

On-line monitoring of supersaturation in sugar crystallisation

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Summary

This paper presents the results of work aimed at a better understanding of supersaturation and its reliable monitoring on-line in batch vacuum pans.

A mathematical model of supersaturation was developed based on the four independent variables which completely specify it, namely: syrup/mother liquor concentration, temperature, purity and the value of Wiklund-Vavrincz saturation function. Calculation results are presented in graphs, which are well suited to tracking both the syrup concentration and the sugar crystallisation phases of a strike.

The shortcomings of the popular sensors presently used for sugar boiling suggest the need for an alternative method. Monitoring of supersaturation is proposed using an on-line refractometer and a mathematical model. The results can be used directly for boiling control.

Practical experience gained during tests using refractometers from K-PATENTS in Hungary and Finland is reported.

Introduction

Sugar crystallisation is a delicate process, which has to be controlled either by a skilled man, or by an automatic control system. Crystallisation can only take place in a solution which is supersaturated, that is it contains more sugar than is required for saturation. This can be achieved either by evaporating some of the solvent, or by cooling the solution and taking advantage of the fact that the amount of dissolved solids at saturation decreases when the temperature decreases. This distinguishes *evaporative* and *cooling* crystallisation.

In addition to the supersaturation of the solution, the presence of crystal seeds is also required. They are most often introduced into the pan at the right moment in the form of a small amount



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of crystal slurry containing fine crystals, but can also be generated in the pan with so called shock seeding. Unfortunately, crystals can be generated unintentionally after seeding if the supersaturation exceeds a safe limit, resulting in the appearance of unwanted conglomerates. Sugar which has crystallised has to be replenished by feeding syrup into the pan as required by the actual crystal growth, in order to maintain crystallisation.

It follows that both supersaturation and crystal content play decisive roles in sugar crystallisation. Unfortunately both are difficult to measure and therefore to control.

It is the pan man's responsibility to make the decision on the right moment of seeding. He also operates the syrup feed valve to replenish the sugar deposited on the crystals. The pan man uses his eyes and fingers to test the viscosity of the syrup sample from the pan. He knows from practice that viscosity has a strong correlation to solids content and thus to supersaturation. Again he uses his eyes to judge the crystal content in the massecuite, to decide the rate of syrup feed to keep the crystals growing.

There are no two identical artists, and no two pan men control the crystallisation in exactly the same way, day in, day out. This is a major driving force behind the automation of sugar crystallisation.

Measurement of supersaturation

Knowledge of supersaturation is very important over the full duration of crystallisation, and there are many publications dealing with the subject.

The first phase of a batch strike, syrup concentration, is characterised by a fast increase in supersaturation. Accurate information in real-time is therefore very important for the exact timing of seeding, if a desired super-saturation is to be reached at the seeding point. It is general practice to decrease it for a while after seeding has been completed and to increase it again later. There are various recommendations on the magnitude of these changes in both directions, but to implement them requires reliable feedback. However, one can rarely find claims that supersaturation is closely monitored on-line and used to control crystallisation in real-time during a full strike.

There is no direct method to measure supersaturation. The different types of sensors in use provide primary data (for example conductivity, boiling point elevation or massecuite density) which are in some way correlated to supersaturation, but depend on several physical parameters of the massecuite and / or the mother liquor. Usually at least some of these parameters are not measured separately. Reliable and accurate equations describing their effect on the primary data are often missing. Charts and data tables on supersaturation are difficult to use and are unsuitable for programming in a control system.

Calculations of supersaturation quite often do not take into account all the variables which determine its actual value and in some cases they are based on assumptions which are clearly unacceptable.

These are the major reasons which may be responsible for the limited real-time use of supersaturation in controlling crystallisation.

The independent variables influencing supersaturation

In order to be able to monitor supersaturation all the variables which have any influence on it have to be taken into account by using appropriate equations, which give a quantitative description of supersaturation (i.e. a

mathematical model). By definition, supersaturation (SS) is a dimensionless number expressed as the ratio of sugar in solution at actual syrup parameters divided by the sugar content at saturation with the same syrup parameters, that is:

$$SS = \frac{\text{actual sugar in solution}}{\text{sugar in solution at saturation}} \quad (1)$$

In the following discussion the term mother liquor and the index (ml) will be used for its different parameters even if there are no crystals in the solution as yet.

The four mother liquor parameters which determine the numerator and denominator of Equation 1 are: mother liquor concentration (C_{ml}), temperature (T_{ml}), purity (Q_{ml}) and the actual value of the Wicklund-Vavrinecz saturation function (F_{sat}). These will be discussed in more detail.

(i) Concentration or solids content of the mother liquor (C_{ml})

More solids (sugar + non-sugars) mean more sugar in solution, i.e. a higher SS.

(ii) Temperature (T_{ml})

Temperature has a profound effect on the solubility of sugar in water. The amount of sugar in solution at saturation (denominator of Equation 1) increases in a highly non-linear way when

temperature is increased.

(iii) Mother liquor purity (Q_{ml})

By definition, purity is the ratio of sugar to all the solids in solution.

In order to be able to determine the amount of sugar in solution a knowledge of purity is required as well as concentration. Due to the deposition of sugar on the crystals and the continuous accumulation of non-sugars, mother liquor purity begins to drop below that of the feed syrup as crystallisation proceeds, after seeding. Despite this obvious change in purity, it is quite often neglected as a contributing factor in attempts to track SS.

(iv) Saturation function (F_{sat})

Syrup quality and non-sugars to water ratio have an effect on sugar solubility as given by the value of the Wicklund - Vavrinecz saturation function (F_{sat}). It depends on the composition and the amount of non-sugars in the solution. These are reflected by the parameters m , b and c and the non-sugars to water ratio in the saturation function¹:

$$F_{sat} = m \cdot NS/W + b + (1-b) \cdot \exp(c \cdot NS/W) \quad (2)$$

where NS/W is the non-sugar to water ratio of the mother liquor.

