

ONLINE MEASUREMENT OF SUGAR CRYSTALLIZATION WITH A PORTABLE NON-INTRUSIVE INSTRUMENT

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Abstract

This paper presents results from a measurement campaign at a sugar refinery with a novel non-intrusive measurement technique for crystallization diagnostics. By measuring batch-to-batch development of key crystal properties, we demonstrate a quick and effective approach for crystallization control, monitoring and troubleshooting. The developed measurement system is based on the photometric stereo technique including a digital color camera and three LED lights illuminating the crystal suspension from three directions. The measurement system can be installed on a standard sight glass of a boiling pan, and it provides real-time measurement results on crystal size distribution and massecuite flowability. As the instrument does not require direct contact with the suspension, the installation can be done while the boiling pan is in operation, and the instrument is truly portable in nature. The portability allows quick studies in various locations, e.g. for studying mixing and homogeneity of the crystal population in the pan. The accuracy of the new technique is demonstrated by comparing crystal size distributions measured with the system to results acquired by sieving and traditional transillumination-based process microscopy.

Introduction

Sugar crystallization batches can be monitored online with camera-based measurement systems. The systems used in this paper measure crystal size distributions in real-time, directly from the process liquid in the boiling pan. The image analysis accompanying the measurement hardware not only provides real-time information on crystal size but also makes it possible to observe the structure of the crystals, including agglomeration, aspect ratio and other measures of shape. This is a major advantage of camera-based 2D measurement techniques over point-wise 1D techniques based on a single laser beam, such as laser diffraction and focused beam reflectance measurement. Furthermore, with the help of advanced image analysis techniques the detected crystals can be classified based on their morphology [1-4]. By measuring the development of key crystal properties in a number of batches, we demonstrate this quick and effective tool for crystallization monitoring, troubleshooting and control.

To observe crystal growth, process microscopes have been utilized in sugar crystallization for decades. Today most pan microscopes are equipped with a camera to view live image of the suspension in the control room. A natural extension of this technology is to utilize automated image analysis to detect crystals and measure their properties from the images [1-5]. In sugar crystallization the properties analyzed include mean crystal size and its standard deviation [5]. The process microscopes that have been used in sugar production so far are typically based on the principle of transillumination: the crystal suspension flows through a narrow gap and the illumination is guided to the opposite side of the gap.

The measurement method presented and used in this paper utilizes front light (FL) instead of the typical transillumination (i.e. back light, BL). In other words, the illumination is guided to the measurement volume from the side of the camera. Thus, the imaging system can be installed on a standard sight glass of a boiling pan without any components inside the boiling pan. This setup has several advantages. First, as the hardware is not in direct contact with the suspension, there is no risk of contamination. Secondly, the installation and any maintenance tasks can be performed while the

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process is running. In addition, the system is easily portable, making it possible to study different locations and boiling pans quickly with minimal effort.

The sampling conditions of the FL configuration differ from the measurement gap of the BL setup. These differences, including the basic principles of crystal size analysis, are discussed in the Results section. The results presented and discussed include time trends for crystal size, crystal growth rate, crystal content, suspension temperature and massecuite density. The measurement technique introduced in this paper provides highly desired possibilities for optimizing crystal growth and final crystal size distribution.

Methods

The results presented and discussed in this paper are based on a two-week measurement campaign at a sugar refinery. Both the newly developed FL system and the traditional BL system were installed on the same boiling pan. The systems are installed at the same level, and the horizontal distance between the measurement heads is about 1 m. During the campaign both systems were continuously recording data. Both systems produce crystal images with a spatial resolution of 3 μm per pixel and a field of view of 4.5 x 3.4 mm^2 . The locations of both systems remained the same over the entire campaign. The two measurement systems are described briefly in the following sections.

Back Light Method

The BL system was built in an existing process microscope using upgraded camera and illumination modules. The camera used to collect the image data is a high-resolution model with a GigE interface. The illumination is provided by a high-power strobe light source, guided to the measurement head with an optical fiber. This combination provides blur-free high-quality images of the suspension throughout the process. With current installation mechanics the measurement gap is fixed at 1.5 mm, which is fairly narrow considering the flow properties of the suspension at the end of each batch. The implications of a narrow measurement gap are discussed later. A picture of the BL setup is provided in Figure 1, and the basic principle of the system is illustrated in Figure 2. An example image captured with the BL system 1 h after seeding is presented in Figure 3.

