

# Sugar crystallization: Look for the devil in the details Part 1

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## *abstract*

**Sugar crystallization is a process of key importance. Currently a large percentage of crystallized sugar is produced under manual control using instruments that arguably do not provide actionable information, and apparently resemble methods used several decades ago. This kind of approach is completely unfit to mass produce quality sugar at competitive cost. The first part of the paper is devoted to discussing some of the many details of crystallization which have been the subject of many investigations since a long time. Some common myths are challenged. Supersaturation and its role in determining product quality, cost of production, crystal growth and seeding are discussed in more detail. This is followed by the discussion of quite a few important details including pan circulation, strike control and crystal size distribution.**

Keywords:

## **Introduction**

The long history of sugar manufacturing brings back memories of times when sugar crystallization relied exclusively on the expertise of the “artisan” pan-men, who acquired their skill and prestige during long years of observation. Quite many decades have passed by without any major change in crystallization practice. Even nowadays there are thousands of vacuum pans in use worldwide which had been designed several decades ago.

The first real changes began to appear towards the middle of the last century. The installation of the motor-driven stirrer was applauded

as a big step to improve circulation in the pan (it is still missing in a large number of pans in use today). The first on-line instrument (the conductivity probe) was followed later on by some other ones (nuclear or microwave density and solids contents (brix), capacitance / conductivity (RF) sensors), which comprise even today the majority of instruments used for crystallization control. In theory the importance of supersaturation as the main driving force of crystallization was soon widely acknowledged. However, due to the fact that none of the instruments listed is able to measure supersaturation on-line, most of its treatment in the relevant professional literature and crystallization practice amounts to nothing else as obligatory lip service.

Mechanical and chemical engineers have played a very important role in the advancement of crystallization practice and sugar manufacturing as a whole. Due to the lack of on-line instruments their research activities have been mostly based on experiments in the laboratory, where one of the most important instruments was the refractometer, which is the undisputed tool even today. The findings of these researchers are well documented in the journals of the profession and in thick volumes of their prestigious books. It has to be noticed, however, that most of these books which can be found on the bookshelves of practicing plant managers and technologists (often with some layer of dust on them) are reprints of the original first editions dating back in quite many cases to several decades. Mechanical construction and even design of complex machinery are often treated to the finest details while the tools, that is the instruments required to diagnose the multitude of problems related to crystallization are completely neglected, or are treated, if at all, on almost “pre-historic” levels. The same is true with the on-line automatic control of crystallization.

This author came to the field of sugar manufacturing more than 25 years ago as an electrical engineer specializing in industrial process control. It was obvious that in order to be able to solve control problems of different parts of the technology they had to be studied very carefully. The importance and role of supersaturation in crystallization control was discovered very soon in the relevant literature. Surprisingly enough, no trace of any instrument capable to monitor supersaturation on-line was found. It was only a few years later when we began to use the only known supersaturation transmitter manufactured by Ziegler Associates, USA. The late J. Ziegler himself was a well-known name not only as a pioneer in automatic control, but his contributions to sugar crystallization control are also held in high regard. His sensing of the lack of the right instrument needed in crystallization control was well understood.

We can witness very considerable changes in the requirements facing the sugar industry of our times. Product quality and cost of production are the major criteria, which determine the fate and survival of a manufacturer today. Mass production of high- and constant quality products at competitive cost is un-conceivable without advanced automatic control based on the use of on-line instruments providing data on those parameters of the product which are really vital. For several reasons laboratory data have only very limited use in closed loop control. On-line monitoring of the right data right in the real process under control is a must, and no data from the virtual reality of the often idealized experiments in the laboratory are able to fill this need.

New tools, skills and know-how are required to diagnose the problems, to discover the “devil in the details”, to open new ways to advanced control methods in a very conservative industry, where old habits die hard.

The first part of the paper describes and discusses most of the key issues on crystallization. With particular reference to 6 case studies part 2 of the paper will elaborate the discussion further.

## Common myths

### “Sugar crystallization is an art” and the myth of the “master boiling”

“While there have been several generations of equipment improve-

ments, several pan floors continue to depend totally upon the sugar boiler’s power of observation and his sense of timing.”<sup>[1]</sup> Simply put: the “artisan” sugar boiler is the undisputed master of operations.

