Sugar crystallization: Look for the devil in the details - Part 2

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abstract

In Part 1 of this paper some of the most important details of sugar crystallization, like product quality, cost of production, crystal growth, pan circulation and seeding were discussed. The role and importance of supersaturation in all of these details were duly emphasized. See ISJ (2008), 110 (1315): 403-413. In Part 2 the emphasis is placed on real-life case studies and on some new tools which can be used to shed light on the inner workings of a crystallizer in real time and use the information for advanced control of crystallization.

Keywords: **crystallization, sugar, sugar crystals**

How to diagnose crystallization problems and use the findings for better control?

This part of the paper will be devoted to the introduction of methods (some of them are probably new), which can be used to provide help in diagnosing and solving crystallization control problems. The same way as a medical examination using advanced instruments and tools may discover health problems of a patient never diagnosed before, there are new tools available to discover the problem areas connected with sugar crystallization. Knowledge of the problem at hand is certainly the first logical step to solve it.

Case studies

Source of data

The case studies to be discussed here are based on data acquired during numerous visits paid to mills in different countries of the world and on on-line data sent to the author for further analysis. The mills will not be identified here; only the geographic areas the data came from: Australia, Hungary, the Middle East, North America, Scandinavia and Southeast Asia.

The instruments and tools used:

- K-PATENTS process refractometer (obligatory).
- Massecuite density or solids content (brix) or stirrer motor power / current transmitter transmitter when SeedMaster 2 was used.
- Notebook PC running the on-line calculation of supersaturation.
- SeedMaster software option for the PR-01-S type K-PATENTS process refractometer [1].
- SeedMaster 2 dedicated crystallization transmitter and automatic seeding device [2], Figure 1.
- Crystal photography.
- Image analyzer software to calculate main crystal data (mean size MV, size distribution and the coefficient of variation CV).

The most important instrument used was the process

Figure 1. SeedMaster 2 serving two vacuum pans simultaneously

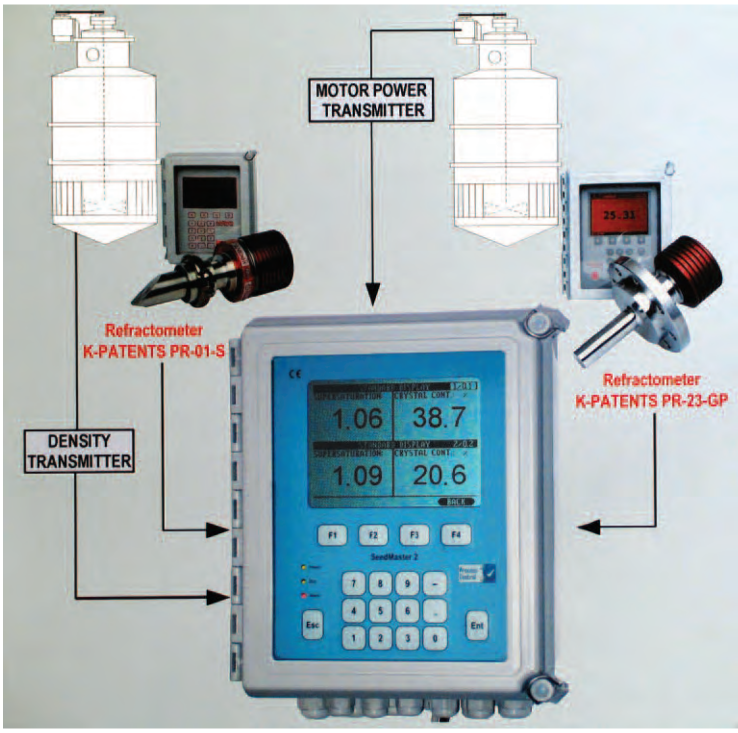


Figure 2. Crystal photography, a no-fuss solution

refractometer. Its use is obligatory whenever the calculation of supersaturation is attempted. The sensor was located in most cases in the pan bottom under the calandria. Refractometer data (syrup / mother liquor concentration and temperature) are used as inputs to the optional SeedMaster software and SeedMaster 2.

Test methods

When diagnosing crystallization problems or when implementing advanced control of crystallization on-line monitoring of supersaturation is a must. The trend of supersaturation (its "profile") tells a lot about crystallization control as practiced in a mill or refinery. Monitoring of several strikes provides information on the repeatability of control as well.

Differences

- in seeding (supersaturation in the seeding point),
- in the magnitude of supersaturation and time spent above the critical limit of supersaturation during nucleation and later (see Figures 5 and 6.a. in Part 1 [3]),
- in the maximum, minimum and average values of supersaturation after seeding has been completed will result in differences in the product parameters (crystal quality and content).

On-line monitoring of supersaturation was first tested running in a notebook PC connected to a K-PATENTS process refractometer. It was soon followed by using the SeedMaster optional software which has already become a standard device in quite many mills in different countries (such as USA, Saudi Arabia, Peru, Colombia and Iran) to monitor supersaturation and to implement reliable automatic seeding in vacuum pans [1]. In a new development SeedMaster 2 (manufactured by Process Control Ltd., Hungary) with a much wider scope of features is available to monitor up to 7 parameters of the massecuite on-line [2] and to implement reliable automatic seeding.

Whenever available, data from some available instruments provided useful additional information.

Finally, crystal macro photography using an inexpensive digital camera supported by a transparent plastic cup provided valuable help in tracing crystallization problems (Figure 2).

Some of the monitored data will be shown on the display (LCD) of the K-PATENTS refractometer (SeedMaster optional software), or on the LCD of SeedMaster 2, while others by trend diagrams made in Excel based on data from the transmitters and / or from SeedMaster 2. Crystal size distribution trends were calculated in Excel using data from an image analyzer program. The length of treatment of the following cases is limited by the space available in this paper.

Case study 1: Refinery in Australia (Figure 3)

Strike control is automatic. Some of the parameters, like steam flow to the calandria are controlled following a pre-set set-point profile based on the results of a series of trials. When there is already considerable crystal content in the massecuite (from the "bend" point on the motor consumption trend) syrup feed is controlled based on motor current.

Strike history:

1. Feed syrup purity: 99.5 %. Type of seeding: Shock seeding.

2. Instruments used: Process refractometer, steam flow, temperature and motor current transmitters.

3. Supersaturation: Seeding at 1.28; maximum: 1.30; strike end: 1.02; average: 1.086.

4. After nucleation has been completed supersaturation was close to the critical limit of nucleation for about 30 minutes.