Front Light Method

The FL system is designed for installation on a standard boiling pan sight glass. A picture of the installation is presented in Figure 4. The imaging principle of the FL probe is presented in Figure 5. The system is based on the principle of photometric stereo [6] utilizing a color CCD camera and three LED lights illuminating the crystal suspension from three directions. The LED lights are monochromatic and each of a different wavelength. The wavelengths are selected to match the RGB filters of the Bayer matrix of the CCD sensor. Thus, the camera produces three images of the crystals (channels R, G and B) that are illuminated from different directions. These three images of the same crystals are used in the analysis phase to separate neighboring crystals as their facets show different colors depending on their orientation. The computational operations performed for the color channels can be seen as a preprocessing step and the actual crystal analysis is based on the same principles as that of the BL images. An example of a crystal image with all the color channels is presented in Figure 6. In the visualization image for the operator, the information of color and illumination direction are typically discarded. The image is converted to gray scale, as presented in Figure 7.

Analysis of the Crystal Images

The crystal measurement is based on the optical imaging of the suspension in the actual concentration followed by automatic image analysis to detect, classify and measure the properties of the crystals. The image analysis consists of image preprocessing, segmentation, feature extraction, object classification and finally crystal modelling and validation. The size of each crystal is determined by the maximum dimension visible in the segmented crystal image.

The system provides crystal size histograms as a function of time. The single histograms are used to compute crystal count [pcs/image], volumetric mean size and standard deviation and

volumetric percentiles of crystal size (e.g. D10, D70, D95 vol-%). The crystal growth rate as [$\mu\text{m}/\text{h}$] is computed from the histogram time series. In addition, the average aspect ratio of crystals [-] is estimated. The statistics can be filtered using time averaging.

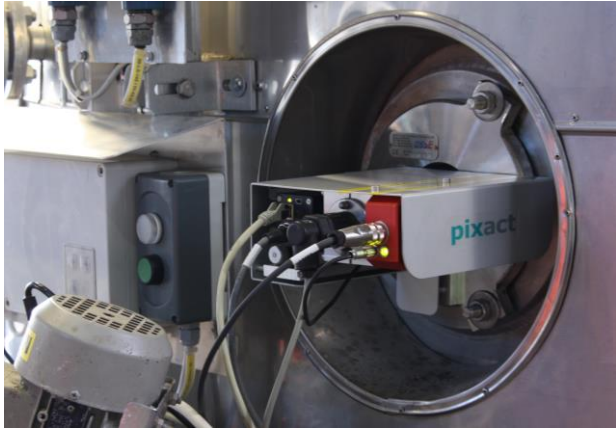


Figure 1. BL installation on the boiling pan.

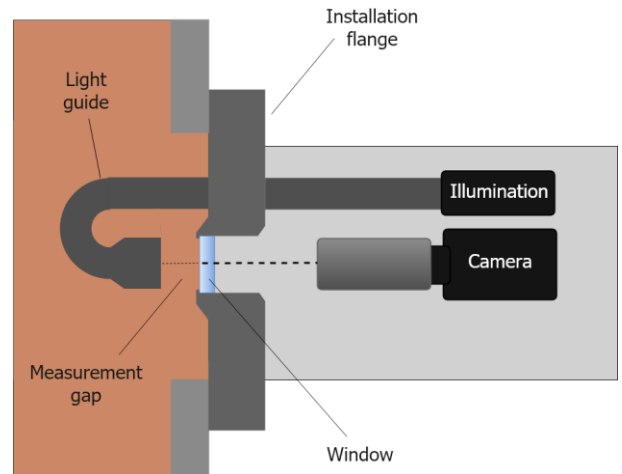


Figure 2. Illustration of the BL setup and imaging principle.

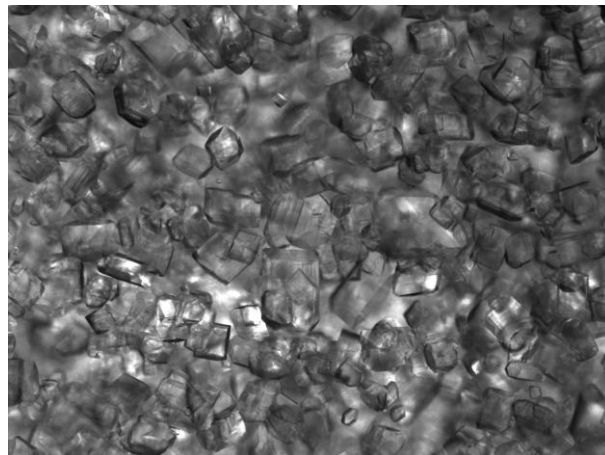


Figure 3. Example image of the crystal suspension 1 h after the seeding acquired with the BL probe.