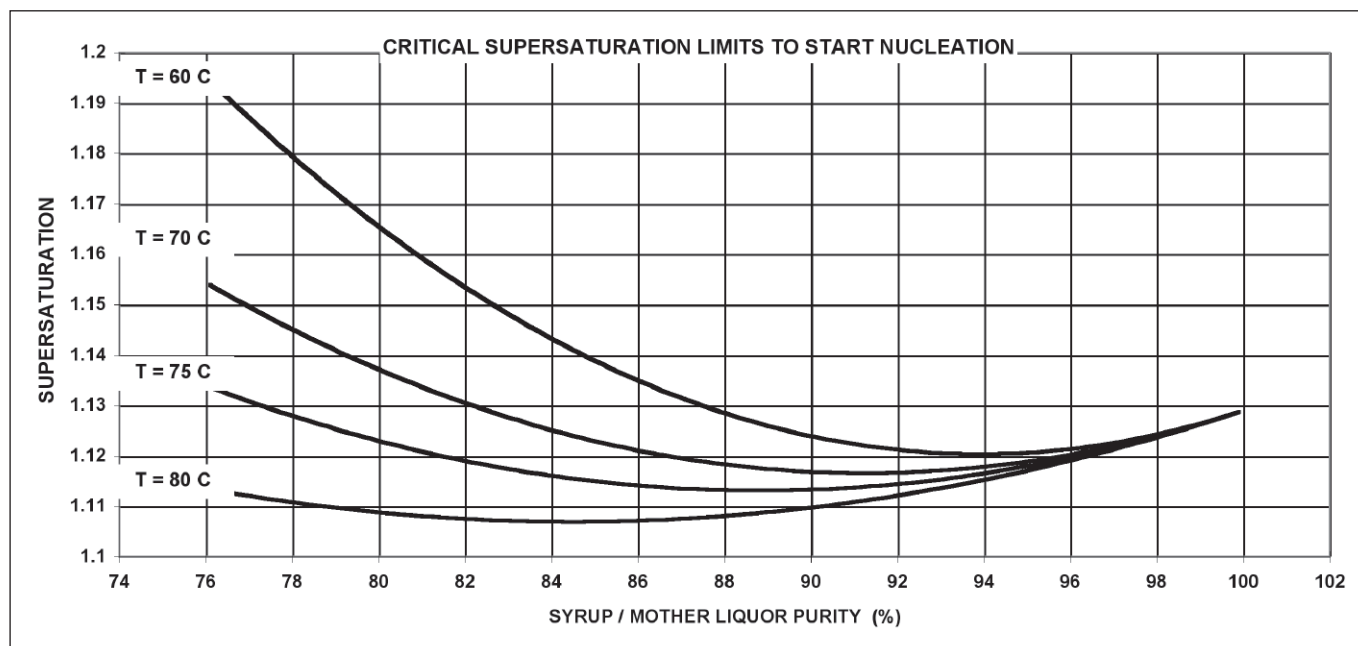
About ten years ago I had the opportunity to monitor the operation of a master boiler during two consecutive strikes in daylight with no change of conditions in a big mill in South East Asia. A process refractometer from K-PATENTS OY, Finland was installed in the bottom of a big vacuum pan and it was connected to my notebook computer running an on-line supersaturation calculation. A mercury thermometer and a vacuum gauge completed the set of instruments in use. The supersaturation profile (trend) in the two strikes differed considerably: in the first one higher peaks and lows (the lows due to excessive use of water) could be observed. Contrary to a typical 45 % claimed by the local laboratory, product crystal content in the first strike was a mere 26 %, while in the second one, probably due to my presence and attention noticed by the pan-man it was a little better with 34 % when the charge was dropped. I decided not to wait for a night strike. It has to be realized: the times are over when the survival of plants depends totally on the human sugar boiler’s skill, mood, time of the day etc.

Automatic control of processes, including crystallization control is commonplace nowadays. Very advanced tools of process control (PLC-s, Distributed Control Systems (DCS)) are available from well known system vendors. Instrument selection and control strategy, however, are sensitive questions. We are witnessing a situation when quite often the level of crystallization control know-how does not exceed that of the local “master boiler” of several decades ago. For some reason it seemed to be evident for quite a few decision-makers that if automation became un-avoidable, then it was logical to let the control system do what the “master” did before: this is the myth of the “master boiling”. Those who have already been confronted with the task of the design, programming and successful commissioning of a sugar crystallization control strategy, probably have been embarrassed to find that the double Dutch provided by one of the “master boilers” (by the way: which one to trust?) could be barely used to tame that rigorous beast, the computer. We can often find monitors in control rooms with really nice screens on the pan-farm, for example, but be prepared for some surprises, too. In quite many cases it turns out that the screens are only used to replace outdated instrument dials and vital control operations are mostly done in manual mode serving the sugar boiler as the old ones did before. Why should the end result regarding production costs, product quality and competitiveness be any different?

Spending a lot of time, effort and money on expensive automation is simply useless if it is based on the lack of the right instruments and on “the sugar boiler’s power of observation and sense of timing” enshrined in the form of an outdated control concept and program. It will only result in committing the same mistakes and causing the same losses made by the “master boiler” before, but from now on repeatedly and automatically, discrediting the very aim of a project.

There are good examples as well. In the control room of a refinery in Scandinavia the single staff operator sitting in front of the monitors during most of the year is replaced during the summer vacation time by an undergraduate from the chemistry or law (!) faculty of the local university. It took quite some time to refine the control program to the level it has nowadays, but it paid off. It is the computer that makes the decisions. The same ones, relentlessly even during the night shift.

**Figure 1.** Supersaturation limits to avoid unwanted nucleation



In our world today mass production faces very strict quality requirements and results in product volumes unconceivable 50 years ago. Only a few percent improvement in the cost of production can be decisive in securing the survival of a plant. This, however, needs new approaches to advanced crystallization control, too. The know-how of a traditional “master boiler” even if it is programmed (and frozen) in a nice computer based control system is just not good enough.

“We use conductivity, density, solids content (brix), RF capacitance, consistency to measure supersaturation”

It is well known that supersaturation is the most important parameter of a crystallization process. It is a multivariable function of several liquid phase (that is: syrup or mother liquor) parameters of the masseccuite. It is evident that these parameters must be measured selectively, that is not disturbed by the increasing crystal content in order to be able to calculate supersaturation on-line during the complete strike. This will be discussed in more detail later.

Independently from the principle of operation the readings of the sensors mentioned above will be governed more and more by the increasing crystal content after seeding has been completed. None of them is able therefore to provide information on the concentration of the liquid phase (only the digital process refractometer can live up to the task), which is one of the important parameters in the calculation of supersaturation. The only other alternative, that is using the measurement of boiling point elevation to track changes in mother liquor concentration is unable to meet the strict accuracy requirements. These sensors are therefore unable to provide reliable information on supersaturation.

Supersaturation can not be determined based on the measurement of a single parameter (see Eq. 1.). Therefore claims similar to the one quoted above are completely false and misleading.

“Supersaturation is only important when seeding”

It is well known that crystallization is possible only if the solution is

supersaturated. Still it is surprising to find in some of the technical papers of the industry opinions stating that supersaturation is only important in the seeding point. Later on, at least according to these statements, when there is already a considerable crystal content the danger of excessive supersaturation, and so the formation of fines and conglomerates can be excluded. In some cases this can be true, but only due to some circumstances, which will be discussed later. The truth is that in order to prevent these undesired consequences supersaturation must not exceed the critical level all over the complete strike. How well this requirement is met will be determined by the parameters of crystallization and the mode of its control (syrup purity, quality and feed, heating steam pressure and vacuum, that is the way a strike is being controlled). If, for example, the speed of evaporation is higher than required, then this can result in excessive supersaturation. The same way: less than desired supersaturation can lead to loss of already crystallized sugar and / or longer boiling time.