5. Later on syrup feed was drastically reduced. Due to the increasing crystal mass (shown by increasing motor current) supersaturation followed a continuous downward path. The drop of supersaturation is due to the appropriate drop of mother liquor concentration. It can be proved that crystal content in the motor

Figure 3. Shock seeding with automatic feed control based on motor current (starting from the current "bend" point)

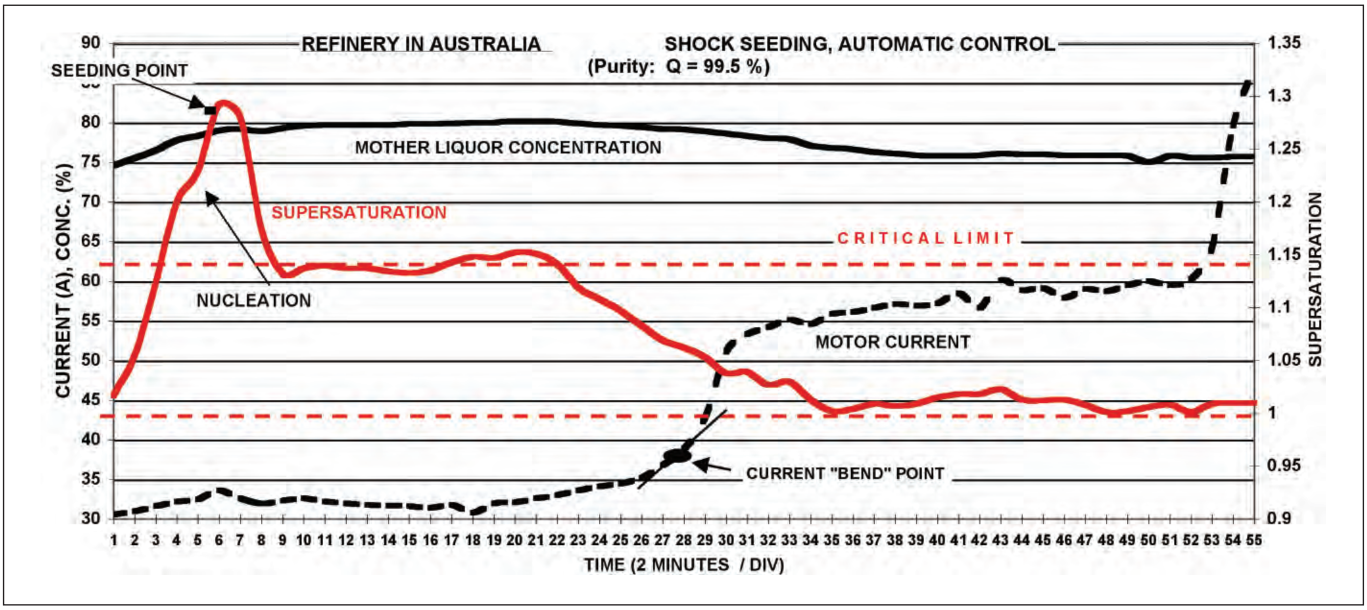
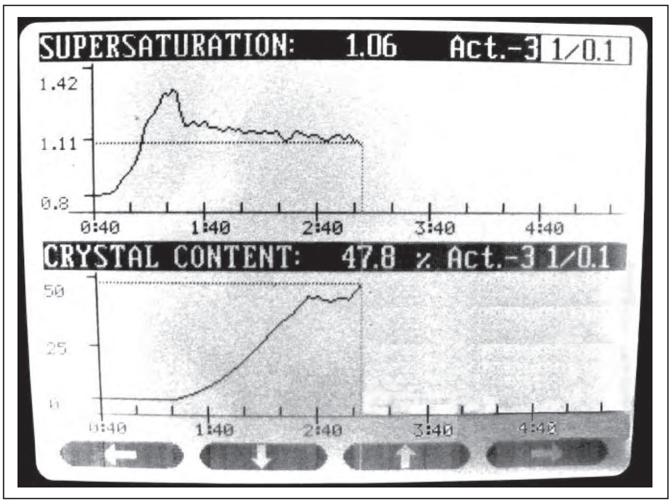


Figure 4. Supersaturation and crystal content by volume trended on the SeedMaster 2 display



current "bend" point is about 32 % by volume.

6. From about 35-40 % crystal content syrup feed was re-started based on motor current data, and steam pressure in the calandria was considerably increased. This, however, could not stop the further decrease in supersaturation which was stabilized in the 1.02-1.04 region for quite a long time till the end of the strike.

Notes:

• Some of the information mentioned above (syrup feed and steam control) is based on verbal communication.

• Supersaturation was calculated in a notebook PC connected to the process refractometer.

Comment:

In the first part of the strike the supersaturation profile was exemplary: shock seeding with good nucleation followed by optimal supersaturation. Later on the drastic reduction of syrup feed and increasing crystal content resulted in a quite dense ("tight") massecuite in the neighborhood of the refractometer sensor head (pan bottom).

Problems identified:

• Fast deteriorating circulation in the pan prevented the feed syrup to reach the bottom of the pan where crystal growth was actually stopped. Production capacity of the pan was only partially used.

• There were no data available on product quality. In such cases wide crystal size distribution and conglomeration can be expected.

Case study 2: Refinery in Scandinavia (Figure 4)

This refinery has implemented full automatic control of crystallization. This results in a supersaturation curve typical of shock seeding. After nucleation has been completed supersaturation has a slowly decreasing slope downwards. The ripples on its trend are due to intermittent syrup feed.

Strike history:

1. Feed syrup purity: 99.2 %

2. Instruments used: Process refractometer, density transmitter, SeedMaster 2.

3. Shock seeding. Supersaturation: Seeding at 1.27; maximum: 1.39; close to strike end: 1.06.

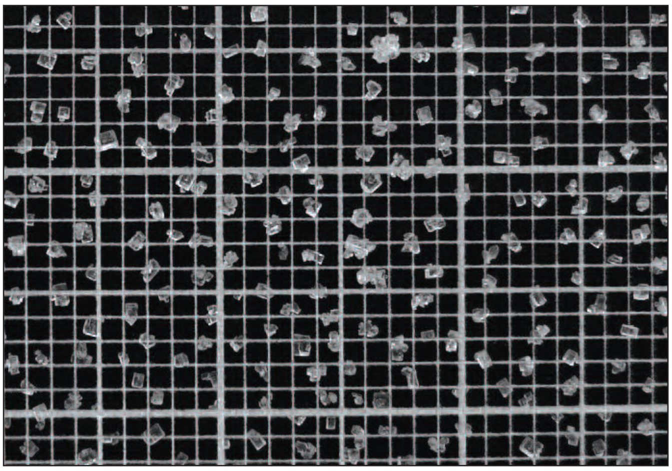
4. After nucleation has been almost completed supersaturation was still close to 1.20, well above the critical limit for some time.

