OPTIMIZATION OF PROCESSES FOR PRODUCTION OF GLUCOSE SYRUPS AND DEXTROSE MONOHYDRATE

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ABSTRACT

The continuous discovery and selection of new enzymes for the starch industry is presented as an opportunity to improve production processes. The implementation of new commercially available enzymatic preparations is only possible after a meticulous technological and economic validation, to show unequivocally that these new enzymes bring more resources into the productive process and the company.

The first goal of this study was the endorsement of a new α-amylase with technical and economic advantages while providing the company with an alternative supplier of this type of enzyme.

The enzyme in test proved to have suitable characteristics when applied in the process of starch liquefaction, namely, the ability to maintain its activity at low concentrations of calcium and low pH.

After the results evidenced the applicability of the enzyme in the process, an analysis of the economic impact of its use was carried out, and it was found that the use of this new enzyme in the process of starch liquefaction allowed a total gain of 43% compared with the use of the current enzyme.

The second goal of this work layed on the yield optimization of the crystallization process of dextrose monohydrate, through the optimization of the process variables.

It was concluded that it would be possible to adjust the characteristics of the feeding syrup of the crystallization and of the cooling profile throughout the section.

Keywords: α-amylase, enzymatic liquefaction, saccharification, dextrose, crystallization, crystal size.

1. INTRODUCTION

1.1. STARCH

Starch is the main substance of reserve in higher plants and provides about 70 to 80% of the calories consumed by man. There are several sources of starch and regular maize is the most common source worldwide (Schenck & Hebeda, 1992). The most significant commercial sources of starch are the cereal grains, legumes grains and tuberous roots. The main five species considered globally as commercial sources of starch are maize, wheat, rice, potato and cassava.

Pure starch is a white, insipid and odorless powder, insoluble in water and alcohol. It consists of a mixture of two structurally different polysaccharides: amylose and amyllopectin.

Amylose is a linear molecule of D-glucose units linked through α-1,4-glycosidic bonds. As amylose, amyllopectin is composed of glucose units connected by α-1,4-glycosidic bonds, differing from the former by presenting α-1, 6 links that form branch points.

The starch’s properties involve their physical, chemical and functional features, with many of them associated with one another. The starch’s solubility is a property of great importance in the context of this work in view of the fact that the enzymes studied do not act on solid starch, but on the gelatinized starch.

1.2. STARCH INDUSTRY

Starch industry extracts its raw material from several sources, processing it in a variety of products, such as native starch, glucose syrup, glucose-fructose syrup and dextrose (anhydrous or monohydrate). From the production processes several sub-products are obtained, such as corn gluten feed, corn gluten meal or germ, whose valorization represents a way to reduce production costs.

1.3. GLUCOSE SYRUPS PRODUCTION FROM STARCH

Glucose syrups are the hydrolysis product of starch. This hydrolysis can be chemical (by acid treatment, temperature, pressure), enzymatical, or the association of both.
The enzymatic hydrolysis of starch consists of two steps: liquefaction and saccharification. Liquefaction corresponds to the complete gelatinization of starch polymer, to enable the action of α-amylase, followed by dextrinization to a degree that would prevent the retrogradation in later steps of the process. In the saccharification step, oligosaccharides from liquefaction are further hydrolyzed in a more complete manner to produce syrup with a high proportion of low molecular weight sugars.

1.4. DEXTROSE PRODUCTION FROM STARCH

Dextrose is a monosaccharide available in two forms: monohydrate with 8.5 percent of crystallization water and anhydrous, which does not contain free moisture (Josly, 1964).

For the production of dextrose monohydrate, the hydrolysate from the saccharification is purified and discolored for later to be concentrated in an evaporator and sent to crystallizers. The crystallizers are horizontal cylindrical tanks equipped with slowly turning agitators and a cooling jacket, filled with cooling water, to induce crystals growth. The process of cooling crystallization can be used when the solubility of the substance to be crystallized increases with temperature, which is the case of dextrose. The resulting magma of crystallizers is named massecuite and is sent to a perforated-screen centrifuge basket to separate crystals from the mother liquor, which is called hydrol. The crystals are then sent to a fluidized bed dryer.

