Improved industrial crystallization by automation

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Abstract

Compared with earlier times in beet sugar factories with manual or semi-manual operation, modern process automation is responsible for great improvements in sugar house processing. The practical experience gained in sugar factories shows the impact of automation e.g. on the improvement of crystal content and crystal quality as well as efficiency of the vacuum pans.

Generally, in industrial crystallization, the focus is on steadily improving the process control, with the aim to achieve additional yield increases. A new concept is presented, demonstrating how an improved process control can be implemented with the aid of supplementary sensors, while particularly observing the metastable limit.

Keywords: Crystallization, automation, yield increase, determination of the metastable limit

1. Introduction

One of the essential criteria in sugar house operation is the quality of the sugar produced. The specific challenge is to ensure controlled crystal growth by preventing the formation of crystal nuclei and crystal agglomeration, since these two processes would lead to a non-controllability of particle size distribution, crystal purity and crystal colour. In addition, problems would result downstream in centrifugation and, because of a higher sugar dust concentration, in the sugar silo.

Controlling sucrose crystal growth is not a new concept, it has, in fact, been described by many authors; Van der Poel et al. (1998), for example, gives detailed information. However, despite the existing theoretical process knowledge, many beet sugar factories outside Europe still run crystallization processes in manual or semi-manual mode, and the sugar quality suffers as a result.

This paper uses an example to describe the automation of a pan boiling process with up-to-date methods and concepts applied. Moreover, it presents a new concept for optimizing the yield of crystallizers, focusing particularly on the metastable limit.

2. Ideal pan boiling

Thanks to the automation of the sugar crystallization process, it is today possible to keep supersaturation during pan boiling in the metastable range of the phase diagram. Seed crystals that are added at the seeding point start growing without any additional formation of false grain. A number of measures have to be taken to keep crystallization within the metastable range.

Experience gained in sugar factories has shown that supersaturation at the seeding point is critical for crystallization. The very low crystal content of some 0.001 % after seeding with slurry results in a very small crystal surface. If supersaturation is too high during or after seeding, this leads to an undesired secondary nucleation ‘shower’; controlled product quality can no longer be ensured.

Ideal pan boiling therefore provides for a stabilization phase after the seeding point (Fig. 1). Low supersaturation and a resulting low crystallization rate are achieved owing to time-limited low heating steam pressure and a low evaporation rate. The slurry particles then grow to an adequate crystal size without secondary nucleation, so supersaturation during boiling-up can be maintained in the metastable range thanks to the adequate crystal surface.

In this ideal pan boiling process, the dry substance content and the massecuite level are linearly raised during boiling-up, until a high crystal content with large space-time yield is reached. Ideally, vapour pressure and heating steam pressure remain at a constant level. During the following tightening phase, the heating steam pressure is increased further, in order to obtain a crystal content of typically 50 – 55 % at the end of the batch.