According to information obtained on the spot strike control operates quite well. Mean crystal size was repeatedly very close to the target value (0.65 mm), while the coefficient of variation was exceptionally good (CV = 20…24). Everything seemed to be in order.

Due to some suspicion based on the supersaturation profile it was decided to collect a sample of crystals right at the sugar drier exit for photography and a later size distribution analysis. Figure 5. shows the product crystals. The size of the small rectangles behind them is 1 x 1 mm.

Having seen the crystal photo the plant manager was really

Figure 5. Product crystals



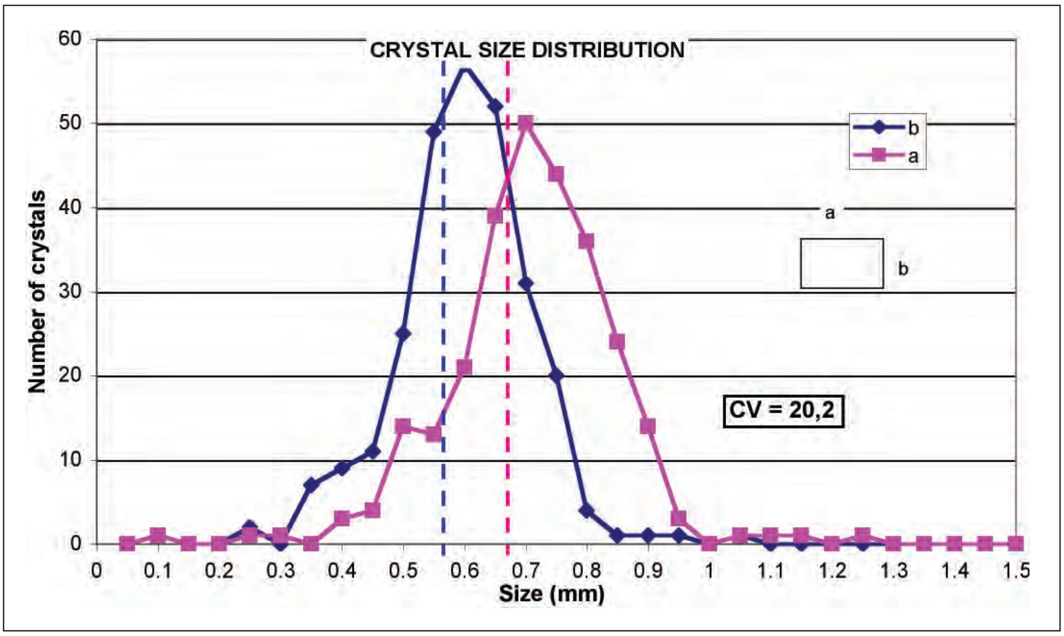
"shocked" and demanded to shoot a new one with a new sample right in front of him on his desk. The result was similar. The size distribution analysis was made later (Figure 6).

Comment:

In Figure 5. a surprisingly large number of twins and conglomerates can be detected. It is interesting to note that their sizes are quite similar. This means that they had roughly the same time to grow to their final sizes, that is they had been formed at about the same time, most probably right after nucleation was supposed to be completed.

The crystal size distribution (Figure 6) confirms the very good MV and CV data ("a" and "b", determined by the image analyzer program are the sizes of the rectangles enclosing the individual crystals). However, even exceptionally good CV data, and MV very close to the target value are no guarantee for faultless crystal quality.

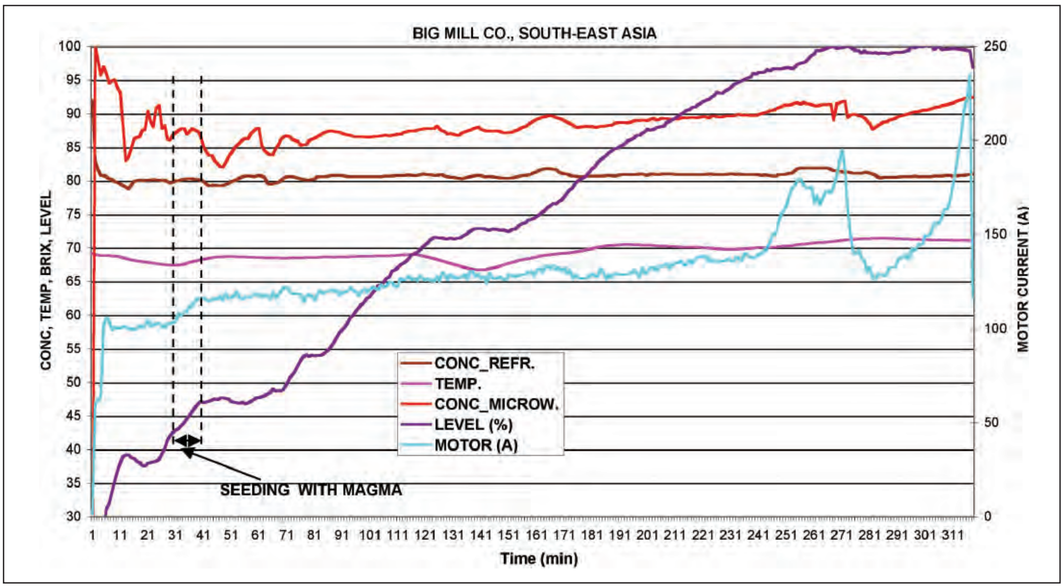
Figure 6. Crystal size distribution



Problems identified:

- Supersaturation was above the critical limit for some time even after nucleation was supposed to be ended. There is no way to detect this problem by relying only on density data used for strike control (the case would be similar with massecuite brix as well).
- The laboratory methods to check crystal size distribution were unable to detect the problem of serious conglomerate formation.
- The gradual decline of the trend of supersaturation towards the end of the strike (well below 1.10) results in longer than optimal boiling times.

