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Robust identification and control of batch processes

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Abstract

An approach is proposed for the robust identification and control of batch and semibatch processes. The batch experiments used for model identification are designed by minimizing the magnitude of the parameter uncertainties, and the effect of these uncertainties on the product quality achievable by optimal control is used as a stopping criterion for the identification procedure. The optimal control approach incorporates a quantification of the impact of both parameter and control implementation uncertainties on the performance of the optimal control policy. The approach is applied to the nucleation and growth of crystals with multiple characteristic dimensions, where the nominal parameters used in the simulation study are quantified from experimental data.

Keywords: Optimal control; Parameter estimation; Batch optimization; Crystallization; Population balance equations; Distributed parameter systems

1. Introduction

Batch processing is used in the production of specialty chemicals, polymers, and pharmaceuticals. Since batch processing requires operations over a range of conditions, relevant process models are highly nonlinear. Many batch processes involve complex products, and the lumped and distributed parameter models for these processes have many unknown parameters that must be identified from experimental data.

The exact identification of the parameters is impossible due to unmeasured disturbances and limited and noisy data. This motivates using model identification techniques that quantify the accuracy of the model parameters (Beck & Arnold, 1977; Cooley & Lee, 2001; Featherstone, VanAntwerp & Braatz, 2000; Miller & Rawlings, 1994). Here an identification and control procedure for batch processes is proposed that takes these model parameter uncertainties into account. Batch experiments used for model identification are designed to minimize the magnitude of the parameter uncertainties quantified in the parameter estimation procedure. A quantification of the impact of parameter and control implementation uncertainties on the product quality is used to decide whether more experiments are needed to obtain a higher accuracy model which can result in more accurate predictions of the product quality, and to define the performance objectives for the lower level feedback control loops that implement the optimal control policy. The objective of the optimal control formulation is to compute the control policy that optimizes the worst-case product quality. The robust identification and control procedure is applicable to the design of finite-time optimal control policies for nonlinear lumped and distributed parameter systems, including both batch and semibatch operations. The approach is evaluated through application to the nucleation and growth of crystals with multiple characteristic dimensions, where the nominal parameters used in the simulation study are quantified from experimental data.

The next section provides background on the classical approach to the identification and control of batch processes. It is discussed how the classical approach does not adequately address model uncertainty. This is

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followed by an approach that incorporates the parameter uncertainty description produced by the model identification procedure to design the optimal control policy to be robust to model and control implementation uncertainties. The approach is demonstrated for the nucleation and growth of crystals with two characteristic dimensions from aqueous solution.

2. Background: the classical approach

The following formulation of the batch identification and control problems will be referred to as the classical approach, since it has been extensively studied and applied for nearly three decades.

2.1. Iterative process of model identification

Model identification is an iterative procedure. The first batch experiment is either designed using engineering judgment on how to excite the dynamics of the system, or is computed using initial estimates of the kinetic parameters. Improved parameter estimates and an associated confidence region are computed from the dynamic data collected from the experiment. The parameter and uncertainty information is used to design the next laboratory experiment (Atkinson & Donev, 1992; Beck & Arnold, 1977; Blau, Klimpel & Steiner, 1972; Reilly, Bajramovic, Blau, Branson & Sauerhoff, 1977; Reilly & Blau, 1974). Parameter estimates obtained from this procedure can be much more accurate than estimates obtained without using optimal model-based experimental design. Once the model parameters are considered to be accurate enough, the simulation model is used by a dynamic optimization algorithm to compute the batch control policy, which can include initial conditions and physical design variables as well as the manipulated variables and setpoints to lower level feedback control loops. The relationships between parameter estimation, optimal experimental design, and batch optimal control are illustrated in Fig. 1. The selection between several hypothesized models can be incorporated into this procedure as well (Blau et al., 1972; Gunawan, Ma, Fujiwara & Braatz, 2002; Matthews & Rawlings, 1998); for brevity this is not discussed here.

