

LOW RAW CRYSTALLIZATION – IS THERE ROOM FOR IMPROVEMENT?

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Abstract:

Low raw crystallization is arguably the most important step in a beet sugar factory. Sugar loss with molasses is the largest single loss; and the sugar recycle with low raw sugars leads to increased energy consumption and reduced throughput. Additionally, every sugar boiling increases the fillmass color by 10-20 %. Therefore, every effort directed to minimization of recycle and improved centrifuge performance makes significant impact to the bottom line. Growing uniform sugar crystals with correct particle size to assure maximum rate of crystal growth as well as molasses exhaustion still remains much of an art due to multiple factors affecting the crystallization process. A crystal population should be grown allowing for sufficient surface area, however, providing large enough crystals to assure proper surging and minimizing non-sugar recycle with low raw sugars. Procedures will be discussed for evaluation of crystal size distribution in the industrial low raw fillmass and seed slurries. Experimental results showing changes in crystal size and coefficients of variation as fillmass is subjected to crystallization and centrifugation will be presented.

Introduction:

Crystallization process in the sugar industry has been studied in much detail. Generations of sugar technologists worked on optimization of all stages of sugar boiling. Standard boiling procedures have been established, pan automation reached very high levels, and sugar boiling became a technical procedure rather than an art. However, not too many sugar factories can demonstrate perfect operation of sugar end, which results in relatively high molasses loss and related negative effects of factory economics. Ever-changing operating conditions in the sugar factories affect the composition and physico-chemical properties of sugar solutions that in turn influence supersaturation. Joint influence of multiple parameters, e.g., steam pressure and vacuum fluctuations negatively affect sugar boiling and subsequent centrifugation. Noteworthy, that although many of US sugar beet mills have an option of recovering sugar from molasses via chromatography, sugar recovered during the first pass has the lowest production cost.

Low raw boiling has direct influence on the downstream operations-cooling crystallization, reheating and centrifugation. Poor quality of low raw sugar negatively impacts color and energy balance as well as economics of a sugar factory due to increased washing, both sugar and non-sugar recycles. Increasing washing rate in the low raw centrifuges leads to high sugar loss with molasses, while reducing non-sugar and color recycle. Affination of the low raw sugar additionally increase sugar recycle into the lower boiling stages rather than returning the sugar to the melter prior to white boiling. Therefore, improvements that can be made to the crystal population in the low raw side of the factory should positively effect overall process economics. Crystal size in the low raw side is rarely if ever monitored in the beet plants.

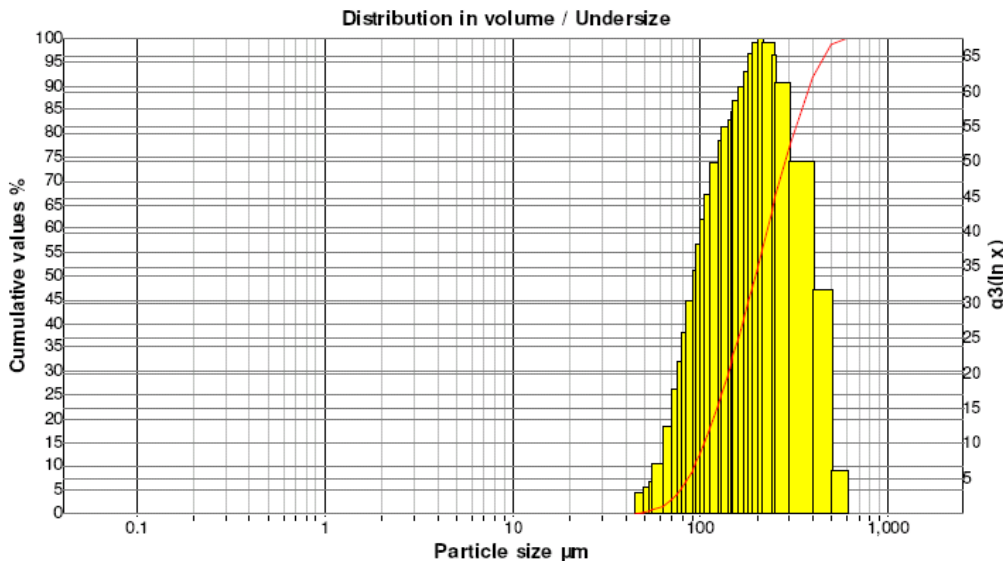
Measurement of Crystal Size Distribution:

Most reported information on crystal size measurement in the beet industry is related to the quality of white sugar product. The accepted method of analysis uses a set of sieves; results are reported in terms of MA (mean aperture) and CV (coefficient of variation) (McGinnis, 1982). MA is often confused with the average crystal size. It actually represents the size of the sieve aperture that is a “cutoff” point for 50 % of the sample’s weight. Another definition of MA is a median of weight distribution.