Figure 7. Typical sensor data



Case study 3: "BIG MILL" Co, South-East Asia

This really big mill uses microwave massecuite solids content (brix) sensors on its huge pans. Strike control is manual.

Strike history:

1. Concentration of the syrup followed by full seeding with magma (mean seed crystal size: 0.6 mm). Feed syrup purity P1 = 83 %.

2. Manual strike control. Later on feed was continued with lower purity syrups. Towards the end of the strike the motor was switched to low

Figure 8. Supersaturation and mean crystal size calculated by SeedMaster 2

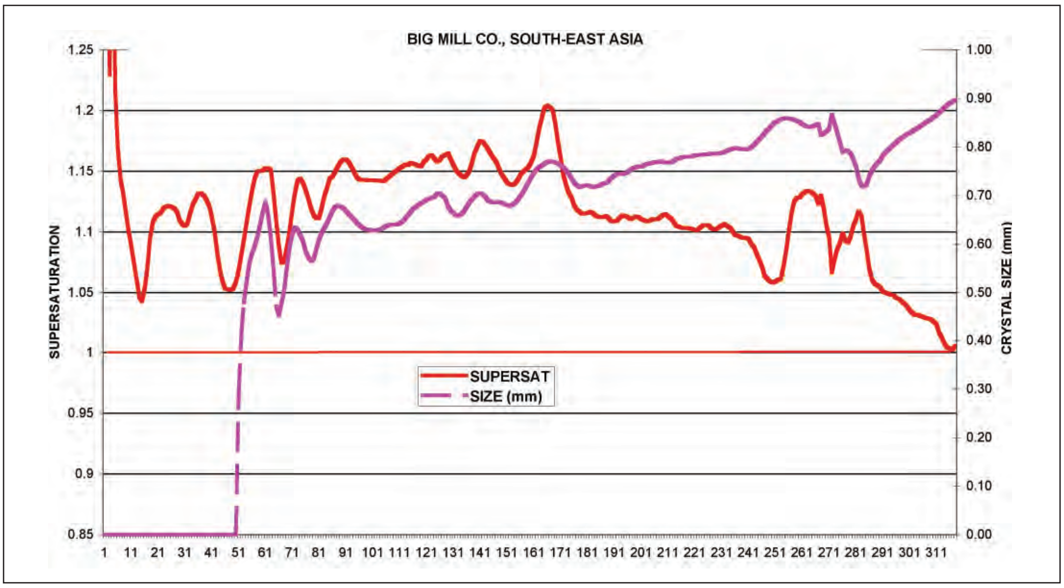
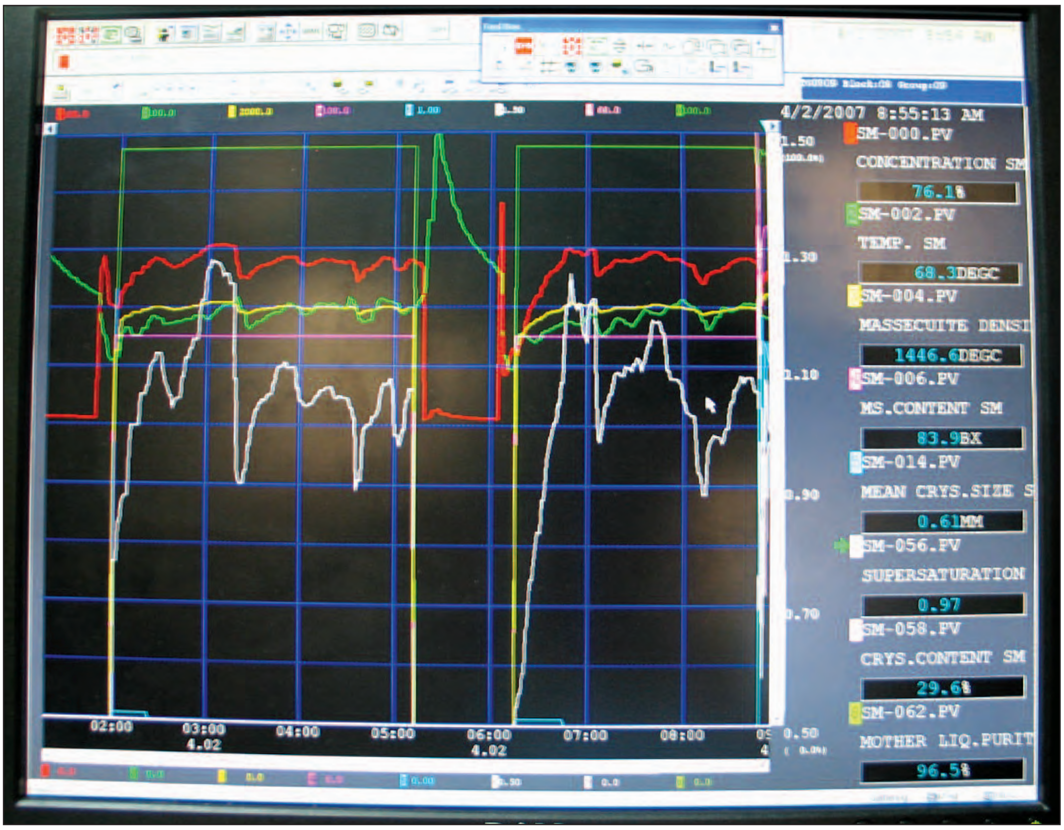


Figure 9. Manual control of refined product strikes



speed operation.

3. Instruments used: Microwave brix, temperature, motor current and level transmitters. The K-PATENTS process refractometer and the SeedMaster 2 device were added to check first the actual control practice.

In Figure 7, typical sensor data while, in Figure 8, calculated supersaturation and crystal size are trended.

Comment:

Based on the available data trended in Figure 7 it is simply impossible to judge the quality of strike control. Due to the fact that the strikes are under manual control, there are considerable differences from strike to strike. On-line data and the ones calculated by SeedMaster 2 prove this point.

Problems identified:

- Disregarding the large fluctuations of supersaturation, the first part of the strike is fairly well controlled. Later on supersaturation exceeds the safe limit somewhat, which might cause some unwanted nucleation.
- Towards the end of the strike supersaturation drops considerably and the strike is finished with no supersaturation at all (1.00).