Figure 4. FL installation on the boiling pan sight glass.

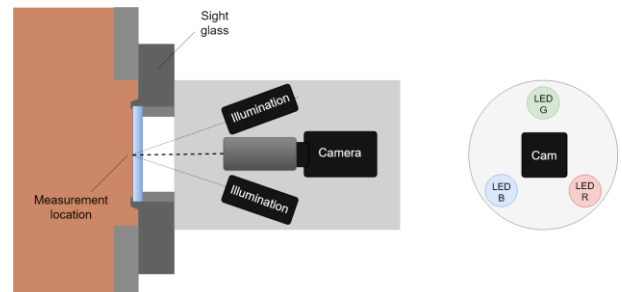


Figure 5. Illustration of the FL setup and imaging principle.

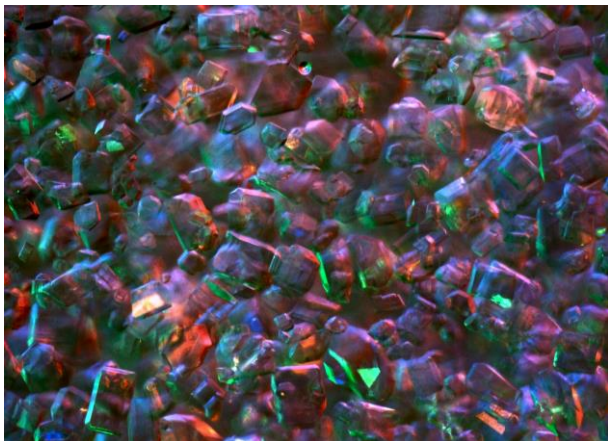


Figure 6. Example image of the crystal suspension 1 h after the seeding acquired with the FL probe.

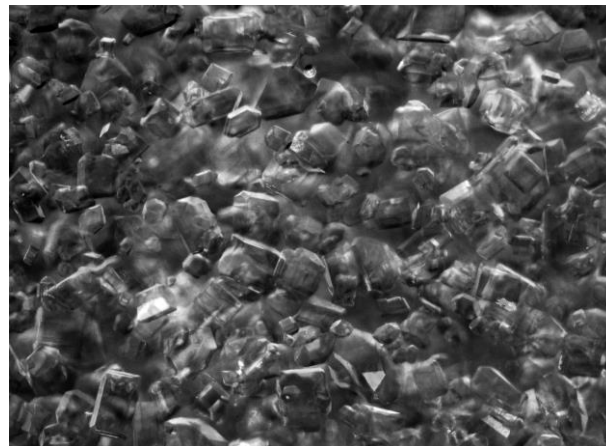


Figure 7. Example image of the crystal suspension 1 h after the seeding acquired with the FL probe converted to gray scale.

Results

The results section starts with a short discussion on the differences in the sampling procedures of the FL and the BL probes. Having established the essential differences, the discussion moves on to the comparison of measurement results obtained with the probes. In total, about 40 batches were measured, but the current paper focuses on nine batches for which sieving data is available. The sieving data shows clear differences between individual boilings, which provides an optimal starting point for the comparison. The comparison with the sieving analysis is mainly based on the FL data. Finally, some interesting observations on the BL data are presented to highlight the role of the seeding phase.

Sampling and Measurement Location

In this paper we compare two measurement systems that utilize a similar method, the imaging of crystal suspensions, but differ from each other in terms of “sample preparation”. The FL probe captures images of the crystal suspension in the vicinity of the sight glass surface. The crystals are affected by wall effects and the flow pattern on the wall. There is a boundary layer flow condition inducing a velocity gradient near the window surface, but the flow pattern is dominated by the large-scale mixing in the tank. This is not the case in the measurement gap of the BL probe, which creates a channel flow between the two windows. The gap causes a contracting flow pattern and strong flow gradient. The flow gradient tends to align the crystals so that instead of being randomly oriented they are aligned with the main flow direction. This is demonstrated for needle shaped crystals in Figure 8. The aspect ratio of sucrose crystals is not high, but the crystals can be expected to be aligned so that the longest chord is facing the camera. This effect causes the results to be biased towards a higher mean size compared to data in which the crystals are randomly oriented. This is similar to the sampling conditions of a laboratory microscope: the sample is imaged when the crystals are laying on a glass surface with the addition of the longest chord of the crystal facing the camera [7]. The problem is illustrated in Figure 9, which presents a model crystal viewed from different directions.

