FULL PAPER

COMPARATIVE RESULTS FROM WHITE BOILINGS USING CONVENTIONAL SLURRY-SEEDING AND SEEDING WITH SEED PRODUCED FROM A CONTINOUS SEED PREPARATION SYSTEM UTILISING SOUND-WAVE-INDUCED NUCLEATION

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Abstract

Sugar technologists have long been striving to develop a robust crystal seed production technology for pan stations, aiming to overcome the limitations of conventional slurry-seeding and enable fully continuous production of sugar.

The Sugar Milling Research Institute NPC (SMRI) has been developing a Continuous Seed Preparation System (CSPS) that can continuously generate and supply seed crystals to continuous vacuum pans and to prepare batches of seed for batch pans. The CSPS utilises Sound-Wave-Induced Nucleation (SWIN) to generate seed crystals from a sucrose solution. A pilot-scale CSPS capable of producing 300 l of seed crystals was built at the SMRI and it successfully produced seed crystals that met the required seed size (>50 µm) and crystal number density (>7 x 10^4 crystals/ml) specifications.

A crucial step in the pilot-scale experimental work was to compare sugar produced from conventional slurry seed with sugar produced from CSPS seed, and to investigate the technical advantages associated with the use of CSPS seed. Replicate white sugar boilings were performed using the SMRI's 51 l steam-heated pilot pan. The pan was seeded with either slurry or CSPS seed, and performance indicators such as mean aperture of the sugar, size coefficient of variation, crystal regularity index, pan yield, and colour transfer were measured. The results indicated that acceptable and comparable pan performance was achieved using CSPS seed. Furthermore, CSPS seed was immediately visible upon addition to the pan, unlike ball-milled slurry which takes 5 to 10 minutes to confirm the presence of sufficient crystals. Replacing slurry with CSPS seed has the potential to reduce boiling times as the CSPS seed crystals (approximately 100 µm) are about 20 times larger in size and 8 000 times larger in mass than slurry crystals (about 5 µm). There will also be a reduction in water and steam usage due to the reduced time spent holding on water in the early stages of crystal growth.

Keywords: Pan boiling, seeding, Mean Aperture (MA), size Coefficient of Variation (CV)

Introduction

Crystallisation is a key process in both raw and refined sugar manufacturing and is typically performed in a pan station through evaporative crystallisation (or ‘pan boiling’). The objectives of pan boiling are to:

- Recover the maximum amount of sugar (i.e. crystalline sucrose).

- Produce product crystals of the required size and with a narrow size distribution to enable efficient curing of the massecuite (Rein, 2007).
- Crystallise sugar in an energy and time efficient manner.

Acceptable pan station performance broadly depends on (1) mother liquor supersaturation control, (2) adequate massecuite circulation, and (3) seed crystals of consistent and suitable quality (Rein, 2007, Love *et al*., 2016). Good instrumentation and pan design are necessary for the first two requirements; however, to this day, the third requirement is very dependent on the skill and/or experience of the operator.

Seed crystals are the starting crystals onto which sucrose from the mother liquor is deposited. Consequently, seed crystals of the required size, uniformity and number are required to provide adequate surface area for crystallisation and to produce sugar of acceptable quality (i.e. size and uniformity). Seed crystals are generally produced in batch pans and the conventional technique of producing seed in South Africa is known as the slurry-seeding technique (Love *et al*., 2016). This technique involves the addition of ball-milled slurry (i.e. dust-like crystals suspended in alcohol) to the pan whilst the mother liquor is in the metastable zone of supersaturation (i.e. the zone where crystal growth occurs without the possibility of secondary nucleation). This technique has a major drawback, determining if seed of a suitable quantity and size distribution has been produced relies mostly on the skill of the operator. Hence, automatic adjustment of seed production requirements to improve pan station performance is difficult to achieve.

Ideally, all the slurry crystals added into the pan should grow into product crystals with no false grain formation or crystal dissolution; this is known as full-seeding. Austmeyer and Schipalke (1983) deemed the slurry-seeding technique to be insufficiently reproducible for practical purposes due to zones of undersaturation in a vacuum pan that cause slurry crystal dissolution. Furthermore, Rahiman *et al*. (2019) reported that the results from an SMRI survey of slurry-seeding of B-massecuite and C-massecuite at 18 South African factories in 1977 (Matic, 1977) revealed that true seeding was not achieved, as there was either substantial dissolution of slurry crystals or a large amount of false grain formation during pan boiling. Reviews of the slurry-seeding technique by Love *et al*. (2016) and Rahiman *et al*. (2019) suggest that unless careful control of the mother supersaturation is achieved and strict ball-milled slurry preparation practices are implemented, full-seeding becomes difficult. Given the shortcomings of the slurry-seeding technique, there is certainly scope to develop a robust seed production technology that would be able to generate seed crystals continuously for continuous pans and/or accumulate batches of seed for batch pans.

The SMRI has been developing a seed production technology to satisfy these process demands. This technology involves the generation of seed through sound-wave-induced nucleation of a sucrose solution (Love *et al*., 2016; Rahiman *et al*., 2019). A pilot-scale CSPS capable of producing approximately 300 l of seed was constructed and installed at the SMRI to undertake the relevant experimental work (Figure 1). According to the knowledge of the authors, there is no continuous commercial seed production technology available and the closest commercial seed production system involves batch seeding production via cooling crystallisation of slurry crystals (Cramer and Lehnberger, 2021).

Figure 1. Pilot-scale CSPS installed at the SMRI

A key step in the pilot-scale experimental work was to compare sugar produced from slurry seed with sugar produced from CSPS seed, and to quantify the relative performance of these two seeding options.

Originally, the intention was to produce sufficient CSPS seed for a full-scale refinery pan at the Tongaat Hulett Sugar (THS) refinery at Rossburgh. This was to be done on a fully-instrumented and controlled fourth refined sugar batch pan (Mncube *et al.*, 2017). This would have provided useful information on the benefits for both conventional batch boiling and for the new crystallisation processes associated with 'Green Refining' (Jensen and Love, 2015). Unfortunately, the logistics of transporting sufficient seed from the SMRI to the refinery without degradation of the seed proved unsuccessful (Rahiman *et al*., 2019), and when the option of testing by using the new SMRI 51 l pilot pan became possible (Rahiman *et al.*, 2023), the decision was made to do the comparisons between slurry-seeded and CSPS-seeded boilings using the pilot pan. To continue to build on the earlier work (and to have feed material that was available throughout the year) the feed material was kept as refinery Jets 3 or 4, depending on their current refinery performance.

