in this mill.*

Production of footing magma for the product pans

It is common practice since a long time in the mill where the tests had been carried out to use a cooling crystalliser to produce footing magma for the product pans. The process starts with concentration of standard syrup in a normal product pan. Part of the concentrated syrup is fed to the cooling crystalliser (typical target parameters: concentration: 77 %, temperature: 64 °C, purity: 94.5 %, supersaturation: 1.07). Having filled the crystalliser, the pan is filled again to seeding level, and using previously prepared footing magma for seeding from the magma receiver continues operation as a normal product pan.

In Fig. 3 trends on different parameters stored during 3 consecutive strikes are shown. The cooling crystalliser was fed with concentrated syrup twice (in strikes No.1 and No.3). The strikes are well controlled by using online supersaturation (SS) data provided by a SeedMaster (SM-2) device. There are two trends (very close to each other) showing total solids content of the massecuite: BRIX1 data were provided by a microwave instrument, while BRIX2 shows calculated test data based on stirrer motor power consumption (the idea of using motor consumption data to calculate massecuite solids content was finally abandoned).

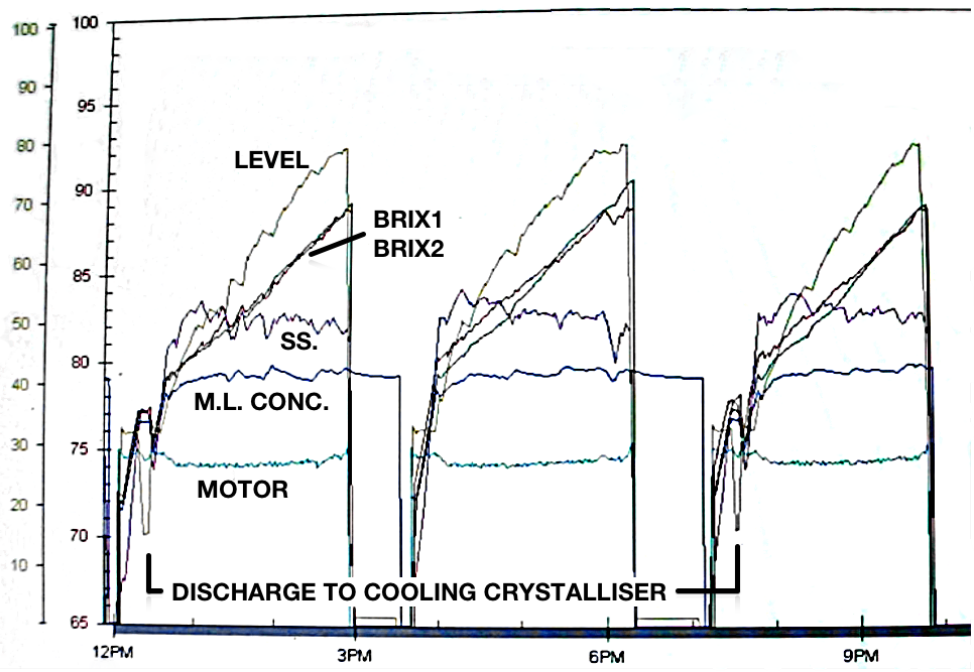

Fig. 3 A-PAN in double role: concentrating syrup for the cooling crystalliser and continuing operation as a normal product pan

Figure 4 and Figure 5 show cooling crystalliser details and refractometer sensor head installation. Cooling water for the crystalliser is provided by a temperature-controlled heat exchanger.

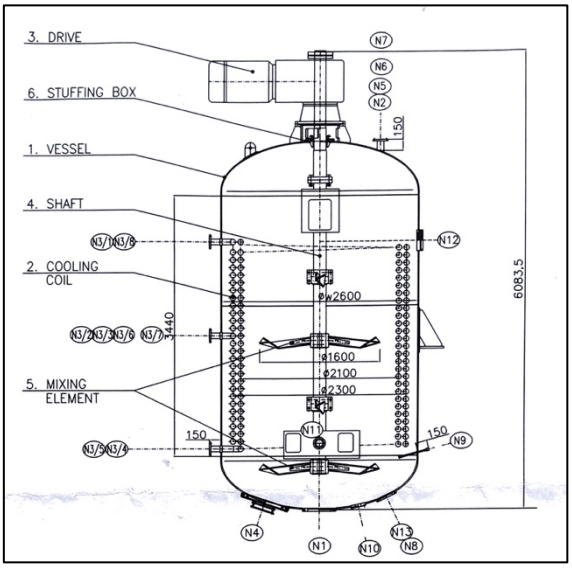

Fig. 4 Cooling crystalliser

Fig. 5 Refractometer sensor head

Instruments

According to local practice crystallisation is controlled by using online temperature data from a temperature transmitter and occasional laboratory data based on massecuite samples.
Testing of the new SM-3 instrument version was based on the use of

- 1 K-PATENTS process refractometer and
- 1 SM-3 device with added new software to serve cooling crystallisation applications.