2.2. Parameter estimation

Let $\boldsymbol{\theta}$ be the vector of model parameters to be determined from experiments. A common formulation for the parameter estimation problem is to minimize:

$$\Phi(\boldsymbol{\theta}) = \sum_{i=1}^{N_m} \sum_{j=1}^{N_{d_i}} w_{ij}(y_{ij} - \tilde{y}_{ij}(\boldsymbol{\theta}))^2 \tag{1}$$

where y_{ij} and $\tilde{y}_{ij}$ are the measurement and model simulation prediction of the ith measured variable at the jth sampling instant, w_{ij} is a weighting factor, N_m is the number of measured variables, and N_{d_i} is the number of sampling instances for the ith measurement. The weights are selected to take the magnitude of the noise for each measurement into account, where larger weights are used for more accurate measurements. Maximum likelihood estimation selects the weights based on the standard deviation of the noise for each measurement (Beck & Arnold, 1977).

The parameter estimates are stochastic variables whose joint distribution can be estimated along with a hyperellipsoidal confidence region that quantifies the accuracy of the parameters:

$$E_\theta \equiv \{\boldsymbol{\theta}:(\boldsymbol{\theta} - \hat{\boldsymbol{\theta}})^T V_\theta^{-1}(\boldsymbol{\theta} - \hat{\boldsymbol{\theta}}) \leq \chi^2_{N_p}(\alpha)\} \tag{2}$$

where α is the confidence level, N_p is the number of parameters, $\chi^2_{N_p}$ is the chi-squared distribution with N_p degrees of freedom, and the parameter covariance matrix V_θ can be estimated using sensitivity equations (Caracotsios & Stewart, 1985; Feehery, Tolsma & Barton, 1997; Li, Petzold & Zhu, 2000) or by using Monte Carlo simulations (Bard, 1974; Featherstone & Braatz, 1998).

It is common in real applications for the matrix V_θ to have a large condition number. In this case, computing the matrix V_θ using sensitivity equations requires the inversion of an ill-conditioned matrix (Beck & Arnold, 1977). Even with double-precision arithmetic, the numerical roundoff errors associated with this matrix inversion step can be large. In this situation it is best to use Monte Carlo simulations to estimate V_θ or to directly estimate E_θ. Monte Carlo simulations avoid the matrix inversion step which causes numerical difficulties for ill-conditioned systems, but involve multiple parameter estimation calculations, which are more computationally expensive.

2.3. Optimal model-based experimental design

Classical model-based experimental design computes the experimental conditions that minimize the volume of the hyperellipsoid Eq. (2), which is equivalent to minimizing (Atkinson & Donev, 1992; Beck & Arnold, 1977):

$$\Psi(\boldsymbol{u}(t)) = \det(V_\theta) \tag{3}$$

where V_θ is the covariance matrix based on the simulation model and current and past data. In Eq. (3), $\boldsymbol{u}$ is the vector of experimental design variables, which can include fixed variables, initial conditions, and functions of time t. This minimization is subject to all experimental constraints $\boldsymbol{u}(t) \in \Omega_{exp}$ where constraints