While pretending to use them to provide information on supersaturation when seeding, the myth quoted above is only used to disguise the fact that all of the single-parameter sensors are useless to provide meaningful and reliable information on supersaturation, and it is even more true after seeding has been completed.

**The role of supersaturation in crystallization**

**Supersaturation limits to avoid unwanted nucleation**

Supersaturation is defined as the ratio of sugar in solution to sugar needed to saturate the solution at the same temperature. It is a multivariable function of several variables and can only be calculated based on acquired data:

$$\text{Supersaturation (SS)} = f1 (C_I, Q_I, T, m, b, c) \quad \text{Eq. 1.}$$

where

C_I:	liquor (mother liquor) concentration	(%)
Q_I:	liquor (mother liquor) purity	(%)

T: temperature ©  
 m, b, c: feed syrup quality parameters ()

**Supersaturation, product quality and cost of production**

The feed syrup quality parameters reflect the difference of the solubility of saccharose in pure and in impure sugar solutions. There is a considerable difference between the impure beet and cane sugar solutions regarding the quality parameters [2]. It is evident from Eq. 1. that no single parameter is able to provide reliable information on supersaturation.

One can find only limited (and often contradictory) data in the literature (mostly fairly old ones) on the critical supersaturation (limit of the metastable zone), above which in the presence of crystals unwanted nucleation occurs. A few of these are listed below.

- J.C.P. Chen : 1.20
- A. VanHook : 1.20
- McGinnis : 1.35
- E. Hugot : 1.17 (A product)
- Webre : 1.25...1.40 (C product)

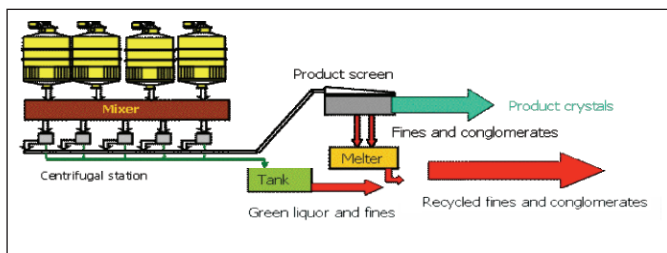
In a recent paper the published equation results in data in the 1.11...1.15 range depending on liquor purity and temperature [3] (Fig 1.).

It is quite amusing to find that most of the different authors who advise the critical limit data do not pay any attention to the lack of reliable on-line information on supersaturation. This can be attributed to:

- misleading claims about sensors in use today, and
- the lack of any instrument capable to fill the need for on-line data on supersaturation. [4], [5]

In the last few years, however, with the introduction of the optional SeedMaster software [6] and the SeedMaster 2 crystallization transmitter and automatic seeding device [7] considerable changes took place and found their way into actual practice. Both rely on the use of the digital process refractometers manufactured by K-PATENTS OY, Finland (for more information on these devices visit [www.processcontrol.hu](http://www.processcontrol.hu) or [www.kpatents.com](http://www.kpatents.com)).

**Figure 2.** Material flow in crystallization



It is well known that excessive supersaturation results in unwanted nucleation (formation of fines) and besides poor circulation it is made responsible for the formation of twins and conglomerates as well. When advised not only to monitor on-line, but also to modify the previous supersaturation profile during the strike, conglomerate content was reduced by 40 % in a refinery in North America.

Besides other parameters the speed of crystal growth is proportional to supersaturation. In order to increase the rate of production plant managers often instruct their staff to reduce boiling time. This will perhaps unknowingly result in supersaturation well above the critical limit. As a first approximation the rate of production is measured by the number of strikes during a shift, or day. Problems with crystal quality and the true yield (the actual amount of product per strike) will turn out only later, if ever. Fines and conglomerates are recycled in the process resulting in considerable losses (Fig 2.).

It is informative to have a closer look at this problem by using material balances on crystallization. We can define the following 2 parameters:

Yield of crystallization (K): amount of crystals produced over amount of dissolved sugar entering the process.

Product yield (Yp): % of final product from the total amount of sugar entering the process.

Let us assume that we have a crystallization station where 1 t of dissolved sugar in the thick juice is crystallized to result in 0.6 t of perfect quality product (obviously an idealization), while 0.4 t sugar leaves the station dissolved in the green syrup (K = 0.6). In this case there is no recycled crystallized sugar, that is product yield Yp = 60 % (Fig 3.).

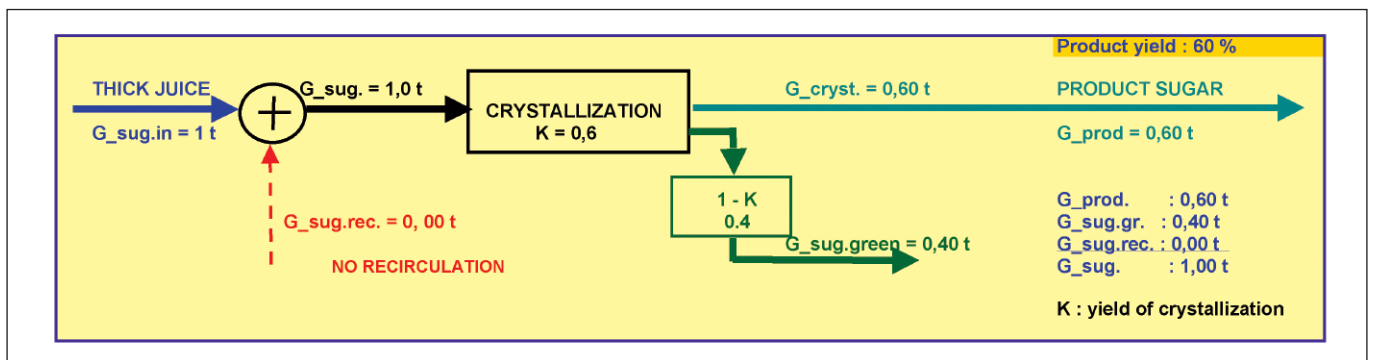
In a more realistic example due to excessive supersaturation the amount of recycled crystal sugar that is composed of:

- crystals which escape through the holes of the centrifuge screen with green syrup, and
- fines and conglomerates removed by screening

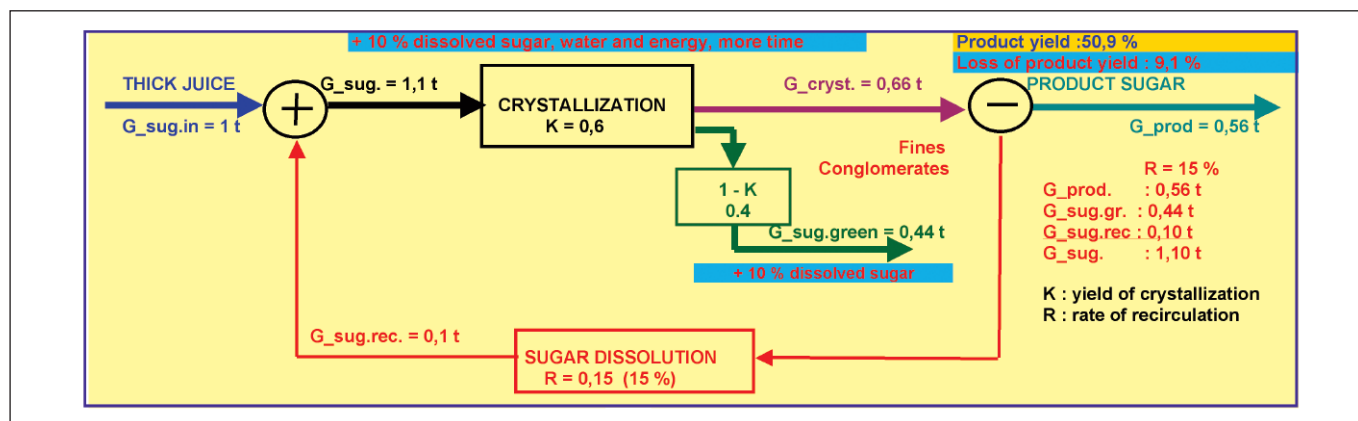
equals 15 % (rate of recycling R = 0.15) of the full amount of crystals produced, while the yield of crystallization remains unchanged (K = 0.6). Fig 4. shows the material flow diagram and data valid for this case.

Assuming the same amount of dissolved sugar in the thick juice (1 t) from the evaporator station it is informative to compare some of the data of the two cases:

**Figure 3.** Material balance in an idealized crystallization process



**Figure 4.** Material balance in a realistic case of crystallization



	Ideal process	Realistic process	Change
Recycled crystal sugar	0.0 t	0.10 t	+ 0.1 t
Sugar input to crystallization	1.0 t	1.10 t	+ 10 %
Yield of crystallization (K)	0.6	0.6	-
Amount of product	0.6 t	0.56 t	- 0.04 t (-6.67 %)
Dissolved sugar in the green syrup	0.4 t	0.44 t	+ 0.04 t
Product yield ( $Y_p$ )	60 %	50.91 %	- 9.09 %

**Summary:**

1. Sugar input to the crystallizer station (load) has increased by 10 % as compared to the ideal case.
2. Despite the increased sugar input and the identical yield of crystallization (K) the amount of final product was decreased by 6.67 % (0.04 t). This leaves the station with the green syrup.
3. Product yield ( $Y_p$ ) was decreased by 9.09 %.
4. Consequences: increased use of water and energy, longer boiling time and reduced real product output resulting in increased cost of production.

The lesson to learn: “pushing” the rate of production by exceeding the critical limit of supersaturation can be counter-productive indeed.

**Supersaturation and crystal growth**

Product quality was not that much an issue fifty or even more years ago as it is today. Quality and the amount of sugar produced during a unit of time are nowadays important parameters of crystallization. The later is determined by:

1. the speed of evaporation as a limiting factor, and
2. the speed of crystal growth.

**Speed of evaporation**

With well designed and maintained equipment the speed of evaporation should not become a limiting factor, that is the heat transfer coefficient between the calandria and the massecuite should not decrease to such a low value which would result in limiting the speed of evaporation. This should be true even if the calandria is supplied with low-grade vapors in order to meet the energy efficiency requirements so natural of our times. It is questionable, however,

how vacuum pans built and designed several decades ago can meet these requirements.

“Scale formation on either side of the heating surface can drastically reduce the heat transfer rate and reduce the evaporation rate to the extent that it becomes rate-limiting. Of the bulk fluid properties consistency of the crystal-syrup suspension is well known to be the main factor determining the overall crystallization rate as it effects both the rate of water evaporation as well as the rate of crystal growth”.

Besides occasional scale formation fast decrease of heat transfer is experienced on a regular basis during a strike due to fast deteriorating movement (circulation) of the massecuite as crystal content keeps increasing. This is due to the fact that the temperature within the calandria tubes drops more and more below the local boiling temperature of the mother liquor due to increasing hydrostatic pressure. Real boiling takes place only in a not too wide layer below the increasing surface level. “At the beginning of the strike there is violent evaporation in the tubes, and this has a pumping effect and increases velocity; but towards the end, when evaporation has dropped to almost nothing, there results stagnation, and not circulation”. Simply put: towards the end of a strike it can happen that the massecuite is not circulating, only vacillating: move or not to move? Taking a look at the lowest window of a pan during this time can often prove this point. Even with pans supplied with stirrers the drop in the heat transfer coefficient is quite large. Hugot reports the following results for pans with impellers in the cane sugar industry:

Heat Transfer Coefficient (HTC,  $W / m^2.K$ ): start: 640 end: 224

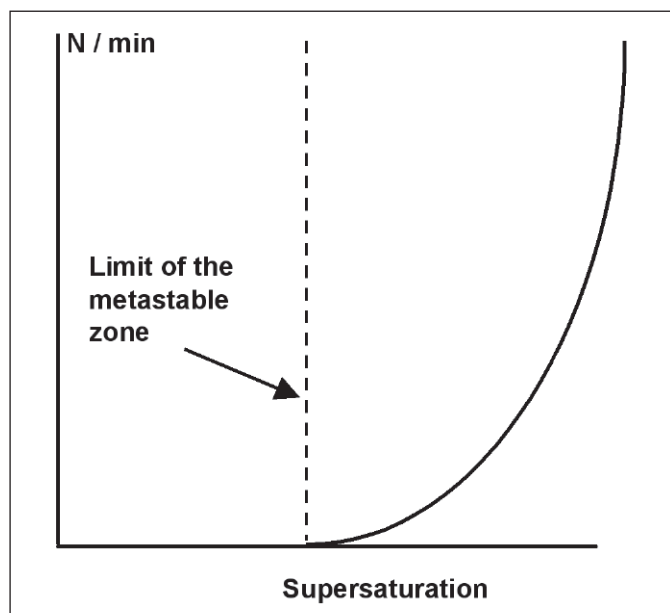
It is no wonder:  $HTC = 640 (W / m^2.K)$  of a technical sugar solution concentrated to ~ 80 % definitely will be influenced very much by increasing crystal content. HTC of crystallized sugar is only  $0.58 (W / m^2.K)$  and as the volume of mother liquor in unit volume of the massecuite keeps decreasing while crystal surface is increasing fast, resistance against heat transfer to the pockets of liquid enclosed in between the crystals goes up dramatically. This drop of heat transfer is even more enforced by the decreasing speed of massecuite flow due to fast increasing consistency.

**Speed of crystal growth**

Literature is awash with publications on the measurement of the speed of crystallization in the laboratory. In most of these attempts



**Figure 5.** Number of crystals formed per minute during nucleation



important parameters, like temperature, flow rate of the 100 % purity syrup, supersaturation etc. were intentionally kept constant. Crystal to crystal interactions are carefully avoided by using a single crystal and measuring its growth in ideal conditions.<sup>[10]</sup> Changes in crystal weight and / or size are documented.

Should, by the same reasoning problems like hunger, for example in large and poor societies be studied by monitoring the behavior of a single man living in ideal conditions alone (well, with his girl-friend...) on an isolated island? Probably not. When controlling sugar crystallization we are faced with the task of providing the best possible conditions of growth for billions of crystals in the pan on a regular basis. For crystals which are very close to each other and are fighting for the precious supply of sugar in the mother liquor, leaving behind and accumulating the “garbage” (non-sugars), which makes their further growth more and more difficult.

For sake of simplicity and disregarding form factors let us assume that the sugar crystals are cubic with a side length “a” (mm). The volume and weight of a single crystal is:

$$V = a^3 \quad (\text{mm}^3) \quad \text{Eq. 2.}$$

$$G = \rho \cdot V \quad (\text{mg}) \quad \text{Eq. 3.}$$

where  $\rho$  is the specific weight of crystallized sugar (mg / mm<sup>3</sup>).

The speed of growth (by weight) is:

$$dG / dt = 3 \cdot \rho \cdot a^2 \cdot da / dt \quad (\text{mg} / \text{min}) \quad \text{Eq. 4.}$$

where

$da / dt$  is the linear speed of growth (mm / min).

According to Eq. 4. the speed of growth by weight is proportional to the quadrate of the side length “a”. In common practice it is translated into statements like “the speed of crystallization is proportional to crystal surface”. This kind of thinking completely disregards the real cause of crystal weight increase: the linear speed of growth. A large crystal surface alone is no guarantee for actual crystal growth. An existing crystal surface is the result of the past; it does not provide reliable information on the present. It is the linear speed of growth which is the really important indicator: if it is negative, as it can be, then we are experiencing dissolution of the already crystallized sugar even when crystal surface is large.

The linear speed of growth has been the subject of studies since a long time. It is well known from the literature that supersaturation is the main driving force of crystallization. Besides that mother liquor purity, temperature and syrup quality are parameters which have considerable effect on crystal growth. Lately crystal content was added to the list of parameters governing it. Based on data acquired by on-line monitoring hundreds of strikes in different countries using process refractometers from K-PATENTS Oy and other instruments, we succeeded in developing reliable calculations to calculate - among others - the linear speed of crystal growth on-line. It is a function of several parameters having the general form:

$$da / dt = f_2(SS, Q_I, T, Y) \quad \text{Eq. 5.}$$

where

Y: crystal content by volume (%)

The actual size of a single crystal is then any time t:

$$a = a_s + \int_0^t f_2 \cdot dt \quad \text{Eq. 6.}$$

where

$a_s$ : crystal size (mm) in the slurry or footing magma when seeding at time  $t = 0$ .

In summing up:

1. the quality of the crystals is mainly determined by the supersaturation and pan circulation maintained all over the strike,
2. while the time needed to reach the required product crystal size is determined by the linear speed of crystal growth (Eq. 5.).

Supersaturation plays a major role in both. Its trend during a strike reflects the complete history of crystallization. If it is supplemented by some other information un-available on-line in common practice, diagnosing crystallization problems and its advanced control becomes easy indeed.

### Seeding methods

#### Traditional (secondary, or shock) seeding

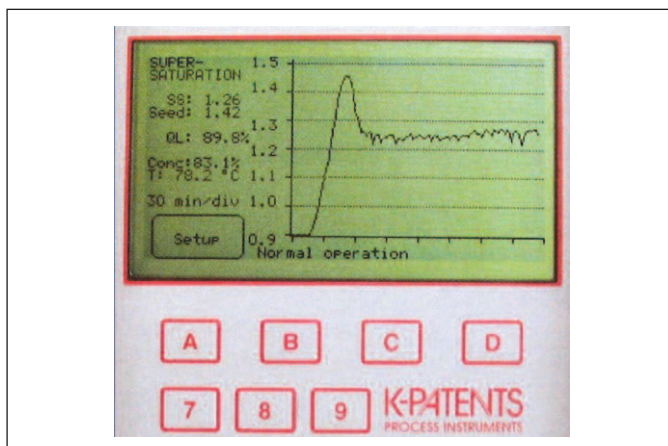
The old traditional method of seeding practiced by the “artisan” pan-men is characterized by the addition of some amount of fine icing sugar, or nowadays slurry to the supersaturated solution. If supersaturation is large enough (in the metastable zone, above the curves of Fig 1.), new crystals will be formed (nucleation). Slurry is a suspension of finely ground sugar mixed for example in isopropyl alcohol.