The sensor head of the refractometer, which provides online data on syrup/mother liquor concentration and temperature, is mounted in the bottom of the crystalliser (Fig. 5).
The instrument configuration is shown in Fig. 6. As can be seen, in this case there is no need to use the optional I/O hardware unit of the SM-3 device. With the addition of a second refractometer sensor head the configuration is ready to serve two crystallisers simultaneously.
It is important to note here that *the only instruments used to provide online data on the process are the K-PATENTS process refractometer and the K-PATENTS SM-3 device.* Data on feed syrup purity and quality (m, b, c parameters [16]) should be provided by the local laboratory.

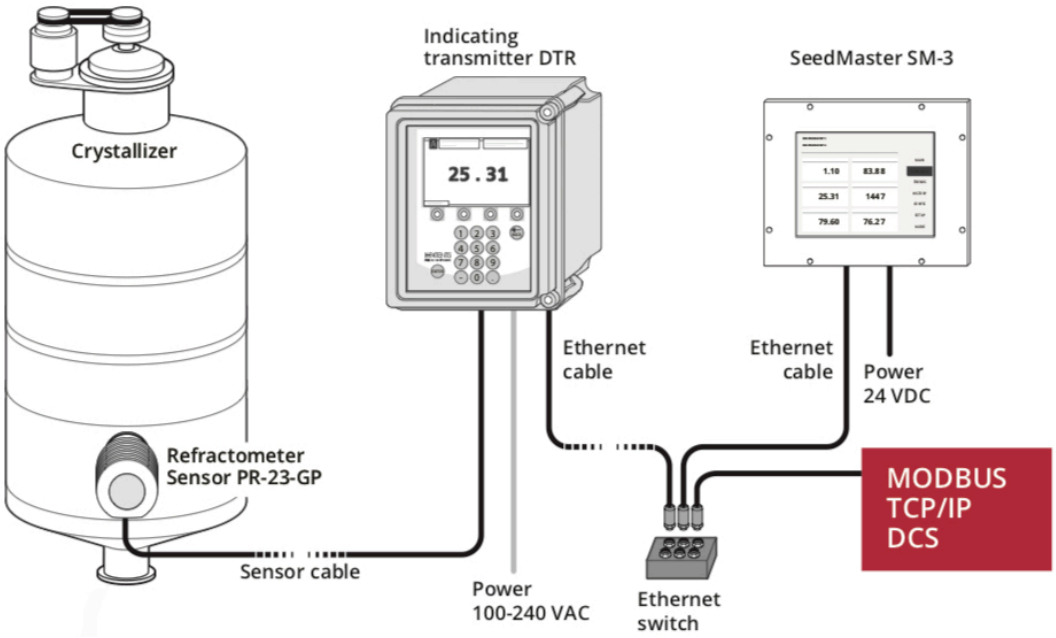

Fig. 6 Instruments used in monitoring batch cooling crystallisation of sugar

The most important parameters provided online (in numerical and/or trend form for the current and 3 previous strikes) by the SM-3 instrument are listed below.

1. Temperature (°C or°F)
2. Mother liquor concentration (%)
3. Supersaturation (--)
4. Mother liquor purity (%)
5. Crystal content (% vol.)
6. Crystal yield (%)
7. Crystal size (mm)
8. Massecuite density (kg/m3)

Product yield is defined as the percentage of dissolved sugar when seeding, converted to crystals during the process of crystallisation. Besides these data, other parameters, like time of crystallisation (from seeding to end), supersaturation when seeding, minimum, maximum and average values, total weight of the crystals in the crystalliser etc. are also available.

Test results

Comparative data on 8 parameters in 10 consecutive strikes

During testing the monitoring of cooling crystallisation, process data on 10 consecutive strikes were collected. What follows is a brief summary of the results with some comments on the recorded data.
As a first step online data provided by the instruments shown in Fig. 6 on parameters No. 1 to No. 8 listed above will be presented.

