THE EFFECT OF DEXTRAN AND INORGANIC SALTS ON THE CRYSTALLISATION OF SUGAR IN A LABORATORY VACUUM PAN

By

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Abstract
Bulk growth rate of sugar crystals in pure sucrose solution at 1.1 and 1.2 supersaturation were calculated at 54°C. It was observed that the growth rates doubled with the increase in supersaturation. The growth rates were determined in the presence of 1.0 g of dextran, starch, potassium chloride, potassium iodide and calcium chloride added one at a time. The growth rate was found to decrease with the addition of dextran, starch and potassium iodide and increase in the presence of potassium chloride. It was found not to vary in the presence of calcium chloride. The effect of these impurities on apparent viscosity was also determined. It was observed that impurities in general increased the viscosity, which was found to be substantial on the addition of dextran, starch and calcium chloride. The effect was, however, negligible on the addition of potassium chloride and potassium iodide. Conductance studies after crystallisation of sucrose solutions containing inorganic salts indicate that some amount of impurity has been carried away by the crystal during the process of crystallisation.

Introduction
There is still no general applicable theory that permits an accurate prediction of the effects of all the factors that govern the industrial process of crystallisation. However, crystal growth rates determined under controlled conditions can be used to predict rates in industrial crystallisers.

It is often observed that minute amounts of impurities exert a profound effect on the growth of crystals as they affect the solubility. Maraundi (1982) has pointed out that an impurity has an effect like lowering the temperature and decreases the overall growth rate, while others indicate that potassium chloride actually reduces the solubility of sucrose.

Mechanical harvesting has resulted in an increase in the quantity of bacterial polysaccharides like soluble dextran in the juice (Cuddihy and Donal, 1999). It causes sugar loss (Donal, 1994) and processing problems (James and Cameron, 1971). Mantovani et al. (1967) have reported that dextran hinders the process by adsorption on the glucose fructose portion of the molecule. Studying the rheological properties with these impurities, along with the growth rates, might monitor this hindrance. The conductance studies have also been undertaken to ascertain the amount of impurity removed by the crystal during crystallisation.

Materials and methods
All chemicals used were of S.D. fine make. Sucrose and potassium chloride were of analytical reagent grade and potassium iodide, calcium chloride, starch and dextran of laboratory reagent grade.

Rheological and conductance measurements
Rheological determinations were done with Brookfield HB DVIII programmable rheometer. (Brookfield Engg. Lab. Inc. Stoughton M.A.) using 304 Ultra Low Adapter. The temperature was controlled with a water jacket in which water was circulated at the required temperature from a Brookfield thermostatic bath (TC-500).

The conductance measurements were carried out with Conductivity Bridge of century instruments.

Viscosity was measured by the addition of 0.2 g of the impurity to 100 g of sucrose solution of 68.5 brix at 50°C. Conductance measurements of sugar solutions were carried out before and after crystallisation at 20°C.

Kinetics of crystallisation
The separation of sucrose from the impure solution was achieved by crystallisation in a 2 L batch vacuum pan (Figure 1) immersed in a water bath operating 10°C higher than the solution boiling point. The pan was initially part filled (about 30%) with sucrose (about 750 g) along with 1.0 g of the impurity. Double distilled water was added to make a total mass of around 1040 g.

Sucrose solution of the required supersaturation at 54°C was prepared by refluxing under vacuum at an approximate temperature of 70°C. The temperature was then reduced by increasing the vacuum. The system is now operating in the metastable zone. The seed material was prepared by sieving commercial granulated sucrose and separating the fraction between 14-28 mesh. The seed was washed with 1% aqueous methanol, pure methanol, dried and stored, to give clean seed surfaces.

Crystallisation was maintained at 54°C for sixty minutes and the massecuite centrifuged. The crystals were washed with methanol and oven dried at 70°C and weighed. Sampling for the brix using an Abbemat refractometer monitored the process.

KEYWORDS: Supersaturation, Apparent Viscosity, Crystal Growth Rate, Impurities, Solubility.
The supersaturation was maintained by adding unsaturated solution or water.

Results and discussion

Rheological and conductance measurements

Apparent viscosities of the sucrose solutions and conductivity data before and after crystallisation are displayed in Table 1. Apparent viscosity was found to increase with the addition of 0.2 g of dextran, starch and calcium chloride, while potassium chloride and potassium iodide had a lesser effect. It was found to be independent of the speed of the viscometer, thereby suggesting Newtonian behaviour of the solutions. Also conductance values of crystallised sugar solutions containing inorganic salts showed that the crystal during the process of crystallisation carried some amount of the impurity away.

Growth rate

The bulk growth rate of the crystals V is defined as the rate of deposition of mass, m, per unit area, A.

\[ V = \frac{1}{A} \frac{d m}{dt} \]  

The growth rate in pure sucrose solution at 54°C was determined at the supersaturation (S) of 1.1 and 1.2. The rate was found to double and had values of 2.8 and 4.1 g/m²/min respectively.

The effect on growth rate at S = 1.1 of the addition of 1.0 g of impurity is reported in Table 2. It decreased substantially on the addition of dextran and starch. Potassium chloride is found to favour the process of crystallisation as the rate was enhanced on its addition. Potassium iodide surprisingly was found to retard the process. This may be due to the effect of the large iodide ion. The presence of calcium chloride hardly brings about any change in the growth rate.