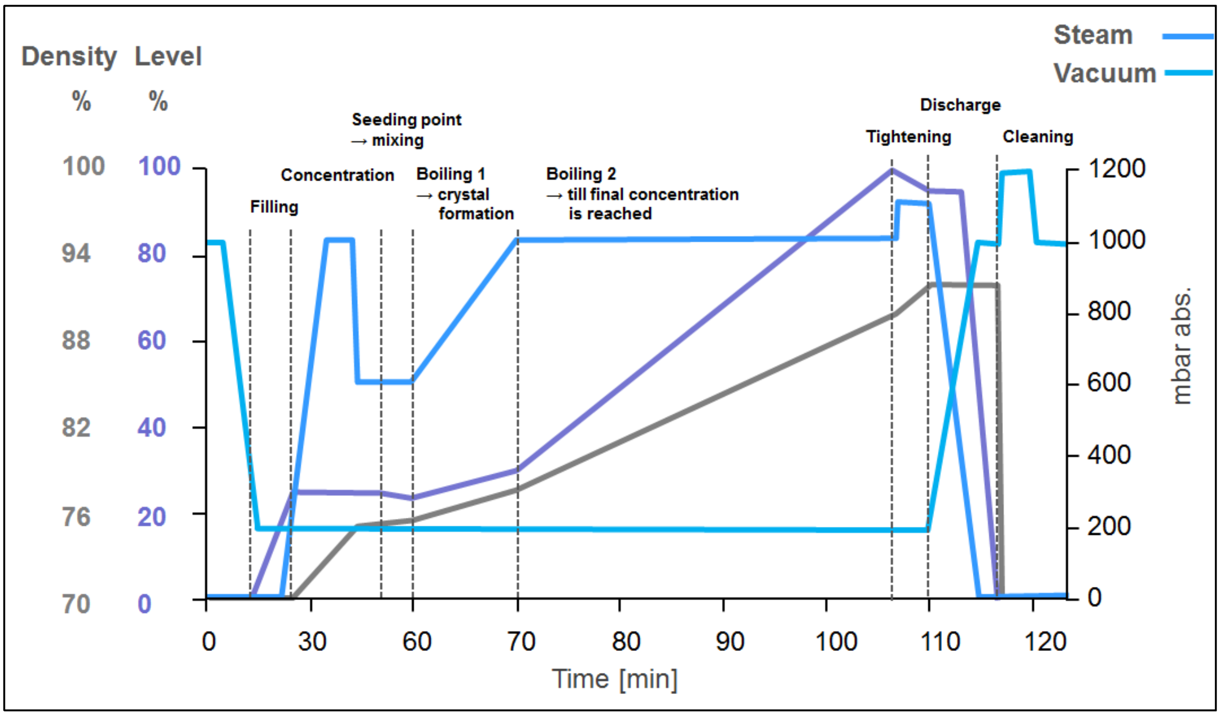

Fig. 1: Process curves of ideal pan boiling.

3. Example of an automation system for pan boiling

In 2014, a white sugar station of a Russian beet sugar factory was equipped with a BMA pan boiling process automation system. This helped gradually raise the crystal content, which was finally approx. 5 % higher compared to the 2013 campaign (Fig. 2). The coefficient of variation for crystal size distribution (CV) was also improved from 44 % to 33 %. As a result, the wash water quantity for the centrifugals could be reduced and crystal preservation improved.

Automating the white sugar pan boiling process also contributed to heating steam savings, since the process could run without water intake during crystallization. This was previously required to dissolve the false grain that had formed. Altogether, automation helped improve the space-time yield by two batches per day.

Thanks to the automatic control of the crystallization process, crystals of a consistently superior quality can be produced; a high throughput and lower workload for the plant operators are additional benefits.