Figure 9 shows a monitor picture in the same mill using the same instruments with a high purity strike. In this case SeedMaster 2 was connected to a control system via its Ethernet communication line transmitting calculated data and the ones measured on-line (some of the data trends were removed to make the screen less crowded). The white trend line (scale on the right side) shows the trend of supersaturation during almost two complete strikes.

In this case shock seeding was practiced with supersaturation in the 1.26…1.28 range.

Problems identified:

- The number of crystals formed during nucleation is proportional to the area between the curve of supersaturation and the critical limit of nucleation. There are considerable differences among the consecutive strikes. This consequently results in differences in product crystal contents from strike to strike.
- In both strikes the extensive use of water results in large drops (down to 0.9!) of supersaturation. Evidently, the pan man was trying to get rid of some of the unwanted crystals (1st strike), while after the first attempt of nucleation in the 2nd strike he found the crystal crop not to be sufficient, therefore it was followed by a second nucleation.

Figure 10. Major strike data in a mill in North America

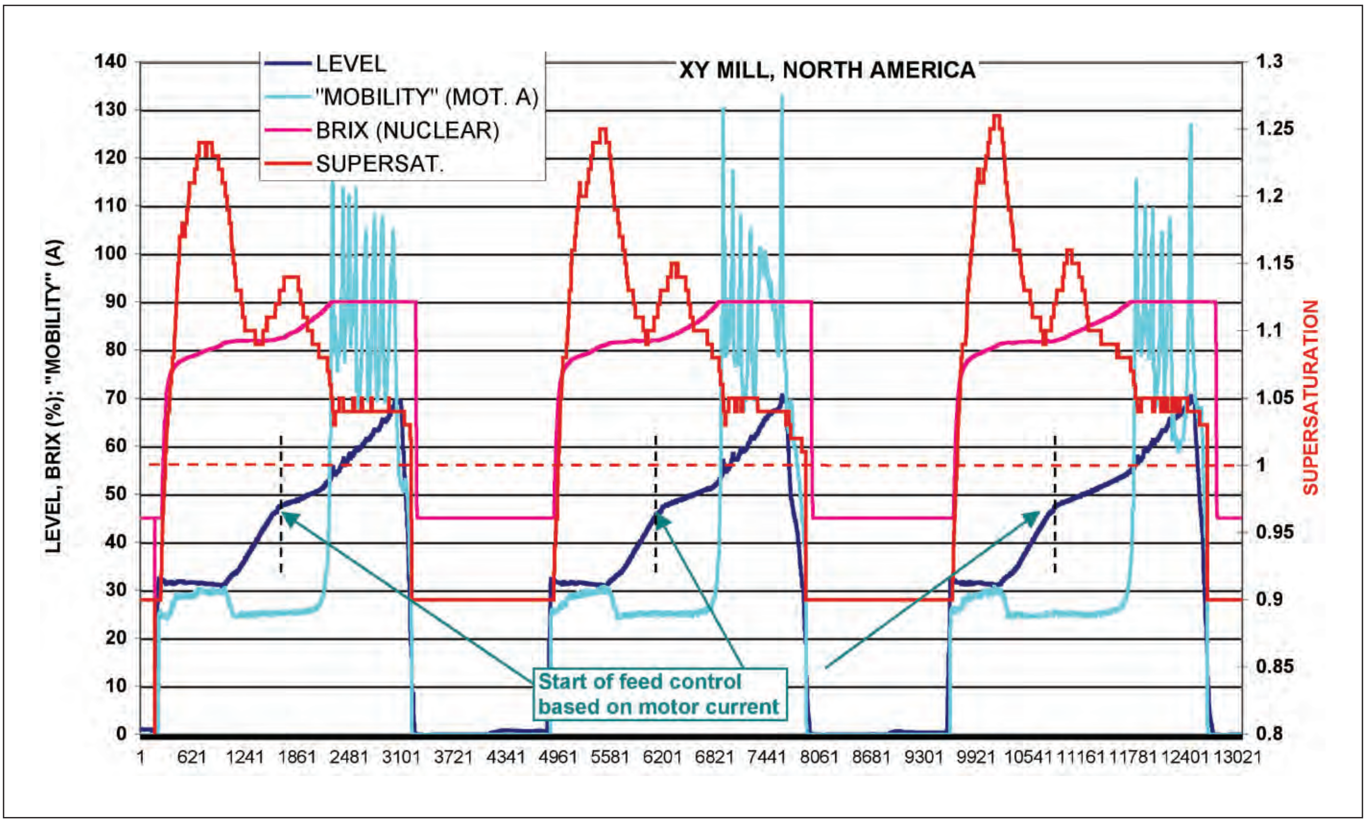


Figure 11. A closer look at the major strike data

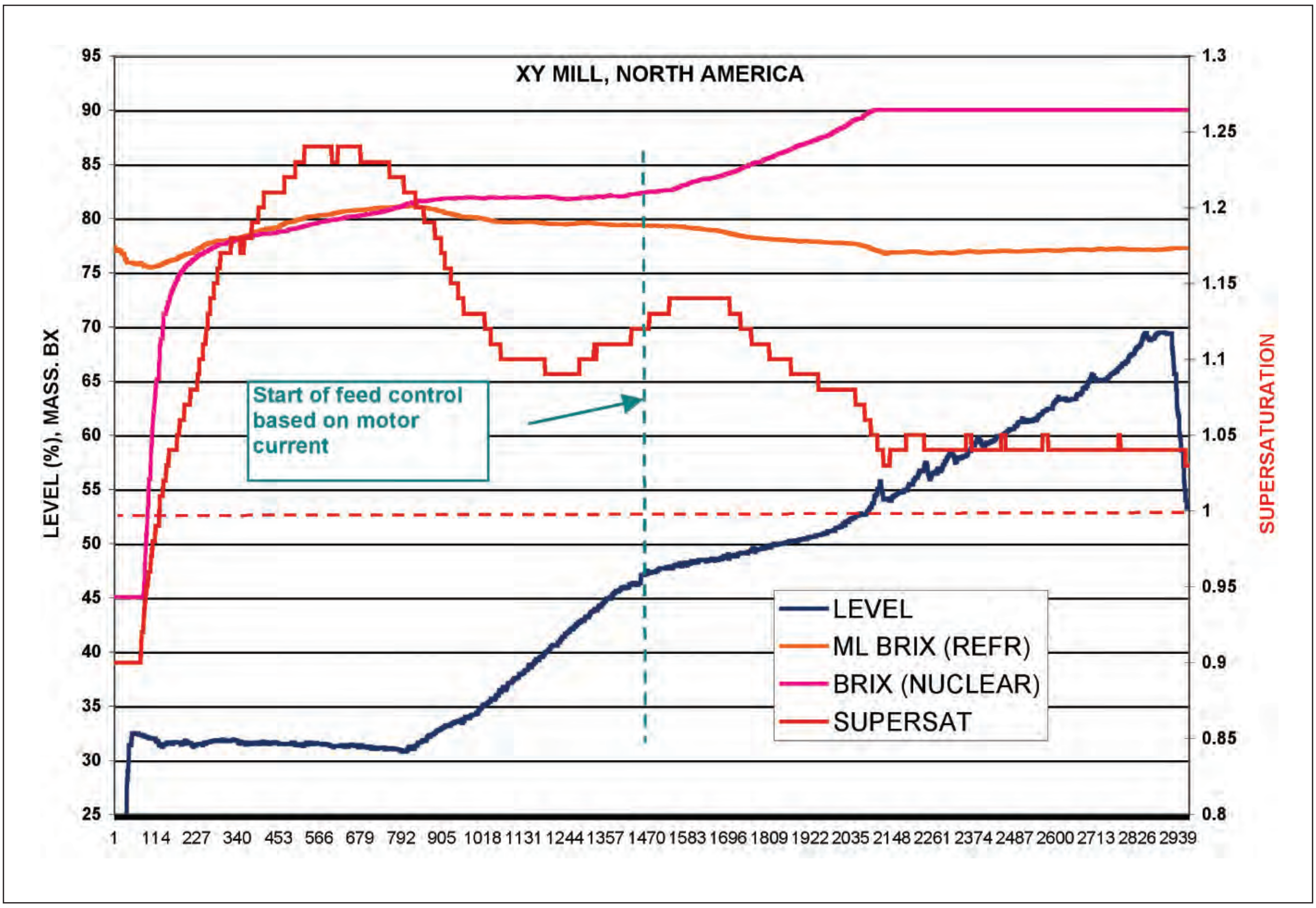


Figure 12. Monitored data

Figure 13. Crystal photograph

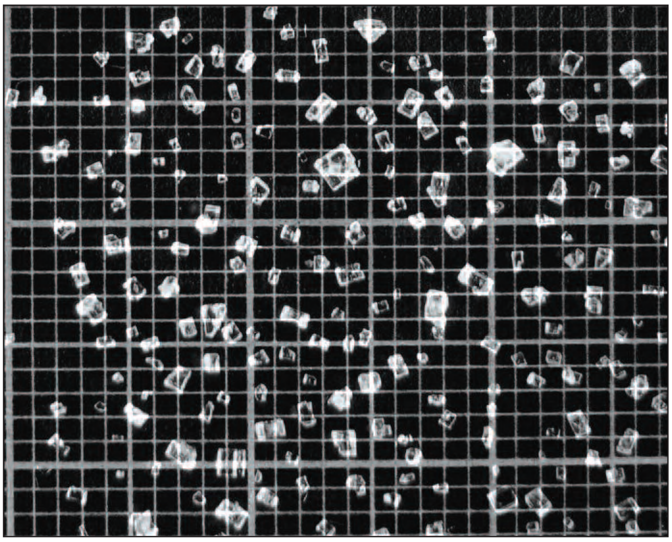
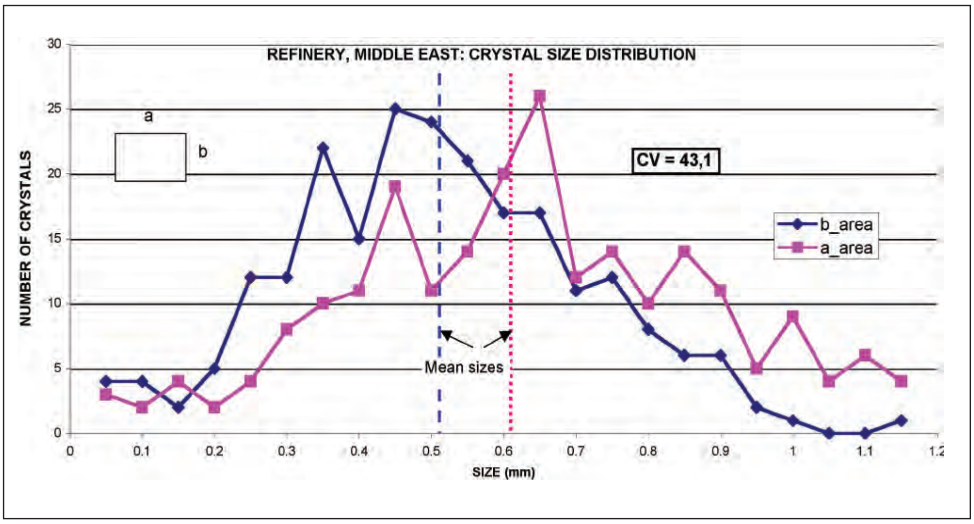


Figure 14. Crystal size distribution