This paper summarises the results of pan boiling experiments using the SMRI's 51 l steam-heated pilot pan (Figure 2) for the comparative boiling tests.

Figure 2. SMRI steam-heated pilot pan

Experimental Methodology

Replicate white boilings were performed using refinery jet (i.e. run-off from a centrifuge that cures refinery massecuite) as the feed material for the slurry-seeded and CSPS-seeded boilings. The refinery jet was collected from the THS refinery. Either Jet 3 or Jet 4 was collected depending on which process stream had a gravity purity between 90 and 93%. The purity of the feed material has a strong impact on the selection of the graining point (particularly for the slurry-seeded boilings) and the crystallisation rate. Therefore, it was desirable to perform the pan boilings with feed material of very similar purity for comparison purposes. The procedures used for both the slurry-seeded and CSPS-seeded boilings are detailed below.

Pan boiling key performance indicators such as mean aperture of the sugar, size coefficient of variation, crystal regularity index, pan yield, and colour transfer were measured. The main success indicator was whether the massecuites produced using the CSPS seed showed equivalent or improved size Coefficient of Variantion (CV) compared to slurry-seeded massecuites. Size CV is an indication of the uniformity of the crystals and is the ratio of the standard deviation to the mean size of the crystal population expressed as a percentage (Rein, 2007). Lower CV values indicate that the crystals are more uniform in terms of size and this is desirable for efficient separation of molasses from the massecuite in the centrifuge station.

Slurry-seeded boiling procedure

The slurry-seeded boiling procedure detailed by Rahiman *et al*. (2023) was used for all slurry-seeded boilings and is summarised in Appendix A. Three slurry-seeded boilings were conducted in 2022 (Table 1). The slurry used for the boilings, labelled ‘SSa’ to ‘SSc’, was prepared using the South African Sugar Technologists’ Association recommended method (Anon, 1985).

Table 1. The slurry-seeded boilings performed

Slurry-seeded boiling name	Feed material (date collected)	Slurry used
SSa	Jet 3 (8/4/2022)	Sourced from a South African raw sugar factory
SSb		
SSc		

CSPS-seeded boiling procedure

The CSPS-seeded boilings comprised of three steps, viz. (1) once-off production of the volume of seed required for at least three pan boilings, (2) storage of the seed, and (3) execution of the CSPS-seeded pan boilings. The quantity of seed required for the pilot pan boilings was calculated based on the seed quality results obtained by Rahiman and Mbuthuma (2023a). To produce massecuites that matched the size of the massecuite crystals produced by slurry seeding, the seed volume required for each CSPS boiling was determined using the sieve analysis data of the sugars and the crystal contents of the massecuites from boilings SSa, SSb, and SSc. The technique used to determine the seed volume is analogous to the alternative crystal number density technique as detailed by Rahiman and Mbuthuma (2023b). Once the total number of crystals in the final massecuite was determined, this value was divided by the crystal number density of the CSPS seed to determine the volume of seed required. An average crystal number density of 1.5×10^5 crystals/ml for the CSPS seed was assumed based on the results detailed by Rahiman and Mbuthuma (2023a). A sample calculation is shown in Appendix B. The average CSPS seed volume required was determined to be approximately 2.7 l.

Production of the CSPS seed

The pilot CSPS was used to prepare the seed for the pilot pan boilings following the procedure described by Rahiman and Mbuthuma (2023a). Approximately 12 l of CSPS seed was collected from each pilot plant run. The sampling bucket was pre-heated before collection to prevent rapid cooling of the seed which would potentially cause secondary nucleation. Two 12 l batches of CSPS seed were produced, and these were used to perform five CSPS-seeded boilings. The first two preliminary boilings used the first 12 l batch of seed (produced on 20 June 2023) and were part of method development while the last three (CSPSa, CSPSb, and CSPSc) used the second batch of seed (produced on 24 October 2023) and were used for comparison purposes.

Storage of the CSPS seed

The seed was stored in a 12 l constant temperature vessel after collection from the pilot CSPS (Figure 3). The storage vessel was controlled at a temperature of 78°C and was equipped with an overhead stirrer for suspending the crystals. The seed was stirred at approximately 300 rpm during seed storage. The seed crystals were expected to grow in the 12 l vessel during the first hour of storage as the seed cooled (88 to 78°C) and the mother liquor supersaturation approached saturation.

Figure 3. Constant temperature seed storage vessel

CSPS-seeded boilings

After reviewing the results of the preliminary set of CSPS-seeded boilings (not shown), it was decided that the pan boiling recipe should be modified to suit the size characteristics of the CSPS seed, rather than using the slurry-seeded boiling recipe. The rationale for this was the unacceptable CV of sugar produced from the preliminary boilings (>35%), and an analysis of the pan boiling logged data revealed that false grain was formed during the boiling process (see Appendix C). Potential reasons for the formation of false grain were considered to include suboptimal graining conditions during seed addition, poor mother liquor concentration control, and poor control of the pan evaporation rate particularly during feeding. If the evaporation rate is too high during the feeding phase, the pan control feeds the jet very quickly to maintain the mother liquor concentration and therefore the volume of the massecuite increases with minimal crystallisation. This reduces the crystal surface area per unit of available sucrose, consequently increasing the likelihood of false grain formation, particularly if there is poor control of mother liquor concentration. The modified boiling procedure used for the CSPS boilings is detailed in Appendix A.

The CSPS-seeded boilings that were performed with the modified boiling procedure are listed in Table 2. A software communication failure during CSPSc allowed an uncontrolled amount of water into the pan and the CSPS seed was dissolved. There was approximately 1.4 l of seed remaining in the seed storage vessel and this was used to complete the boiling with the volume of pan feed being adjusted accordingly, to compensate for the low volume of seed (i.e. a lower volume of final massecuite was produced to compensate for the low volume of seed available). Therefore, approximately 39 l of massecuite was produced in CSPSc instead of 75 l.