2. MATERIALS AND METHODS

2.1. ANALYTICAL METHODS

2.1.1. DETERMINATION OF DRY SUBSTANCE THROUGH REFRACTIVE INDEX

The refractive index of a substance is the relationship between the speed of light in vacuum and in substance, and depends on its composition, concentration and temperature. Knowing the composition and temperature of the substance, its refractive index is a measure of dry substance (DS). The thermostat set point should be adjusted so that the reading temperature on the refractometer (Index Instruments, Mod GPR-11-37) could be 20 ± 0.2 °C. It should be confirmed that the surface of the prism is clean before disposing approximately 0.1 ml of homogenized sample in the center of the prism. It is important to ensure that the sample does not contain air bubbles.

2.1.2. DETERMINATION OF DEXTROSE EQUIVALENT BY OSMOMETRY

Dextrose Equivalent (DE) is a measure of the reducing power of a hydrolysate expressed as D-Glucose on a dry basis. The DE of a carbohydrates aqueous solution is related to the molecular weight of the sugars present in the solution, and can be determined by the measurement of the solution’s freezing point depression. For this purpose the osmometer (Advanced Instruments, Model 3320) is used. These readings are given in miliOsmolalities (mOsm). The presence of salts in the sample affect the readings, so to determine the DE of the solution it is necessary to correct the miliOsmolalities readings, by taking into account the conductivity of the solution, given by equation 1.

\[ mOsm_{corrected} = mOsm_{read} - \text{conductivity} \times 0.02 \]  

In addition to the correction of the miliOsmolality, one has to determine the value of the solution’s reference miliOsmolality. To determine this value it is necessary to dilute the sample with demineralized water to obtain a solution with a refractive index (RI) in the range from 1.34937 to 1.35247 measured at 20 °C, so that the table where the value of reference miliOsmolality is can be consulted. The value of DE can then be calculated by the use of equation 2.

\[ DE = \left( \frac{mOsm_{corrected}}{mOsm_{ref}} \right) \times DE_{ref} \]
2.1.3. SUGAR COMPOSITION DETERMINATION BY HPLC

The sugar composition of samples is determined using a liquid chromatograph HPLC (Waters, Model 717 plus). The elution is done with water through the chromatography column (Aminex HPX-87C, Bio-Rad) with resin bed of cation-exchange in calcium form. The sample, previously filtered, is diluted to a refractive index of 1.3370 and demineralized through a treatment with a mixture of anionic (Amberlite IRA92) and cationic resins (Amberlite 252Na). Then the mixture is filtered through a filter paper to separate the resins, and the concentration is adjusted until the mixture has a value of refractive index of about 1.3358. After adjusting the concentration, the sample is filtered through a filter-disc (Millipore's) from 0,22 μm for a vial, filling it halfway and covering it with a suitable seal to prevent dust from entering the sample and from splashing occur in the automatic injector.

2.1.4. pH DETERMINATION

pH is a measure of free solution acidity or alkalinity on a scale of 0 to 14, and is determined by measuring the difference of the potential between two electrodes immersed, due to migration of ions from solution loaded positively or negatively. The electrode (WTW, Model 340i) should be carefully washed with water between each measurement and when it is not in use must remain immersed in a solution of 3M KCl (potassium chloride, p.a, from Panreac).

2.1.5. STARCH TEST BY IODINE REACTION

Determination of starch's presence in the solution is based on the reaction of iodine with starch, developing a blue color when amylose is present. The reaction with amylopectin produces a red color. This method consists in collecting 10 ml of sample, previously filtered, to a test tube, to which is added a 0.02 N iodine (I₂, p.a, from Panreac) solution drop by drop and with stirring, until the sample is stained. If the sample turns yellow, the starch test is negative. If the sample turns brown, then the starch test is positive.