Massecuite temperature and mother liquor concentration

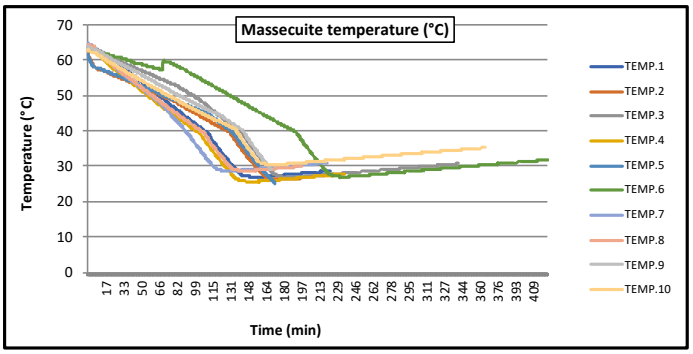

Fig. 7 Temperature

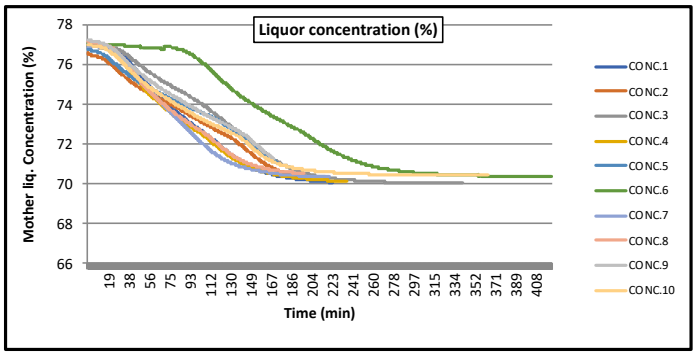

Fig. 8 Mother liquor concentration

According to the trends on temperature (Fig. 7) crystallisation is carried out roughly in the 62...64 to 28...30 °C range of temperature. When reaching the low end of the range cooling is not continued any more, but dropping the massecuite may be delayed due to lack of receiver capacity.
Liquor concentration changes in the 77 to 70 % range.
Trends on temperature and liquor concentration during the 10 strikes were fairly similar, except those which belong to strike No. 6.

Supersaturation and mother liquor purity

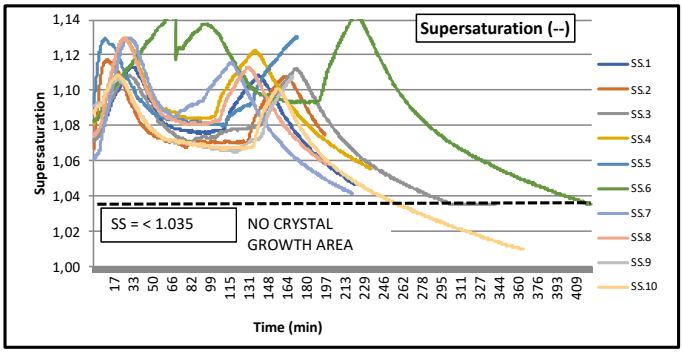

Fig. 9 Supersaturation

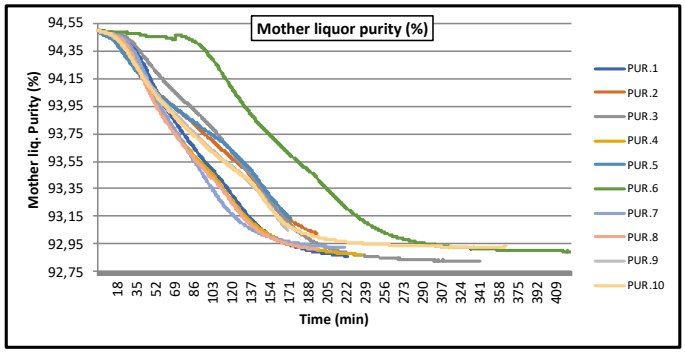

Fig. 10 Mother liquor purity

Supersaturation, when using the SM-3 instrument is defined as *sugar in solution over sugar at saturation (both at the same temperature).* After having completed seeding the crystals begin to grow and purity of the mother liquor drops accordingly. It is natural that this drop in purity is taken into account when calculating supersaturation in the SM-3 device. The trends on supersaturation (Fig. 9) show data in the SS = 1.01 to 1.14 range, where spontaneous nucleation will not take place in the bulk of the massecuite (feed syrup purity: ~ 94.5 %). In their presentation [8] the authors correctly identified the danger of unwanted nucleation in the vicinity of the pipe section where the cooling water enters the crystalliser. The danger is increasing as the rate of temperature change increases. The cooling crystalliser shown in Fig. 3 has multiple (distributed) cooling water entries which reduces the risk of nucleation.
Based on the data on supersaturation it is clear that full seeding with slurry is practiced in this mill.
In three from the ten strikes supersaturation reaches, or even is much less than SS=1.035, the value which is the high limit of the „NO CRYSTAL GROWTH AREA" (see Eq. 3).
Trends of the characteristic crystal size (Fig. 13) clearly show that when supersaturation approaches (drops to) the high limit of the NO GROWTH area, crystal growth will be ended accordingly.

Crystal content and crystal yield

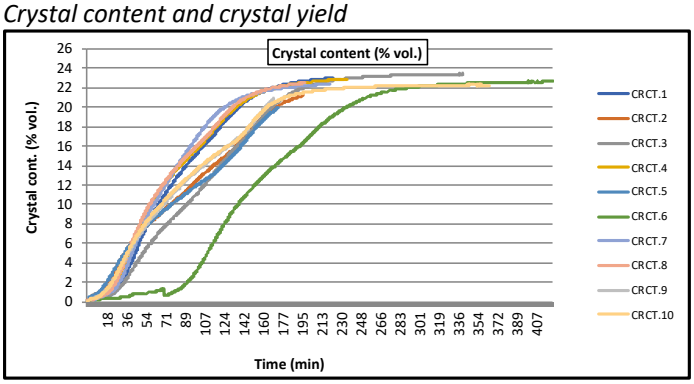

Fig. 11 Crystal content (% vol.)

