Crystallization of beet sugar

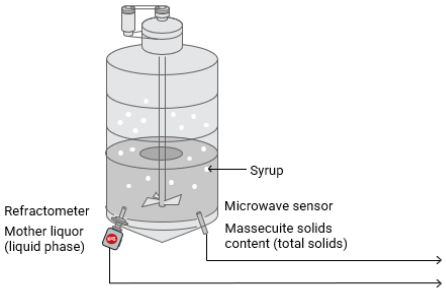
Supersaturation, mother liquor concentration

Benefits of R.I. measurement

The KxS DCM-20 refractometer enables beet sugar factories to transition toward fully predictive, supersaturation-based crystallization control—unlocking significant efficiency gains and quality improvements.

When supersaturation becomes measurable in real time:

- strikes become faster and more stable
- MA increases and CV narrows
- product quality improves,
- steam consumption decreases
- model-based contradictions are eliminated
- and process control becomes scientific rather than experiential



DCS implemented supersaturation calculation

Supersaturation function = f (C, Q, T, m, b, c)

C : syrup / mother liquor concentration (%)
Q : syrup / mother liquor purity (%)
T : temperature (C)
m, b, c : syrup quality parameters ()

Process description

In beet sugar production, crystallization takes place in batch vacuum pans where thick juice is concentrated by boiling under reduced pressure. Once the syrup reaches the appropriate concentration, the massecuite is seeded with fine sugar crystals that provide the nuclei for larger crystals to grow. As the strike progresses, sucrose crystallizes out of the mother liquor until the desired crystal size is achieved. The resulting *massecuite*—a mixture of crystals and syrup—is discharged and spun in centrifuges to separate the sugar from the mother liquor. The crystals are then washed, dried, cooled, and conveyed to storage silos.

Overview of crystallization and Supersaturation

Beet sugar crystallization is a delicate and highly dynamic process where small variations in operating conditions can have significant consequences on product quality, energy consumption, and overall efficiency. The operational objective is to produce uniform sugar crystals with a narrow particle size distribution while minimizing strike time, steam consumption, and recycle ratio. These goals require precise control of *Supersaturation*, the fundamental driving force behind crystallization, yet most industrial crystallizers operate without access to reliable Supersaturation data.

Instead, refineries rely heavily on:

- massecuite Brix (microwave or nuclear sensors)
- vacuum and temperature readings
- steam, and feed valve positions
- historical operating practice
- manual observation
- operator intuition.

The result is variability. Even the same operator may run a strike differently depending on shift conditions, fatigue, or perceived pan behavior.

A central challenge in industrial crystallization is that supersaturation cannot be inferred from any single measurement.

Supersaturation depends simultaneously on:

- dissolved sucrose concentration in the mother liquor, and
- the fraction of undissolved crystals in the massecuite.

As crystallization progresses, the massecuite transitions from a homogeneous liquid to a dense, multiphase system with rising solids content, shifting dielectric properties, non-uniform temperatures, and declining circulation. Under these conditions, no single sensor can characterize the entire crystallization environment.

Accurate supersaturation calculations require two complementary measurements:

- **Mother liquor concentration** (liquid-phase Brix) – via refractometer
- **Massecuite solids content** (total solids: dissolved + undissolved sugar) – via microwave sensor

These variables behave differently and must be measured separately.

Mother liquor concentration reflects the **sucrose dissolved in the liquid phase**. This parameter requires a measurement technology that selectively measures the liquid phase. A DCM-20 digital process refractometer uniquely fulfills this role: KxS DCM-20 is the only refractometer on the market proven to remain reliable and immune to crystal interference under real crystallization conditions, owing to its advanced optical image detection combined with a proprietary pattern-recognition algorithm specifically developed for distinguishing liquid-phase reflections from scattering caused by suspended crystals.

Massecuite solids meter measures the **total solids (liquid + crystals) content** in the system, based on changes in dielectric properties. It complements the refractometer by quantifying solids loading.