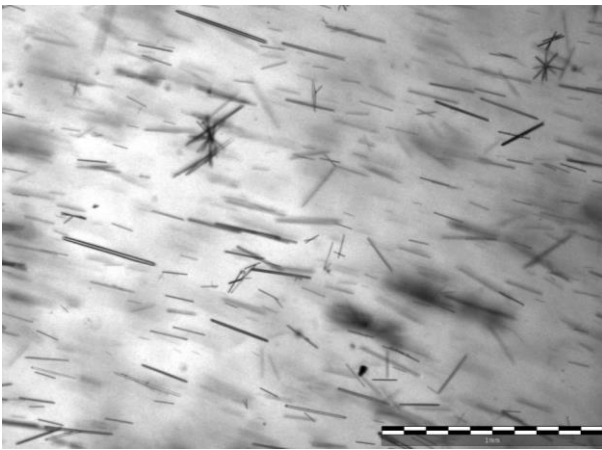


Figure 8. Example image of needle shaped crystals in the measurement gap of the BL probe.

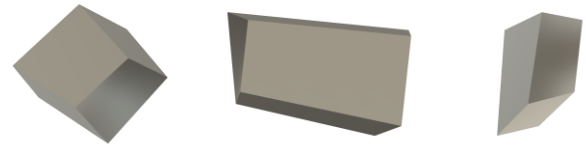


Figure 9. Different projections of a 3D crystal model.

Unbiased measurement of crystal size is possible only when the crystals are entirely visible. This means that the suspension measured needs to have a relatively low crystal content in order for the crystals not to overlap. As the number of overlapping crystals rises, the margin of error increases. The image analysis algorithm used in this study attempts to solve the problem by removing some detections of partly visible crystals from the calculations. Naturally, this is an ambiguous task, and the results always contain partially visible crystals.

The final aspect related to sampling is the high viscosity of the suspension caused by an increase in crystal content. As the thick suspension might not be mixing as efficiently, pockets may form in the suspension that do not reflect the overall crystal population. When the suspension velocity approaches zero, such a pocket may remain in the measurement location for an extended time period. Thus, temporal variation in the measured mean size can be observed especially at the end of the batch. Figure 10 presents the crystal flow velocity trends in the measurement volumes of BL and FL probes. Local flow velocities are measured by both, BL and FL probes utilizing the cross-correlation method for consecutive image frames. The fluid velocity measurement in crystallization processes has been presented by the authors in a previous paper [8]. The velocity trends reveal that the suspension flows fluently across the sight glass and provides good sampling conditions for the FL probe, whereas in the measurement gap of the BL probe, the flow velocity decreases rapidly already at 30 minutes after the seeding.

Based on this discussion, it can be concluded that

- The measurement probe should be placed in a region of efficient mixing (see also [5]),
- The probe should be designed so that it has minimal effect on sampling (large gap, or no gap at all), and
- The most accurate results are obtained at low concentration or by using sample dilution.

In practice, it is difficult to realize all three of these conditions simultaneously.

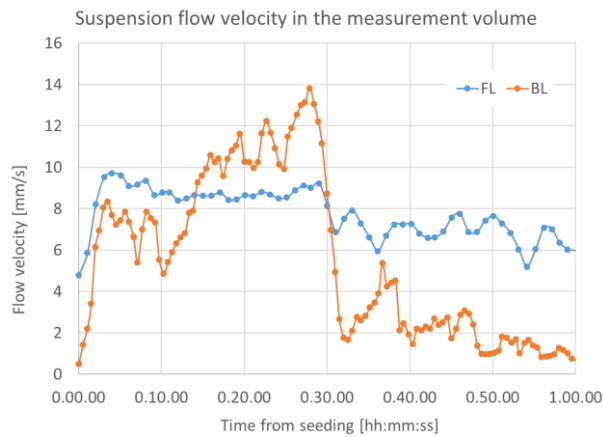


Figure 10. Examples of measured flow velocity trends during a batch.