This, however, was more than enough, so he decided to use water again. This was a typical trial and error mode of control.

• The extensive use of water and the hectic way the strikes are being controlled result in long boiling times (more than 3 hours with a high purity strike!), waste of energy, poor product yield and rhapsodically changing product quality.

Case study 4: Sugar mill in North America

In this mill full automatic control of crystallization was implemented. Figure 10 shows the major monitored data with supersaturation calculated on-line by using the K-PATENTS PR-01-S refractometer and the optional SeedMaster software.

Strike history:

1. Concentration of the syrup followed by shock seeding. Maximal supersaturation is close to 1.25.

2. Nucleation is stopped by intensive syrup feed until it drops to about 1.10.

3. Having completed nucleation syrup feed control is based on motor current consumption. This results in slower syrup feed and level increase until crystal content exceeds about 40 % by volume. During this time supersaturation shows a brief increase up to 1.15, followed by a fast decrease to about 1.05.

4. When crystal content exceeds about 40 %, use of the motor as a sensor results in a type of feed control similar to on-off control: due to a large “draught” of syrup directed by fast increasing current it drops fast to a still high value. As most of the fresh syrup entered moves probably upwards in the downtake, its effect in the neighborhood of the impeller will disappear fast, resulting again in a fast increase of motor current. This kind of operation continues till the end of the strike.