The value of F_{sat} can change due to

changes in the type of beet processed, the fertiliser used or the deterioration caused by freezing or storage of the beet. Therefore, mother liquors having the same concentration, temperature and purity, but different saturation function values, can hold different amounts of sugar in solution, resulting in different SS data. The syrup parameters m , b and c depend on local factors and have to be determined locally.

SS can be expressed by the general equation:

$$SS = f(C_{ml}, T_{ml}, Q_{ml}, F_{sat}) \quad (3)$$

Based on the requirement of not neglecting anything which influences SS, a set of equations (a mathematical model) has been developed using these independent variables. This is in contrast to similar attempts to calculate SS, which generally neglect one or more of the variables or make assumptions which are at least arguable.

Presentation of supersaturation data

In the following discussion results based on the use of the mathematical model will be presented in the form of 2- and 3-dimensional graphs. Tabulated data will be used to show typical sensitivities (partial derivatives) of SS on the different independent variables.

Due to its multivariate nature,

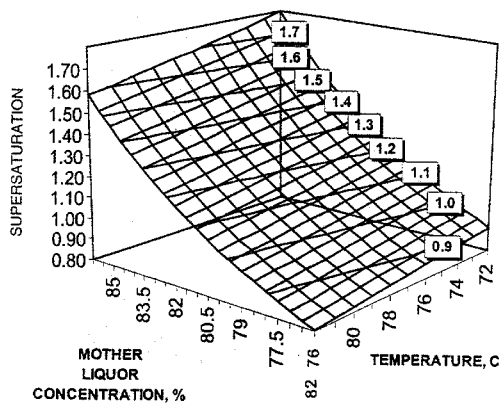


Fig. 1. Supersaturation vs. mother liquor concentration and temperature (purity = 94%, $m = 0.145$, $b = 0.811$ and $c = -1.8$)

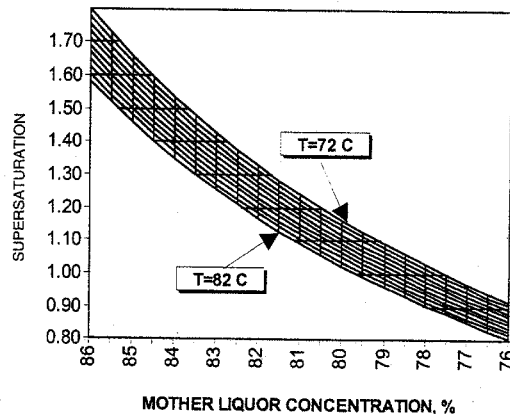


Fig. 2. Supersaturation vs. mother liquor concentration and temperature (purity = 94%, $m = 0.145$, $b = 0.811$ and $c = -1.8$)

displaying the results in graphical form is difficult. It will be useful to separate the process of sugar boiling in a batch vacuum pan into two main phases:

- (1) syrup concentration, when unsaturated syrup fed into the pan becomes supersaturated - due to evaporation - and ready for crystallisation;
- (2) crystallisation, when controlled growth of the crystals introduced during seeding of the pan takes place.

The availability of a mathematical model is very important for at least two reasons.

- (a) It is very useful in gaining a better understanding of how SS is controlled by the different mother liquor parameters, and
- (b) If the required data are available, it can be used to calculate SS in real time.

The two phases will be examined separately and results will be presented in graphical form to track the history of a strike in an illustrative way.

Syrup concentration phase

During concentration the mother liquor purity Q_{ml} is constant and equals the feed syrup purity Q_k , usually known from laboratory data. In the same way the m,

b and c parameters of the saturation function are also known, or if not, typical data can be used.

For the figures which follow, purity (Q_{ml}) was 94 %, while m, b and c were selected to equal those determined during October, 1994 in the Mezőhegyes Sugar Mill, Hungary (0.145, 0.811 and -1.8, respectively).

Using these data and the set of equations developed, SS can be calculated versus mother liquor

concentration and temperature. Results form a smooth inclined surface as shown in Fig. 1. The calculated data and the figure are valid as long as the assumptions of constant mother liquor purity and saturation function hold, that is in the concentration phase.

Appropriate projections of the surface shown can be used one by one to study the effect of the two independent variables (concentration and temperature) on SS during this phase of the process.

Table I. Sensitivity of supersaturation on mother liquor concentration
Parameters: m = 0.145, b = 0.811, c = -1.8

	Temperature °C	Concentration %			
		77	80	83	86
Purity 94%	72	0.0547	0.0723	0.0998	0.1461
	74	0.0533	0.0705	0.0973	0.1425
	76	0.0520	0.0687	0.0949	0.1389
	78	0.0507	0.0670	0.0925	0.1354
Purity 90%	72	0.0526	0.0691	0.0944	0.1356
	74	0.0513	0.0674	0.0920	0.1322
	76	0.0500	0.0657	0.0897	0.1289
	78	0.0487	0.0640	0.0875	0.1256

Table II. Sensitivity of supersaturation on mother liquor temperature
Parameters: purity = 94%, m = 0.145, b = 0.811, c = 1.8

Concentration %	Temperature, °C			
	72	75	78	81
77	0.0121	0.0118	0.0115	0.0112
81	0.0155	0.0151	0.0148	0.0144
85	0.0206	0.0202	0.0197	0.0192