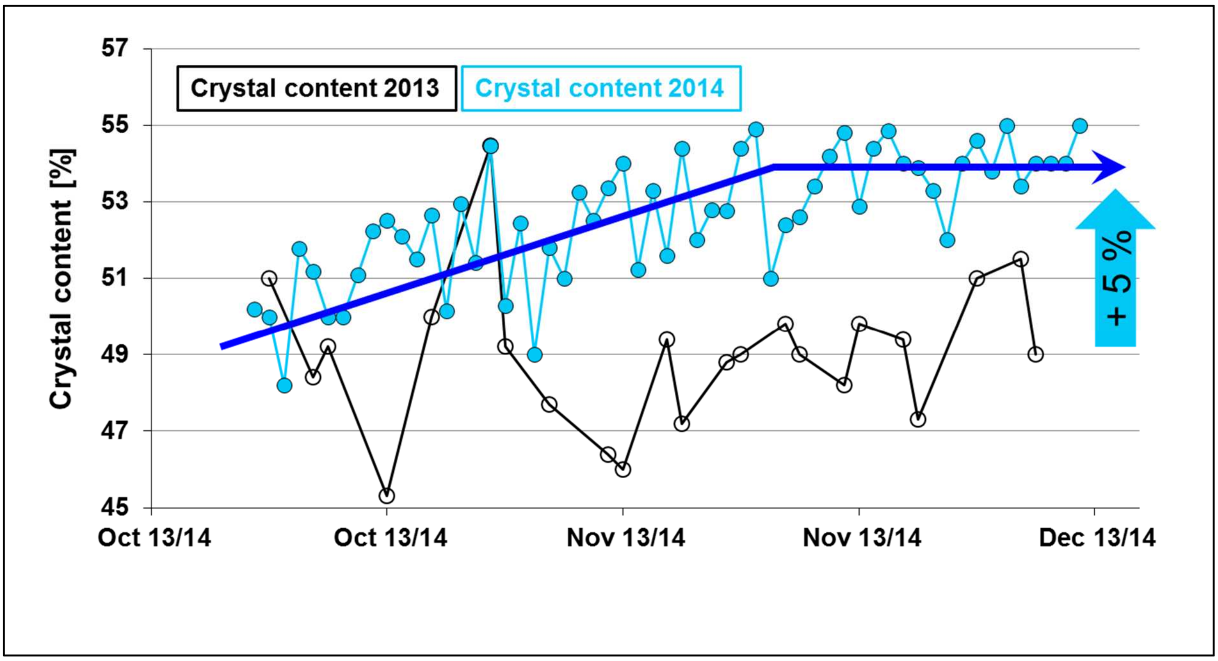

Fig. 2: Crystal content of white sugar vacuum pans in a Russian beet sugar factory without (2013 campaign) and with automated crystallization (2014 campaign).

4. Novel BMA concept for controlling crystallization processes

In industrial crystallization, the focus is on the continuous improvement of process operation, with the aim of achieving additional yield increases at a constant or improved crystal quality.

The novel BMA concept for controlling crystallization processes is based on the fact that the empirically optimized crystallization processes in the sugar industry are executed at reduced supersaturation levels. They include safety factors aimed at excluding secondary nucleation. In the literature, very wide variances are given for the metastable limit. Roza et al. (2013) quotes a range of 1.20 – 1.40. In practice, however, Roza (2011) says, crystallization processes run within the supersaturation range below 1.10 – 1.12 (high purity syrups) or 1.12 – 1.15 (low purity syrups).

The metastable range depends on many parameters, some of them time-variable, with varying effects, for instance

- Type and concentration of non-sugars
- Presence, number and size of crystals (seed crystals, incrustations)
- Inhomogeneities (temperature, concentration)
- Structure and design of the crystallizer
- Construction materials used (surface energy)
- Rate of supersaturation build-up (evaporation, cooling)
- Rate and size of mechanical energy input

The metastable limit is thus no system constant; it is in fact variable depending on the current conditions in a sugar factory and cannot be predicted. Therefore, one has to work with supersaturation levels that are too low rather than too high.

However, if it was possible to determine the specific metastable limit of a pan boiling process, this would open up the possibility of controlling towards higher supersaturation and thus the chance for a higher space-time yield.

An additional problem is the fact that supersaturation cannot be measured online. It is a complex, multi-variable function that depends on the following four parameters:

a) Dry substance content of the mother liquor
b) Sugar content of the mother liquor
c) Boiling temperature
d) Wiklund parameters

The parameters a) and c) can be measured, whereas b) and especially d) have to be determined in the laboratory, which takes considerable time. No real-time statements can therefore be made, but the process situation can only be evaluated retrospectively.

To solve this problem, the known approaches for controlling crystallization processes were identified in a literature survey. Fig. 3 was prepared following Nagy et al. (2013) with three main groups:

- model-free approaches
- model-based approaches
- hybrid-based approaches

While model-based and newly hybrid-based approaches have found only limited application in an industrial context, model-free approaches are preferred there. They involve the direct use of PAT (process analytical technology)-based measuring signals as feedback control in the crystallization process.

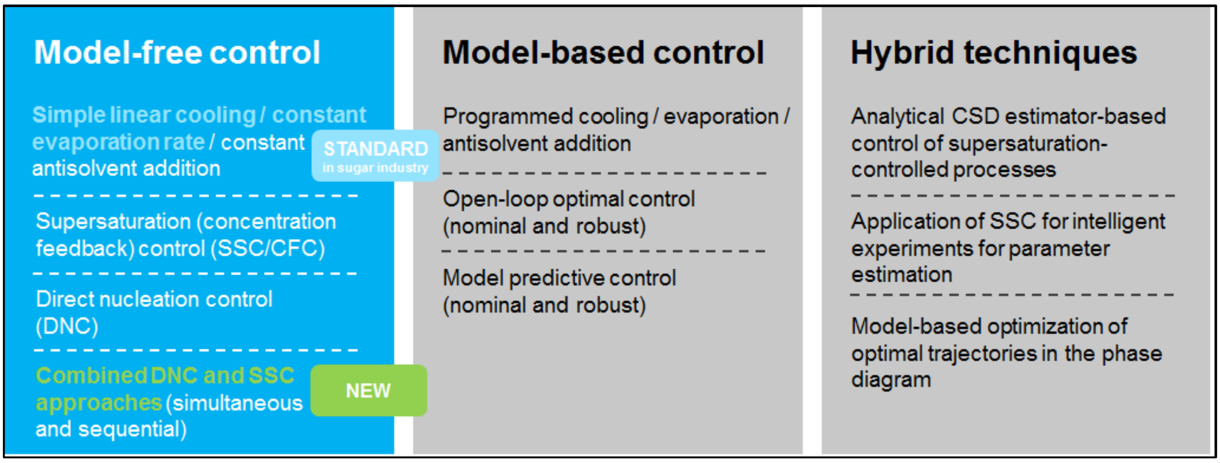

Fig. 3: Survey of crystallization control approaches.

