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Batch-to-batch model improvement for cooling crystallization ☆

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ABSTRACT

Two batch-to-batch model update strategies for model-based control of batch cooling crystallization are presented. In Iterative Learning Control, a nominal process model is adjusted by a non-parametric, additive correction term which depends on the difference between the measured output and the model prediction in the previous batch. In Iterative Identification Control, the uncertain model parameters are iteratively estimated using the measured batch data. Due to the different nature of the model update, the two algorithms have complementary advantages and disadvantages which are investigated in a simulation study and through experiments performed on a pilot-scale crystallizer.

1. Introduction

Cooling crystallization is a separation and purification process often performed in batch mode in the pharmaceutical, food and fine chemicals industries for the production of high value-added products (Myerson, 2002). In a batch cooling crystallization process, a solution consisting of a solute dissolved in a solvent is loaded at high temperature into a vessel called crystallizer, and is subsequently cooled down. The crystallizer temperature is manipulated by circulating a cooling medium inside the jackets surrounding the crystallizer. Due to cooling, the equilibrium concentration of the solution (i.e. the solubility) is lowered and part of the solute is transferred from the solution to the solid crystalline phase. The content of the crystallizer is no longer a clear solution, but a two-phase fluid slurry consisting of the solution and the solid crystals. The concentration of the solute in the solution decreases, while the amount of solid crystals increases. When the final temperature corresponding to the desired yield is reached, the solid crystals are separated from the solution and the batch ends.

In industrial batch cooling crystallizers, the crystallizer temperature is often the only process variable that is controlled (Fujiwara, Nagy, Chew, & Braatz, 2005). The jacket temperature is the manipulated variable used to steer the crystallizer temperature. Since accurate on-line temperature measurements can be readily obtained, the crystallizer temperature is controlled in a closed-loop setting. In this configuration, the desired cooling profile is given as set-point to a feedback temperature control loop.

However, even when the temperature is effectively controlled, the crystal product of a batch might not show all the desired properties. In fact, even though the temperature is an important process variable, it is not the one most closely related to the crystallization dynamics. The process variable having the most direct influence on the crystallization process is the supersaturation, often defined as the difference between the solute concentration and the solubility at a given temperature. Supersaturation is the driving force for physico-chemical phenomena involved in crystallization such as the birth and the growth of crystals (Myerson, 2002), and its trajectory throughout the process influences several aspects of the final product including chemical purity, polymorphic state, crystal size and shape (Barrett, McNamara, Hao, Barrett, & Glennon, 2010; Sanzida & Nagy, 2013). In general, operating at too high supersaturation has to be avoided since it leads to a degradation of the product quality. Conversely, operating at low supersaturation leads to a slow growth of the crystals and therefore to a low production rate. A trade-off between product quality and productivity is often defined by aiming at a constant supersaturation during the batch time (Fujiwara et al., 2005; Gutwald & Mersmann, 1990; Mesbah, Nagy, & Huesman, 2012).

In a batch cooling crystallization process, the supersaturation can be manipulated by changing the crystallizer temperature, since the latter determines the solubility. Supersaturation control strategies for batch cooling crystallization have been widely investigated in the literature (Fujiwara et al., 2005; Nagy, Chew, Fujiwara, & Braatz, 2008; Vissers et al., 2012; Xie, Rohani, & Phoenix, 2002). In general, supersaturation control has been shown to give better performance

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compared to temperature control only, particularly in terms of consistency of the product quality.

In most of the supersaturation control strategies, a nominal model of the supersaturation dynamics is used to design the model-based controller. Therefore, the quality of the model has a direct influence on the tracking performance that the model-based controller can achieve. Unfortunately, the models describing the process often suffer from severe uncertainties. Due to these uncertainties, the performance delivered by the model-based controller can significantly deteriorate in the case of a model-plant mismatch.

This paper investigates the opportunity of using input/output data collected from previous batches in order to improve the model, and consequently the performance delivered by a model-based control solution. In particular a situation is considered in which the concentration measurements collected throughout a batch (which are required in order to compute the supersaturation) are available for control only at the end of the batch. This situation can occur in an industrial environment, where the measurements are often obtained through the off-line analysis of samples collected throughout the batch. The quality of the model is even more important than in the case of feedback control. Indeed, feedforward control solutions are in general more sensitive to model-plant mismatches than the ones based on on-line feedback.

In Forgione, Mesbah, Bombois, and Van den Hof (2012a, 2012b) the authors introduced a novel batch-to-batch (B2B) control strategy conceived in order to track efficiently a supersaturation profile in a batch cooling crystallization process, under the presence of disturbances and model uncertainties. The findings presented in those previous contributions, which were obtained using a simulation model of the process, are here complemented with the results of real experiments performed on a pilot-scale crystallization setup which confirm the applicability of our method. To the best of the authors' knowledge, these are the first B2B control experiment for a batch cooling crystallization process documented in the literature.