Front Light vs. Back Light

First, the development of mean crystal size measured with the two measurement systems is compared. Figures 11 and 12 illustrate the measured crystal growth for three example batches. These batches were selected as they represent different type of development patterns and final crystal sizes. In batch no. 3, the crystals remain small whereas in batch no. 8 the crystals grow faster and the end mean size is larger. Batch no. 6 represents a typical development pattern. All 40 batches measured during the campaign were between batches no. 3 and 8. These same three batches are also used when comparing online data with the sieving analysis. Data is presented only for a one hour period starting from the seeding because of poor mixing resulting in large variations for some batches after this time. The typical boiling time is about 1 hour 30 minutes.

The results measured with the FL probe are shown in Figure 11. The crystal size appears to remain rather constant during the first few minutes after the seeding. After 10 minutes the measured mean crystal size starts to increase in a rather linear manner. The same batches measured with the BL probe are presented in Figure 12. These trends show slightly different of behavior during the first 20 minutes of boiling. The BL probe shows a more pronounced non-linear development in the trend about 15 minutes after the seeding. The initial trend starting from the seeding levels off and the growth continues following another linear trend with a slower growth rate. At 20 minutes the measured mean crystal size is larger with the BL probe than with the FL probe. The reason for this difference is not obvious, but can be speculated to be related to for example the flow pattern in the measurement gap, which tends to align the crystals as discussed above. However, the differences between the individual batches are well defined and follow very similar development patterns with both techniques excluding the differences in the very beginning of the batch. Note also that the measured final crystal size is similar with both techniques. Figure 13 presents time trends for suspension temperature, level, massecuite density and seeding for batch no 6, which displays average crystallization behavior. In Figure 13 the shape of the massecuite density trend curve is in line with the measured crystal size trends (batch 6 in Figures 11 and 12).

A more detailed comparison of the results obtained with the FL and BL probes is carried out by comparing measured mean crystal sizes at 30 minutes and 1 hour after the seeding. The comparison at 30 minutes is presented in Figure 14. The correlation is relatively good considering the fluctuations seen in the time trends. The same comparison at 1 hour is presented in Figure 15. At this point, the correlation is somewhat lower, which is probably related to the increasing variation towards the end of the batch.

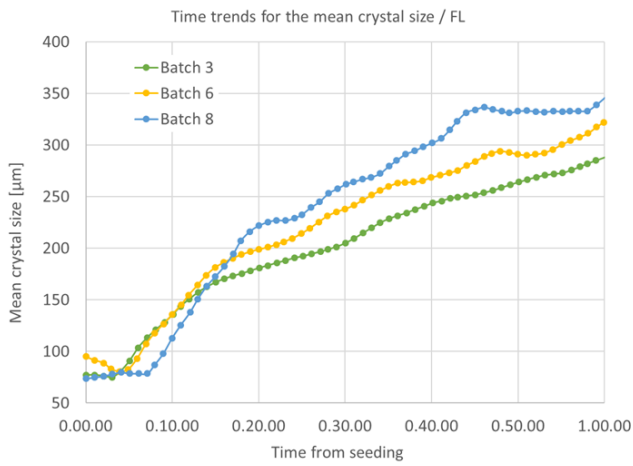


Figure 11. Development of mean crystal size for three batches measured with the FL probe.

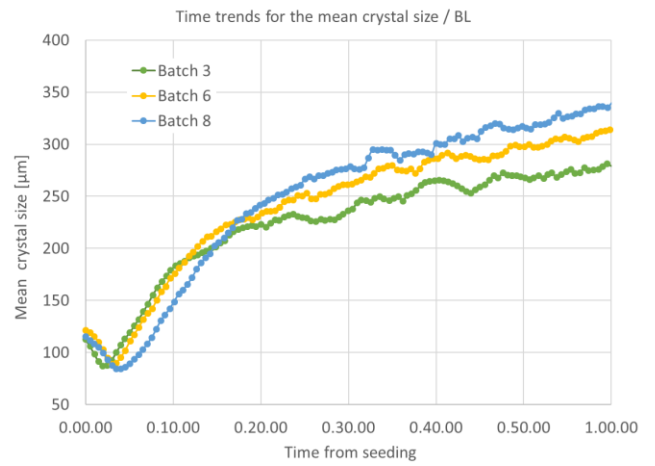


Figure 12. Development of mean crystal size for three batches measured with the BL probe.