The model-free approaches are subdivided into four subgroups, which we shall look at in more detail below:

a) Typical “feedback control strategies” (e.g. PID, cascade) are designed in such a way that they adhere to simple heuristic operating rules, such as a linear cooling rate, a constant evaporation rate, or a constant anti-solvent addition rate (Rawlings et al., 2002).

b) Supersaturation control (SSC), frequently also referred to as concentration feedback control (CFC):
SSC tries to control the crystallization process by following a desired operating curve (trajectory) in the phase diagram. This operating curve has to be determined empirically, with a focus on the non-formation of nuclei. The process state at any one time is determined by concentration and temperature measurements, so the desired concentration (supersaturation) can be followed in the phase diagram. Most applications use constant supersaturation (absolute or relative) for the setpoint. The SSC approach controls the crystallization process in the phase diagram, making it a more direct approach than process control by following, e.g., time-defined temperature curves or dry substance content levels of the massecuite. However, the crystal properties are not directly checked. This means, for example, that false grain or agglomerates caused by malfunctions cannot be detected and thus represent a problem for the downstream process (Naygy et al. 2013).

c) Direct nucleation control (DNC)
In the DNC approach, the number of crystals and the crystal size distribution are directly measured and controlled. DNC detects any increase or decrease in the number of crystals. To keep the number of crystals identified at a setpoint, the supersaturation of the mother liquor is increased or reduced; in some cases the mother liquor even has to be undersaturated. DNC is characterized by controlled crystal growth and dissolution cycles (GDCs), which are achieved by cooling/heating, the cyclic addition of anti-solvents/solvents, or a combination of both. DNC can be used for consistent in-situ nucleation, the elimination of agglomerates, the elimination of false grain, and the optimization of the crystal size distribution (more homogeneous and larger crystals) (Naygy et al. 2013).

d) Combination of direct nucleation control (DNC) and supersaturation control (SSC)

The sugar industry uses almost exclusively conventional model-free approaches of type a) ("best practice concept" or "master boiling concept“) for controlling the pan boiling and cooling crystallization processes.

Recently, a small number of beet and cane sugar factories have also started using approaches based on type b) (cf. Roza 2013). Here, the solubility curve and the metastable limit have to be predefined. This permits definition of a trajectory in the phase diagram for nucleation-free crystallization, along which the process is run. Knowledge of the current non-sugar/water ratio is also crucial. If the purity and mass flow rate of the feed solution are known, as well as the boiling temperature and water evaporation rate, the current non-sugar/water ratio and thus the current supersaturation can be calculated. This involves use of a process refractometer as PAT.

The novel BMA concept for controlling pan boiling processes relies on a type d) model-free approach. The benefit this offers is that it basically requires no a priori knowledge of the mother liquor properties, such as the solubility curve, the metastable limit or the purity of the feed solution.

The metastable limit for every crystallization process in its respective vacuum pan is determined via DNC. This can be done, for example, as shown in Fig. 4, which shows an iterative multi-batch process for pan boiling. In a sequence of several batch processes with different levels of supersaturation, the still unknown metastable limit is gradually approached. As secondary nucleation is sometimes triggered, and sometimes not, this iterative approach helps determine the limit. In this way, an optimal operating point is reached, which, as expected, is higher than in conventional process operation in the sugar industry.

The PAT used is a special, highly sensitive sensor that is able to detect nucleation very quickly throughout the process. The secondary nucleation ‘shower’ triggered in some batches can be recognized early and dissolved by immediate water intake. These nuclei thus do not become part of the crystal product and represent no risk for centrifugation. The water intake should lengthen the batch process by no more than about 30 minutes. For an iteration process consisting of 4 – 6 batches, only 2 – 3 hours more would therefore be required; it can be assumed that the iteration process has to be repeated only every 2 – 4 weeks.