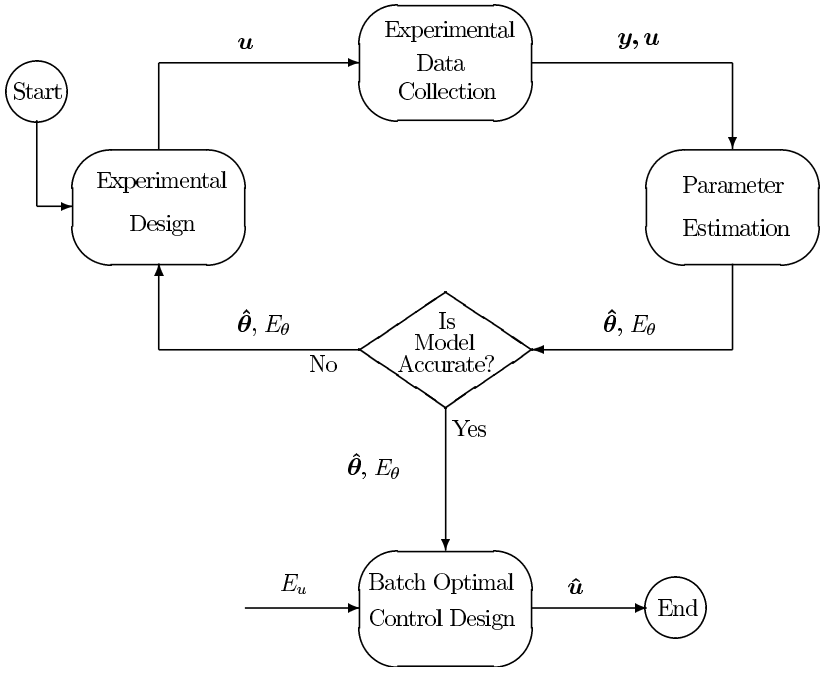

Fig. 1. Iterative process of model identification: **u** represents all experimental design variables (e.g. initial conditions, temperature profile), **y** represents the measurements (e.g. solute concentration), $\hat{\boldsymbol{\theta}}$ is the vector of estimated kinetic parameters, E_θ is a confidence region for the parameters, $\hat{\boldsymbol{u}}$ is the optimal control policy, and E_u is the uncertainty in the implementation of the optimal control policy.

on the states are included by parameterizing the state variables in terms of $\boldsymbol{u}(t)$.

2.4. Optimal control

The batch optimal control problem is formulated as:

$$\min_{\boldsymbol{u}(t)\in\Omega_{control}} \Upsilon(\boldsymbol{u}(t)) \tag{4}$$

where Υ is some desired performance objective at the end of the batch computed from the simulation model, and $\boldsymbol{u}(t)$ contains all variables that can be optimized during batch operations, including initial conditions, manipulated variables, setpoints to lower level feedback control loops, and equipment specifications (e.g. volume, number of units, etc.). The performance objective is typically in terms of a product quality variable such as purity, or can be a monetary objective such as profit or a weighted sum of several product quality and economic variables. The constraints $\Omega_{control}$ ensure that the optimal control policy $\boldsymbol{u}(t)$ can be implemented in practice. While many techniques have been proposed for computing the solution to the optimal control problem, probably the most common approach is using sequential quadratic programming that iteratively calls the batch simulation model (e.g. see discussion by Feehery & Barton, 1999 and references cited therein).

3. Robust identification and control

3.1. Critique on the classical approach

There are several drawbacks to the classical approach for batch identification and control. First, the stopping criterion for determining whether the model parameters are sufficiently accurate is not directly linked to the accuracy of the performance objective computed by the simulation model. Hence the assessment of whether the model is sufficiently accurate is based on engineering guesswork rather than a rigorous assessment in terms of the main purpose of the batch identification procedure, which is to produce a simulation model able to accurately predict the performance objective which is to be optimized by the optimal control algorithm. Second, when computing the optimal control policy the classical approach does not take into account the parameter uncertainty E_θ identified in the parameter estimation step. While it is common to run a few simulations with different model parameter values, this does not take into account the correlations in the model parameter uncertainties that are common in practical problems. A limited number of simulations may not locate a critical combination of parameter uncertainties that has a significant effect on the performance objective. Third, the classical approach ignores control implementation uncertainties, that is, that the optimal control policy cannot be implemented exactly due to disturbances, nonminimum phase behavior, and mea-

surement noise which limits the performance of lower level feedback control loops used to implement the optimal control policy. Past work has shown that control implementation uncertainties are critical for some batch processes (Ma & Braatz, 2001; Ma, Chung & Braatz, 1999).

3.2. Uncertainty analysis for batch processes

Addressing the aforementioned weaknesses of the classical approach requires rigorously taking model parameter and control implementation uncertainties into account in the identification and control algorithms. This need to address robustness of batch control policies to model uncertainties has been recognized by a number of researchers (e.g. see Belanger, 1966; Eaton, Rawlings & Edgar, 1988; Ma & Braatz, 2001; Ma et al., 1999; Miller & Rawlings, 1994; Phenix, Dinaro, Tatang, Tester, Howard & McRae, 1998; Takamatsu, Hashimoto & Ohno, 1970; Visser, Srinivasan, Palanki & Bonvin, 2000 and references cited therein).

One approach to relating model parameter uncertainty to its effect on the performance objective is to fix a perturbation of optimal performance Υ_{opt} inside some allowable region first, then to utilize sensitivity analysis to estimate the size of parameters' confidence region (Takamatsu et al., 1970). While this formulation does not address all of the weaknesses of the classical approach, it is especially notable given the earliness of the contribution. Some more recent papers have focused on developing efficient algorithms for computing the distribution of the performance objective based on the distribution on the parameters (see Tatang, 1995 and references cited therein). These techniques apply to computing distributions on any state variables, or functions of state variables, and so can provide deep insight into the effects of uncertainty on the dynamics of the system. Here we focus on the effects of uncertainty on the performance objective, since its minimization is the goal of batch optimal control.