Table 2. CSPS-seeded boilings performed using seed produced on 24 October 2023

CSPS-seeded boiling name	**Pan boiling date**	**Feed material (date collected)**	**Volume of seed added**
CSPSa	25/10/2023	Jet 4 (27/09/2023)	Approximately 2.7 l
CSPSb	26/10/2023		
CSPSc	27/10/2023		Approximately 1.4 l

Sampling and analysis

A single CSPS seed sample was analysed before each CSPS-seeded boiling to determine the crystal size, size CV and crystal number density as per the methods described by Rahiman and Mbuthuma (2023b). At the end of the pan boilings, massecuite, molasses, Nutsch molasses (i.e. mother liquor) and sugar samples were collected, which were used to determine the following performance parameters (Rahiman *et al*., 2023):

- Massecuite brix (°Bx)
- Pan yield (%)
- Mean aperture (MA) of the sugar (mm)
- Size coefficient of variation (CV) of the sugar (%)
- Crystal regularity index (CRI) of the sugar (%)
- Colour transfer (i.e. the ratio of the affinated sugar to massecuite colour expressed as a percentage)
- Average linear crystal growth rate over the boiling cycle (µm/h).

A Gamma distribution function (Metwalli, 2023; Anon, 2024) was fitted to the cumulative sieve analysis results of the sugar produced in the pan boilings. The cumulative distribution was transformed into a probability distribution to assist with visualising the size distribution of the sugar.

Data recording

A digital log of the pan boiling operational parameters was saved at the end of each pilot pan boiling. The key events that occurred during the pan boiling were manually captured to assist with troubleshooting.

Statistical analysis of results

A Student's t-test was performed (at a 95% confidence interval) to determine if there were any significant differences between the performance parameters of the slurry-seeded and CSPS-seeded boiling groups.

Results and Discussion

In analysing the results of the tests, an attempt has been made to extract the maximum insight from the information collected. These efforts must be understood in the context of the following:

- Pan boiling is well known to be very demanding in terms of skill and technique to achieve repeatable performance that meets the required standards (a major reason for developing the CSPS for industrial use).
- To enable a comparison between slurry-seeded and CSPS-seeded pan boiling, it was necessary to develop full boiling procedures for both techniques and to be able to conduct the boilings with an acceptable level of repeatability.
- The scale and time required for each pan boiling is particularly demanding. The 51 l pilot pan has proven that it is probably the smallest size of equipment that can provide a reasonable approximation to full-scale operation (Love and Walthew, 2002; Mncube and Love, 2003), but still requires much preparation in the research laboratory.
- A particular aspect of the testing was to evaluate the relative performance of the two seeding systems in terms of crystal size distribution (and CV in particular). Published data (Rein *et al*., 1995; Wright and White, 1969) show that there are substantial variations in the data on crystal size distribution, even when analysing data from pans boiled by skilled industrial pan boilers.

Comparison of the refinery jets used for the pan boilings

The compositions of the refinery jets collected for the pilot pan boilings are shown in Table 3, along with the analytical precision for each analyte. The results indicated that the compositions of the various refinery jets were very similar (i.e. the differences between the analytes were within the analytical precisions of the measurements).

Table 3. Analyses of the refinery jets used for the pilot pan boilings

Analyte	**Jet 3 (08/04/2022)**	**Jet 4 (27/09/2023)**	***Analytical precision**
Sucrose (%)	59.5	59.4	±1.1
Brix (°Bx)	65.0	65.0	±0.9
Gravity purity (%)	91.6	91.4	±2.5
Conductivity ash (%)	0.9	0.9	±0.2
Fructose (%)	0.5	0.6	±0.3
Glucose (%)	0.7	0.6	±0.3
Colour [**ICUMSA units (IU)]	5755	5590	±440

*The analytical precisions for sucrose[1], brix[2], gravity purity[1] (using the Monte Carlo simulation technique), conductivity ash[1], fructose[1], glucose[1] and colour[2] were provided through personal communication.
** International Commission for Uniform methods of Sugar Analysis (ICUMSA)

CSPS seed quality

The quality results for the CSPS seed samples measured before each CSPS-seeded boiling are shown in Table 4. The average quality of the CSPS seeds met the specifications, apart from the size CV of the crystals, particularly Batch C. The size CVs of the seed in batches A and B were deemed acceptable for pan boiling, however, the size CV of the seed in Batch C was much higher than the target. As shown in Table 4, the average crystal number density varied between the batches [the seed used in CSPSa (i.e. Batch A) had the lowest crystal

[1] Personal communication with Stephen Walford (SMRI), 2020.
[2] Personal communication with Anayshree Naicker (SMRI), 2023.

number density and the seed used in CSPSc (i.e. Batch C) had the highest] and this corresponded inversely with the differences in the seed crystal sizes, suggesting the possible formation of new crystals, particularly in Batch C. Furthermore, the size CV of seed used in CSPSc was 75%, indicating that there was substantial deterioration of the seed quality over time. The photomicrograph of the seed used in CSPSc (see Appendix D) suggests that there may have been spontaneous nucleation which may explain why the average size CV of Batch C, stored for three days, was substantially higher compared with batches A and B which were stored for one and two days, respectively (Table 2). A possible cause for the spontaneous nucleation may have been the adverse storage conditions as a consequence of the low seed volume present in the storage vessel (approximately 1.4 l).

Although the size CV of seed used in CSPSc was unacceptable in terms of the required target, this arose because of false grain formation during storage and was addressed by appropriate washing. The boiling procedure included the option of washing of false grain after seed addition (see Appendix A) to deal with a situation such as this. A factory-scale implementation of CSPS seed production would not require seed storage of anywhere near the length of time required for these replicate boilings. It is envisaged that the application of good engineering practices can prevent the occurrence of adverse seed storage conditions and that the washing of false grain formed in the seed would only be required in exceptional circumstances.

Table 4. CSPS seed quality results of seed batches removed from the 12 l storage vessel for pan boiling

Seed quality parameter	**Target**	**Batch A**	**Batch B**	**Batch C**
Crystal size (µm)	>50	154	138	126
Size CV (%)	<50	51	52	75
Crystal number density (crystals/ml)	7.0×10^4 to 2.0×10^6	7.8×10^4	9.1×10^4	*1.0×10^5
Boiling for which seed was used		CSPSa	CSPSb	CSPSc

*The seed and Nutsch samples for the seed used in CSPSc were not collected and therefore the crystal number density was not determined using the alternative crystal number density technique. The crystal number density for the seed used for CSPSc was determined by extrapolation using the crystal size and crystal number density values for the seed samples used for CSPSa and CSPSb.