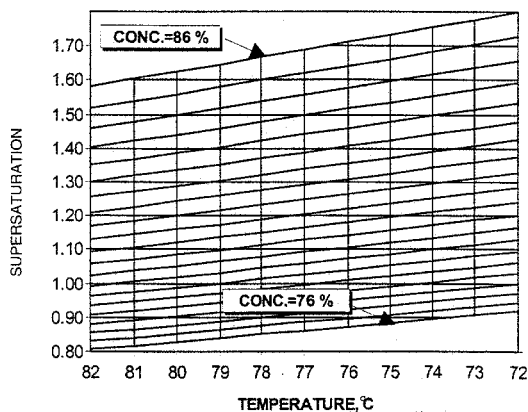


Fig. 3. Supersaturation vs. mother liquor temperature and concentration (purity = 94%, m = 0.145, b = 0.811 and c = -1.8)

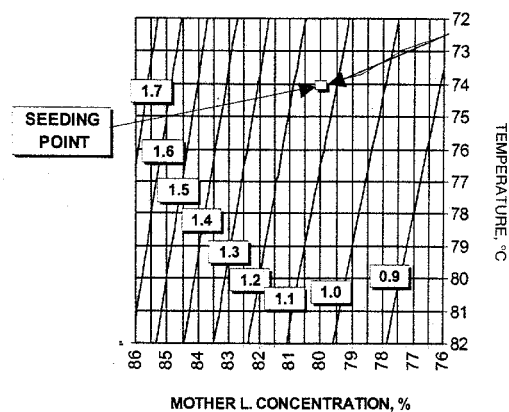


Fig. 4. Supersaturation vs. mother liquor concentration and temperature (purity = 94%, m = 0.145, b = 0.811 and c = -1.8)

Table III. Sensitivity of supersaturation on mother liquor purity

Parameters: Temperature = 74°C, m = 0.145, b = 0.811, c = 1.8

Concentration %	Purity %				Change %
	96	92	86	82	
77	0.006	0.008	0.010	0.011	77.6
80	0.007	0.010	0.013	0.014	97.7
83	0.009	0.014	0.018	0.019	119.1
86	0.012	0.020	0.026	0.027	133.1
Change %	85.1	142.7	151.9	142.9	

Note: Calculated change data are accurate, while tabulated ones contain some rounding error

(a) SS vs. mother liq. concentration (C_m)

Fig. 2. can be generated as a projection of the surface of Fig. 1. along an axis parallel to that of temperature. It clearly shows that the sensitivity (i.e. the partial derivative, or the slope of the curve) of SS on concentration is not constant, but increases with increasing concentration. The series of near parallel curves are for different mother liquor temperatures.

Table I shows numerical data on the sensitivity of SS which give the change in SS due to a unit change of the independent variable (concentration, temperature or purity). It can be seen that the sensitivity on mother liquor concentration increases by over 160% at different (but constant) temperatures in the 77 to 86% concentration range. Reducing the purity of the mother liquor

from 94% to 90% results in a 5 - 6% drop in the sensitivities, but the change over the 77 to 86% concentration range is only slightly different (158%).

(b) SS vs. mother liquor temperature (T_m)

Fig. 3. is a projection of the surface shown in Fig. 1 along an axis parallel to that of concentration. The lines for different mother liquor concentrations show only a very slight drop in their slopes as temperature decreases (the second derivative of SS on temperature is close to zero). Table II shows there is a considerable change (71%) in the sensitivity of SS on temperature at different (but constant) concentrations in the 77 to 85% range.

(c) The combined effect of C_m and T_m

Finally, Fig. 4. is the top view of the

surface and a possible trajectory of temperature and concentration leading to the seeding point is shown (the arrow in the upper right corner). During the concentration phase this trajectory enters at 72.5°C temperature, 76% mother liquor concentration and SS = 0.92. It gradually moves on to the seeding point at 74°C and 80.0%, where SS = 1.135, and seeding can take place.

During syrup concentration the mother liquor purity is constant and equal to the feed syrup purity, known from laboratory data; the same applies to the saturation function parameters m, b and c. Therefore, if measured data on mother liquor temperature and concentration are available, a figure of the type shown in Fig. 4. is well suited to displaying the exact history of a strike from the start of syrup feed into the pan up to the seeding point. It should be noted, however, that for different feed syrup purities and saturation function parameters the figures would be different.

It is to be noted that, when approaching the seeding point due to increasing concentration, the sensitivity of SS on concentration and temperature increase considerably, which makes the accurate timing of seeding important.

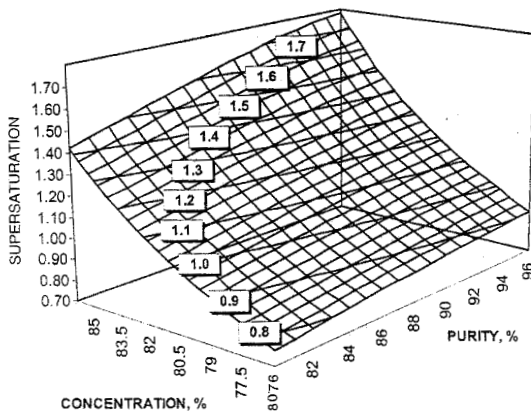


Fig. 5. Supersaturation vs. mother liquor concentration and purity (temperature = 74°C, m = 0.145, b = 0.811 and c = -1.8)

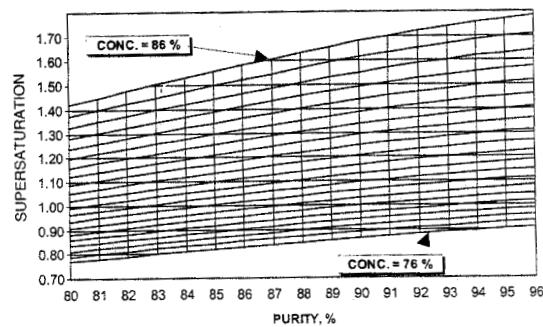


Fig. 6. Supersaturation vs. mother liquor concentration and purity (temperature = 74°C, m = 0.145, b = 0.811 and c = -1.8)