Here we summarize an approach that explicitly uses the model parameter uncertainty E_θ and control implementation uncertainty E_u to compute bounds on the performance objective (Ma & Braatz, 2001; Ma et al., 1999; Matthews, 1997). More specifically, the approach computes bounds on the performance objective for all parameters and control policies described by the parameter and control implementation uncertainty sets. The approach uses a combination of series expansions and structured singular value analysis to quantify the worst-case performance for the batch crystallization system. The approach is based on the fact that an arbitrary polynomial objective function maximized over an uncertainty set can be written exactly as a skewed structured singular value problem (Braatz & Russell, 1999; Braatz, Young, Doyle & Morari, 1994), and that stability is not an issue for control problems defined only over a finite time horizon. The analysis approach has been applied to unidirectional crystal growth, using experimentally determined models for the nucleation and growth kinetics. The upper and lower bounds for the skewed structured singular value μ_s for these problems was found to be extremely tight as is typically found when μ_s is used to analyze the worst-case performance of continuous-time lumped parameter systems. This approach provides additional information than just using sensitivity calculations to characterize robustness (Belanger, 1966; Caracotsios & Stewart, 1985; Eaton et al., 1988; Takamatsu et al., 1970), since this approach computes explicit bounds on the performance objective.

Let $\hat{\boldsymbol{u}}(t)$ be the nominal control policy, and $\delta\boldsymbol{u}(t)$ and $\delta\boldsymbol{\theta}$ represent the perturbations around nominal values $\hat{\boldsymbol{u}}(t)$ and $\hat{\boldsymbol{\theta}}$, respectively. Then the worst-case performance is:

$$\Upsilon_{max}(\hat{\boldsymbol{u}}(t)) = \max_{\substack{\delta\boldsymbol{\theta}\in E_\theta \\ \delta\mathbf{u}(t)\in E_u}} \Upsilon(\hat{\boldsymbol{u}}(t)+\delta\boldsymbol{u}(t),\ \hat{\boldsymbol{\theta}}+\delta\boldsymbol{\theta}) \tag{5}$$

The parameter uncertainty set E_θ is determined from the parameter estimation algorithm, while the control implementation uncertainty set E_u is determined from an analysis of the disturbances and achievable performance for the feedback controllers that implement the optimal control policy.

3.3. Robust optimal identification and control

The aforementioned analysis tools provide a quantifiable link between the model parameter and control implementation uncertainties and their effect on the performance objective for batch optimization. These analysis tools can be used to modify the classical approach to remove the weaknesses described in Section 3.1.

First, the analysis tools provide a criterion for determining whether the model parameters are sufficiently accurate to stop the iterative model identification procedure. The stopping criterion can be that the performance objective satisfies a robustness specification ($|\Upsilon_{max}-\Upsilon| \le \varepsilon$, where ε is specified by the engineer), or that the worst-case performance objective satisfies some specification ($\Upsilon_{max} < \gamma$, where γ is specified by the engineer). With this approach, robustness of the performance objective is used to determine whether the model is sufficiently accurate.

Second, the analysis tools can be used to explicitly take into account the confidence region E_θ identified from the model identification procedure in the subsequent batch optimal control formulation. This approach replaces the nominal performance objective with the

worst-case performance objective:

$$\min_{\hat{\boldsymbol{u}}(t)\in\Omega_{control}} \Upsilon_{max}(\hat{\boldsymbol{u}}(t)) \tag{6}$$

This min–max formulation is a direct generalization of the robust control design formulations used for linear and weakly nonlinear lumped parameter systems (Morari & Zafiriou, 1989; Skogestad & Postlethwaite, 1996; VanAntwerp & Braatz, 2000; Zhou, Doyle & Glover, 1995) to highly nonlinear lumped and distributed parameter systems.

Third, control implementation uncertainties is directly included in the robust optimal control formulation, since the analysis tools handle both control implementation and model parameter uncertainties. This allows the effect of disturbances on the performance objective to be quantified, so that it can be determined whether it is worthwhile to design the batch process and its control system to further reduce disturbances.