Why both measurements are needed

Before seeding, both instruments show the same Brix value (pure liquid).

After seeding, they diverge:

- The **refractometer** remains stable, reflecting true mother liquor concentration.

- The **microwave meter** increases steadily as crystal content grows.

The difference between these two signals contains essential information about the strike's evolution, growth rate, and solids loading.

Customers may combine the DCM-20 refractometer with their existing microwave meters for massecuite solids control. KxS supports the implementation of supersaturation formulas and DCS integration.

Scientific basis of Supersaturation

Supersaturation (SS) is defined as:
SS = C_actual / C_saturation(T)

where:

- **C_actual** is the concentration of sucrose in the mother liquor
- **C_saturation(T)** is the saturation concentration at the same temperature

Crystallization only occurs when **SS > 1.0**, and the magnitude of supersaturation determines whether the dominant process is nucleation, crystal growth, dissolution, or agglomeration.

Supersaturation is a multivariable function: **SS = f(C, P, T, m, b, c)**

where:

- **C** = mother liquor concentration
- **P** = purity of the mother liquor
- **T** = temperature
- **m, b, c** = syrup quality parameters affecting solubility

This multivariable nature means that supersaturation cannot be reliably inferred from single-phase measurements such as massecuite Brix or density. These measurements integrate contributions from both the crystal and liquid phases and are highly sensitive to viscosity, mixing conditions, and crystal content.

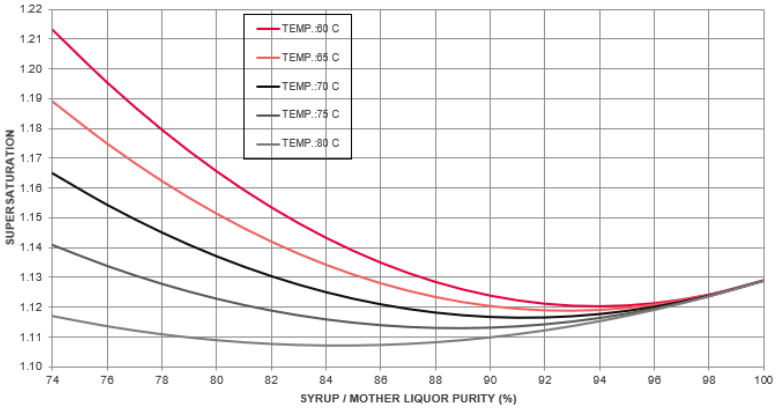


Figure 1. Critical SS limits to start nucleation and to avoid unwanted nucleation. Source: M.Saska: Boiling point elevation of technical sugarcane solutions and its use in automatic pan boiling. International Sugar Journal 2002, VOL.104, No.:1247

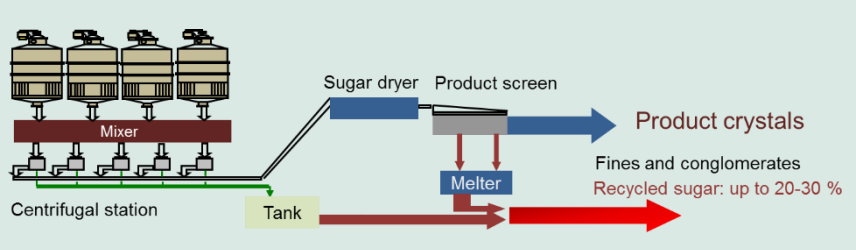


Figure 2. All sugar produced in crystallization station goes through screens which separate fine and large crystals from the product. The rejected fines and large crystals are melted and recycled back to crystallizers. Source: L. Rozsa, J. Rozsa, S. Kilpinen, E. Mielonen, M. Kivenheimo: On-line Monitoring of Crystallization Control Practices; SIT Annual Technology Meeting 2016, New York

Accurate supersaturation calculation requires:

- Selective liquid-phase concentration measurement
- Precise temperature measurement
- Appropriate purity and solubility correlations
- Massequite solids content

The metastable zone in crystallization is narrow, and even small deviations can trigger nucleation (Figure 1).