A few conglomerates (i.e. crystals that consist of three or more crystals that are fused together) and some agglomerates (i.e. crystals clinging to one another) were observed in the CSPS seed (Appendix D). It was expected that the agglomerates would separate during the CSPS-seeded pan boilings; however, the crystals forming conglomerates would not be expected to separate as these crystals are strongly fused with one another. The conglomerates may have formed during storage and a possible cause could be suboptimal mixing in the storage vessel. Conglomerate formation would be expected particularly in pure solutions where circulation and supersaturation control are poor (Rein, 2007). An assessment of the CRI of the sugars obtained from the pilot pan boilings could indicate whether or not the level of conglomerates in the seed may have been a problem (this will be detailed in the section that follows). The level of conglomeration and agglomeration in the CSPS seed may be reduced by improving the circulation within the seed storage vessel.

Comparison of pan boiling performances

Tables 5 and 6 summarise the average results obtained from the slurry-seeded and CSPS-seeded boilings, respectively. Furthermore, the targets for each performance parameter

(Rahiman *et al*. 2023) and the Relative Standard Deviations (RSD) for each boiling group are reported.

Table 5. Summary of the slurry-seeded pan boiling results

Parameter	Target	SSa	SSb	SSc	Average (RSD; %)
Final massecuite brix (°Bx)	90-92	89.9	91.2	92.2	91.1 (1.27)
Yield (%)	≥40	41	44	49	45 (9.05)
MA (µm)	≥600	610	670	750	677 (10.38)
CV (%)	≤35	32	32	30	31 (3.69)
CRI (%)	≥30	39	39	22	33 (29.44)
Colour transfer (%)	≤5%	2.2	1.9	2.0	2.0 (7.51)
Overall linear growth rate (µm/h)	≥140	78	101	110	96 (17.13)

Table 6. Summary of the CSPS-seeded pan boiling results

Parameter	Target	CSPSa	CSPSb	CSPSc	Average (RSD; %)
Final massecuite brix (°Bx)	90-92	88.7	90.2	89.7	89.5 (0.85)
Yield (%)	≥40	34	45	41	40 (13.92)
MA (µm)	≥600	910	980	750	880 (13.40)
CV (%)	≤35	32	33	33	33 (1.77)
CRI (%)	≥30	36	37	38	37 (2.70)
Colour transfer (%)	≤5%	3.4	3.8	2.4	3.2 (22.53)
Overall linear growth rate (µm/h)	≥140	99	112	209	140 (42.93)

Final massecuite brix and pan yield

The average massecuite brix obtained in the slurry-seeded boiling group (Table 5) was within the target range; however, the average brix in the CSPS-seeded boiling group was lower than the minimum target value of 90°Bx (Table 6). A brix balance and a visual inspection of the boiling massecuite are used to determine when the massecuite is ready for discharge and therefore some variation may be expected, particularly if the pan boiler is concerned that the massecuite may not discharge from the pan. However, there was no significant difference between the average slurry-seeded and CSPS-seeded massecuite brix values (p value = 0.124).

The average pan yields obtained from the respective boiling groups met the target specification (≥40%); however, the average yield for the CSPS-seeded boilings was five units lower than for the slurry-seeded boilings. Pan yield is strongly affected by the final massecuite brix (i.e. increasing the massecuite brix generally increases the pan yield) and therefore it was expected that the CSPS-seeded boilings would have a lower pan yield. However, there was no significant difference between the pan yield from both boiling groups (p value = 0.312).

Mean aperture (MA), crystal regularity index (CRI) and coefficient of Variation (CV)

As shown in Tables 5 and 6, the MAs of the sugars produced in both pan boiling groups were above the minimum acceptable size (600 µm). The average sugar MA obtained for the CSPS-seeded boiling group was substantially larger than for the slurry-seeded boiling group, although they were not found to be statistically different (p value = 0.062), probably on account of the magnitude of the RSDs (13.40% for the CSPS boiling group and 10.38% for the slurry-seeded group). The large range of MAs for the CSPS group for only three boilings would have influenced this, especially the MA of 750 µm for the CSPSc boiling compared to those of 910 µm and 980 µm for the CSPSa and CSPSb boilings, respectively The large MAs of the CSPS boilings were expected as the actual average seed crystal number density (9.0 x 10^4 crystals/ml) for CSPSa and CSPSb was lower than the assumed value (1.5 x 10^5 crystals/ml) used to determine the volume of seed required (see Table 4). In practice, these differences would be managed by appropriate adjustment of the controlled variables; hence, the large MAs obtained in CSPS-seeded boilings were not flagged as a concern.

The average size CVs for the sugars obtained from the slurry-seeded and CSPS-seeded boiling groups were acceptable (≤35%). On average, the size CV of the slurry-seeded sugar was slightly better than the CSPS-seeded sugar (also seen in Appendix E), however, there was no significant difference in the size CV between the respective boiling groups (p value = 0.148). According to Rein (2007), generally, and all things being equal, a lower CV sugar should be achieved if crystals have a large MA. Therefore, CSPSa and CSPSb would be expected to have low CVs, as these boilings had the highest MA values. However, the MA of the sugar from CSPSc was similar to that produced in the slurry-seeded boiling group and was acceptable (33%), despite starting with a CSPS seed CV of 75%. This result shows that sugar produced from the CSPS seed can match the size uniformity of sugar produced from slurry-seeding. The practical implication of using CSPS seed is that the seed is immediately visible after its addition to the pan, unlike slurry crystals, which may survive or dissolve, depending on the graining conditions. Furthermore, it is expected that the use of CSPS seed would result in shorter boiling times compared to slurry-seeded massecuites as the seed crystal size (about 100 µm) is substantially larger than the crystal size of slurry (approximately 5 µm).