During nucleation new crystals will be continuously formed. Their number depends very much on the actual value of supersaturation and on the time of nucleation. Fig 5. taken from<sup>[1]</sup> shows the number of new crystals formed per minute versus supersaturation. This kind of information certainly is not much of a help, but shows the difficulty characterizing the method.

Due to the very sharp increase of the curve in Fig 5. it is very difficult (practically impossible) to have the same number of crystals from strike to strike. This, however, even when the final crystal content is constant when the charge is dropped will result in different mean crystal sizes from strike to strike.

Fig 6a. shows a typical example of shock seeding as monitored

**Figure 6a.** Shock seeding



by the optional SeedMaster software of the K-PATENTS PR-01-S type process refractometer.<sup>[6]</sup>

In this case the supersaturation set point for automatic seeding was 1.42 and it was maintained after nucleation well in the metastable zone between 1.23 to 1.26 all over the strike. Though the curve looks nice due to fully automatic control of crystallization, the consequences of excess supersaturation after nucleation cause problems with product quality.

#### Full seeding

The basic idea of full seeding is very simple: enter the number of crystals when seeding equaling the one in the end product. This means that:

**Figure 6b.** The K-PATENTS PR-01-S process refractometer



- no new crystals should be generated during the complete strike, and
- no crystals should be lost from those entered during seeding.

These two requirements simply state that supersaturation from seeding to the end of the strike must be reliably kept between 1.00 and the critical limit needed to avoid nucleation any time during the strike. In order to have the shortest possible time of crystallization supersaturation should be close but always on the safe side below the supersaturation limit. It is obvious that this task can be solved only based on the on-line monitoring of supersaturation.

Full seeding can be implemented in two ways: with slurry or with footing magma. The only difference between the two methods is the difference in the crystal sizes and the difference between the required quantity of slurry, or footing magma. In both cases knowledge of the mean seed crystal size is important.

#### Seeding with slurry

The mean crystal size with slurry seeding is usually in the 5...10 micron range. The crystals are prepared locally by ball mills or are provided by companies producing them. It is rather difficult to have reliable data on crystal size. When using ball mills it is typical to give the time of grinding instead (4-6 hours). The first step is to calculate the number of crystals needed in the slurry by taking into account the product crystal size, the useful volume of the pan and the target crystal content when dropping the charge. It is then followed by calculating the (dry) weight of the seeding sugar needed for a batch of slurry.

It is quite common to find cases when carefully prepared slurry is being used to implement shock seeding.

#### Seeding with footing magma

A way to produce footing magma is the following:

- concentrate syrup in a vacuum pan to reach supersaturation 1.05...1.08;
- transfer the concentrated syrup into a cooling crystallizer and seed it with slurry;
- drop the 1st magma with crystals 0.07... 0.1 mm mean size into a magma receiver with stirrer;
- use the 1st magma to seed a vacuum pan containing concentrated syrup (supersaturation: 1.05...1.08);
  - continue crystallization up to 0.25...0.3 mm mean crystal size (2nd magma);
  - use the 2nd magma to seed the product pans.

The use of footing magma to implement full seeding has the following advantages:

1. Control of the cooling crystallizer is simple, the risk of the formation of fines (nucleation) and conglomerates is small.
2. One batch of footing (2nd) magma can fill the need for quite a few product batches.
3. There is no need to use water during the product strike. It is easy to control the process.
4. Production of the 2nd magma and that of the end product is a simple process: only the size of

the crystals has to be increased. Naturally enough: further nucleation must be prevented by monitoring supersaturation and keeping it close to the critical limit on the safe side.

5. Due to better sugar quality centrifuging of the product is easier and needs less water.

In conclusion: full seeding implemented by using footing magma is the preferred method of seeding.

### Changes in crystallization control

Closed-loop automatic control dates back well over half a century. Its role in the sugar industry was restricted to some simple problems like level control. The early pneumatic instruments and controllers were supplemented by those using transistors, and now we are witnessing the era of computers, PLC-s and DCS based sophisticated control systems. It is natural that these developments have resulted in considerable changes in the way of crystallization control.

1. Due to the fact that closed-loop on-line control is based on information available in real time, the need for instruments capable of providing on-line data on the parameters of the process under control has increased dramatically. Though it is logical that when, for example, level has to be controlled, the use of a level transmitter is a must, in most of the crystallizers in use today, control is based on instruments which are unable to provide useful information on the most important parameter of crystallization: supersaturation. One can often find very misleading statements regarding the monitoring of supersaturation. Some control system vendors claim to use "supersaturation sensors" in their control scheme, but it turns out from the small print in a remote corner of their brochure that the sensor is actually a density or brix sensor. It is even more surprising to read that the test crystallizer of a prestigious research institute is furnished with "conductivity, microwave and RF supersaturation sensors". In view of the multivariable nature of supersaturation (see Eq. 1.) these claims are completely ungrounded.

2. The role of the local laboratory has decreased dramatically. The reasons are simple:

- Real time control of crystallization requires new data at every 10 seconds or so. It is impossible to meet this requirement based on sampling and on traditional methods of the laboratory.
- Reliability of laboratory data. Human intervention in acquiring important data has to be always treated with care. One example: in quite a few mills it is customary to collect samples from the pan and bring them to the laboratory to analyze for example mother liquor concentration. If there is a process refractometer installed in the pan, it usually turns out that its reading differs more or less from the one determined by the lab refractometer. The list of reasons can be quite long: different location of measurement, evaporation from the sample used in the laboratory, difference in temperature, different ways of calibration of the refractometers, mood of the operator in a laboratory etc.

3. Process control of complex processes requires familiarity with modern instrumentation and automatic control theory and practice coupled with the use of computers. Needless to say, familiarity with the fine details of the technology is a must. This, however, should be

based on a critical use of accumulated know-how and most of all, on data acquired on-line from the real process under control.