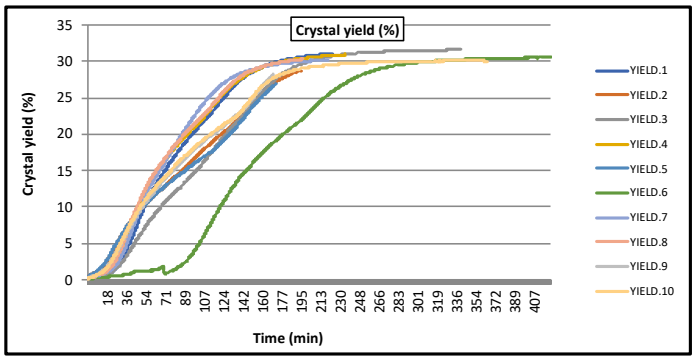

Fig. 12 Crystal yield (%)

Figures 11. and 12. show similarly looking trends on two closely correlated crystal parameters.

Definitions of crystal content and crystal yield:

$$CRCT = 100 * \frac{Volume\ of\ crystals\ in\ unit\ volume}{unit\ volume}\ (\%\ vol) \qquad \text{(Eq. 1)}$$

$$YIELD = 100 * \frac{SUGAR.seed - SUGAR.t}{SUGAR.seed}\ (\%) \qquad \text{(Eq. 2)}$$

Where:

SUGAR.seed : sugar in solution when seeding (g / 100 g water)
SUGAR.t : sugar in solution at time *t* (g / 100 g water)

It is evident that both parameters depend on the *difference between the sugar concentrations SUGAR.seed and SUGAR.t.* High yield can be achieved by using fairly high concentration feed syrup and continuing crystallisation to a fairly low temperature value. However, there are constraints to be taken into account:

- In order to prevent unwanted nucleation, use of high concentration feed syrup demands fairly high feed syrup temperature as well. Temperature however has its own high limit at about 73...74 °C in order to prevent caramelisation of sugar. It is also clear that reliable online information on supersaturation is needed all the time.
- The rate of crystal growth can be described by the equation [17]:

$$\frac{da}{dt} = K * (SS - 1.035) * 0.277 * e^{0.0186*T} * e^{-1.75*\frac{NS}{100}} * (1 - y)^2 \left(\frac{mm}{min}\right) \qquad \text{(Eq. 3)}$$

Where:

K : Overall crystallisation growth rate, function of several parameters
SS : Supersaturation (--)
T : Temperature (°C)
NS : Non-sugar concentration (g / 100 g water)
y : Crystal content by volume (abs. v.)

According to the equation the growth rate of crystal size drops fast as the operating temperature decreases. So to continue cooling crystallisation to too low temperature will not bring much increase neither in crystal content, nor in product yield, but the time of crystallisation will be increased considerably.

Crystal size and massecuite density

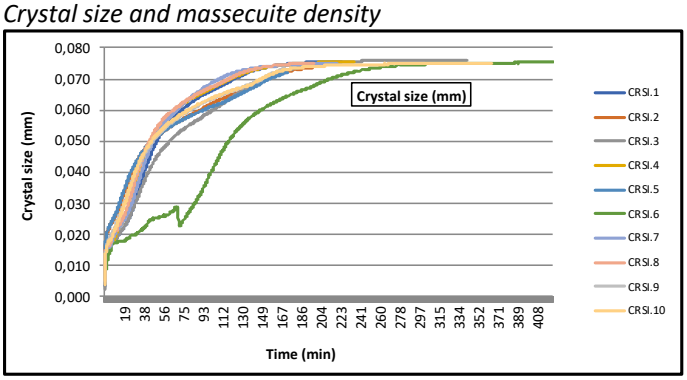

Fig. 13 Crystal size (mm)

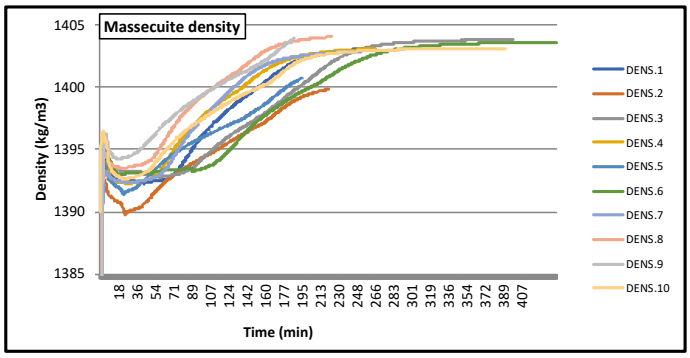

Fig. 14 Massecuite density (kg/m3)

Fig. 13 and Fig. 14 show the changes in *characteristic crystal size* and massecuite density, respectively.