The average sugar CRIs for the slurry-seeded and CSPS-seeded boiling groups were acceptable (≥30%). The average CRI of the sugars from the CSPS-seeded boiling was four units greater than the average CRI of the sugars from the slurry-seeded boiling group. This indicates that on average there was a lower level of conglomerates in the sugars obtained from the CSPS-seeded boiling when compared to the slurry-seeded boilings. The sugar from the SSc boiling had a substantially higher quantity of conglomerates (CRI = 22%) compared to the sugar from the other slurry-seeded boilings (Table 5) and therefore may explain some of the above-mentioned difference in the average CRIs between the boiling groups. Conglomerate formation is typically a problem in refinery pans, forming under high supersaturation conditions (>1.2 supersaturation coefficient) and where circulation is poor. It was observed that the concentration control of the mother liquor, via boiling point elevation (BPE) control, was suboptimal during the tightening phase of the massecuite which lasted for approximately one hour (Figure 4 - see area encircled by a red ring). This event may have contributed to the high level of conglomerate formation in SSc. However, there was no significant difference in the average CRIs between the two boiling groups (p value = 0.543).

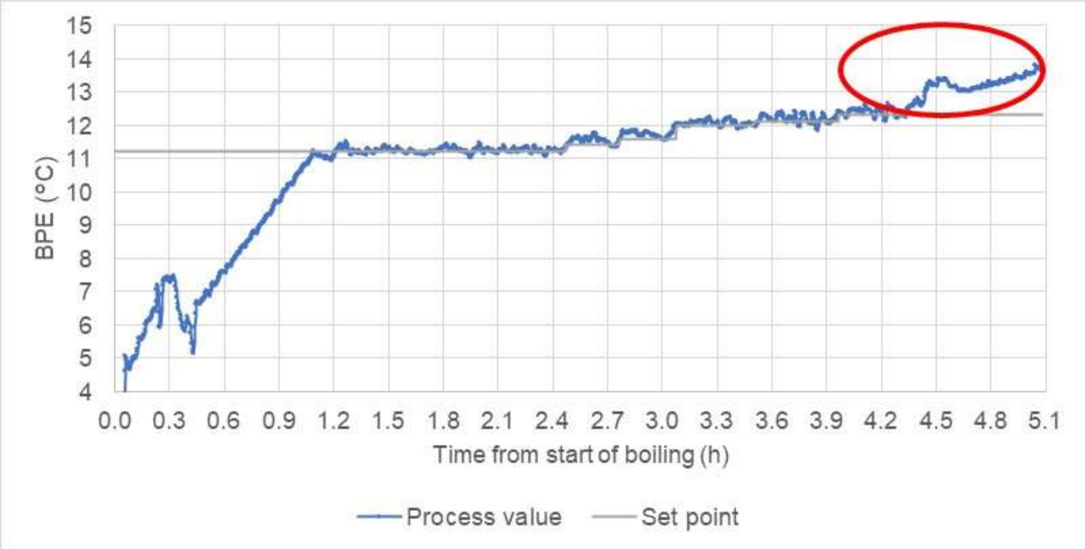

Figure 4. BPE elevation control during the SSc boiling

Colour transfer

Although the affinated sugar colour transfer for the sugars obtained from the CSPS-seeded boiling group was on average significantly higher (1.2 units, p value = 0.042) than that for the sugars from the slurry-seeded boiling group, the average affinated sugar colour transfers for all boilings were acceptable (≤5%).

Crystal linear growth rate

The average overall linear growth rate for the slurry-seeded boiling group (96 µm/h) was below the minimum acceptable value of 140 µm/h (Rahiman *et al*., 2023) whereas the average crystal growth rate for the CSPS-seeded boilings (140 µm/h) was acceptable. The overall crystal growth rate was substantially higher in CSPSc compared to the other CSPS-seeded boilings (Table 6), and this was attributed mainly to the improved circulation in CSPSc. The massecuite volume in CSPSc was 39 l instead of 75 l (as per procedure) and therefore it would be expected that the circulation would be better as the boiling level was closer to the calandria in CSPSc. Hence, the overall crystal growth rate obtained in CSPSc inflated the average crystal growth rate obtained in the CSPS-seeded boilings, giving a false impression of an improvement in crystal growth rate when CSPS seed was used. If the overall crystal growth rate of CSPSc is excluded, the average crystal growth rate for the CSPS-seeded boiling was 105 µm/h, and this is slightly higher than the crystal growth rate obtained in the slurry seeded boilings (96 µm/h). However, there was no significant difference in the overall linear growth rate between the two boiling groups (p value = 0.298). The overall linear growth rate results for both boiling groups suggest that there is room for improvement in the boiling recipe and this may be achieved by improving the mother liquor concentration control and/or circulation during the boiling.

Assuming (a) a final product crystal size of about 600 µm and an average growth rate of 100 µm/h (Rein, 2007) for the slurry-seeded and CSPS-seeded boilings and (b) that the crystal size of CSPS-seed is at least 100 µm (see Table 4) and the mean crystal size of ball-milled slurry is 5 µm (Rahiman *et al.*, 2012), the use of CSPS-seed would be expected to reduce the slurry-seeded boiling time by approximately 16% as the CSPS seed is 20 times larger than the slurry crystals. This excludes the additional time-saving required for the establishment of the grain by slurry-seeding. A techno-economic report that details the quantitative (additional sugar made and energy savings) and qualitative benefits of using CSPS-seed is currently being compiled by the SMRI. The techno-economic report will assist with decision-making regarding further development of the CSPS technology.

Conclusions

Sugars of acceptable quality (MA and size CV) were obtained in the CSPS-seeded boilings compared to industry targets, particularly when the improved CSPS pan boiling recipe was applied. The average sugar MAs for the slurry-seeded (650 µm) and CSPS (880 µm) boilings were above the minimum acceptable size of 600 µm, although there was a substantial difference between the CSPS and the slurry-seeded boiling groups. The main reason for this was that the crystal number density of the seed used in CSPS-seeded boilings was on average approximately 1.6 times less than what was assumed to determine the volume of CSPS seed required, but this was not flagged as a concern. The average sugar size CV values for the slurry-seeded and CSPS-seeded boiling groups were acceptable (<35%) and not significantly different. Average sugar size CVs of 31% and 33%, respectively, were obtained from the slurry-seeded and CSPS-seeded boiling groups.