5. Instruments used: Nuclear massecuite brix probe, K-PATENTS process refractometer with the SeedMaster software option, level and motor current transmitters.

Comment:

Figure 10 documents excellent reproduction of all of the major data monitored during three consecutive strikes. Figure 11 provides some more detailed information on strike control. This case shows some similarity to the one already discussed (Case 1).

Problems identified:

• When after a fairly fast increase of

Figure 15. Indicating transmitter of the refractometer and SeedMaster 2

level syrup feed control based on motor current begins, this results in a slower increase of level. Not much later the fresh sugar supply is unable to cover the increasing demand, therefore a gradual decrease of mother liquor concentration and supersaturation can be observed. However, the massecuite solids content (brix) probe reports continuing and quite fast crystal growth. This is a contradiction which would need more information (for example: location of the refractometer and brix sensors) to clarify. It is also not known why the brix sensor output was limited ("frozen") from strike to strike at exactly 90 Bx during the last third of the strike. The massecuite brix sensor probably needs a calibration check.

• It is logical to assume that fast deteriorating circulation in the vicinity of the refractometer sensor head prevents the fresh supply of sugar to reach that part of the pan where it was located.

Case study 5: Refinery in the Middle East

The fairly new and large refinery has microwave solids content (brix) sensors on its pans. It has purchased recently a PR-01-S type process refractometer from K-PATENTS complete with the optional SeedMaster software in order to monitor supersaturation on-line during batch sugar crystallization.

Plant information and data:

PR-01-S sensor location: under the calandria in the pan bottom.

Feed syrup purity:	99.7 %
Crystal content (end of strike):	55 %
Target product mean crystal size (MV):	0.60…0.62 mm
Coefficient of variation (CV):	35…41

Strike history:

1. Concentration of the syrup followed by shock seeding. Set-point for seeding: 80 Bx measured by the microwave probe. Supersaturation maximum during nucleation: 1.26…1.39. Length of nucleation: different from strike to strike (see Figure 12).

2. Nucleation is stopped by a combination of intensive syrup feed (see the drop of mother liquor concentration measured by the refractometer) and increase of absolute pressure (and temperature, not shown here) in the pan.

3. In the second half of the strike supersaturation regularly hovers in the 1.06…1.08 range strike time changes between 90 to 120 minutes.

4. Instruments used: microwave probe to measure massecuite solids content (brix), K-PATENTS process refractometer with the SeedMaster software option.

Besides the real-time data shown in Figure 12, a crystal photo (Figure 13) of the product and the crystal size distribution (Figure 14) based on it, provided useful additional information.

Comment:

This case is in quite many respects similar to Case 2 (refinery in Scandinavia). Feed syrup purities, product target crystal sizes, the type of seeding methods used are very similar. The end results, however, show large differences.

Problems identified:

• Trended data in Figure 12 proves the point that constant syrup brix in the seeding points (measured by any type of instrument) will not guarantee constant supersaturation in these points. In this case the 80 Bx set-point for seeding results in supersaturation data rhapsodically scattered in the 1.15…1.25 range. Remember: Supersaturation is a multivariable function of several parameters, no single measurement can provide it.

• Concentration data readings of the two instruments (microwave probe and refractometer) in the seeding points (there are no crystals as yet) should be the same. In this case (as in quite many others, too), the readings of the microwave probe from strike to strike are 2 to 3 % larger than those of the refractometer. This reflects a serious calibration and stability problem with the microwave instrument. The process refractometer is calibrated by the manufacturer using authorized standard calibration liquids assuring + / - 0.1 % accuracy and it is not influenced by sensor head location.

• The supersaturation trend of any strike in Figure 12 gives the impression of uncertainty, lack of real control unlike the trend in Case 2 (see Figure 6a in Part 1 [3]). Looking at the series of similar trends enforces this feeling even more.

• The fairly fast drop (after approximately the first half of the strike is over) of supersaturation well below 1.10 and decreasing to about 1.06 towards the end of the strike makes the time spent for crystallization longer than necessary. The low supersaturation readings are closely connected to the low readings of mother liquor concentration measured by the refractometer (about 77.5 % during this time). Lacking more information it can only be suspected that the basic reasons are difficulty in the supply of fresh syrup to the increasing crystal mass coupled with deteriorating heat transfer and evaporation.

• The crystal photo and the crystal size distribution confirm the original information from the plant. The coefficient of variation (CV) is far from being acceptable.

Figure 16. Supersaturation and crystal content on the SeedMaster 2 display

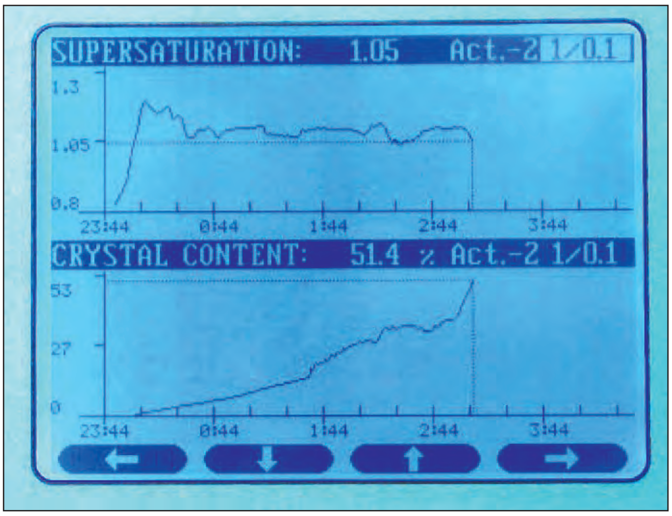
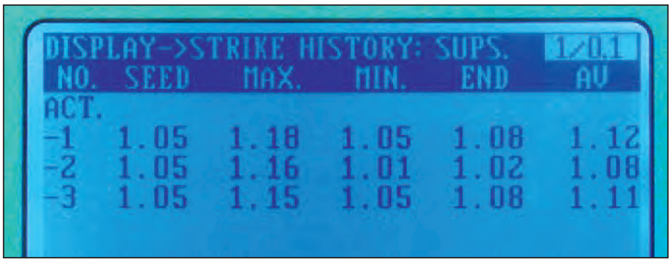


Figure 17. Strike history (supersaturation) for the actual (inactive) and 3 previous strikes



Case study 6: Beet sugar mill, Hungary (Figure 15)

This mill was one of the testing grounds of the SeedMaster 2 Crystallization Transmitter and Automatic Seeding device (CT&SD). Supersaturation and other calculated data acquired and shown here did not play any active role (except when seeding) in the actual strike control implemented by a DCS. However, they certainly had an indirect effect in inducing some changes later.