One can often make disturbing observations when visiting mills in different countries. When sitting down to have a closer look at one of the nice-looking screens on the monitor in a control room one can often find control loops left in the manual mode. Sometimes it turns out that the control strategy reflected by the control program is anything but up-to-date. There are small, but still disturbing niceties, too: in a large refinery the trend on the monitor was titled "Masseccuite density". When asked about the type of sensor in use it turned out that the trend displayed was based on data from the current transmitter of the stirrer motor. Inconsistent use of terms and engineering units is quite common.

The subject of automation in sugar manufacturing is discussed in more detail in.<sup>[13]</sup>

### A few details in short

#### Concentration of the feed syrup

Several years ago concentration of the feed syrup was usually in the 65-68 % range. In modern practice it is rather 72-73 %, which results in a more efficient use of energy (most of the water is evaporated in the evaporator station). In order to prevent unwanted nucleation in storage tanks, pipes, valves etc. the temperature of the syrup must not drop to the value where supersaturation exceeds the limit level (see Fig 1.). Heat insulation and controlled heating can help to solve this problem.

#### Pan circulation

"Good quality crystal crops have always been dependent upon rapid circulation of the crystal slurry. Ebullition or the rapid formation and rise of vapor bubbles from the heat transfer surface stimulates the movement of the syrup upward".<sup>[1]</sup> It should be noted here that as the masseccuite level keeps increasing, the zone of ebullition moves upwards and leaves the calandria, that is the heat transfer surface. Its role in pan circulation decreases fast.

The effect of masseccuite consistency on circulation and on the heat transfer in a vacuum pan has already been discussed before (see: Speed of evaporation). Construction of the traditional vacuum pan does not show much change since that of those built several decades ago. Had computer technology shown such a rate of development, we would still use computers filling rooms and having memories with ridiculous size.

It is well documented that bad pan circulation is – besides excessive supersaturation – a major cause of conglomerate formation. It is therefore very important to improve it as far as possible. Unfortunately, in a lot of pans feed syrup enters the pan from a pipe located above the calandria and directing syrup downwards in the downtake. This practice is anything but logical. As the difference in the specific weights between the feed syrup and masseccuite increases, syrup entering the pan will go upwards, that is it will definitely hinder the desired flow in the downtake. Feed syrup should enter the pan from several locations along a ring-pipe mounted under the calandria, close to the wall of the pan. This solution instead of hindering it can considerably improve circulation.



## Sensor location

Sensors (any type of sensor) should be located under the calandria. Locations close to the feed syrup inlet should be avoided in order to prevent acquisition of data from an un-representative diluted volume. Fast response of any type of sensor to the sudden opening of the feed valve is rather cause for concern, and not an advantage. Pan designs in most cases completely disregard instrumentation requirements. For more information on sensor location selection see <sup>[6]</sup> and <sup>[7]</sup>.

## Fluctuating vacuum

Rhapsodically changing vacuum, due to changes of the temperature generates similar changes in supersaturation (see Eq. 1.). Individual vacuum system, individual control of vacuum in a common system and individual scheduling the start of strikes in a pan farm are the tools which can be used. If individual vacuum control is possible, it can be put to good use in controlling supersaturation if it is monitored on-line. Use of high-concentration feed syrups helps in reducing the load on a vacuum system.

## Hold on water and other uses of water

Disregarding emergency cases holding a strike on water should be avoided by well designed automatic scheduling of strike starts.

Use of water in order to dissolve unwanted fines, a method used by the pan men as a natural way to get rid of any trace of poor control should be punished. It increases not only the amount of heating steam but the time of crystallization as well.

## Strike control

Besides massecuite circulation in the pan, strike control after seeding has been accomplished is also an ever-green problem. The two are actually closely related.

To put it simply: which strategy is better:

- control the strike so that the crystals are as close to each other as possible (“tight boiling” with high consistency), or
- strike control preferring to have a rather “loose boiling” (lower consistency)?

If crystal mass is the same, massecuite level in the first case is lower, resulting in higher crystal content (by volume). The difference between the two naturally disappears at the end of the strike. Both methods have advocates.

**Tight boiling.** Those who prefer higher massecuite consistency usually argue that due to the closeness of the crystals the concentration difference between a crystal surface and any point in the mother liquor is smaller; therefore the danger of excessive supersaturation is smaller as well. This is quite true. The smaller concentration gradient, however, is the result of the fact that crystals very close to each other are fighting for the small store of sugar in the mother liquor pockets among them, depleting it and due to increasing consistency make transportation of a fresh supply of sugar to them more and more difficult. If the stored (in the mother liquor) plus fresh supply is not sufficient to keep space with crystal growth, concentration of the mother liquor and therefore supersaturation begins to

drop. It will drop not only because of the drop of sugar content, but also because the fast drop of purity in the small volume among the crystals as well. At the same time, as already discussed, the rate of evaporation is decreasing due to increasing consistency. It is even more true for pan locations where there is no evaporation at all due to increasing hydrostatic head and boiling temperature. The end result: there is really no danger of excessive supersaturation, on the contrary, it will be decreased, but the speed of crystal growth will suffer even to the point of complete stop in a saturated mother liquor. We have experienced similar phenomena when due to high crystal content and poor feed supply crystal growth was completely stopped in the bottom of a pan (see Case 1 in Part 2).

**Loose boiling.** Strike control resulting in smaller consistency (loose strike) has the following advantages:

1. “With respect to crystal growth rate, a more fluid slurry delays the mother liquor purity drop. Also, a more fluid slurry enhances turbulence and a reduction of diffusion resistance”.<sup>[1]</sup> With a smaller drop of purity the speed of crystal growth will be reduced less.
2. Due to smaller massecuite consistency circulation in the pan is better and the danger of conglomerate formation is definitely smaller.
3. Mixing the fresh supply of feed syrup is easier, therefore homogeneity of the massecuite (identical parameters in a large volume) is better, therefore all data acquired on-line are more representative and reliable. This is true for calculated data like supersaturation, too; therefore its automatic control has a much better chance. Due to improved homogeneity and larger mother liquor to crystal volume ratio, good supply of sugar for the crystals can be maintained longer (a larger part of it is already there), which results in better crystal size distribution.
4. Due to better pan circulation heat transfer between the calandria and the massecuite is better (see: Speed of evaporation). The temperature difference  $\Delta T$  between the heating steam and the massecuite can be smaller to maintain the same rate of heat transfer.