Other performance indicators such as the pan yield, sugar CRI, and colour transfer were acceptable for both the slurry-seeded and CSPS-seeded boilings when compared to the respective targets. The overall growth rates obtained for both the slurry-seeded and CSPS-seeded boilings suggests that there is room for improvement, particularly concerning mother liquor supersaturation control and circulation, as the results obtained were generally lower than the benchmark growth rate of 140 µm/h. The practical advantage of using the CSPS seed is that the crystals are immediately visible in the pan after they are added, unlike slurry crystals, which may survive or dissolve because of the sensitivity to the graining point and circulation conditions in the pan. Therefore, it is expected that CSPS seed would assist pan boilers to approach full-seeding (i.e. only the seed crystals that are added grow without crystal dissolution nor false grain formation) more closely when compared to using slurry. Furthermore, it is expected that replacing slurry with CSPS seed will reduce pan boiling time as the CSPS seed crystals (approximately 100 µm) are about 20 times larger in size and 8 000 times larger in mass than slurry crystals (about 5 µm). There will also be a reduction in water and steam usage due to the reduced time spent holding on water in the early stages of crystal growth.

The SMRI is currently evaluating the techno-economics of the CSPS and considering the further development of this technology.

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APPENDIX A

The slurry-seeded and improved boiling procedures used for the CSPS-seeded boilings.

Slurry-seeded boiling procedure

(1) Initial charge phase:
- Weighed 155 kg 65°Bx refinery jet.
- 72 kg graining charge
  - 4 ml of antifoam added to graining charge.

(2) Brixing up phase:
- Operating vacuum pressure controlled at 30 kPa (abs).
- Evaporation rate: 13-15 kg/h.

(3) Holding and graining phase:
- 11.0-11.2 graining BPE set point.
- 5-8 min to see grain.
- 30 min for grain to be established.

(4) Feeding phase:
- 11.2-12.3 BPE set point range during feeding.
- Evaporation rate at start: 10-13 kg/h
  - Feed set point changed as level increased such that the existing crystals grew with little/no new crystal formation.

5) Tightening and strike phase:
- 90-92°Bx target
  - Determine using condensate mass.
- Visual observation of massecuite tightness.

Improved boiling procedure

(1) Initial charge phase:
- Weighed 155 kg 65°Bx refinery jet.
- 72 kg graining charge
  - 4 ml of antifoam added to graining charge.

(2) Brixing up phase:
- Operating vacuum pressure controlled at 30 kPa (abs).
- Evaporation rate: 10-15 kg/h.

(3) Holding and seeding phase:
- Seed at a BPE of 10.8°C.
- Wash out any false grain.
- 30-45 min for grain to be established whilst on water (BPE ramped to 11.2°C).

(4) Feeding phase:
- 11.2-12.3 BPE set point range during feeding.
  - Evaporation rate at start: 10 kg/h
  - Feed set point changed as level increased such that the existing crystals grew with little/no new crystal formation.

5) Tightening and strike phase:
- 90-92°Bx target
  - Determine using condensate mass.
- Visual observation of massecuite tightness.

APPENDIX B

The mass of a single sucrose crystal may be determined by using Equation 1 (Bubnik and Kadlec, 1992).

$$M_{crystal} = \propto \times \rho \times L_s^3 \text{ … Equation 1}$$

Where $M_{crystal}$ is the mass of a crystal (g), ∝ is the volume shape factor, ρ is the density of the sugar crystal (g/cm^3) and L_s is the average sieve aperture size (cm). Bubnik and Kadlec (1992) report 0.75 and 1.587 g/cm^3 for the volume shape factor and density of a sucrose crystal, respectively.

The number of crystals, $N_{crystal}$, may be determined by dividing the mass of crystals of a specific size by the mass of a single crystal (Equation 2).

$$N_{crystal} = \frac{x_{crystal} \times CC \times M_{masc}}{M_{crystal} \times 10} \text{… Equation 2}$$

Where $x_{crystal}$ is the mass percent of the crystals of a specific average aperture size, CC is the crystal content (%) and M_{masc} is the mass of massecuite (kg).

The sieve set used in the sieve analysis of the refined sugar included 850, 600, 500 and 350 µm sieves, followed by the pan. The standard consecutive size above the 850 µm sieve was the 1000 µm sieve. Furthermore, the refined sugar produced in the pilot pan boilings was typically affinated using saturated alcohol and a screen with a slot size of approximately 60 µm. The largest and smallest aperture sizes were assumed to be 1000 µm and 60 µm, respectively. These assumptions allowed for calculation of the average sizes on the 850 µm sieve and the pan.

The mass of massecuite may be determined using a brix balance (Equation 3).

$$M_{masc} = \frac{B_{feed} \times M_{feed}}{B_{masc}} \text{… Equation 3}$$

Where B_{feed} is the brix of the feed (°Bx), B_{masc} is the brix of the massecuite and M_{feed} is the mass of feed (kg). It was assumed that the brix input from slurry was negligible since only 0.6 mL of slurry was added into the pan.

The seed volume was determined using Equation 4.

$$V_{seed} = \frac{\sum N_{crystal}}{Crystal\ number\ density \times 1000} \text{ … Equation 4}$$

Where V_{seed} is the volume of seed (l), $\sum N_{crystals}$ is the total number of crystals on each sieve (including the collection pan) and the crystal number density is expressed in crystals/ml.

Table B1 shows the mass of massecuite calculated for SSb.

Table B1. Mass of massecuite for SSb

Variable	Unit	Value
M_{feed}	kg	151.50
B_{feed}	°Bx	65.25
B_{masc}	°Bx	91.45
M_{masc}	kg	108.10

The crystal content of the massecuite was determined to be 40.67%. Table B2 indicates the average aperture size (L_s) on each sieve, the mass percent of crystals ($x_{crystal}$) on each sieve, the number of crystals of a specific aperture size, the total number of crystals and the crystal number density.

Table B2. Determination of the volume seed based on the SSb sieve and crystalline mass data

Sieve size	**L_{sieve}**	**Mass on sieve**	**$x_{crystal}$**	**$M_{crystal}$**	**Mass of sugar of a specific size in the massecuite**	**$N_{crystal}$**
(µm)	**(µm)**	**(g)**	**(%)**	**(g)**	**(kg)**	**(crystals)**
850	925	19.75	19.40	9.42×10^{-4}	8.53	9.05×10^{6}
600	725	44.63	43.84	4.54×10^{-4}	19.27	4.25×10^{7}
500	550	16.22	15.93	1.98×10^{-4}	7.00	3.54×10^{7}
355	428	14.40	14.14	9.30×10^{-5}	6.22	6.69×10^{7}
Pan	208	6.81	6.69	1.06×10^{-5}	2.94	2.77×10^{8}
	Sum	**101.81**	**100.00**		**$\sum N_{crystal}$ (crystals)**	**4.31×10^{8}**
					Crystal number density (crystals/ml)	**1.53×10^{5}**
					Volume of CSPS seed to be added (l)	**2.81**

The volumes of seed determined from the data collected in SSa, SSb and SSc were 3.45, 2.81 and 1.85 l, respectively.