2.1.6. DETERMINING THE CONTENT OF CALCIUM BY TITRATION WITH EDTA

The method of determining the calcium content by titration with EDTA (Riedel – deHaën) consists in the measurement of 10 ml of sample for a 250 ml erlenmayer flask, to which is added about 40 ml of water, 2 ml of 8N KOH (potassium hydroxide, p.a, from Panreac) and a few milligrams of Murexide indicator (BDH Chemicals). Subsequently, this solution is titrated with 0.01 M EDTA, until the red color turns to purple. With the value of the volume of EDTA spent and knowing that 1 ml 0.01 M EDTA = 0.4008 mg Ca, the calcium content is determined by the equation 3.

\[
[Ca], \text{mg/l} = \frac{V_{\text{EDTA}} \times 40.08}{E}
\]  

2.2. EXPERIMENTAL METHODS

2.2.1. INDUSTRIAL TEST: ENZYMATIC LIQUEFACTIION WITH ENZYME CLEARFLOW AA

The industrial test consists of the substitution of the enzyme Liquozyme Supra by Clearflow AA, in the process of enzymatic liquefaction of starch. Throughout the test, all analysis, monitoring and recording of variables process control must be carried out, such as: volumetric flow and density of milk starch, pH, percentage of dry substance, dosage and volumetric flow of enzyme, concentration of calcium, DE, starch test. In the beginning of the test, the same conditions as those used at the time with the enzyme Liquozyme Supra were considered. By the initial results, the dosage of enzyme ought to be adjusted to obtain syrup with 14DE. After stabilization of DE, the value of calcium should be adjusted up to 20 ppm. The pH of the milk starch should also be adjusted to the optimal range of the new enzyme. The remaining process variables are kept within established limits. It is also important to control the hydrolysate 95 from the test deposits, to ensure that the modification of the process conditions does not change the final product quality. Therefore, the starch test must be executed, and DE and sugar composition in hydrolyzate samples should be collected at the saccharification deposits thus these parameters may be controlled. The test should be discontinued if there is any change in the quality of the final product.
2.2.2. Optimization of the Dextrose Monohydrate Production Process

The aim is to determine and compare the theoretical yield and real yield of the crystallization process of dextrose monohydrate, to conclude what steps should be taken in order to optimize the process. The theoretical yield is calculated based on the feeding of crystallization. This value represents the maximum yield that could be achieved if there were no product losses along the production line. For this calculation, it is necessary to know the percentage of dry substance and the concentration of D-Glucose in the syrup feeding crystallization. Knowing these two variables and the temperature of the crystallizer C5, it is possible to determine the theoretical yield using equation 4.

\[
\eta_{\text{theoretical}} = \frac{DS \times DP1}{100 - DS} \times DS
\]

where DS – percentage of dry substance in the syrup feeding crystallization; DP1 – concentration of D-Glucose in the syrup feeding crystallization; GT – solubility of dextrose to the temperature of the crystallizer C5, expressed in g dextrose/g water.

Real yield is determined by mass balances to the production process of dextrose monohydrate.

3. Results and Discussion

3.1. Optimization of the Dextrose Monohydrate Production Process

3.1.1. Correction of the Process Parameters Using Liquozyme Supra

In a first approach, the data for the production conditions of glucose syrup for a period of six months was gathered. This analysis demonstrated that the process required some adjustments to make possible the comparison of optimal processing during the test with the new enzyme. These changes were associated to the reduction of enzyme specific dosage added in the liquefaction, and the reduction of calcium chloride solution concentration.

The first step was the adjustment of the dosage of enzyme added, the other process variables were kept constant, until achieving a syrup with a DE around 14. After the correct dosage of enzyme was determined, the calcium concentration was adjusted, slowly reducing the volumetric flow rate of calcium chloride solution added.

The optimal operation conditions of the process with the enzyme Liquozone Supra determined during the test are summarized in Table 3.1.

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>DE</td>
<td>%</td>
<td>14</td>
</tr>
<tr>
<td>Dry substance</td>
<td>Brix</td>
<td>32,6</td>
</tr>
<tr>
<td>Flow rate of the milk starch</td>
<td>m3/h</td>
<td>6,4</td>
</tr>
<tr>
<td>Density of the milk starch</td>
<td>°Be</td>
<td>20,5</td>
</tr>
<tr>
<td>pH of the milk starch</td>
<td>–</td>
<td>5,9</td>
</tr>
<tr>
<td>Specific concentration of</td>
<td>Kg/Ton</td>
<td>0,60</td>
</tr>
<tr>
<td>enzyme</td>
<td>DS</td>
<td></td>
</tr>
<tr>
<td>Calcium concentration</td>
<td>ppm</td>
<td>72,14</td>
</tr>
</tbody>
</table>