Strike history:
1. The task of the pan used during the tests was to produce high quality crystal footing (second magma) for the A product pans of the mill. Seeding method: Full seeding with first magma produced in a small cooling crystallizer (seed crystal size: 0.07 mm, supersaturation when seeding: 1.05). Product crystal size (target): 0.25 mm, feed syrup purity: 95.6 %.
2. Mode of control: level control and massecuite solids content (brix) control.
3. Instruments used: K-PATENTS process refractometer, microwave brix, level and motor current transmitters. The SeedMaster 2 (its provisional name was CT&SD) was an addition to monitor pan operations. Supersaturation, massecuite brix, crystal content etc. data were calculated based on refractometer data (mother liquor concentration and temperature) and on the current consumption of the stirrer motor. The output of the microwave probe was used only to compare its readings with refractometer data before seeding and with similar massecuite brix data calculated by SeedMaster 2 (Figure 18).

Figure 15 shows the K-PATENTS refractometer and SeedMaster 2 installation right at the vacuum pan used for seed magma production.

Comment:
When crystal footing is used to implement full seeding its quality is very important in order to produce high quality product crystals. One batch of footing magma can cover the need for 20-30 product batches. As can be seen from the supersaturation data shown in the following figures, it was more or less kept below the critical limit. This results in quite low fines content (4-6 %) as determined by the laboratory.

Figure 16 shows one of the many display screens that characterize the man-machine interface of SeedMaster 2. All measured and calculated data, including besides the ones shown massecuite density, solids content, mother liquor purity and mean crystal size can be trended for the actual and three previous strikes pairing them according to the selection made by the user.

In the strike history display of Figure 17, supersaturation when seeding, maximum, minimum, strike end and average (from seeding till the end of strike) values are displayed for the actual (here: Inactive) and three previous strikes.

Problems identified:
1. The fluctuation of supersaturation (Figure 16) is caused by temperature changes due to fluctuation of the absolute pressure (or vacuum) in the pan served by a common vacuum system. There were no means to implement individual vacuum control.
2. While seeding was done exactly at the same supersaturation (1.05) from strike to strike by making use of supersaturation data from SeedMaster 2, there are some changes in the minimum, maximum, strike end and average data as documented by the strike history screen (Figure 17). These are due again to changes of massecuite temperature.
3. Readings of the microwave probe before and in the seeding point were by about 1.8 % larger than those of the K-PATENTS process refractometer (Figure 18). What was even more disturbing: The difference was not constant, but showed rhapsodic changes from strike to strike in the 1.6…2.3 range.

Summary

1. The case studies show that from the above six mills, five still use the traditional shock seeding. Despite the fact that all of them have some kind of automatic control system for strike control, there are large differences in the way shock seeding is implemented. There is only one (Case 2), where shock seeding is really under control, though further control of the strike could be improved in order to avoid conglomerate formation. In most cases there are very serious problems with shock seeding: Poor repeatability and lack of convincing control are due to wrong instrument selection, manual control or control programs based on outdated concepts. Case 6 proves the advantage of full seeding by using footing magma coupled with fairly good strike control.
2. In most of the strikes (exception: see Figure 6a in Part 1 [3]) a general tendency can be detected: This is the decline of supersaturation in the second part of the strike. It is due to the:

- decrease of mother liquor purity, and

Figure 18. Some of the important data trended on the DCS monitor

• to the drop of mother liquor concentration at the refractometer sensor head location (pan bottom).

The first one can almost be neglected with high purity syrups (refinery product), but certainly not with lower purity ones. The substantial drop of concentration, however, is due:

• to depleting the sugar in the mother liquor (intentionally or unwittingly),

• to problems with fresh syrup supply because of deteriorating massecuite circulation, or

• to limited speed of evaporation due to fast decreasing heat transfer.

3. Constant and reproducible product parameters (crystal quality and crystal content at the end of the strikes) require reproducible seeding (full seeding) and crystal growth (supersaturation profile) from strike to strike.

Conclusion

Sugar crystallization is a delicate process. Its control was left for a long time to the "artisan" pan men with almost no instruments at all. The local laboratories in the mills were kept busy by taking samples from the pans and analyzing them to monitor the process, diagnose the problems at hand and advise the pan man how to do it better next time. In most cases it was almost hopeless: Parameters and situations can change quite fast during crystallization, and when the diagnosis was ready it was already far too late. The chain of human actions: Data acquisition with very limited means, problem specification (diagnosis) and control operation was much too slow, error-prone and unreliable. This kind of approach is unable to detect the large number of difficulties due to a similarly large number of differences in the details: Differences in machinery, instruments, syrup quality, mode of seeding, strike control etc. Most of the instruments in use today are unable to provide the kind of real-time information needed not only to understand what is going on in the process and why, but also to use it for reliable on-line control of crystallization. Efforts to study the problems by creating a virtual reality in the isolated world of the laboratory and speculations based on question-able simplifications suggest solutions to existing problems that should be treated with suspicion.

Sugar crystallization should be controlled automatically, with a minimum, if any human intervention in real time. This naturally should be based on real-time information on the most important parameters of the process, that is on parameters that really count: most of all on supersat-uration and crystal content which were previously un-available in real time. These data are very important because they can provide real insight in the details of the process going on; not in a generalized, typified, standardized, (often idealized) crystallizer, but right in the real one operating with all of its peculiarities and limitations on the plant floor of the mill. After all, it is the real one, and not a text-book version which should produce good quality crystals by billions in every batch, from strike to strike.

The times are over when a doctor had to rely only on his stethoscope to diagnose some ills of his patient. The Computer Tomograph (CT), Magnetic Resonance Imaging (MRI) device etc. are able to show the inner workings of the human body in fine details in real time, never imagined possible before. The same way, while still using some of the type of information accustomed during the long history of sugar manufacturing, it is time to switch to new ways to diagnose and solve problems better, when product quality and cost of production are at stake. The tools are available: The process refractometer already became the workhorse, among others of the sugar industry, too. When combined with the use of the SeedMaster software or the more sophisticated SeedMaster 2 device, the stage is set to have real insight in the inner workings of a crystallizer in real time, and to implement more advanced ways of sugar crystallization control.

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