The next section will describe a case study, in which this robust identification and control approach is implemented.

4. Case study: industrial crystallization

Advances in sensor technology and increased global competition have motivated recent activity on the batch control of industrial crystallization processes (see Braatz, 2002; Rawlings, Miller & Witkowski, 1993 and references cited therein). For crystals of organic molecules it is common for the growth rate to be different for different faces, which leads to needle-like crystals or other shapes (Ma, Tafti & Braatz, 2002; Ma, Braatz & Tafti, 2002; Matthews & Rawlings, 1998). While hypothesis mechanisms provide expressions for the kinetics for most crystallization kinetic phenomena (Nyvlt, Sohnel, Matuchova & Broul, 1985; O'Hara & Reid, 1973; Randolph & Larson, 1988), the parameters in these expressions must be estimated experimentally.

4.1. Process model

The distribution in crystal dimensions and other state variables can be modeled using a population balance equation (Hulburt & Katz, 1964; Randolph & Larson, 1988). Here the model equations are written for a batch crystallization process in which the crystals are characterized by two characteristic dimensions, r_1 and r_2. This type of model is appropriate for rod-like crystals, for example Ma and Braatz (2001). It is assumed that nucleation and growth are the dominant kinetic processes. The driving force for the kinetics of these processes is the relative supersaturation:

$$S = (C - C_{sat})/C_{sat} \tag{7}$$

where, C is the solute concentration and C_{sat} is the equilibrium solute concentration. Let $f(r_1, r_2, t)$ be the crystal size distribution function:

$$f(r_1, r_2, t)dr_1dr_2 = \text{the number of particles in the system in the range } r_1 \pm dr_1/2 \text{ and } r_2 \pm dr_2/2 \text{ at time } t \tag{8}$$

The volume of the batch crystallizer is assumed to be constant throughout any given experiment and spatial nonuniformity in the crystallizer is assumed to be negligible.

With size-independent growth rate along each dimension and nucleated crystals of negligible size, the material balance for the crystals is described by the population balance equation:

$$\frac{\partial f}{\partial t} + G_1\frac{\partial f}{\partial r_1} + G_2\frac{\partial f}{\partial r_2} = B(C, T)\delta(r_1)\delta(r_2) \tag{9}$$

where G_1 and G_2 are the growth rates in the r_1 and r_2 dimensions, B is the nucleation rate, and $\delta(\cdot)$ is the Dirac delta function.

The method of moments replaces the population balance equation with a small number of ordinary differential equations (Hulburt & Katz, 1964):

$$\frac{d\mu_{00}}{dt} = B$$
$$\frac{d\mu_{ij}}{dt} = iG_1\mu_{(i-1)j} + jG_2\mu_{i(j-1)}, \quad i+j>0 \tag{10}$$

where, the ij cross-moment is:

$$\mu_{ij} \equiv \int_0^\infty \int_0^\infty r_1^i r_2^j f(r_1, r_2, t)dr_1dr_2. \tag{11}$$

It is assumed that seed crystals are initially present in the crystallizer, and the cross-moments at $t=0$ are computed from the seed distribution. The model description is completed by an expression for the equilibrium solute concentration as a function of temperature (given by Togkalidou, Fujiwara, Patel & Braatz, 2001), expressions for the nucleation and growth kinetics, and a material balance on the solute.

4.2. Parameter estimation problem

The most widely adopted kinetic models are in power law form (Nyvlt et al., 1985):

$$B = k_b S^b \mu_{21} \tag{12}$$
$$G_1 = k_{g1} S^{g_1} \tag{13}$$
$$G_2 = k_{g2} S^{g_2} \tag{14}$$

where, S is the relative supersaturation defined in (Eq. (7)), and k_b, b, k_{g1}, g_1, k_{g2}, and g_2 are kinetic parameters. Hence the vector of model parameters is:

$$\boldsymbol{\theta} = \begin{bmatrix} g_1 \\ k_{g1} \\ g_2 \\ k_{g2} \\ b \\ k_b \end{bmatrix}. \tag{15}$$

Three additional parameters, the activation energies for nucleation and the two growth axes, are required when there are significant temperature variations.