Consequences of Supersaturation deviations

In general, high supersaturation means faster crystal growth and vice-versa. The range of control for supersaturation is very narrow. If supersaturation is too high, the crystals form either small fines or stick together into conglomerates, both of which are unwanted in the final product, and need to be melted, concentrated, recycled and re-crystallized.

SS too low (SS < ~1.05–1.10)

When supersaturation is insufficient to drive crystal growth, mass transfer slows dramatically, resulting in:

- Reduced growth rate
- Partial crystal dissolution
- Extended strike time caused by slow mass deposition
- Increased steam consumption associated with prolonged boiling

SS too high (SS > ~1.15)

When supersaturation exceeds the metastable zone, spontaneous nucleation occurs, causing:

- Formation of fines and conglomerates
- Poor crystal size distribution
- Higher recycling ratio

In some cases, up to 30 % of sugar crystals are rejected and must be remelted and reprocessed, causing waste of time and energy (Figure 2).

Crystal quality parameters: MA and CV

Product quality in beet sugar is often evaluated using two key population parameters:

Mean Aperture (MA)

MA describes the average size of sugar crystals. High MA indicates large crystals; low MA indicates small, slow-growing, or dissolved crystals.

Coefficient of Variation (CV)

CV is a measure of the width of the crystal size distribution.

Low CV = narrow distribution (desired)
High CV = broad or variable distribution (undesired)

Crystals reflect the history of the strike; every supersaturation excursion leaves a permanent signature on MA and CV. This makes supersaturation control central not only to process efficiency but to final product quality.

Real-time Supersaturation measurement with KxS DCM-20 digital refractometry

The KxS DCM-20 refractometer measures the refractive index of the mother liquor—selectively and continuously.

Advantages of digital refractometry

- High accuracy (±0.1% concentration or better)
- Fast response suitable for dynamic control
- Drift-free operation
- Fully selective to the liquid phase
- Reliable over entire strike
- Immune to crystal content

These characteristics make it uniquely suited to supersaturation calculation.

Supersaturation values are computed in the DCS, combining refractometer concentration, temperature, and known solubility functions. Factories may integrate existing microwave instruments to assist in level control and endpoint determination.

Seeding practices

Seeding establishes the initial crystal population and directly influences MA, CV, and overall crystal morphology.

Full seeding: Introduces the complete crystal population at once.

- Best performed at SS = 1.08–1.10
- Requires well-prepared slurry or footing magma
- Achieves predictable population density and CSD
- Minimizes spontaneous nucleation when SS is controlled

Shock seeding: Supersaturation is briefly driven above the nucleation limit to produce new nuclei.

- Highly sensitive to exact timing and magnitude of SS spike
- Generates many small crystals (higher CV)
- Less predictable and more operator-dependent

Regardless of method, Supersaturation must be visible to ensure seeding is executed in the correct thermodynamic region.

Installation in crystallizer

Because mother liquor temperature and concentration gradients increase as crystal content rises, the measurement location significantly influences data quality. In industrial crystallizers, three installation positions have been proven suitable: above the calandria, below the calandria, and at the bottom of the vessel (see Figure 3).

The appropriate location depends on the crystallizer's circulation pattern and crystallizer design and syrup inlet position.

A counter flange adapter is recommended to be used to eliminate dead pockets where massecuite could remain between strikes. Such dead zones can crystallize independently, contaminating subsequent measurements and introducing bias into supersaturation calculations

For best performance, the counter flange should be welded directly to the pan wall—without a weld-neck extension. This ensures that the sensor is positioned deeper inside the vessel, rather than recessed in a side pocket or dead space, allowing the prism to reach the liquid and guaranteeing full flow across the prism surface for representative measurement.

Figure 3. Three installation positions: Above calandria, below calandria, and bottom.