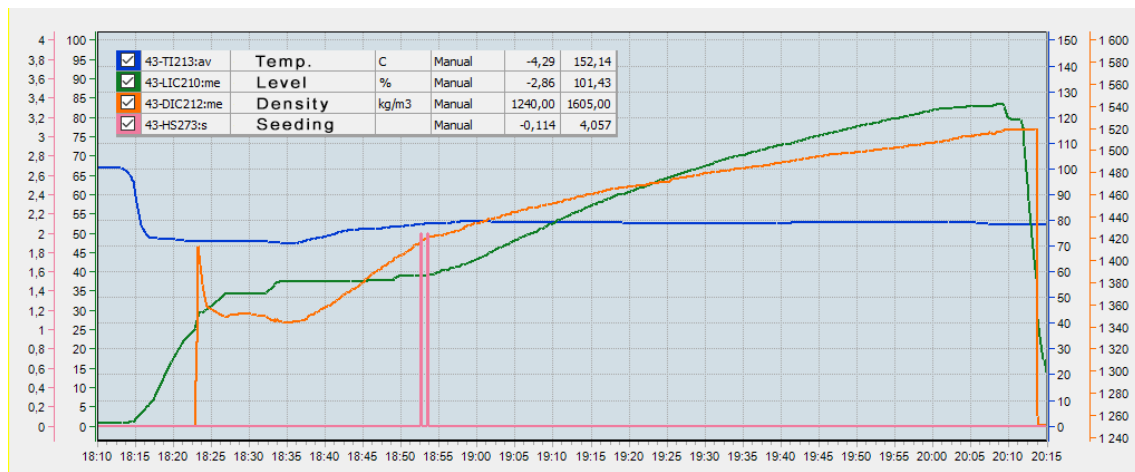


Figure 13. Trends from the factory automation logged for batch no 6.

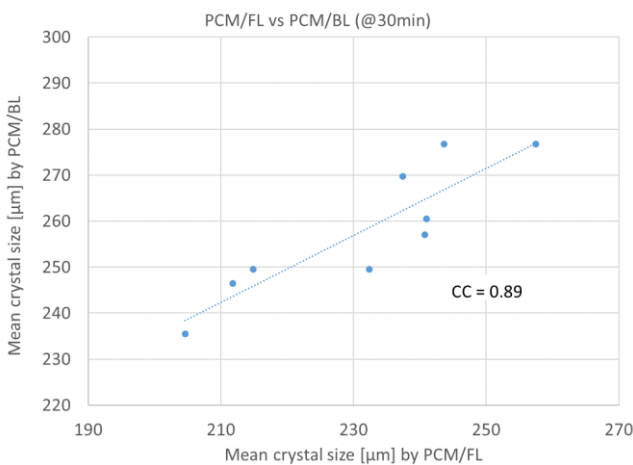


Figure 14. Comparison of mean crystal size measured with the FL and BL techniques at 30 min after the seeding.

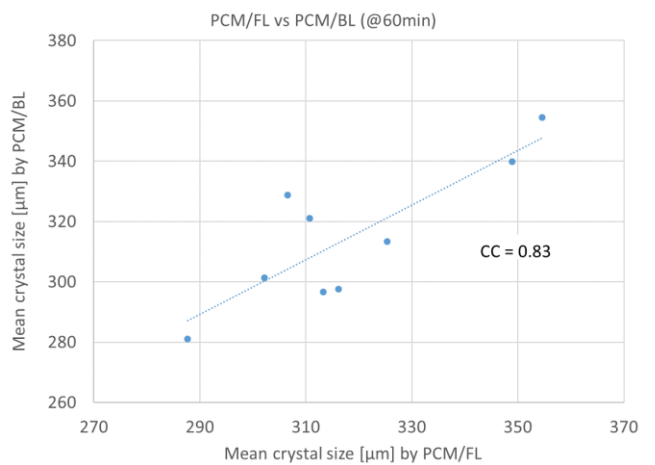


Figure 15. Comparison of mean crystal size measured with the FL and BL techniques at 1 h after the seeding.

Comparison with the Sieving Results

Next the crystal size distribution measured online is compared to that obtained with sieving analysis. The sieving sample is taken from the same batch, but it is the final product after washing and drying. Figure 16 presents the crystal size distributions from the sieving analysis for the same three batches (3, 6 and 8). Batch no. 3 shows a narrow peak in the small range with more fines. Batch no. 6 is a typical in spec batch. Batch no. 8 shows higher variation with more coarse crystals. Batches 3 and 8 represent unwanted batches with too small and too large mean sizes. Figure 17 presents crystal size distributions measured for the same batches online with the FL probe. This data is again taken one hour after the seeding, because poor mixing in the late stages results in a lot of variation in some batches.