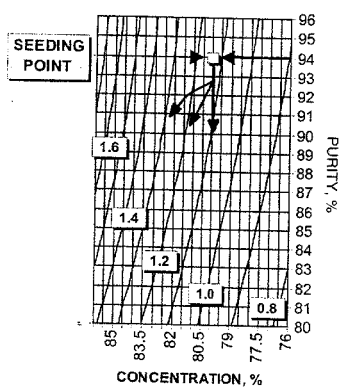


Fig. 7. Supersaturation vs. mother liquor concentration and purity temperature ($T = 74^{\circ}\text{C}$, $m = 0.145$, $b = 0.811$ and $c = -1.8$)

Crystallisation phase

Actual crystallisation should begin only after having reached the seeding point, characterised by the desired SS set-point, as planned. After seeding with slurry has been completed, the crystals will begin their growth and the crystallised sugar has to be replenished by a controlled input of feed syrup. Non-sugars, however, will still be in solution, and with the continuing feed more and more non-sugars will accumulate, resulting in a gradual drop of mother liquor purity (Q_{ml}). Therefore the assumption of constant purity is not valid and in this phase Figures 1 - 4 have only limited use. To show how SS varies in this phase, concentration C_{ml} and purity Q_{ml} are selected as independent variables, while temperature T_{ml} is constant in the figures which follow. The saturation function parameters do not change.

The calculated results are shown as a surface in Fig. 5. Like Fig. 1 this reflects the sharp dependence of SS on mother liquor concentration over a wide range of purity.

Table IV. Sensitivities of supersaturation on the independent variables and their relative values, near the seeding point

Sensitivities	$\partial SS/\partial C_{ml} = 0.0705/\%$	$\partial SS/\partial T_{ml} = 0.0143/^{\circ}\text{C}$	$\partial SS/\partial Q_{ml} = 0.009/\%$
Relative value	100%	20.3%	12.8%

(d) SS vs. mother liquor purity (Q_{ml})

In Fig. 6 the effect of mother liquor purity on SS at different concentrations is shown.

It can be seen that the sensitivity of SS on purity (while concentration is kept constant) increases considerably as purity decreases (the second derivative is quite large). Similarly, sensitivity on purity shows a marked increase for higher concentrations.

(e) The combined effect of C_{ml} and Q_{ml}

Finally, Fig. 7 shows the top view of the surface in Fig. 5. Assuming that boiling of the strike was accomplished at a constant temperature of 74°C , the history of the complete strike can be displayed by the trajectory shown in the figure (only one of many possible ones). In practice seeding is generally followed by a drop in mother liquor concentration and SS, implemented by a good drink of feed syrup. Crystal content and the change in mother liquor purity are small at this stage. After some time crystallisation can continue, depending on the control policy implemented, as shown by the different arrows.

One policy would be to continue sugar boiling along a line of constant mother liquor concentration. This, however, can lead to SS dropping below 1.0, resulting in the dissolving of sugar already crystallised. On the other hand boiling along a curve of increasing or even constant SS needs increasing mother liquor concentration, if temperature remains constant.

(f) SS vs. saturation function parameters

The last independent variable of Equation 1 is the saturation function. It is determined by the non-sugars to water ratio and the m, b and c parameters

(Eq. 2). They reflect feed syrup quality and can be determined in the laboratory. Unfortunately, partly due to the time and effort required for a laboratory analysis and partly due to neglect, they are only rarely measured and used in practice, even in mills and refineries boasting of advanced control of crystallisation. The result is a general lack of data, which makes the evaluation difficult.

If there are no data on these parameters, setting $m = c = 0$ (with any value of b) will result in a constant saturation function value $F_{sat} = 1.0$. In this case the SS calculated by the mathematical model will very closely approach that calculated using Grut's sugar solubility data.

In some typical factory data recorded at the Mezöhegyes Sugar Mill in Hungary there were only two sets of saturation function parameters available:

	m	b	c
October, 1994	0.145	0.811	-1.8
November, 1994	0.191	0.745	-1.8

Using the same mother liquor parameters as before ($C_{ml} = 80\%$, $T_{ml} = 74^{\circ}\text{C}$ and $Q_{ml} = 94\%$) we can calculate:

October, 1994,	SS = 1.135
November, 1994,	SS = 1.149

This means that mother liquors with identical concentration, temperature and purity data had a difference in their calculated SS amounting to $= 0.014$, solely due to their m, b and c parameters. Looking at the sensitivity data already determined, it can be said that this change is approximately equal to a 1°C change in mother liquor temperature.

This difference may be regarded as relatively small, but it is not known how large it could be because of the lack of more factory data. It can be said, however, that a change of at least this size may occur.

(g) Typical sensitivities in the vicinity of the seeding point

It is instructive to compare the effects of the mother liquor parameters in the vicinity of the critical seeding point. Let

the seeding point be characterised by:

$$C_{mi} = 80.0\%$$

$$T_{mi} = 74.0^{\circ}\text{C}$$

$$Q_{mi} = 94.0\%$$

$$\text{Supersaturation} = 1.135$$

Assume m, b, and c are as above (Mezőhegyes Sugar Mill, October, 1994.)

Table IV shows the sensitivities of SS on the independent variables. Besides these sensitivity data, an absolute change of SS near the seeding point of SS = 0.014, solely due to changes in the saturation function parameters over a month was noted above.

This comparison clearly shows that mother liquor concentration has by far the strongest effect, and therefore is the most important parameter to measure during the full course of a strike.

Local variations in a vacuum pan

According to McCabe's rule, all crystals experience the same rate of growth in a massecuite characterised by uniform SS. It follows that, in order to have crystals of the same size in the end product, it would be ideal to have the same supersaturation in the whole of the vacuum pan. How far away from reality this is will be determined by the local differences in the four mother liquor parameters.

During syrup concentration, due to the lack of crystals and because of good circulation, mother liquor purity, concentration and the parameters of the saturation function are the same throughout. This, however, is not true for temperature.

