**Typical end products**
- Crystallized sugar.

**Chemical curve:** R.I. per BRIX at Ref. Temp. of 20˚C

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**Introduction**

The crystallization process takes place in vacuum pans, which boil the thick juice. When the juice reaches the correct concentration, it is seeded with sugar crystals, which provide the nucleus for larger crystals to grow. When the crystals reach the required size, the process is stopped and the resultant mixture of crystal sugar and syrup, known as massecuite, is spun in centrifuges to separate the sugar from the mother liquor. The sugar crystals are washed and, after drying and cooling, conveyed to storage silos.

**Application**

Crystallization has a major effect on product quality and production costs. Supersaturation is the driving force of crystallization and crystal growth, and the speed of crystallization depends on this parameter.

Too low supersaturation increases the strike time. Too high supersaturation results in poor crystal quality (fines and conglomerates). These crystals are melted, concentrated, recirculated and crystallized again, which wastes time and energy and decreases the yield of sugar per strike, while increasing water usage and production costs. To decrease the amount of recycled sugar the particle size and particle size distribution must be as close to the target values as possible.

Besides supersaturation, the other important parameters are: crystal content, mother liquor purity and massecuite solids content. Instead of using a single probe as the main instrument, a measurement of the liquid phase (syrup and mother liquor concentration) as well as a measurement of the syrup and massecuite solids content, are required for successful control of the supersaturation.

**Control of crystallization**

Crystallization can only take place if the solution is supersaturated. Supersaturation is a multivariable function of several parameters of the liquid phase only (syrup or mother liquor concentration) as well as a measurement of the syrup and massecuite solids content, are required for successful control of the supersaturation.

No single instrument can provide on-line data on supersaturation. Conventional sensors used to monitor crystallization provide data of a single massecuite parameter only.
However, two sensors can provide information on the important massecuite parameters. These sensors are not influenced by other process parameters:

1. Process Refractometer. The refractive index technology is successfully used for selective concentration measurement of the liquid phase over the complete crystallization strike.

2. A sensor for measurement of the total solids content (brix of the massecuite).

**Instrumentation and installation**

Vaisala K-PATENTS® SeedMaster SM-3 is a unique third generation crystallization transmitter and seeding device to be used with the Process Refractometer. The SM-3 allows for accurate in-line and real-time monitoring of supersaturation and crystal content over the complete process of crystallization, and implementation and control of automatic or manual seeding. The SM-3 can be connected to one or two Process Refractometers and to one or two crystallizers. The SeedMaster SM-3 provides the following tasks:

1. Electronic data capture on massecuite parameters.

2. On-line calculation and transmission of massecuite parameters for the advanced control of sugar crystallization with control system.

3. Organization and storage of strike history data archive.

4. Advanced communication with the control system.

5. Automatic seeding of the vacuum pans.

6. Serves as user interface for the pan and control system operators.

The mounting location of the refractometer sensor should be carefully evaluated. Despite the use of stirrers in crystallizers, circulation of the massecuite becomes sluggish when the crystal content increases. This means that the syrup/mother liquor concentration and temperature, which have a considerable influence on supersaturation, will not be the same in the full massecuite volume. This is a limitation for all types of sensors.

In general, Vaisala K-PATENTS® PR-23-GP refractometer should be installed in a location where the measured sample is representative for the largest volume of the syrup or massecuite (in terms of sugar content and temperature). The preferred installation position is under the calandria using a counter flange adapter for process connection to minimize dead space around the flange. This is critical for the product quality as the leftovers may grow from batch to batch resulting in a negative effect on the product quality and in controlling the strike (see Figure 1).

**Figure 1. Recommended sensor locations.**

<table>
<thead>
<tr>
<th>Instrumentation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>SeedMaster SM-3 for multiparameter sugar crystallization monitoring and automatic seeding.</strong></td>
<td>Crystallization transmitter and seeding device is used with the Process Refractometer PR-23-GP. It allows for accurate in-line and real-time monitoring of supersaturation and crystal content over the complete strike, and implementation and control of automatic or manual seeding.</td>
</tr>
<tr>
<td><strong>Process Refractometer PR-23-GP</strong></td>
<td>is an industrial refractometer for crystallizers. Installation through a flange connection and Counter flange adapter -AP for vacuum pan installations.</td>
</tr>
<tr>
<td><strong>Prism wash system with warm water</strong></td>
<td>The integral prism wash system helps to avoid crystals deposit or scaling on the prism surface. Prism wash system components are a refractometer with integral water wash nozzle mounted at the refractometer probe, a warm feed water source (hot condensate), and an indicating transmitter with built-in relays for driving the water valve and controlling the wash.</td>
</tr>
<tr>
<td><strong>Measurement range</strong></td>
<td>Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.</td>
</tr>
</tbody>
</table>
BEET SUGAR, CANE SUGAR

Introduction

This note explains the methods and calibration procedure for measuring successfully the massecuite solids content and mother liquor concentration in sugar vacuum pans over the entire crystallization process. Both parameters need to be measured and controlled, as they influence the quality of the sugar crystals.