3.1.2. Industrial Test with Clearflow AA Enzyme

The industrial test consists in the substitution of Liquozone Supra by Clearflow AA enzyme comprising, at the beginning, the same operation conditions of the process, established in the previous chapter. From the results obtained, which indicated a higher conversion degree for the same conditions, the dosage of enzyme was adjusted, with the intention that the DE parameter would fit within the range of specification. After the determination of the correct enzyme dosage in order to stabilize the DE, the flow rate of calcium chloride solution added was reduced to obtain a calcium concentration in the syrup around 20 ppm. In the meanwhile DE values were always kept within the predefined range.

The optimal operation conditions of the process with the enzyme Clearflow determined experimentally are summarized in Table 3.2.
Table 3.2. Optimal operation conditions of the process with the enzyme Clearflow AA.

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Syrup</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DE</td>
<td>%</td>
<td>14</td>
</tr>
<tr>
<td>Dry substance</td>
<td>Brix</td>
<td>32.9</td>
</tr>
<tr>
<td>Process</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flow rate of the milk starch</td>
<td>m³/h</td>
<td>6.4</td>
</tr>
<tr>
<td>Density of the milk starch</td>
<td>°Be</td>
<td>20.5</td>
</tr>
<tr>
<td>pH of the milk starch</td>
<td>–</td>
<td>5.7</td>
</tr>
<tr>
<td>Specific concentration of enzyme</td>
<td>Kg/Ton DS</td>
<td>0.44</td>
</tr>
<tr>
<td>Calcium concentration</td>
<td>ppm</td>
<td>26.05</td>
</tr>
</tbody>
</table>

3.1.2.1. COMPARATIVE ANALYSIS OF THE SPECIFIC ENZYME CONCENTRATION EFFECT

The right dosage required depends on process parameters such as quality of starch, pH, temperature and reaction time, the solids level and the presence of enzyme stabilizers. Comparing the use of two enzymes in the process (Figure 3.1), for the same specific enzyme concentration, the Clearflow AA achieves a higher DE. In other words, to accomplish the desired value of DE, it is required a lower specific concentration of Clearflow AA (0.44 kg/tonne DS) compared to the necessary when using Liquozyme Supra (0.60 kg/tonne DS).

![Specific enzyme concentration effect](image)

Figure 3.1. Analysis of specific enzyme concentration required to obtain certain values of DE.

3.1.2.2. CALCIUM CONCENTRATION EFFECT

During the test, it was confirmed that calcium acts as a stabilizer for both enzymes as, for a given dosage of enzyme and keeping the remaining process variables constant, the addition of a higher concentration of calcium to the process results in higher DE syrup. By the analysis of Figure 3.2 conclusions are that reducing the calcium concentration DE values of the syrup are also reduced. However, it is possible to decrease the calcium concentration within certain range of values, without interfering manifestly in the amount of DE, i.e. for each of the enzymes there is a range of values of calcium concentration in which the catalysts can maintain its activity.

Comparing the use of the two enzymes in the process (Figure 3.2), for the same concentration of calcium, Clearflow AA has permitted a higher DE. Therefore when the Clearflow AA is used, and to achieve the desired DE value, the concentration of calcium required is lower (20 - 35 ppm) when compared to Liquozyme Supra (60 - 75 ppm).

![Calcium concentration effect](image)

Figure 3.2. Comparative analysis of calcium concentration effect in the enzymatic starch liquefaction.

3.1.2.3. pH EFFECT

During the test it was possible to confirm that the optimal pH for each enzyme is in fact within the range indicated, taking the value of 5.9 in the case of Liquozyme Supra, and 5.7 in the case of Clearflow AA. The advantage of working in the lowest range of pH is related to the fact that, after liquefaction, the syrup pH has to be lowered to the optimum performance value of the saccharification enzyme (pH 4.0). As the syrup pH is lower, the
amount of hydrochloric acid solution required to perform this adjustment is less too.

3.1.2.4. Final Product Quality

To prove that the alteration of enzyme applied to the process of liquefaction, would not change the quality of the final product, the process of saccharification of all the syrup in the test was followed (data not shown). There were no non-conformities recorded during the industrial test, regarding the quality of the final product.