In Fig. 8, three trends in temperature, based on measured data in the Porkkala Refinery of Finnsugar (Finland), are shown. Two of these lines run very close to each other and show data from a dedicated temperature sensor and from the temperature sensor of a K-PATENTS PR-01-S type on-line refractometer. Both sensors were located in the same horizontal plane in the bottom of the pan. The third line was plotted using data from another dedicated sensor located above the

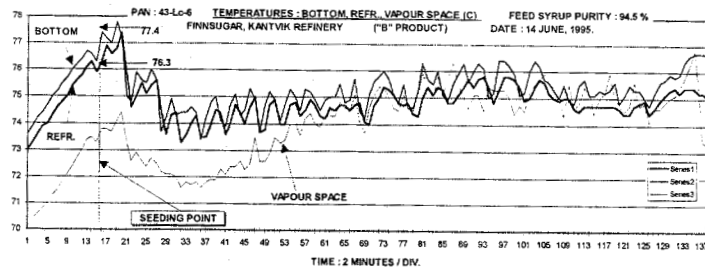


Figure 8

massecuite surface in the vapour space. It can be seen that in the concentration phase (despite the relatively low massecuite level) there was about 2.5°C difference between the pan bottom and vapour space temperatures, the latter being lower. After seeding of the pan was completed, the temperature difference (despite the high massecuite level) gradually decreased to under 1°C.

The fast fluctuations in all temperature data were the direct results of feed syrup control as it was practised in the refinery: the feed control valve was operated almost like a simple on-off valve, resulting in large feed input volume fluctuations. These certainly had an effect on massecuite temperature, density, stirrer motor current (not

shown), mother liquor concentration and supersaturation. This latter can be quite large in the vicinity of the feed syrup inlet point within the pan. On-off feed syrup control, advocated by one leading process control company, will certainly induce large local fluctuations, which should be reduced rather than induced.

Fig. 9. shows a typical temperature and supersaturation profile² along the vertical axis of a pan. Temperature is lowest and SS is highest on the surface of the massecuite, while the opposite is true in the upper plane of the calandria. Too low SS in this latter part of the pan will not only reduce the rate of crystal growth, but can result in dissolving sugar, if it drops occasionally below unity.

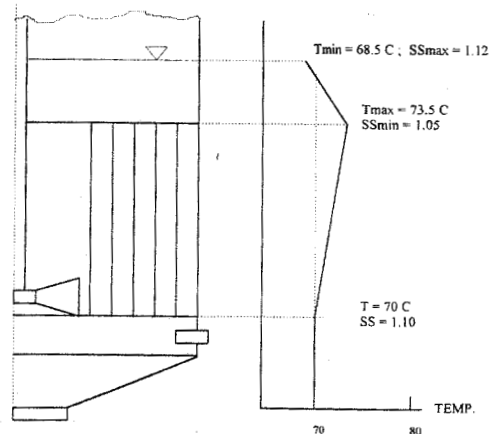


Fig. 9. Typical temperature profile and supersaturation during the first phase of white sugar boiling²

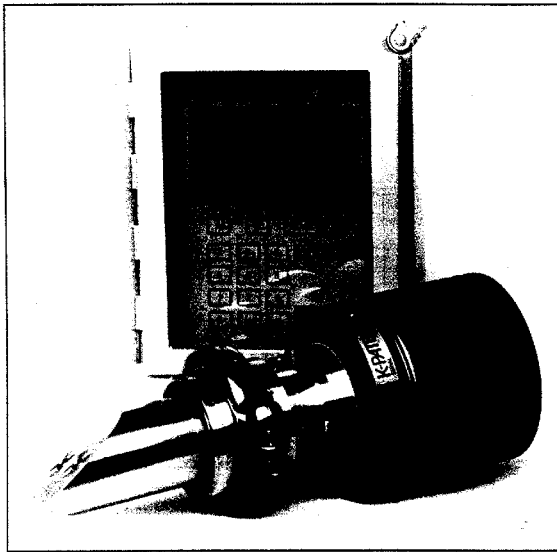


Fig. 10. The K-Patents process refractometer

Based on the considerations above, uniformity of SS in a vacuum pan depends mostly on the efficiency of circulation and the way feed input is being accomplished and controlled.

Validation of the mathematical model

It is natural that the SS data calculated by the mathematical model have to be tested to prove their correctness. The easiest way is to compare these data with those obtained by the use of charts prepared by different investigators or companies, or by using sugar solubility data directly, like the ones collected by Charles, Grut or Vavrincez (the charts are also based on one or other of these data tables). Charts are usually prepared for different (constant) syrup purities and show lines of SS versus syrup concentration and temperature.

Before giving comparative data, however, two notes are in order.

The mathematical model is based on the solubility data given by Vavrincez (and tentatively approved by ICUMSA'). This can be changed, but in our opinion it is the best to use.

The charts and the sugar solubility

tables do not take into account the saturation function, that is they treat supersaturation or solubility as a function of the other three variables only. Tables V and VI give SS data determined at 75°C for purities 90% and 94%, respectively.

Models 1, 2 and 3 are the mathematical model as follows:

	m	b	c
Model 1:	0.145	0.811	- 1.8
Model 2:	0.191	0.745	- 1.8
Model 3:	0	0.800	0

Grut refers to SS calculated using Grut's data of sugar solubility [1].

Charles refers to SS determined by using charts based on Charles' data of sugar solubility. Due to the limitations of the

charts, some of these data are the results of interpolation.

It will be seen that the largest difference between Model 1 and Model 2 is 0.03. Model 3 uses a constant saturation function value equal to 1.0 (see Equation 2). Model 3 and Grut data are almost always identical.

The differences between Model 1/ Model 2 and Grut/Charles are due to the fact that Grut's and Charles' solubility data do not take into account the Wicklund-Vavrincez saturation function.