A more detailed view on some parameters

Due to the wealth of online data provided by the SM-3 instrument it is possible to get a more detailed insight into the inner workings of the cooling crystallisation as practiced in the mill. For this reason two strikes, strike No. 7 and No. 8 were selected.

Strike No. 7

The main massecuite parameters

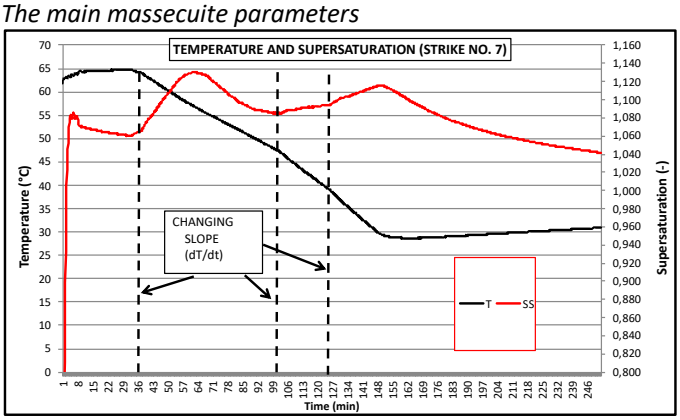

Fig. 15 Temperature-controlled supersaturation

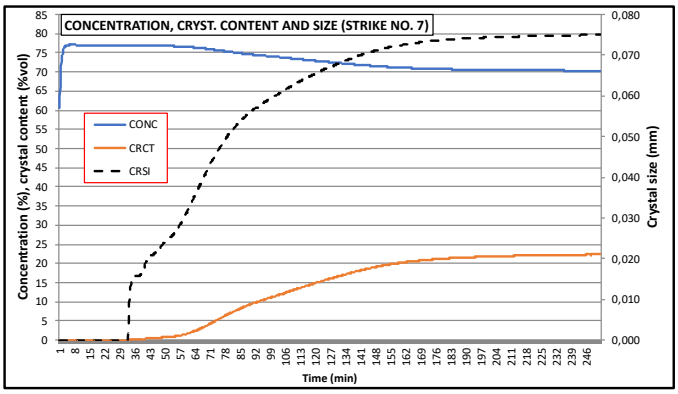

Fig. 16 Concentration, crystal content and size

According to the trends in Fig. 15, supersaturation is controlled by changing the rate of cooling (slope of the temperature curve) 3 times during the strike. The approximate cooling rates (dT/dt) are:

– 0.25, - 0.35 and - 0.4 (°C / min)

Supersaturation does not exceed SS = 1.13 in the bulk of the massecuite.

Mother liquor concentration, crystal content and size data (Fig. 16) show decreasing change in these parameters towards the end of the strike.

Strike No. 8

The main massecuite parameters

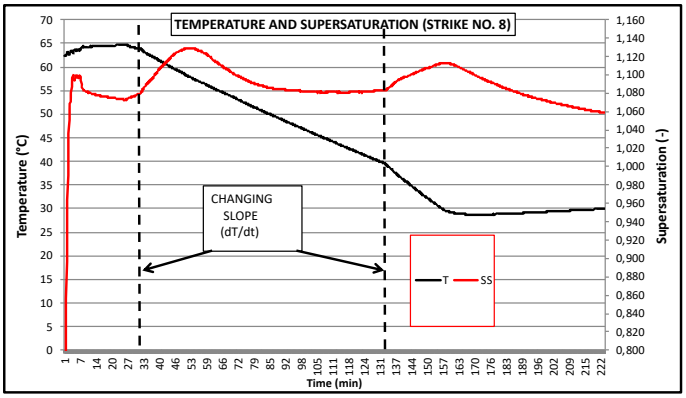

Fig. 17 Temperature-controlled supersaturation

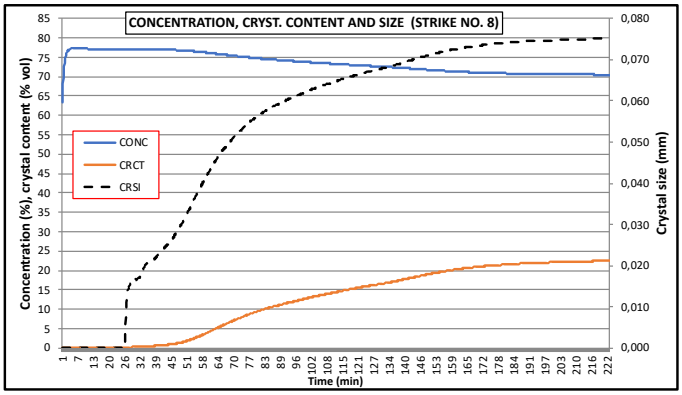

Fig. 18 Concentration, crystal content and size

The approximate cooling rates (dT/dt) are:

- 0.25 and - 0.4 (°C/ min)

Supersaturation does not exceed SS = 1.13 in the bulk of the massecuite.

