MODELING AND OPTIMISATION OF THE DEXTROSE MONOHYDRATE CRISTALLISATION PROCESS

— research paper —

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Abstract: The aim of the present work was to develop a simple mathematical model for the cooling crystallisation of α -dextrose monohydrate in vertical crystallisers and to use it for the optimisation of the plant operation. The system of differential algebraic equation representing the model was numerically simulated using the Scilab environment. Multiple simulation with different values for the cooling water temperature were performed in order to select the ones that assures the growth of the crystal beyond an technological imposed minimal size (500 µm). The results of the simulation with optimal values were presented. For a crystalliser with three individually temperature controlled cooling sections, the temperature of the cooling water at the inlet of the first section was found to be 25.5°C, whereas for the next two sections, the optimal cooling water temperature at the entrance was found to be 20.5°C.

Keywords: dextrose monohydrate, cooling crystallisation, model, optimisation,

INTRODUCTION

Crystalline dextrose monohydrate (α D-glucose) is obtained from supersaturated solutions of D-glucose at temperatures below 50°C (in the interval 30-50°C) from solutions with 60-80% dry weight. Crystallisation at higher temperatures gives anhydrous α and β forms. The driving force of the crystallisation process is the supersaturation which has to be maintained below 10% for avoiding spontaneous nucleation with forming of fine particles (Schenck and Hebeda, 1992).

Because spontaneous nucleation in the glucose solutions needs too longer nucleation time, crystallisation of glucose monohydrate is realised by seeding in coolers (Nyvlt and Kraus, 1993). Cooling temperature has to be correlated with the crystallisation speed in order to assure a continuous

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growth for the crystals. The cooling profile of crystal mass can be natural, linear or controlled; the natural profile could determine secondary nucleation and the linear profile allow the aggregates forming (Steele, 2007). For these reasons, a controlled temperature profile is needed.

The crystallisation models for the continuous dextrose coolers are relatively few. (Sima and Harris, 1999a) and (Sima and Harris, 1999b) propose a CFD model for an vertical sucrose crystallisers. A generic modelling framework for solution crystallisation processes implemented in a commercial modelling and optimisation tool gPROMS is presented in (Bermingham, 2003). A zone network model described in (Kramer et all, 1999) is used to account for the spatial distribution.

The goal of this research is to establish the optimal cooling regime in a DSSE continuous cooler which allows the continuous growth of glucose crystals to optimal values for separation, meaning larger as $500 \,\mu\text{m}$.

CRYSTALLISER GEOMETRY

The optimisation of the dextrose monohydrate crystallisation was performed for a vertical continuous cooling crystalliser type BMA, which is a cylindrical tower equipped with oscillating cooling tubes (Hempelmann, 2008). The cooling system consists of standardized block-type cooling elements, through which cooling water flows counter-current to the glucose massecuite (a mixture of crystals and glucose syrup). The massecuite flows top-down and the cooling water flows bottom-up.

The entire cooling system has a hydraulic oscillating movement in vertical direction; this movement, combined with the symmetrical arrangement of the cooling tubes, ensures an optimum residence time spectrum and cooling rate. Consequently, temperature and concentration fields in the crystalliser can be considered to be stationary.

The chosen system has three individually temperature controlled cooling sections. Each section is 6 m high.

MATHEMATICAL MODEL

The mathematical model is a system of differential algebraic equations which describe the cooling crystallisation process of dextrose monohydrate. The equation system is valid for all the three sections of the crystalliser. It was obtained by writing the balance equations for an elementary volume of height dz. Figure 1 presents the heat and mass fluxes for the three sections.



Figure 1. Heat and mass fluxes for the three sections of the cooling crystalliser \dot{M}_m - mass flow rate of glucose massecuite, considered constant on all three sections; N_c - seed crystals number, in 1 kg of syrup; T_e - temperature of glucose syrup at the apparatus inlet; $T_{e,i}$ -temperature of glucose massecuite at the exit of section *i*; $\dot{M}_{a,i}$ - mass flow rate of cooling water of section *i*; $T_{i0,i}$ - cooling water temperature at the beginning of section *i*; $T_{i,i}$ - cooling water temperature at the end of section *i*;

From the balance equations, three differential equations were derived:

- Glucose concentration rate of change

$$\frac{dC}{dz} = -r \tag{1}$$

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- Massecuite temperature rate of change

$$\frac{dTe}{dz} = \frac{Q_{cryst} - Q}{Cp_{glc} \cdot \dot{M}_m}$$
(2)

- Cooling water rate of change

$$\frac{dTi}{dz} = -\frac{Q}{Cp_{water} \cdot \dot{M}_{w,i}}$$
(3)