The same size classes are used for discretization in both graphs, although the two techniques are not measuring the same characteristic length. Thus, a perfect match between the distributions cannot be expected, but the general appearance of the two plots is similar. The higher number of fines in batch no. 3 and higher number of coarse crystals in batch no. 8 are clearly visible. The modes of the size distributions are located at smaller scales with the online measurement. This is expected as the data is not measured at the end of the batch. Naturally, the bias towards smaller values due to the overlapping crystals and random orientation of the crystals also affects the measured size.

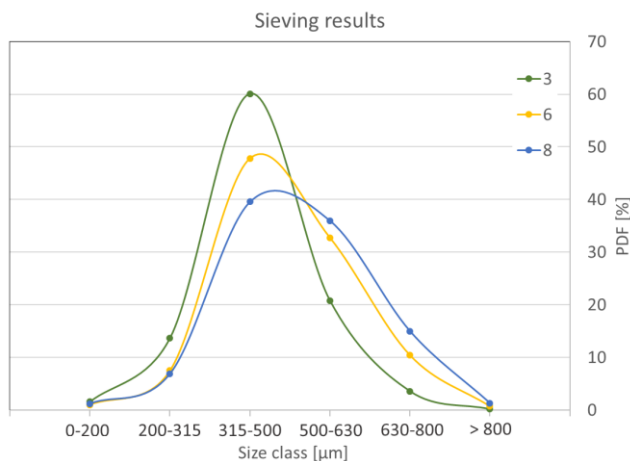


Figure 16. Sieving results for the three selected batches.

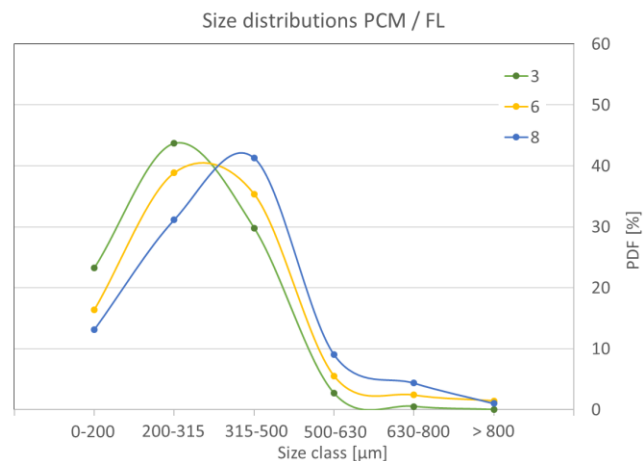


Figure 17. Size distributions measured online with the FL probe for the three selected batches.

Figure 18 presents a comparison between all the sieving samples and the mean size measured online with the FL probe 30 minutes after the seeding. A clear correlation between these two can be observed. Some points deviate more from the trend, but in general the agreement is good. The same comparison with the online data measured 1 hour after the seeding is presented in Figure 19. The correlation here is somewhat lower. The decreasing correlation can be explained by increasing fluctuations in the size trends towards the end of the batch.

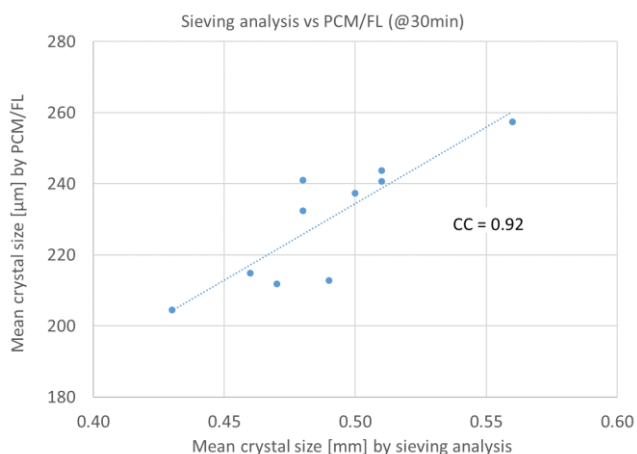


Figure 18. Comparison of the mean crystal size measured with the FL probe 30 min after the seeding and by sieving.