When looking at Fig. 15 and Fig. 17 it is clear that the temperature of the massecuite is being controlled by linearly cooling the massecuite according to several pre-selected temperature profile sections. During the complete time of crystallisation 2 to 4 different sections were detected during the 10 strikes, when the rate of the temperature change (dT/dt) was different. It is assumed that this kind of control is based on operator (manual) intervention and usually results in 2 peaks in the trend of supersaturation.

Process monitoring by using the K-PATENTS process refractometer and SeedMaster SM-3 instrument configuration

Process monitoring by advanced instruments serves two tasks:

1. Provides reliable online data, not available before (like supersaturation) for the advanced automatic control of the process.
2. It provides important information on the main parameters for the process operator.

The trends shown in figures Fig. 7 to Fig. 18 show a wealth of important data of 10 consecutive strikes in a condensed form. These data can be transmitted by using standard digital communication methods to the local DCS or PLC in order to control the process of crystallisation. Naturally, the data can be displayed on the monitors of these devices in numerical and trend forms as well.

The SeedMaster SM-3 device has a large digital memory as well, which can be used to store a large amount of data (for example: STRIKE HISTORY data for 100 previous strikes and much more). This means that

- there is no need to use the often scarce DCS or PLC memory and processor capacity for this task, and
- there is no need to do any programming of these devices.

The SM-3 has its own well designed man-machine interface (HMI) which can be used in the control room or even at remote locations. A single SM-3 device can serve 2 crystallisers simultaneously. Trended data may come from the current strike, or can be selected from the strike history archive as well.

What follows is a very limited introduction of the HMI features using some data from the process monitoring test presented before.

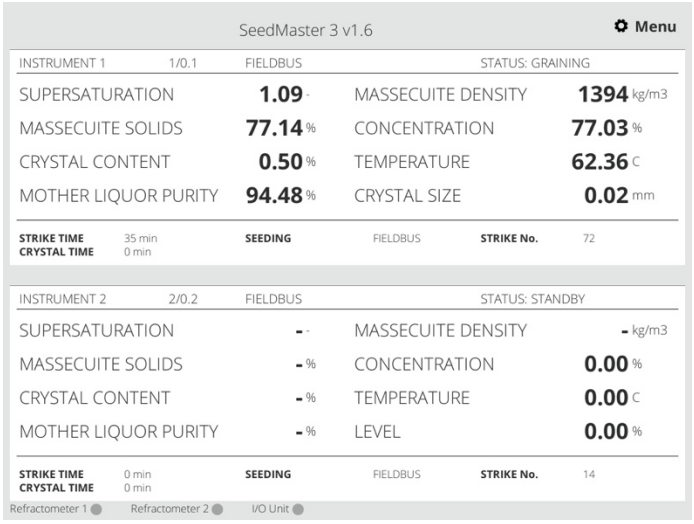

Fig. 19. Main display showing numerical data on 8 parameters for up to 2 crystallisers