The disadvantage of a loose massecuite is, however, that the hydrostatic head is larger, therefore under identical conditions boiling of the massecuite starts at a little bit higher pan level. Massecuite consistency plays a major role in these considerations. A brief example:

Let us assume that with equal mass of crystals the difference of pan levels and with them the difference in crystal contents by volume is 15 %, which is not too large. In this case the ratio of massecuite consistencies at different crystal contents is:

Crystal content (% by volume)	Ratio
23 (20)	1.18
34.5 (30)	1.41
46 (40)	1.81

With 46 % crystal content (“tight” massecuite) the consistency in the pan is by 81 % larger than it would be with identical crystal mass but 15 % higher massecuite level (resulting in 40 % crystal content). It can be seen that the way a strike is being controlled plays a very important role in massecuite consistency.

It is interesting to note that the appropriate hydrostatic pressures below levels 2.5 m and 2.875 m (15 % increase) are:

- Level 2.50 m and crystal content 46 %:  $P = 0.3660$  bar
- Level 2.875 m and crystal content 40 %:  $P = 0.4179$  bar

The increase in hydrostatic head:  $\Delta P = 0.0519$  bar which results in about 2.2°C increase in boiling temperature at the indicated levels

below the massecuite surface.

Another important point in strike control is sugar exhaustion of the mother liquor. It is quite common to find examples when towards the end of a strike the feed valve is closed and boiling is continued until for example, the current or power consumption of the stirrer motor signals the end of the strike. The rate of evaporation in most cases is rather small during this time, therefore due to the decreasing sugar content of the mother liquor supersaturation and with it the speed of crystallization slowly drops until the strike is ended. This will definitely increase the effective time of crystallization.

There is another strategy, too, which aims to have as short strike times as possible with still high final crystal content. It is achieved by maintaining high supersaturation (but always safely below the limit) all over the strike in order to have high speed of crystal growth, and leaves the exhaustion of the sugar content of the mother liquor to the charge receiver, where a type of cooling crystallization takes place. To be effective, the charge receiver will be busy for quite a long time. An example of this strategy is shown in Fig 6a.

As can be seen selection of a strike control strategy is a matter of compromise. It depends very much on local circumstances and preferences. One thing, however, is common to both: advanced control needs on-line data on parameters which are really important.

### Crystal size distribution and crystal content

**Size distribution.** According to McCabe's  $\Delta L$  law the total linear growth of all crystals in the massecuite over the same time interval and along similar axes is the same, if they have similar and invariant geometric shapes and supersaturation is the same for all crystals in all locations in the massecuite. The linear growth rate is independent of crystal size.<sup>[14]</sup>

It follows from this law that in order to have narrow size distribution repeatedly from strike to strike the crystals in the seeding slurry or footing magma when full seeding is practiced

- should have the same mean crystal size and narrow size distribution from strike to strike, and
- supersaturation should be the same on every side of every crystal.

When traditional shock seeding is practiced the situation is more complex. In this case nucleation takes some time (for example 20 minutes) depending on the value of supersaturation (see Fig 5. and Fig 6a.). During this time not only new nuclei are formed, but the crystals used in the slurry to initiate nucleation begin to grow in size fast due to high supersaturation. It is logical to assume that the small new nuclei formed towards the end of nucleation and the crystals coming with the slurry and growing fast during this time will have considerable differences in size when the period of nucleation ends. From this point on, according to McCabe's law and if the conditions are met their further growth will be identical; therefore the size differences will show up in the end product as well.

Crystal size distribution is an important parameter of the end product. It can be artificially improved by screening the product to meet individual customer requirements. This, however, will not solve the problems with centrifuging, conglomerate formation and the loss of already crystallized, but recycled crystal sugar (see Fig 4). Consequences of poor crystallization control practice can not be avoided later on.

**Crystal content.** Massecuites, when the charge is dropped may

have the same mean crystal size and size distribution, but different final crystal content by volume. This means that the linear growth of the crystals was the same, but the number of crystals in a unit volume of the massecuite was different.

If shock seeding was practiced this means that:

- the number of crystals produced during nucleation was different, or
- there was further (unwanted) nucleation later on, or
- some of the crystals were lost later on due to the use of water feed and / or increased massecuite temperature (supersaturation drops below 1,0).

If full seeding was practiced differences in crystal content despite identical mean crystal sizes are due to:

- changes in the number of crystals in the seed slurry or magma, or
- loss of some of the crystals later on (use of water, increased temperature).

Finally, differences in crystal contents may result from both major causes:

- differences in mean crystal sizes of the product due to differences in crystal growth and
- differences in the number of crystals in the massecuite.

It is important to note here that besides good seeding practice supersaturation and strike control play very important roles in determining final crystal content and the real rate of production.

### Summary

1. Supersaturation is the most important parameter of sugar crystallization. It has a major role in determining product quality and the cost of production as well. Its control over the complete process of crystallization is vital if good product quality and cost efficiency are at stake.
2. Massecuite circulation in a vacuum pan plays a very important role in crystallization. The rate of heat transfer, crystal size distribution, conglomerate formation and crystal growth all depend on it. It is closely connected to massecuite consistency and consequently to the way a strike is being controlled.
3. Constant product quality and cost of production requires a high degree of repeatability of strike control. Traditional manual methods are unable to meet this requirement. Advanced methods of control should be based on the on-line measurement and calculation of parameters which are really important; most of all: supersaturation and crystal content.
4. Crystallization control is a case of optimization when the task is to find the ideal compromise among a multitude of often conflicting parameters and details. It is not easy to find the right solution. Traditional methods (on-line and laboratory) are unable to provide real help to diagnose the ills and to solve the problem repeatedly and reliably from strike to strike.

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