Use of the saturation function with valid, or even approximate m, b and c parameters, typically results in a saturation function value somewhat less than 1.0 (high purity syrups) reflecting the reduced sugar solubility in impure solutions. This results in larger SS data than other methods.

Based on the calculated data and the observations above the mathematical model of SS is considered to be validated.

On-line monitoring of supersaturation

Acquisition of input data

It has been shown that the mathematical model provides reliable calculated data on SS, if data on the four independent variables are available. The sensors used most often for monitoring SS are conductivity measurement, boiling point elevation and massecuite density. Detailed examination reveals, however, that all these have limitations and are unable to provide reliable information on mother liquor concentration during the full course of a strike. This examination will be the subject of a separate article to be published in a later issue of this journal.

Table V. Supersaturation data. Parameters: temperature = 75°C, purity = 90%

Concentration	77%	80%	84%
Model 1	0.90	1.08	1.42
Model 2	0.92	1.10	1.45
Model 3	0.87	1.04	1.37
Grut	0.87	1.04	1.37
Charles	0.89	1.05	1.14

Table VI. Supersaturation data. Parameters: temperature = 75°C, Purity = 94%

Concentration	77%	80%	84%
Model 1	0.94	1.12	1.48
Model 2	0.95	1.14	1.50
Model 3	0.91	1.09	1.43
Grut	0.91	1.09	1.43
Charles	0.91	1.09	1.47

The refractometer, invented by Ernst Abbe over a century ago, is used in laboratories everywhere, for accurate measurement of solids concentration in solutions. Its on-line industrial version can be applied to crystallisation control in the sugar and other industries. This, however, requires a refractometer design, the accuracy of which is unaffected by the presence of vapour bubbles and crystals. The K-Patents PR-01-S type refractometer (see Fig. 10) meets these requirements.

Mother liquor concentration alone is not a direct measure of SS. Despite their favourable ratings in several tests it is understandable that the use of on-line refractometers in crystallisation control has been rather limited. In one test⁶ the K-Patents refractometer was shown to give a remarkably good indication of concentration but it was concluded that its cost would preclude its use unless it could lead to better SS control. In order to monitor this on-line, however, real-time information on all the independent variables and the regular calculation of SS is required. Besides mother liquor concentration, the PR-01-S measures temperature, so it is capable of providing two of the variables required (if preferred, a dedicated temperature sensor can be used).

The second important parameter used to control sugar boiling is crystal content, or rather, a variable which is correlated to it, due to the difficulties of direct measurement. This information plus laboratory data on feed syrup purity can be used to calculate mother liquor purity during a complete strike and entered into the mathematical model. Finally, saturation function parameters m , b and c should be available as laboratory data. If not, typical values can be used⁷.

Fig. 11 shows the basic concept of the on-line monitoring of supersaturation.

Practical experience

Despite its relative novelty, every opportunity to gain practical experience in the application of supersaturation

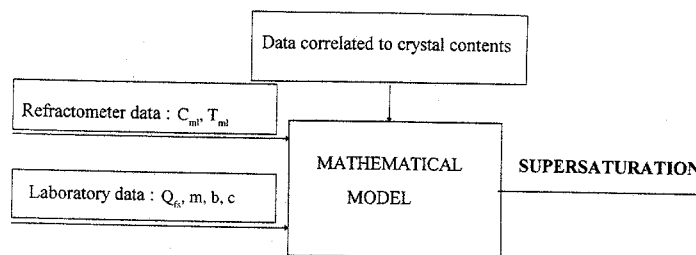


Fig. 11. On line monitoring of supersaturation

monitoring has been used. It has been developed to the point where, by running a real-time PC program package or a notebook PC connected through the serial communication line to the refractometer, SS data can be calculated on site within two minutes.

The first tests of the calculation were carried out in two mills in Hungary and a refinery in Finland.

Kaba Sugar, Hungary

With close to 8000 tons/day beet processing capacity, Kaba Sugar (a joint venture with Tate & Lyle) is the largest sugar mill in Hungary. The sophisticated ABB MOD300 type Distributed Control System (DCS) controls the operation of the sugar house with its 14 vacuum pans, implemented by PROFICON. The

instrumentation of a pan includes consistency and SS transmitters (boiling point elevation measurement) manufactured by Ziegler, USA, but there are experiments with other types (nuclear and radio frequency probes, and refractometer). There were problems with the supersaturation transmitters, which are no longer used.

Processing data collected by the DCS on one strike (mother liquor concentration from the refractometer and temperature from a dedicated transmitter) soon revealed that they had fairly high SS in the pan in the early stage of crystallisation (SS=1.37).

Mezőhegyes Sugar Mill, Hungary

In contrast, this is a mill with less than half the capacity (3300 tons of beet/day)

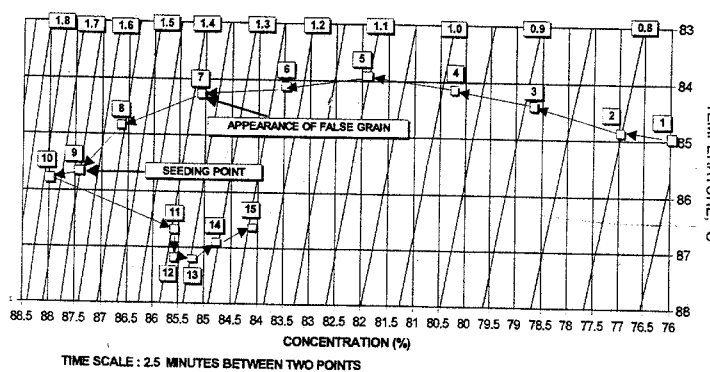


Fig. 12. Supersaturation vs. mother liquor concentration and temperature - Mezőhegyes
 $Q = 91\%$, $m = 0.145$, $b = 0.811$, $c = -1.8$ (October 1995)

and with no automatic control of sugar boiling. Besides the pan manometer, the only instruments are the conductivity transmitter, used by the pan man to determine the seeding point, the pan microscope and a recently acquired refractometer.