APPENDIX C

Figure C1 illustrates the photomicrographs of samples collected during the slurry-seeded boilings. The times (hour) indicated are from the start of slurry-seeding.

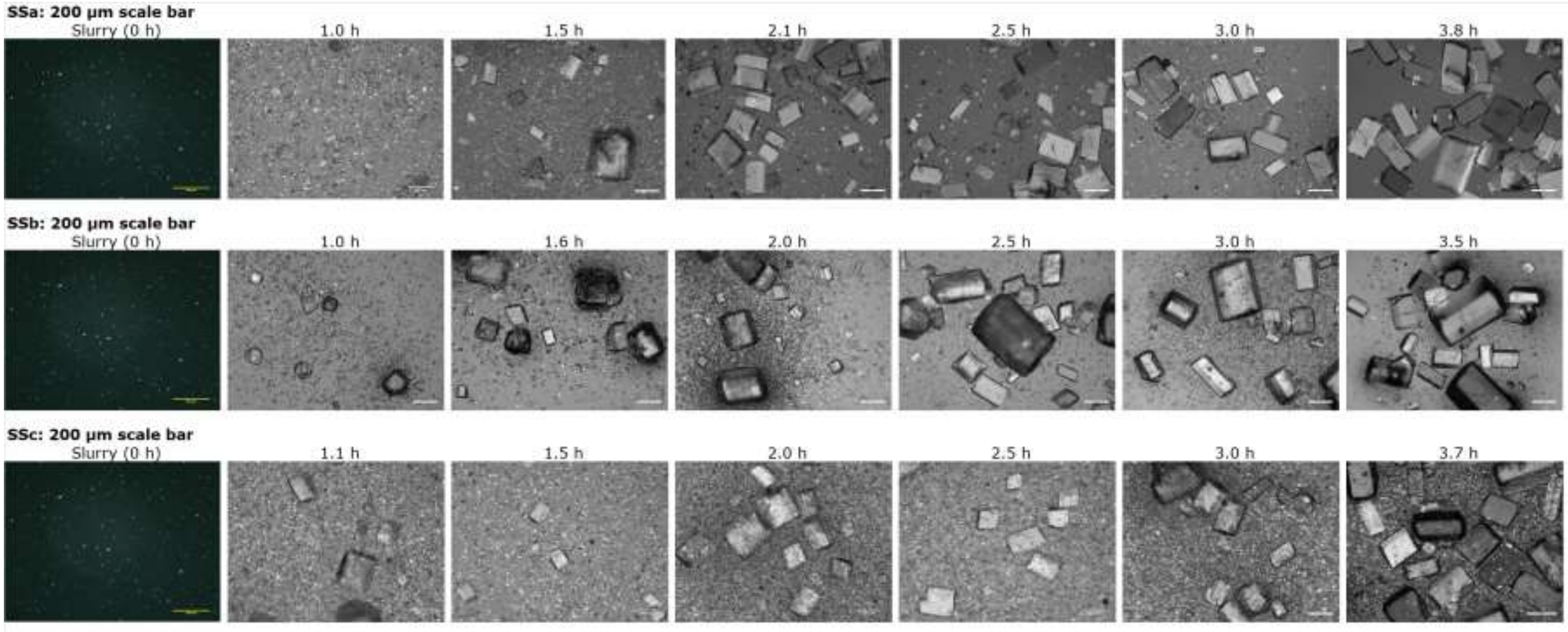


Figure C1. Crystal growth during the slurry-seeded boilings

Figure C2 illustrates the photomicrographs of samples collected during the CSPS-seeded boilings. The times (hour) indicated are from the start of CSPS-seeding. The photomicrographs enclosed by a red rectangle show the false grain formed during the pan boiling.