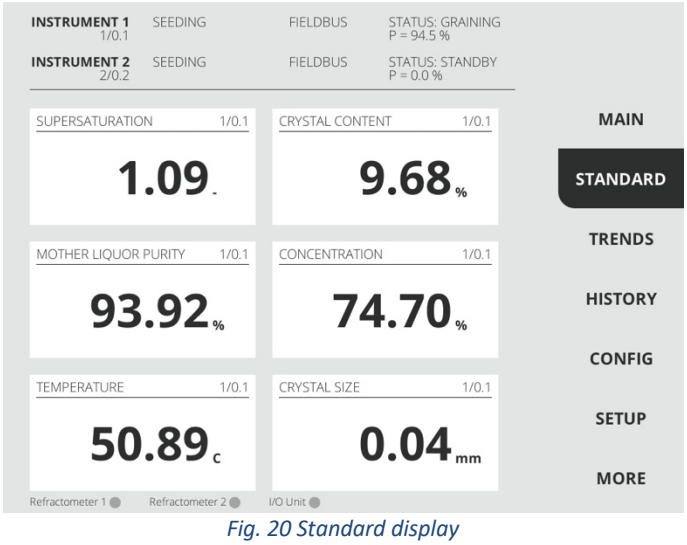

Fig. 20 Standard display

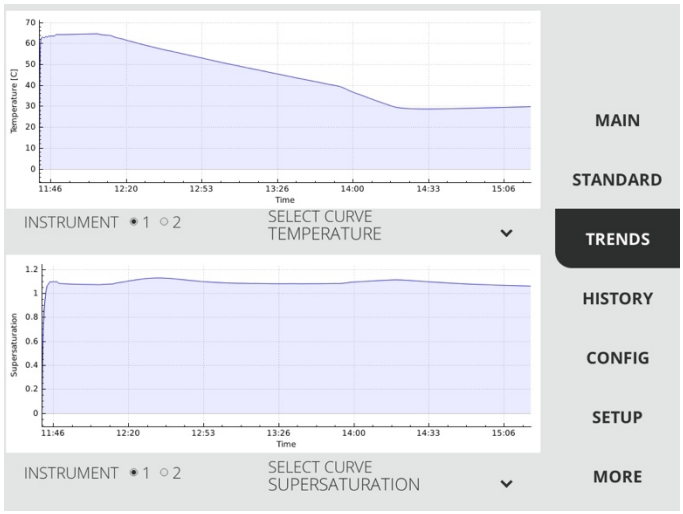

Fig. 21 Temperature and supersaturation trends

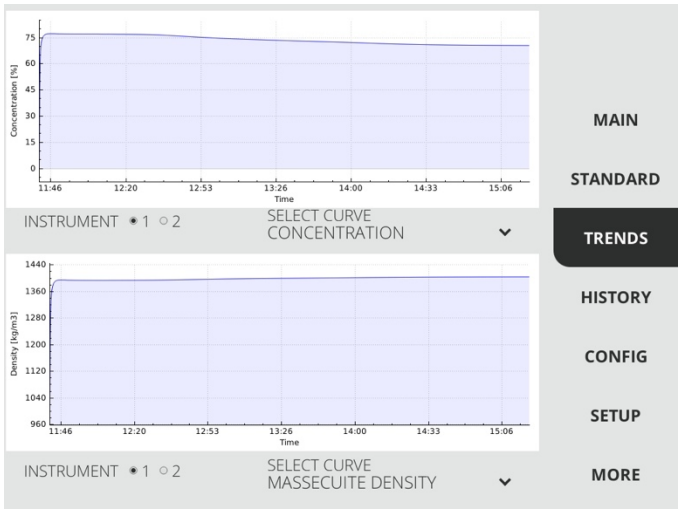

Fig. 22 Trends of density and mother liquor concentration

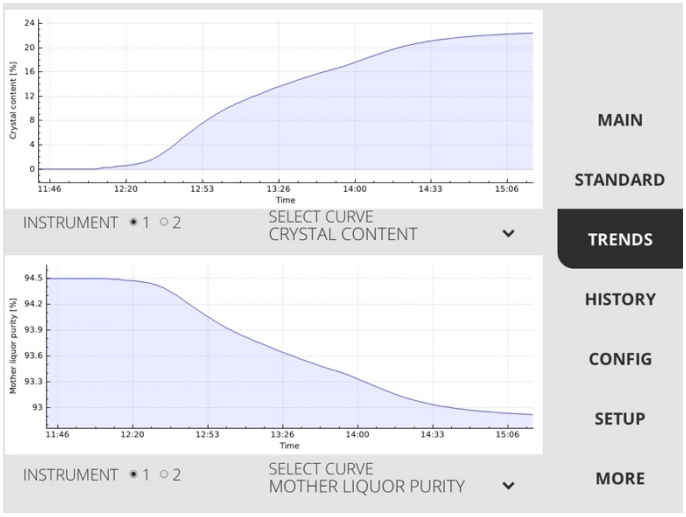

Fig. 23 Crystal content and mother liquor purity

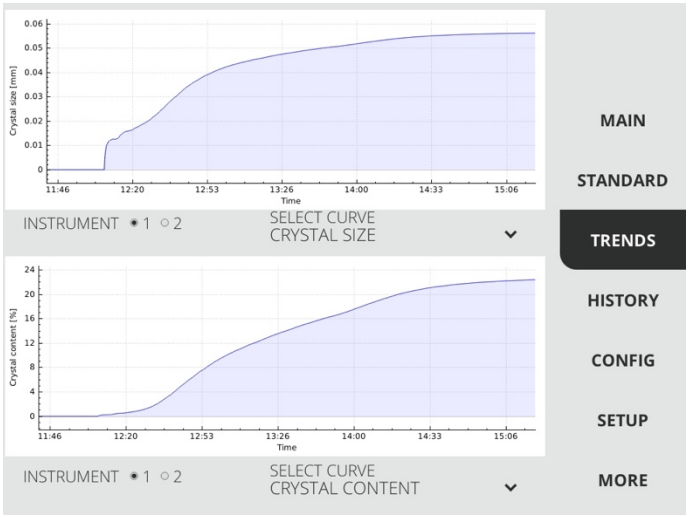

Fig. 24 Crystal size and crystal content

Conclusion

Production of good quality footing magma for the product pans is an important step in sugar manufacturing. The correct operation and control of the process needs reliable online data on its most important parameter that is on supersaturation.
Besides supersaturation, however, there are quite many important parameters which by taking into account the local constraints, can be used to select the *optimal operating parameters* (for example: feed syrup concentration and temperature, temperature range of the cooling water, seeding practice etc.). These parameters have a very important effect on the final parameters (crystal size and content) of the magma to be used for seeding in the product vacuum pans.
It is well known that advanced control of crystallisation needs reliable online data on supersaturation. It is also well documented, that supersaturation is a function of several variables (liquid concentration, temperature, purity (which is not constant during the process) and quality parameters), therefore reliable data on it can be provided only by calculation, taking into account all of these parameters.
The SeedMaster Instruments are well known and used with batch vacuum pans in quite many mills and refineries of the world where supersaturation-based control is implemented. This presentation reports on the results of a series of tests carried out in a mill in Central Europe with a SM-3 instrument developed by Zutora Ltd. and manufactured by K-PATENTS Oy. The instrument has additional software developed for the monitoring of batch cooling crystallisation of sugar. The main features:

- Online monitoring of cooling crystallisation of sugar needs a K-PATENTS process refractometer measuring liquid concentration and temperature, and a single SM-3 instrument.
- A single SM-3 device can serve
    - 2 batch (evaporative) vacuum pans, or
    - 1 batch vacuum plus 1 batch cooling crystalliser simultaneously.
- Online data provided with batch vacuum pans:
    - Supersaturation
    - Massecuite solids content

    - Crystal content
    - Crystal size
    - Mother liquor purity
    - Massecuite density
    - Concentration
    - Temperature
    - Level
- Online data provided with batch cooling crystallisers:
    - Supersaturation
    - Crystal content
    - Crystal size
    - Crystal yield
    - Mother liquor purity
    - Massecuite density
    - Concentration
    - Temperature
    - Level

Development of the new features of the SM-3 instrument was aimed to provide an instrument capable to serve the needs of advanced vacuum and/or cooling crystallization control, based on reliable online data on supersaturation and other important process parameters. Implementation of the actual control solution will be the subject of a further communication.

References

1. D.J. Radford, M.G.S. Cox: The Use of Electrical Properties Measured at Radio Frequencies for Pan Boiling & Brix Control Zuckerindustrie, 111 (1986) Nr.10.
2. L. Rozsa: Sensor performance in monitoring of supersaturation International Sugar Journal Vol. XCIX No. 1182, June 1997
3. L. Rozsa: Online monitoring of supersaturation in sugar crystallisation International Sugar Journal Vol.. XCVII No. 1176, Dec. 1996
4. D.J. LOVE *et al. :* COMBINING TEMPERATURE MEASUREMENT WITH OUTPUTS OF A RADIO FREQUENCY PROBE Proc. Int. Soc. Sugar Cane Technol., 24: 124-129, 2001
5. M. Saska: Boiling point elevation of technical sugarcane solutions and its use in automatic pan boiling International Sugar Journal 2002, Vol. 104, 1247 pp 500-507
6. L. Rozsa: SENSOR SELECTION: STILL AN ISSUE IN SUGAR CRYSTALLISATION CONTROL Philippine Sugar Technologist's (PHILSUTECH) Convention, Bacolod City, Philippines, 2003
7. M. Forgione *et al.:* Batch-to-batch model improvement for cooling crystallization CONTROL ENGINEERING PRACTICE, August 2015.
8. C. Mayhew *et al:* Developments in the repeatability of Cooling Crystalliser performance Sugar Industry Technologists Annual Meeting, Bonita Springs, USA 2018.
9. L. Rozsa: The SeedMaster device: For on-line supersaturation measurement and automatic crystalliser seeding International Sugar Journal Vol. C, NO. 1200, Dec. 1998, pp 601-607
10. L. Rozsa: SeedMaster 2: A universal crystallization transmitter and automatic seeding device International Sugar Journal Vol. CVIII, 1296, Dec. 2006, pp 683-695
11. L. Rozsa: On-line monitoring and control of supersaturation and other massecuite parameters in vacuum pans: A control engineering approach Sugar Industry Technologists Annual Meeting, Montreal, Canada, 2011
12. L. Rozsa: Advanced Control of Crystallisation in Vacuum Pans (in Russian) IX International Sugar Forum, Kursk, Russia, 2012
13. L. Rozsa, G.M. Arriaza, M.T. Romero: Advanced Control of Crystallization Based on the Direct Use of On-line Data on Supersaturation: Theory and Practice Sugar Industry Technologists Annual Meeting, Guanghzou, CHINA, 2013
14. E. Mielonen, L. Rozsa, J. Rozsa: Optimization of Sugar Crystallization Process Using Supersaturation Based Control Practice Philippine Sugar Technologist's (PHILSUTECH) Convention, Lahug Cebu City, Philippines, 2016
15. L. Rozsa, J. Rozsa, S. Kilpinen, E. Mielonen: Selection of the operating parameters in Sugar Crystallization Control Sugar Industry Technologists Annual Meeting, Bonita Springs, USA, 2018
16. L. Rozsa: Sucrose solubility in impure sugar cane solutions. International Sugar Journal, 2000, VOL.102, NO. 1217
17. L. Rozsa: Crystal growth and crystallisation control tactics in industrial sugar crystallisers. Part 1. Crystal growth. International Sugar Journal, October 2016.