By reading concentration and temperature data using the refractometer display during the concentration phase of a strike in October 1995, the trajectory of the SS was plotted manually on the spot on a pre-calculated form (using appropriate laboratory data) as shown in Fig. 12.

Readings were taken at 2.5 minute intervals until some time after seeding has been completed. It was interesting to observe by the pan microscope the appearance of false grain at point No. 7 (which had to be washed off later), where SS was already close to 1.4. The pan man still continued waiting for the conductivity signal to reach the set-point for seeding, which finally took place in point No. 9 at the astonishingly high value of $SS = 1.65$. After further increase it was brought back to the almost normal 1.2 - 1.3 range by feeding in more syrup within about 10 minutes.

It could be argued that the conductivity set-point for seeding was obviously wrong in this case. However, due to the multivariable nature of conductivity it is not easy to determine the correct conductivity set-point, when syrup purity, the composition of non-sugars and/or temperature may change.

After this experience the pan men were instructed to use the pre-printed forms and refractometer concentration and temperature readings to track SS during syrup concentration and do the seeding at $SS = 1.15$. This procedure, which still leaves room for human error and cannot be accepted as a substitute to good automation practice, soon resulted in better crystal size distribution (less small crystals) and yield, reduced use of water and an improved product colour.

In the last two days of the campaign refractometer data were acquired and

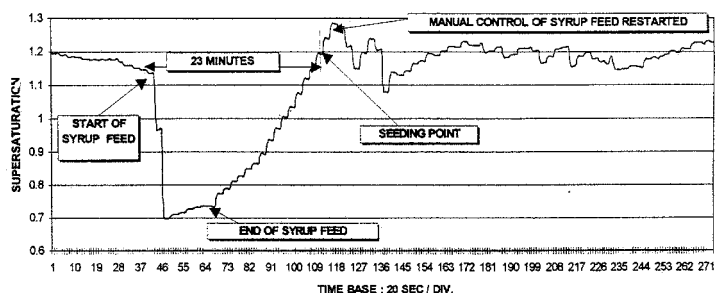


Fig. 13. Supersaturation vs. time ($Q = 92.6\%$) - Mezöhegyes sugar mill, Hungary, November 1995

processed by a PLC+PC combination and supersaturation data were treated on-line on the PC monitor (see Fig. 13).

The high supersaturation at the beginning was the result of a dense syrup film left on the refractometer prism in the empty pan. It was washed off when feeding of the pan started. As can be seen it is not an easy task to control SS manually in a pan having tens of tons of massecuite and there is still room for improvement. However, if real feedback is available, even manual control can be considerably improved, as the comparison of Figs. 12 and 13 testifies.

Finnsugar Porkkala Sugar Refinery

During June 14 - 15, 1995, two complete strikes (A: purity 96%, and B: purity 94.5 %) were monitored in the same pan (capacity 29 tons). Concentration and temperature data from a brand new and calibrated K-PATENTS refractometer, mounted in the bottom of the pan, were acquired on-line at regular intervals through the serial line connected to a

notebook PC. Other data (massecuite and vapour space temperatures, massecuite density etc.) were read at the same 2 minute intervals from the monitor of the DAMATIC DCS used to control boiling. Using these data SS was calculated and plotted later off-line (local m , b and c parameters of the saturation function were not available, so typical values $m = 0.178$, $b = 0.820$, $c = -2.1$ were used). The availability of data on massecuite density correlated to crystal content made the on-line calculation of mother liquor purity possible, as required by the mathematical model.

Fig. 14 shows concentration data as measured by the density probe and the refractometer during the B product strike. In Figs. 15 and 16, the calculated SS data are shown for the two complete strikes.

Calculated data had been validated by comparing them at two points (seeding point and a selected control point) to similar data calculated by using Grut's table of sugar solubility.

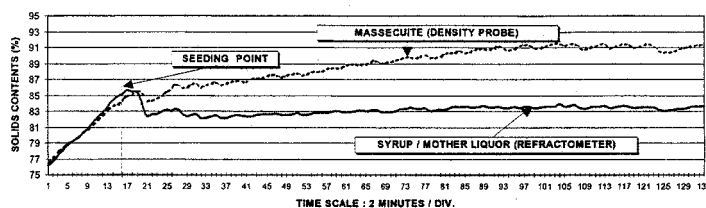


Fig. 14. Syrup/mother liquor and massecuite solids contents - Feed syrup purity: 94.5% ("B" product), Finnsugar Porkkala Refinery, June 14, 1995

	SS (seeding point)	SS (control point)
Strike 1		
Mathematical model :	1.58	1.30
Using Grut's data :	1.52	1.26
Strike 2		
Mathematical model :	1.39	1.24
Using Grut's data :	1.35	1.20

Data calculated by the mathematical model, as expected, are larger than those calculated by using Grut's table.

Conclusion

Bucholz and Bruhns¹⁰ sum up some of the problems experienced during the campaign in 1992 in Germany as follows:

"Cases of imprecise automatic control of supersaturation at seeding in the crystallisation process have led to difficulties in achieving the desired sugar quality and crystal size distribution. The resultant false grain hampers molasses separation in the centrifugals, reduces product quality and increases energy consumption, as well as lowering throughput and creating operational problems such as vibrations."

Complaints like this support our experience that the time-consuming determination of the seeding point and the crystallisation parameters by the traditional trial and error method, based on the measurement of primary data like density, conductivity, RF resistance and capacitance, viscosity/consistency etc., is unable to fill the need for reliable feedback on supersaturation.