Methods of crystal size measurements depend on sample preparation and analytical methods. Evaluation of particle population by visual techniques (Rozsa, 2008) is difficult for many reasons. The accuracy is influenced by sample preparation, limited resolution, software issues, overlapping of the images, evaporation of the sample, potential crystallization in the thin films. Most accurate particle characterization can be achieved by particle size analyzers using various laser techniques (Malvern, Cilas, Horiba, Ankersmid are among major suppliers). Sophisticated statistical software allows to accurately calculating the functions representing average crystal size and CV. Reproducibility of laser methods is usually very good.

Differences exist between various particle analyzers using different principles of measurement (Etzler, 1995). This topic is too broad to be included in the present paper. A typical histogram of particle size distribution is shown in Figure 1. The example represents a sample of C-sugar with average particles size of 250 micron.

Figure 1 Typical histogram of particle size distribution (by volume)



Most published work on crystal size measurement in the beet industry has been reported on the characterization of seed slurries and fondants (AVH Symposium, 2005). Measuring the average size of fondant samples is important for calculating number of seed crystals as potential centers of crystallization. Samples of seed fondant can be analyzed directly in iso-propanol slurries. The values of fondant crystal size are reported within the range from 1 to 10 micron. The results are highly method dependent and are influenced by the analysis of experimental data.

For characterization of seed slurry (fondant) the number distribution should normally be used. Representation of the same crystal sample in numbers show different particle size compared to volumetric distribution. An example illustrating the difference in median size for

volume and number distribution of the same fondant is shown in Table 1. Particle size distribution is represented in the table using following characteristics: D10, D50 and D90, mean and CV. Values of D10 and D90 stand for crystal size, below which 10 and 90 % of the total crystal volume is located, respectively. D50 is, thus, a median of the distribution (or MA). Both tables 1 and 2 contain data measured for a sample of powdered sugar ground in a ProSep ball mill. Ball mill parameters (rpm, number of balls and milling time) are omitted from the current discussion, because they are meaningful only if mills of the same design are compared. Mills of various designs need to be tuned for each specific case to target a desired quality of seed slurry.

Table 1 Comparison of particle size distribution reported in number and volume

Sample	D10	D50	D90	Mean	CV
Fondant(Volume)	2.9	9	17.7	9.7	60
Fondant(Number)	0.9	1.4	2.9	1.9	92

The data in Table 2 represent the fondant parameters as a function of grinding time. If fine fondant is used as a starting material, the median particle size changes within the accuracy of measurement during grinding. The coefficient of variation, however, changes dramatically. Experimental data showing importance of the initial CV of fondant on crystal development in the low raw pan are discussed later in this paper.

Crystal size of fillmass is rarely studied because of difficulties of sample preparation and analysis. It has been mostly reported in the cane sugar literature, because C-sugars (equivalent to low raw sugars in the beet industry) are used as seed magma for boiling higher purity pans. Analysis of crystal size in fillmass is much more tedious, and large error may be introduced due to residual syrup supersaturation in a sample. Particle counting using washed and dried sugar samples have been applied; a method is described in SASTA manual (2005). It is difficult to apply this procedure to analyze the seed development in low raw pans. Iswanto et al. (2007) used ethanol pre-saturated with sucrose to separate crystals from the mother liquor. Some researchers additionally centrifuge and affinate the sugar samples (Broadfoot, 2008). Our experience has shown that treating fillmass with alcohol containing solution typically hardens the fillmass rather than enhancing dispersion. Instrumental methods work well for crystal size analysis provided that samples are sufficiently dispersed in a solvent.

Table 2 Fondant grinding experiments (data represent the distribution by number)

Sample name	Median size , micron	CV, %
10XX powdered sugar (Starting material)	2.5	102
1 hr	3.5	81
2 hr	4.1	69
3 hr	3.7	77
6 hr	4.2	62
12 hr	3.7	55
Samples shown for comparison		
6X Powdered sugar	3.0	114
Commercial fondant 1	3.0	59
Commercial fondant 2	2.4	76

A procedure has been developed, where a saturated sugar solution was prepared by mixing measured amount of sucrose in water for about 24 hours and then filtering the syrup through a 5- micron filter to remove traces of fine crystals. Small samples of fillmass (3-5 grams) were soaked in a saturated sugar solution and carefully mixed with a spatula to avoid crystal breakage. Depending on the quality of fillmass, sample preparation usually takes about 20-30 minutes. No additional sample handling or centrifuging was required. Prepared sample was introduced into a particle analyzer preloaded with clean saturated sugar solution. This procedure proved to be reliable and had been used for the last two seasons for sample preparations.

Crystal Development In Low Raw Pans:

Experimental work has been performed in a beet sugar factory that was at the time processing a mixture of thick juice and chromatographic extract. A set of fillmass samples were taken from a low raw pan as the crystal population was being developed. After the low raw pan was dropped, time delayed samples were taken from the Lefeuille crystallizers and the mingler that stored feed material to low raw centrifuges. Samples of low raw sugar, affinated low raw sugar and molasses were taken as well as the fondant powder.

Fondant samples were analyzed directly in the iso-propanol suspension; the fillmass samples were dispersed in saturated sugar solution at room temperature according to the procedure described earlier in this paper. A Cilas 1180L laser diffraction particle size analyzer was used. The summary results are presented in Table 3.