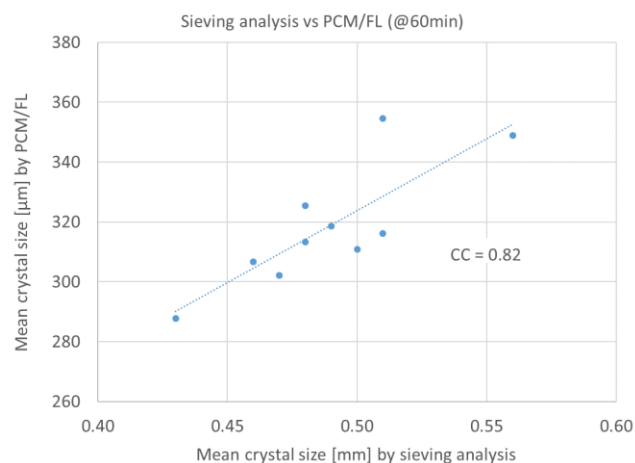


Figure 19. Comparison of the mean crystal size measured with the FL probe 1 h after the seeding and by sieving.

Currently, the resolution of the FL probe is not sufficient for very detailed analysis of the crystal suspension right after the seeding. The BL technique is preferred in this phase to provide accurate information on the crystal suspension. The seeding phase is known to be one of the most critical parameters in sucrose crystallization. The number of detected crystals typically hits a peak at 3-5 minutes after the seeding. After this, the number of detections starts to fall monotonously towards the end of the batch. This is partly related to the overlapping crystals and the feature of the analysis algorithm that removes crystals that are mostly hidden by overlapping crystals from the analysis. However, the peak value of crystals detected at the beginning of the batch appears to predict the final crystal size distribution accurately. The peak number of crystals detected with the BL system vs. the mean crystal size measured by sieving is presented in Figure 20. The agreement is even better than for the measured mean crystal size at 30 minutes after the seeding.

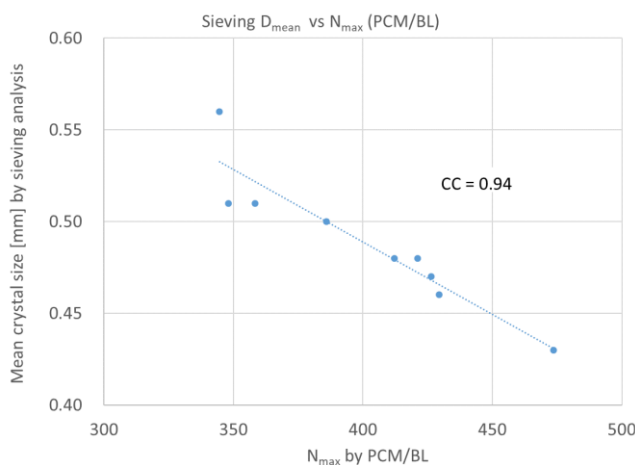


Figure 20. Comparison of the maximum number of crystals detected after the seeding vs. the mean crystal size of the sieving analysis.

Conclusions

The FL process microscopy system presented in this paper is able to monitor crystal growth throughout the sugar crystallization batch. It provides better sampling conditions for crystals than the traditional transillumination-based measurement system. The narrow measurement gap of the traditional BL method is associated with problems in the sampling of the crystal mass, the alignment of the crystals along the flow and finally, the analysis of the latter part of the process as the flow of the suspension in the gap slows down. The FL method avoids these issues by monitoring the crystals through a sight glass of the boiling pan without any components submerged in the suspension.

The FL method utilizing a three-color photometric stereo reconstruction enables the analysis of highly dense crystal masses in the latter part of the crystallization process. Overlapping crystals can be recognized based on the differences in crystal orientations, which are visualized by the crystal color in the images. On the other hand, the BL method performs better in the early stage of the process when the crystals are very small. The weak light reflectance of microcrystals makes it difficult for the FL setup to detect crystals that are smaller than 20 μm in diameter. Future development of the FL method aims at increasing the brightness of the crystal images.

The results presented in this paper demonstrate that data recorded even just a few minutes after the seeding can be used to predict the final crystal size. An increase in the number of seeding crystals decreases the final crystal size. The mean crystal sizes measured with both methods (BL and FL) correlate extremely well with the sieving results. Various methods can be used to predict crystal growth during the boiling.

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