It may be concluded that impurities exert a profound effect on the crystallisation of sucrose. Large molecules like dextran and starch are found to retard the process of crystallisation, while the inorganic salts are in general found to help the crystal growth.

Acknowledgment

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### Table 1—Apparent viscosity and conductivity of 100 g sucrose solution of 68.5 brix containing 0.2 g of impurity.

<table>
<thead>
<tr>
<th>Impurity</th>
<th>Apparent viscosity (cP)</th>
<th>Conductivity (µ mhos)</th>
<th>Before</th>
<th>After</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure sucrose</td>
<td>52.5</td>
<td>1.0</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Dextran</td>
<td>62.5</td>
<td>1.5</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>Starch</td>
<td>71.7</td>
<td>0.8</td>
<td>0.6</td>
<td></td>
</tr>
<tr>
<td>Potassium chloride</td>
<td>59.1</td>
<td>40.8</td>
<td>5.4</td>
<td></td>
</tr>
<tr>
<td>Potassium iodide</td>
<td>59.1</td>
<td>13.5</td>
<td>2.1</td>
<td></td>
</tr>
<tr>
<td>Calcium chloride</td>
<td>67.2</td>
<td>48.8</td>
<td>6.2</td>
<td></td>
</tr>
</tbody>
</table>

### Table 2—Growth rate variation in the presence of impurity at S = 1.1.

<table>
<thead>
<tr>
<th>Impurity</th>
<th>Growth rate g/m²/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure sucrose</td>
<td>2.82</td>
</tr>
<tr>
<td>Dextran</td>
<td>1.10</td>
</tr>
<tr>
<td>Starch</td>
<td>1.21</td>
</tr>
<tr>
<td>Potassium chloride</td>
<td>3.21</td>
</tr>
<tr>
<td>Potassium iodide</td>
<td>1.68</td>
</tr>
<tr>
<td>Calcium chloride</td>
<td>2.26</td>
</tr>
</tbody>
</table>

REFERENCES


L’EFFET DU DEXTRANE ET DES SELS INORGANIQUES SUR LA CRISTALLISATION DU SUCRE DANS UN APPAREIL À CUIRE À ÉCHELLE DE LABORATOIRE

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Résumé
Le taux de croissance de la masse des cristaux de sucre dans une solution de saccharose pure à une supersaturation de 1.1 et 1.2 fut calculée à 54°C. Il fut observé que le taux de croissance doublait avec une augmentation de la supersaturation. Les taux de croissance furent déterminés en présence de 1.0 g de dextrane, d’amidon, de chlorure de potassium, d’iode de potassium et de chlorure de calcium, ajoutés l’un après l’autre. Le taux de croissance diminuait avec l’addition du dextrane, de l’amidon et de l’iode de potassium et augmentait en présence du chlorure de potassium. Le taux de croissance ne changeait pas en présence du chlorure de calcium. Les effets de ces impuretés sur la viscosité apparente furent aussi déterminés. Il fut observé qu’en général, les impuretés augmentaient la viscosité, laquelle était substantiellement avec l’addition de dextran, d’amidon et de chlorure de calcium. L’effet était cependant négligeable avec l’addition de chlorure de potassium et d’iode de potassium. Des études de conductivité après cristallisation d’une solution de saccharose contenant les sels inorganiques indiquent qu’une certaine quantité d’impuretés a été emportée par le cristal durant le procédé de cristallisation.

Mots clés: Supersaturation, viscosité apparente, taux de croissance des cristaux, impuretés, solubilité.

EL EFECTO DE LAS DEXTRANAS Y LAS SALES INORGANICAS SOBRE LA CRISTALIZACION DE AZUCAR EN UN TACHO AL VACIO DE LABORATORIO

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Resumen
Se calculó, a 54°C, la tasa aproximada de crecimiento de cristales de azúcar en soluciones de sacarosa pura con un grado de sobresaturación de 1.1 y 1.2. Se observó que la tasa de crecimiento se dobla con el incremento de la sobresaturación. Por otra parte se determinó la velocidad de crecimiento del cristal en presencia de 1.0 g de dextrana, almidón, cloruro de potasio, yoduro de potasio y cloruro de calcio, adicionando uno a uno cada uno de estos compuestos. Se encontró que la tasa de crecimiento del cristal decrece con la adición de dextrana, almidón y yoduro de potasio y se incrementa en presencia de cloruro de potasio. La tasa de crecimiento no varió en presencia de cloruro de calcio. También se determinó el efecto de estas impurezas en la viscosidad aparente, observándose que en general las impurezas la incrementan, presentándose los mayores incrementos cuando se adicionó dextrana, almidón y cloruro de calcio. El efecto fue sin embargo despreciable cuando se adicionó cloruro de potasio y yoduro de potasio. Los estudios de conductividad posteriores a la cristalización de las soluciones de sacarosa que contenían sales inorgánicas indican que cierta cantidad de impurezas ha sido retenida por el cristal durante el proceso de cristalización.

Palabras claves: Supersaturación, viscosidad aparente, velocidad de crecimiento del cristal, impurezas, solubilidad.