3.1.3. Comparative Costs Analysis

The economic evaluation of a process is one of the key points of any study, since it is a decision factor of the feasibility of it. Thus, a comparative analysis of costs of the production process, with the use of each enzyme, was carried out.

The immediate effect on the costs of the process is related to the price differences of the used enzymes, since Clearflow AA has a lower price than the Liquozyme Supra. Moreover, the necessary dosage of Clearflow AA to obtain values of DE within the range of specification is lower than the dosage of Liquozyme Supra. There is also a gain on the reduction of the dosage of calcium chloride solution added to the process. On the other hand, the difference between the optimal pH of the starch milk for each enzyme also allows the decrease of the production costs, since there is a dosage reduction of solutions such as sodium carbonate and hydrochloric acid used to adjust the pH during the process. It follows, therefore, that the use of the enzyme Clearflow AA in the process of enzymatic starch liquefaction, compared to the use of the enzyme Liquozyme Supra, allows a total gain of 43%.

It should also be noted that there are additional gains in the downstream purification process of the syrup, namely in the ion exchange chromatography, which were not quantified. This savings are based on reduced consumption of reagents used in the regeneration and balance of the column, lower consumption of resin due to longer cycles, lower water consumption and therefore less waste water treatment.

3.2. Optimization of the Dextrose Monohydrate Production Process

3.2.1. Determination of the Crystallization Theoretical Yield

The theoretical yield of crystallization is calculated based on the percentage of dry substance and glucose concentration of the syrup that feeds the crystallizers (C1 and C2), and on the dextrose solubility at crystallization temperature, according to equation 4. To perform this calculation, one must assume a value of supersaturation of the solution. The coefficient of supersaturation should not exceed 1.60 at the beginning of crystallization and cooling profile ought to result in a final ratio of 1.05 when the crystals are collected in the centrifuge (Blanchard, 1992).

3.2.2. Determination of the Crystallization Real Yield

The real yield of crystallization was determined by mass balances to the unit operations involved in the production of dextrose monohydrate.

Figure 3.3 is a schematic representation of the production of dextrose monohydrate. The calculation basis used to carry out the mass balances is a ton of hydrolyzate 95 fed to the process. In Table 3.3 are characterized the various currents.

![Figure 3.3. Schematic representation of the production of dextrose monohydrate.](image-url)
Table 3.3. Characterization of the currents of the production process of dextrose monohydrate. The quantities of each component in the currents were determined by mass balances.

<table>
<thead>
<tr>
<th>Current</th>
<th>Total mass (Kg)</th>
<th>Glucose mass (Kg)</th>
<th>Other sugars mass (Kg)</th>
<th>Water mass (Kg)</th>
<th>DS (%)</th>
<th>DP1 (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Input of hydrolyzate 95</td>
<td>1000,0</td>
<td>323,8</td>
<td>17,9</td>
<td>658,3</td>
<td>34,0</td>
<td>95,0</td>
</tr>
<tr>
<td>2 Input of recirculated hydro</td>
<td>304,7</td>
<td>125,4</td>
<td>23,9</td>
<td>155,4</td>
<td>49,0</td>
<td>83,0</td>
</tr>
<tr>
<td>3 Evaporator feed</td>
<td>1304,7</td>
<td>449,2</td>
<td>41,8</td>
<td>813,7</td>
<td>38,0</td>
<td>91,0</td>
</tr>
<tr>
<td>4 Cristalization feed</td>
<td>644,5</td>
<td>449,2</td>
<td>41,8</td>
<td>153,5</td>
<td>76,0</td>
<td>91,0</td>
</tr>
<tr>
<td>5 Condensed water</td>
<td>660,2</td>
<td>–</td>
<td>–</td>
<td>660,2</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>6 Massecuite</td>
<td>644,5</td>
<td>449,2</td>
<td>41,8</td>
<td>153,5</td>
<td>76,0</td>
<td>91,0</td>
</tr>
<tr>
<td>7 Washing water of the centrifuge</td>
<td>86,3</td>
<td>–</td>
<td>–</td>
<td>86,3</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>8 Output of hydro from centrifuge</td>
<td>439,4</td>
<td>228,1</td>
<td>41,4</td>
<td>169,9</td>
<td>49,0</td>
<td>83,0</td>
</tr>
<tr>
<td>9 Wet dextrose</td>
<td>291,5</td>
<td>221,1</td>
<td>0,4</td>
<td>70,0</td>
<td>76,0</td>
<td>99,8</td>
</tr>
<tr>
<td>10 Output of dextrose monohydrate</td>
<td>241,8</td>
<td>221,1</td>
<td>0,4</td>
<td>21,3</td>
<td>91,0</td>
<td>99,8</td>
</tr>
<tr>
<td>11 Water</td>
<td>48,7</td>
<td>–</td>
<td>–</td>
<td>48,7</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