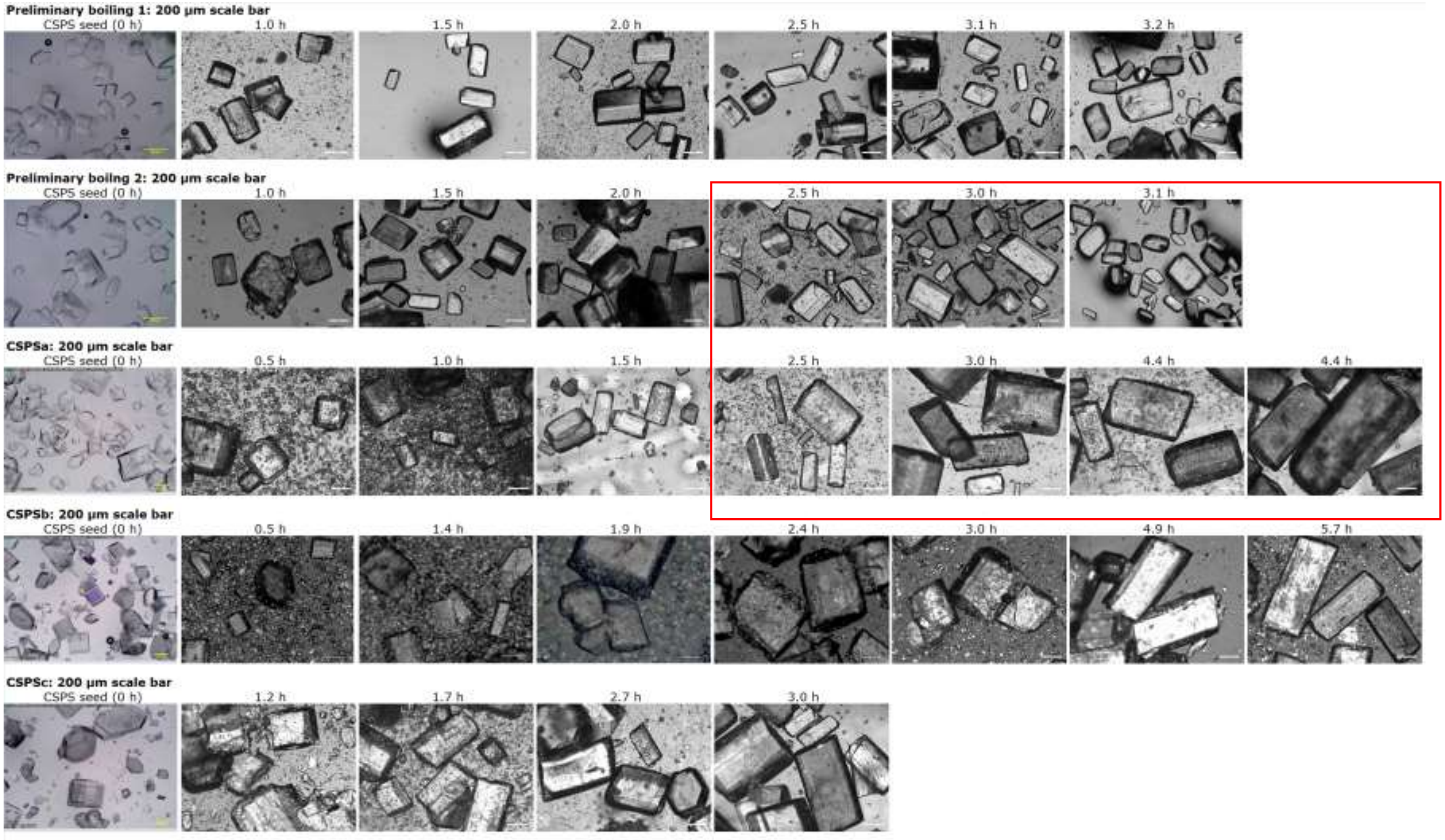


Figure C2. Crystal growth during the CSPS-seeded boiling

APPENDIX D

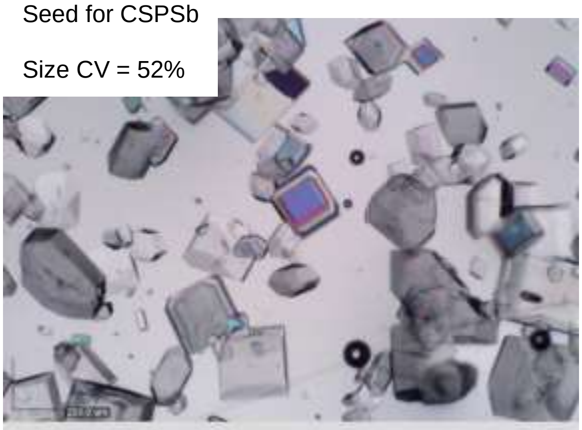
The photomicrographs of the CSPS seeds used before each CSPS-seeded boiling are shown below.

Seed for CSPSa

Size CV = 51%

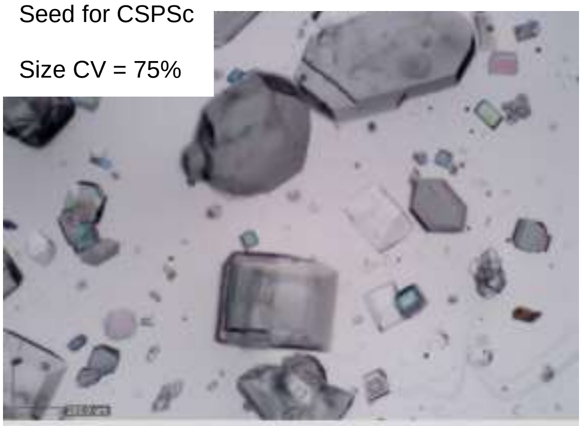
Seed for CSPSb

Size CV = 52%



Seed for CSPSc

Size CV = 75%



APPENDIX E

Figure E1 illustrates the gamma distribution probability curve for the sugar produced from the slurry-seeded boilings.

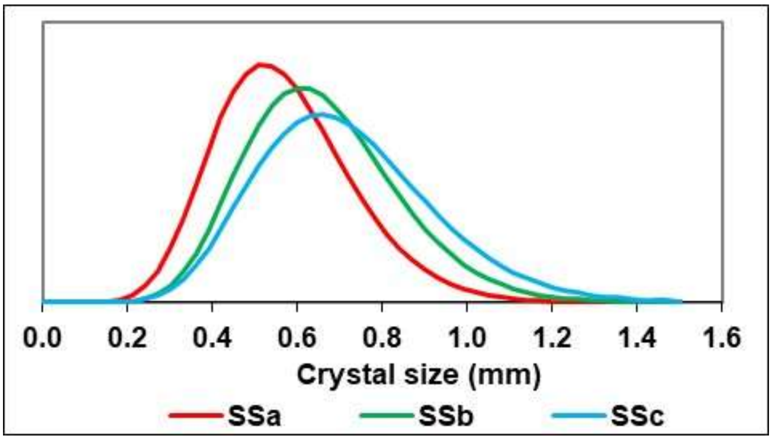


Figure E1. Mass-based gamma distribution probability curves for the slurry-seeded sugars

Figure E2 illustrates the gamma distribution probability curve for the sugar produced from the slurry-seeded boilings.

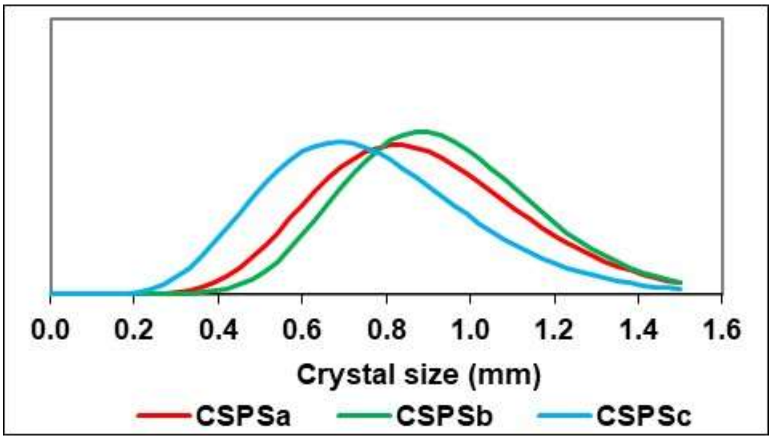


Figure E2. Mass-based gamma distribution probability curves for the CSPS-seeded sugars