Table 3 Crystal development in a low raw pan

Sample	D 10	D 50	D 90	Mean	CV
Fondant(Volume)	2.9	9	17.7	9.7	60
Fondant(Number)	0.9	1.4	2.9	1.9	92
After seeding					
Fillmass, 20 min	38	81	123	81	40
Fillmass, 55 min	73	100	143	105	26
Fillmass, 180 min	72	113	179	120	36
Fillmass , as dropped	19	129	248	135	68
Out of Lefeuille crystallizer	51	148	275	160	56
Into Law Raw Centrifuge	77	156	324	179	56
Low Raw Sugar	81	147	288	169	53
Affinated Low Raw Sugar	77	192	363	104	87

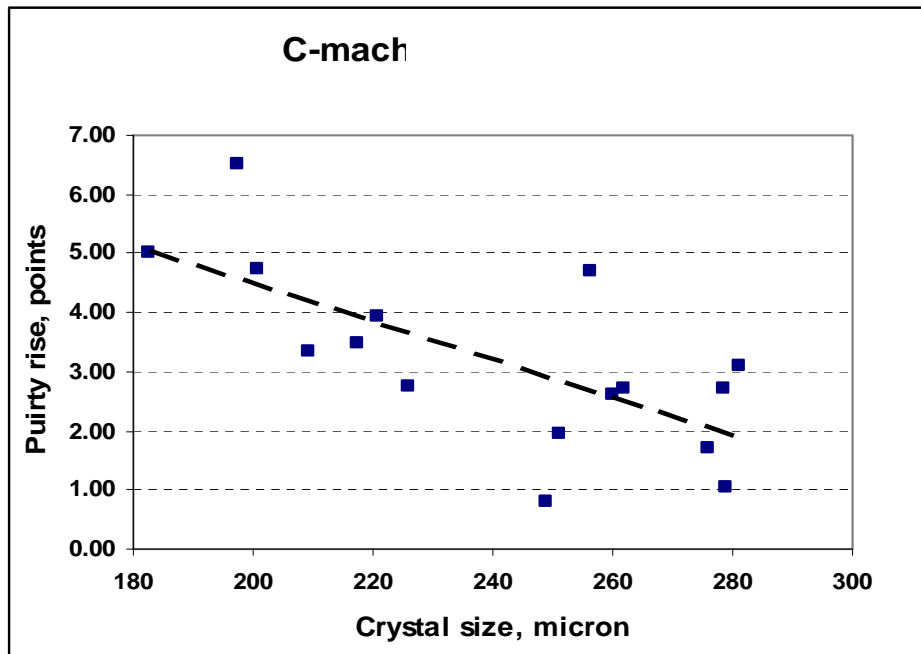
As expected, crystal linear dimension increased significantly over first 20-30 minutes of operation, then crystal size gradually increased during boiling period. Coefficient of variation (CV), however, after initial decrease, increased gradually and reached its maximum level towards the end of boiling cycle. Initial CV decrease may be caused by dissolution of fine grain which should not be happening at normal operating conditions, where saturated or

supersaturated solutions exist in the pan. Following gradual increase may be an indication of formation of false grain especially towards the end of the cycle when the pan is brixed up. Later in cooling crystallizers and the mingler crystals continued growing, while the CV was staying at approximately same level. Noteworthy that a similar pattern of crystal size changes was observed in our experiments with graining strikes in the cane sugar mills.

The crystal size of the low raw sugar reached about 150 microns, while 10 % of the low raw crystals particles are smaller than 77 microns (see D10 value in Table 3). At these conditions, significant portion of crystals could pass through centrifuge screens that typically range between 40 and 60 microns for low grade machines. Observed large difference between crystal size in low raw and affinated raw sugars infers that many fine crystals had been washed off through the centrifuge screens.

Efforts should be made to grow larger crystals with better uniformity to reduce molasses losses and improve purging of low raw sugars. The graph in Figure 2 shows data illustrating a trend of molasses purity rise across the C-centrifuges as a function of crystal size. The experimental data were obtained in a raw sugar cane mill during the 2008 grinding season. Although C-boiling in raw cane mill process is different from low raw boiling in a beet plant, the characteristics of centrifuging process is very similar. Crystal size of about 250 micron is more desirable to provide acceptable purity rise across the centrifuges. The graph may be used as an illustration that significant benefits can result from optimization of crystal size in the low raw pans.

Figure 2 Purity rise across the centrifuges as a function of median crystal size



Additional studies are planned that will attempt to link the changes on crystal development with levels of supersaturation and other boiling parameters.

Conclusions:

1. Crystal size analysis is a good tool for troubleshooting of low raw sugar boiling, cooling crystallization and centrifugation operations.
2. A simple procedure for fillmass sample preparation has been developed and tested for crystal size analysis of crystal development in low raw pans
3. Most non-uniformity in the low raw crystal size is introduced in the pans, especially during the last stage of boiling
4. Optimization of crystal size in the low raw pans can result in reduction of molasses losses and improvement of overall plant operation via improvement of purging characteristics across the continuous centrifugals.

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