The real yield of the process is the ratio of the mass of glucose obtained at the end of the process and the mass of glucose entering the crystallizers. Thus, the real yield is given by equation 5.

$$\eta_{\text{real}} = \frac{\text{final glucose mass}}{\text{glucose mass fed to the crystallization}} \times 100 \quad (5)$$

3.2.3. COMPARATIVE ANALYSIS BETWEEN THEORETICAL AND REAL CRYSTALLIZATION YIELD

The results of the mass balances show that the dry substance percentage of the syrup that feeds the crystallization is 76.0% and the glucose concentration of the same syrup is 91.0% (Table 3.3). Introducing these variables in Equation 4, the theoretical yield of crystallization can be calculated, taking the value of 59.0%. On the other hand, the real yield of crystallization, given by equation 5, is 49.3%.

It is important to note that the real yield, calculated by mass balances, corresponds to an overall efficiency of the process and not just the single process of crystallization. In turn, the theoretical yield only concerns the crystallization. Thus, in a comparative analysis of the theoretical yield and actual yield, the loss of product in the unit operations downstream the crystallization should be considered.

The next items are explanatory of the various factors that influence the production process of dextrose.

3.2.3.1. PROCESS VARIABLES INFLUENCING THE CRYSTALLIZATION YIELD

At the beginning of the crystallization is essential to determine the solubility of the working substance and its variation with temperature. In the case of dextrose monohydrate, the solubility increases with temperature and is therefore appropriate to choose the process of crystallization by cooling. However, the solubility does not explain how this substance is assembled. Instead, the kinetics of the crystal formation provides that answer ie, the nucleation, growth and their speeds. The degree of supersaturation (DS) of a solution measures the deviation from equilibrium and is a prerequisite for any crystallization process being, in fact, its main driving force (Lang et al., 1999). The product’s particle size profile depends on the relationship between the processes of nucleation and growth. For low values of GS, the growth rate is higher than the rate of nucleation: the tendency for the growth of already formed nuclei is higher than the formation of new nuclei. For higher values of GS, the tendency for the formation of new nuclei predominates over the growth of the nuclei already formed.

The characteristics of the syrup that feeds the crystallization is of great importance for the correct functioning of the process. The dry substance, the glucose concentration and the solution’s temperature must be carefully controlled to ensure that the supersaturation is...
maintained in the optimal range. In fact, an increase of dry substance of syrup feeding crystallization corresponds to an enhancement in crystallization yield. To raise this parameter it is required a bigger amount of steam introduced into the evaporator. This increased amount of steam leads to an increase in energy costs, and therefore it is required a compromise between the gain that can be achieved by rising the amount of dry substance of syrup and losses resulting from increased energy costs. Monitoring the glucose concentration of syrup that feeds crystallization is essential and therefore it is important to note the quality of the hydrolyzate and hidrol that feeds this section, for proper functioning. To increase the yield of crystallization, keeping all other variables constant, the process should have higher concentrations of glucose, which in other words, means reducing the recirculation flow of hidrol.

### 3.2.3.2. LOSSES OF PRODUCT IN SEVERAL UNIT OPERATIONS AFFECTING CRYSTALLIZATION YIELD

At the end of the crystallization, the suspension is sent to a basket centrifuge to separate the crystals from hidrol. In the centrifugation is also important a correct crystals shape and size. If, in one hand, a suspension from an inefficient crystallization, tends to have very fine crystals that can dissolve during the washing step, on the other hand, a suspension with a higher crystal phase yield is very viscous and difficult to sent to the centrifuge, and moreover, the crystals tend to break and lead to finer crystals that difficult the performance of this equipment. It is also necessary to take into account the shape of the crystals. The formation of aggregates of crystals or crystals with shape of needles should be avoided, since they are very difficult to dehydrate in the centrifuge.