Because the α D-glucose crystals are hexagonal or acicular plates, the expression after (Myerson, 1952) can be used to describe the dependence of the crystal length variation from the crystallisation parameters trough an Arrhenius type equation. It results the forth differential equation for the crystal length rate of change

$$\frac{dL}{dz} = Kg \cdot e^{-\frac{Eg}{R \cdot T}} \cdot \frac{C - C_{sat}}{v}$$
(4)

where:

dL/dz - rate of change of the crystal size;

- *L* crystal chord length;
- *Kg* crystal growth rate constant;
- *Eg* activation energy for the crystal growth;
- *R* universal gas constant;
- *T* system temperature;
- *C* glucose concentration;

 C_{sat} - glucose concentration at saturation for temperature T;

dC/dz - rate of change of glucose concentration;

r - crystallisation rate;

 Q_{cryst} - crystallisation heat;

 λ - crystallisation latent heat;

dTe/dz - rate of change of massecuite temperature;

dTi/dz - rate of change of cooling water temperature;

 Cp_{water} - specific heat capacity of water;

 Cp_{glc} - specific heat capacity of massecuite;

The correlation between the mass growth rate and crystal length rate is given by:

$$r = \frac{dM_c}{dz} = N_c \cdot \frac{\pi}{2} \cdot \rho_c \cdot L^2 \cdot \frac{dL}{dz}$$
(5)

where:

dMc/dz - rate of change of the crystal mass;

 ρ_c - density of the glucose crystals.

The heat flux through the heat exchange surface is:

$$Q = K \cdot A \cdot (Te - Ti)$$
(6)

where:

- *Q* heat changed between the cooling agent and the glucose massecuite;
- A heat transfer area;
- v massecuite speed.
- *K* global heat transfer coefficient;

NUMERICAL SOLUTION

The SciLab environment was used for the numerical simulation of the differential equations system. The program has three modules:

PROPERTIES.SCI : assure the computing of crystal solution and water properties (Cp_{glc} , Cp_{water} , λ and C_{sat}) as function of temperature;

SCT.SCI : describes the equations system;

SEGMENT.SCI – integrates numerically the differential equations described by SCT.SCI and represents the results graphically for one section of the crystalliser.

By solving the equations system, for each of the four variables a function was obtained which describes their variation on the section length.

For each section, one of the initial conditions — the initial temperature of cooling water — is important from the process control point of view. The equations system for each section was solved for many values of this initial condition, in order to find its optimal value. The values of the cooling water temperature were chosen in the range used industrially, not lower as 20° C. For the first section the other initial conditions (Te, C, L) were kept constant in all the simulation cases. Like in the real case the crystalliser is feed with dextrose syrup at 51° C, with 72% glucose (95% purity, 76% dry mater content); the syrup is seeded with crystals standardised by sieving at 120μ m. For the next sections, the values resulted from calculations at the exit of the previous section are used as initial conditions.

RESULTS AND DISCUSSIONS

Figure 2 presents the results of the simulation in which the final crystal size was larger as $500 \ \mu m$.



Figure 2. Simulation results for the variation of glucose concentration (C), temperature of massecuite (T_e), temperature of cooling water (T_i) and crystal length (L) with the length for the three sections of crystalliser

The crystallisation process is irregular on the height of the cooling crystalliser. The highest growth rate is the first section, where the crystal size grows from 120 μ m to 420 μ m for a cooling water inlet temperature of 25.5°C. The crystallisation rate is not constant on the whole height of this section. On the first third, where the difference between the temperature of the cooling agent and the massecuite is low, an exponential growth occurs. On the rest of the section, a quasi-linear growth is obtained. This result is in accordance with the BSF theory regarding the evolution of the crystallisation rate at low supersaturation (Schiweck et al., 2000).

In the sections 2 and 3, the optimal value for the cooling agent is 20.5°C, the lowers value acceptable for the crystalliser operation. The massecuite temperature is evolving differently because it starts with different values in each section and it has high influence on the massecuite physical properties.

The model was validated by comparing with the available experimental data from BMA (Braunschweigische Maschinenbauanstalt AG) (data not shown).

CONCLUSIONS AND FUTURE WORKS

In this research, an application in SciLab based on differential algebraic equations was written. Through simulations with the SciLab environment, the evolution on each section of the crystallisation process for different values of the initial conditions can be visualised. In this way, the application is a useful tool for the choice of the optimal working regime of the real plant. The proposed mathematical model describes correctly the variation of the crystal length, together with the glucose concentration, temperature of massecuite and temperature of cooling water, with the crystalliser height.

The simplicity of the model makes it suitable for the integration in optimisation and control frameworks, especially for large scale plants with multiple processing units.

The next step will be the extension of the model with a population balance model.

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