A system controlling sugar boiling using data from these sensors will give consistent and reproducible results only as long as the variables (syrup purity, composition of non-sugars, temperature, crystal content and size) influencing the measured properties listed above do not change. How long this requirement is met will be observed only by the change in product quality (see the complaint above), which is, unfortunately, too late.

Therefore, instead of substitutes of questionable value, it is time to close the

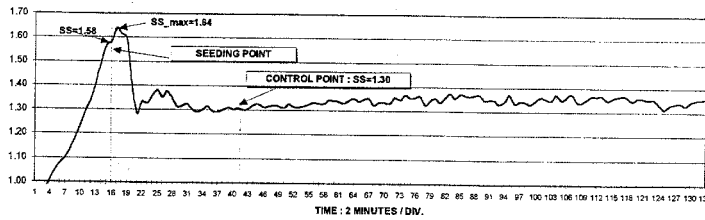


Fig. 15. Supersaturation - Feed syrup purity: 94.5% ("B" product), Finnsugar Kantvik Refinery, June 14, 1995

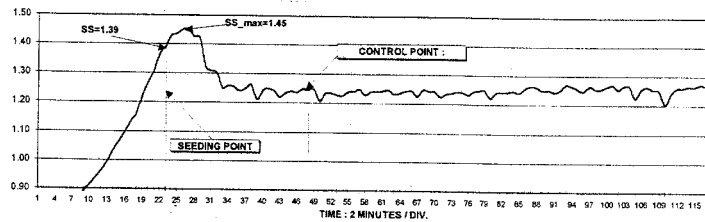


Fig. 16. Supersaturation - Feed syrup purity: 96.0% ("A" product), Finnsugar Kantvik Refinery, June 15, 1995

loop and provide feedback directly on supersaturation. The combination of the on-line industrial refractometer and the mathematical model can provide the missing link.

Monitoreo en línea de la supersaturación en la cristalización del azúcar (Resumen)

Este documento presenta los resultados de un trabajo dirigido a mejorar el conocimiento de la supersaturación y el monitoreo en línea en tachos al vacío discontinuos. Se desarrolló un modelo matemático de supersaturación basado en cuatro variables independientes con completa especificación, a saber: concentración de jarabe/licor madre, temperatura, pureza y la función de saturación. Los resultados de los cálculos se presentan en gráficas, lo cual sirve muy bien para determinar la concentración de jarabe y las fases de cristalización.

Las deficiencias de los sensores populares actualmente utilizados en el proceso de cocción sugieren la necesidad de un método alternativo y el monitoreo

de la supersaturación se propone utilizando refractómetros en línea y un modelo matemático. Se reporta que se ha ganado experiencia práctica durante las pruebas utilizando refractómetros K-Patents en Hungría y Finlandia.

Monitoring en ligne de la sursaturation dans la cristallisation de sucre (Résumé)

Cet article présente les résultats des investigations qui visent une meilleure compréhension de la sursaturation et le monitoring efficace en ligne de la sursaturation dans les appareils à cuire discontinus. On a développé un modèle mathématique de la sursaturation, basé sur les quatre variables indépendantes qui la déterminent, à savoir la concentration du sirop/égout-mère, la température, la pureté et la fonction de saturation. Les résultats des calculs se présentent en visualisation graphique, laquelle convient à suivre la concentration du sirop et les phases de cristallisation.

Les défauts des détecteurs que l'on utilise à présent dans la cuite semblent

indiquer la nécessité d'une autre méthode et on propose le monitoring de la sursaturation au moyen d'un réfractomètre en ligne et un modèle mathématique. On fait un compte-rendu de l'expérience pratique acquise avec les réfractomètres de K-Patents en Hongrie et en Finlande.

On-line-Überwachung von Übersättigung bei Zuckerkristallisation (Zusammenfassung)

Erläutert werden die Untersuchungsergebnisse mit dem Ziel, die Übersättigung besser zu verstehen und ihre zuverlässige On-line-Überwachung in periodischen Verdampfungskristallisatoren zu ermöglichen. Entwickelt wurde ein mathematisches Modell von Übersättigung auf der Basis der vier

unabhängigen Veränderlichen, die sie völlig bestimmen, nämlich: der Konzentration von Sirup/Mutterlösung, Temperatur, Reinheit und Sättigungsfunktion. Errechnete Ergebnisse werden in Diagrammen präsentiert, die sehr günstig die Sirupkonzentration und die Kristallisationsphasen eines Zyklus verfolgen. Die Nachteile von zur Zeit weitverbreiteten Sensoren, die man bei der Zuckerkristallisation verwendet, deuten auf den Bedarf an einer anderen Methode hin, und die Verwendung eines On-line-Refraktometers und eines mathematischen Modells zur Überwachung von Übersättigung wird vorgeschlagen. Es wird über praktische Erfahrungen aus Testen mit einem K-Patents-Refraktometer in Ungarn und Finnland berichtet.

References :

- 1 McGinnis: "Beet Sugar Technology", 3rd Edition. 1982.
- 2 Austmeyer & Frankenfeld: *Zuckerindustrie*, 1985, **110**, (2), 122 - 130.
- 3 Hempelmann & Austmeyer: *ibid.*, 1987, **112**, (8), 695 - 698.
- 4 Radford & Cox: *ibid.*, 1986, **111**, (10), 922 - 932.
- 5 Rozsa: *Cukoripar*, 1995, **48**, (4), (Hungarian).
- 6 Peacock: *I.S.J.*, 1995, **97**, 571 - 577.
- 7 Hugot: "Handbook of Cane Sugar Engineering".
- 8 Miller & Skippen: *Proc. 11th Conf. Australian Soc. Sugar Cane Tech.*, 1989, 175 - 183.
- 9 K. Buchholz & Bruhns: *I.S.J.*, 1994, **96**, 12A - 13A; *Zuckerindustrie*, 1993, **118**, 321 - 338.

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