Crystals discharged from the centrifuge are sent to a fluidized bed dryer. The air used for drying, after leaving the dryer goes through a cyclone to recover fine particles by the force of gravity, allowing the output of clean air to the atmosphere through the chimney. In this process there are losses of product, which contribute to the reduction of process yield.

### 3.2.4. SUGGESTIONS OF FUTURE ACTIONS

To optimize the yield of the crystallization process of dextrose monohydrate production, it is suggested to perform an industrial test to determine the real crystallization yield. During the test there should be a daily report of the variables: percentage of dry substance of the syrup that feeds crystallization, glucose concentration of this syrup and temperature of the crystallizers C1 to C5. Each of these parameters should be adjusted to obtain a maximum yield of crystallization. It should also be performed a characterization of the particle size profile of the product to observe how changes in the cooling profile during crystallization influence the size of the crystals and also to ensure that their size complies with the specification.

To achieve an improvement in dextrose production, there should also be measures regarding the losses of product in the centrifuge and cyclone. The centrifuge consists of three components: a basket with holes of 5 mm for the disposal of hidrol, a support network and a screen filter. To avoid loss of product during washing in the centrifuge, it is suggested the installation of a network with lower staging area or a different configuration in terms of holes in order to retain finer crystals. Another alternative would be to increase the centrifugation speed in order to reduce the amount of wash water added. There should also be a quantification of the product lost through the chimney, to perceive whether this loss is significant. If it is justified, a hypothesis to optimize the cyclone operation would be the installation of a powder retrieval system, composed of a baghouse that captures the dextrose and would then be recycled to the process.

### 4. CLOSING REMARKS

The industrial trials conducted to test the enzyme Clearflow AA allowed conclusions about its applicability in enzymatic liquefaction process. It was concluded that the enzyme under test meets the conditions necessary for its approval.

By the comparison of the enzymes Clearflow AA and Liquozyme Supra, it was found that to achieve the desired degree of conversion, there is a lower specific consumption of Clearflow AA, it is required a lower concentration of calcium...
to stabilize the enzyme and its optimal pH is inferior than with Liquozyme Supra, which allows working at lower pH values.

It should be noted that during the test there were no non-compliances regarding the quality of the final product.

After the demonstration of the applicability of the enzyme in the process, a comparative analysis of costs of the use of each enzyme, was carried out. It was concluded that the use of the new enzyme in the process of starch enzymatic liquefaction allows a total gain of 43% compared to the use of actual enzyme. This gain comes from the need for a less specific enzyme concentration, a lower dosage of calcium chloride solution added to the process and dosage reduction of solutions such as sodium carbonate and hydrochloric acid used to adjust the pH during the process. It should also be noted that, despite not having been kept account, there are additional gains in the downstream purification process of the syrup, namely in the ion-exchange chromatography. This savings are based on reduced consumption of reagents used in the regeneration and balance of the column, lower consumption of resin due to longer cycles, lower water consumption and therefore less waste water treatment.

The analysis of the production process of dextrose monohydrate has shown that the real yield of crystallization is lower than the theoretical yield. The reason for the difference in these values are parameters such as the characteristics of the syrup that feeds the crystallization, the profile of cooling during crystallization, and also losses of product during centrifugation and in the cyclone located downstream of drying product.

An increase of dry substance of syrup feeding crystallization corresponds to an enhancement in crystallization yield, and to increase this parameter it is required a greater amount of steam introduced into the evaporator. The glucose concentration of this syrup is a parameter that influences the yield of crystallization. To increase the yield of crystallization the process should have higher concentrations of glucose, which in other words, means reducing the recirculation flow of hidrol.

An effective cooling during crystallization is of great importance for the proper functioning of the process, as it requires a temperature profile appropriate to maintain the value of solution supersaturation constant. Furthermore, this temperature profile determines the crystal size of the product.

Given that there are losses of product along the production line, it is suggested to carry out a study to account losses of dextrose during washing in the centrifuge and losses through the chimney of the cyclone

REFERENCES