The DNC approach therefore provides an optimized and nucleation-free operating point, while requiring only slightly more effort for every crystallization process in the respective vacuum pan. The new operating point determined in this way then serves as the reference variable based on the SSC method up to the next iteration process.

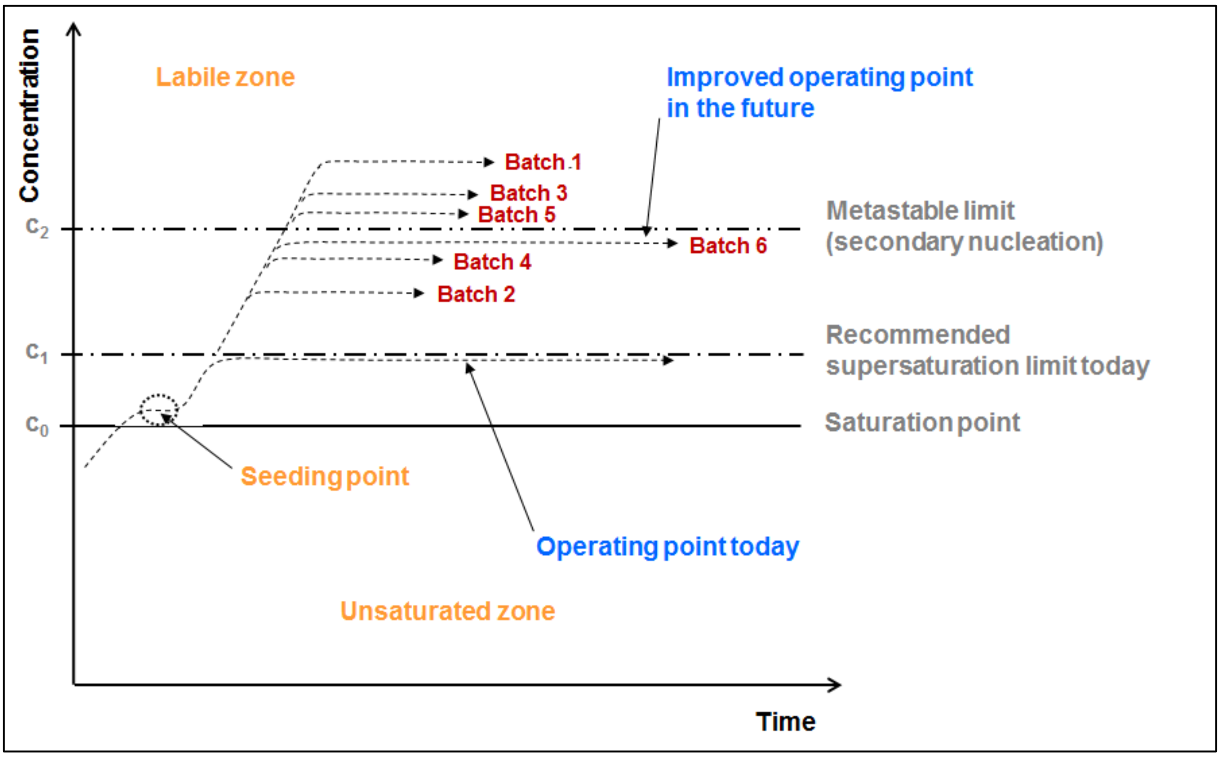

Fig. 4: Iterative multi-batch process for determining the metastable limit in pan boiling.

5 Conclusions and outlook

Automation of the pan boiling process, especially at the beginning of the crystallization process is essential for a high crystal yield, good sugar quality and low energy consumption. BMA's pan boiling automation based on the ideal pan boiling process ensures that undesired secondary nucleation is reliably and consistently prevented, leads to higher space-time yields, and allows for reproducible process operation.

The new BMA concept for online determination of the metastable limit for every crystallization process in its respective vacuum pan provides a way of identifying the optimized and nucleation-free operating point in every case. The DNC/SSC concept thus ensures higher space-time yields and greater economic efficiency compared to conventional control concepts for crystallization processes in the sugar industry.

The studies performed so far in the laboratory and in sugar factories show a very good traceability and response behaviour of the new sensor, especially in nucleation processes. The new concept will be implemented in in an improved BMA pan boiling automation system in the near future.

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