Massecuite solids content, or total sugar content, is typically determined using e.g. microwave measuring technology whereas mother liquor concentration (dissolved sugar) is measured with a refractometer. The common measurement scale is Brix.

Refractometer

Vaisala K-PATENTS® Process Refractometer is successfully used for selective measurement of liquid phase over the complete crystallization strike. Due to the unique digital principle, the refractometer measures the true concentration of the mother liquor, without being influenced by the sugar crystals or bubbles in the pan. Moreover, the refractometer does not require re-calibration.

Massecuite solids content meter

A microwave sensor can measure only the total solids (liquid and undissolved solids phase). Microwave probes are based on the measurement of attenuation and phase shift of microwave radiation. Both are related to the length travelled by the radiated signal, and the density and dielectric characteristics of the medium.

Phase shift is the result of decreasing speed of propagation. Due to the fact that water has a high dielectric constant compared to sugar and the accompanying non-sugars, the water content (and, consequently, solids content) is the major parameter, which determines the dielectric properties of the medium. As an output the microwave probes provide density or solids content of the massecuite.

Particularities of the crystallization process

Varying process conditions present a challenge to measuring massecuite solids content accurately. Process medium changes during different production phases from liquid to massecuite and consists of both liquid and crystals. Generally, calibrating the massecuite solids content meter can be quite easy either for the liquid phase or for massecuite phase, but not for both. This means that the massecuite solids content sensors cannot solely produce reliable results as they do not cover the whole processing range.

For accurate results, the calibration must cover the full range from pure liquid to the point where Vol. 55 % of the massecuite is crystals. However, if the vacuum pan is also equipped with a refractometer, the calibration difficulties can be mostly avoided.

In the beginning of the strike the process medium is pure liquid. At this point the refractometer and the massecuite solids content meter should give the same measurement value (Figure 1).

The crystals are introduced only after the pan has been seeded. After seeding the massecuite solids content increases as the crystal content increases, whereas refractometric concentration stays rather constant (±3 Brix). Improved accuracy in massecuite solids measurement can be achieved when a refractometer is combined with a microwave sensor. Moreover, a refractometer can offer calibration reference value...
for the massecuite solid content meter at the seeding point.

The recommended control practice is to use a refractometer to measure the concentration from the beginning of the strike until the seeding point. The refractometer provides accurate and repeatable seeding.

Calibration procedure

The Process Refractometer is factory calibrated according to International Commission for Uniform Methods of Sugar Analysis (ICUMSA) Brix table. The factory calibration should be verified against the production laboratory when commissioning the instrument. The laboratory reference values should be taken in the beginning of the strike when there is only liquid present in the vacuum pan. Only a small BIAS adjustment might be needed to match the refractometer and the production laboratory.

Typically, the massecuite solids content meter needs regular calibration and calibration checks. The best calibration result is typically achieved when the microwave density meter is calibrated on a narrow range for either liquid sugar or massecuite. In vacuum pans the recommended calibration practice is to calibrate the microwave density meter from seeding point to the end of the strike, which means that the reference samples should be taken at the seeding point, end of the strike and one sample in between (minimum three samples).

Table 1. Example of calibration table.

<table>
<thead>
<tr>
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</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>77.6</td>
<td>77.5</td>
<td>78.5</td>
<td>-0.1</td>
<td>0.0</td>
</tr>
<tr>
<td>2</td>
<td>79.5 (seeding)</td>
<td>79.4</td>
<td>80.9</td>
<td>-0.1</td>
<td>1.4</td>
</tr>
<tr>
<td>3</td>
<td>85.6</td>
<td>78.5</td>
<td>86.3</td>
<td>-</td>
<td>0.7</td>
</tr>
<tr>
<td>4</td>
<td>90.5</td>
<td>77.9</td>
<td>91.3</td>
<td>-</td>
<td>0.8</td>
</tr>
</tbody>
</table>

Table 2. Example of calibration table after calibration procedure. Refractometer offset adjustment +0.1 Massecuite solids meter offset adjustment -0.9.