The B2B control strategy exploits the fact that the concentration measurements are only available off-line, but the temperature measurements are readily available on-line. In fact, a higher-level B2B supersaturation control algorithm is combined with a lower-level feedback PI temperature controller. Based on the desired supersaturation profile and the off-line concentration measurements from the previous batches, the B2B algorithm updates a model of the process dynamics. Subsequently, it uses the updated model in order to compute an improved reference temperature profile T^r. This profile is set as reference to a lower-level PI temperature controller in the next batch. The role of the PI controller is to suppress the system disturbances as efficiently as possible in order to decrease their influence on the supersaturation dynamics.

Two B2B algorithms, namely Iterative Identification Control (IIC) and Iterative Learning Control (ILC), are presented. While IIC is based on a parametric model update, ILC performs a more flexible, nonparametric model correction. Due to the different nature of the model update, the two algorithms have complementary advantages and disadvantages, which are investigated in this paper.

The ILC algorithm used in this paper is based on the two-step procedure first introduced in Volckaert, Diehl and Swevers (2010). After a batch, the model of the dynamics from the reference temperature T^r to the supersaturation S is updated using a nonparametric additive correction term, which depends on the difference between the measured supersaturation and the supersaturation predicted by the model for the previous batch. This correction term is obtained in such a way that the updated model matches more closely the actual supersaturation measured during the previous batch. Subsequently, the improved reference temperature is computed using the updated model in order to minimize the supersaturation tracking error for the next batch. In the literature, other applications of ILC for supersaturation control in batch cooling crystallization have been presented in Zhang, Nguyan, Xiong, and Morris (2009), Sanzida and Nagy (2013). In both algorithms, a new temperature trajectory is designed based on a linear time-varying perturbation model of the nonlinear supersaturation dynamics. Compared to those papers, the advantage of the approach presented here is twofold. First, the presence of a lower-level PI controller, which is an asset in the presence of disturbances on the temperature dynamics. The ILC algorithm alone, which is a feedforward control solution, could not compensate for these real-time disturbances. In addition, these disturbances could be easily confused by the algorithm with parts of the actual process dynamics since ILC is based on a nonparametric model correction. Second, the use of the nonlinear, first-principles model of the process in the algorithm, as opposed to the linearized model used in Zhang et al. (2009) and Sanzida and Nagy (2013), which is an acceptable approximation of the dynamics only along the time-varying working point. Besides, it has to be mentioned that while in Zhang et al. (2009) and Sanzida and Nagy (2013) only simulation results have been reported, in this paper experimental results are also included.

In the IIC algorithm, estimates of the uncertain physical parameters are refined after a batch according to a Maximum a Posteriori criterion which combines the information coming from the measurements collected during the most recent batch with the previous parameter estimates. By doing this, the accuracy of the model increases after each batch, since the parameter estimates are obtained using an increasing amount of information. Next, as in the ILC algorithm, the reference temperature T^r for the next batch is optimized off-line using the updated model in order to follow the desired supersaturation set-point.

The remainder of the paper is organized as follows. First, a model for the batch cooling crystallization process is presented in Section 2. Subsequently, the B2B control framework is discussed in Section 3. The framework is applied in a simulation study in Section 4 and on the pilot-scale crystallizer in Section 5. Finally, overall conclusions and directions for future research are discussed in Section 6.

2. The batch cooling crystallization model

A model of the batch cooling crystallization process is presented in this section. This model is used extensively throughout this paper. First, it is used in Section 3 for the design of the B2B control algorithms. Second, the data-generating system used in Section 4 to represent the crystallizer in the simulation study is a numerical implementation on this model. Finally, the B2B control algorithms based on this model are applied on the real pilot-scale crystallization setup in Section 5.

As previously discussed in the Introduction, in a batch cooling crystallization process a chemical solution is cooled down in a crystallizer. The jacket temperature T_J is the manipulated variable used to steer the crystallizer temperature T.[1] By cooling, the solubility of the solution is lowered, and part of the solute is transferred from the solution to the solid, crystalline phase. Therefore, the concentration C of the solute within the solution decreases. The batch cooling crystallization process is often represented using the so-called *moment model* (Randolph & Larson,

[1] In practice, the jacket temperature T_J is not directly manipulated, but it is controlled by a low-level control loop. The set-point of the low-level controller is the variable that is actually accessible (see the description of experimental set-up in Section 5.1). However, the dynamics of this low-level control loop is usually much faster compared to the other system dynamics, and for this reason it is ignored in the modeling.