4.3. Optimal experimental design problem

For the two-dimensional crystallization process considered here, **u** represents the temperature and seed characteristics, where the seed is characterized by its initial mass M_{seed}, two mean characteristic lengths $\bar{r}_1$ and $\bar{r}_2$, and the percentage widths W_1 and W_2 (see Fig. 2). The temperature must stay within the operating range of the crystallizer. The experimental design constraints are listed in Table 1.

4.4. Optimal control problem

In a well-mixed batch seeded crystallizer, the final crystal product is determined by the supersaturation profile, the initial seed mass, and the seed crystal size distribution. In this paper, we only consider the case where supersaturation is created by reducing the temperature $T(t)$, although other methods of achieving supersaturation such as antisolvent addition (Charmolue & Rousseau, 1991) can be formulated in a similar manner. Hence the optimal control variables are the mass, mean characteristic lengths, and widths of the distribution for the seed, and the temperature profile. The classical optimal control formulation is a generalization of (Chung, Ma & Braatz, 1999; Miller & Rawlings, 1994):

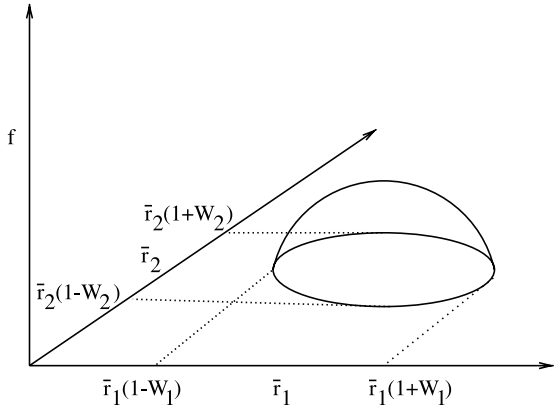

Fig. 2. Seed crystal size distribution: $\bar{r}_1$ and $\bar{r}_2$ are the mean characteristic dimensions; W_1 and W_2 are the widths of the distribution.

Table 1
Experimental design and optimal control constraints

	Minimum	Maximum
Temperature (°C)	23.0	33.0
$dT(t)/dt$ (°C/min)	−0.5	-1.0×10^{-9}
Seed mass (g)	5	40
Percentage width W_1	0.5	0.95
Percentage width W_2	0.5	0.95

$$\begin{aligned} \underset{T(t),M_{seed},\bar{r}_1,\bar{r}_2,W_1,W_2}{\text{minimize}} \quad & \Upsilon \\ \text{subject to} \quad & g_1(t) = T_{min} - T(t) \le 0 \\ & g_2(t) = T(t) - T_{max} \le 0 \\ & g_3(t) = \frac{dT(t)}{dt} - R_{max} \le 0 \\ & g_4(t) = R_{min} - \frac{dT(t)}{dt} \le 0 \\ & g_5 = C(t_{final}) - C_{max} \le 0 \end{aligned} \tag{16}$$

where, Υ is some desired characteristic of the crystals at the end of the batch (details below), which is computed using the cross-moments simulation model. The temperature constraints $g_1(t)$ to $g_4(t)$ ensure that the temperature profile stays within the operating range of the crystallizer. The constraint g_5 is the minimum yield constraint, as the final solute concentration specifies the amount of crystals produced. The bounds on the temperature and rate of change of temperature (T_{min}, T_{max}, R_{min}, R_{max}) and the constraints on the seed characteristics are the same as used in the experimental design and are given in Table 1.

Several objectives have been recommended to favor downstream operations or product quality for one-dimensional crystallizers (Ajinkya & Ray, 1974; Braatz & Hasebe, 2002; Eaton & Rawlings, 1990; Jones, 1974; Rawlings et al., 1993). These objectives can be used for multidimensional crystallization with slight modification. Controlling the aspect ratio of two-dimensional crystals can be another useful optimal control objective (Ma & Braatz, 2001). In this paper, maximizing the average length of the crystals is selected as the objective, which can be calculated directly from the cross-moments:

$$\bar{r}_2 = \frac{\mu_{01}}{\mu_{00}} \tag{17}$$

4.5. Specific system under investigation

The measurements selected for this study are solute concentration, the first-order moments μ_{10} and μ_{01}, and the second-order moments μ_{11}, μ_{20}, and μ_{02}. These moments can be measured using on-line video micro-

scopy or off-line optical microscopy (Braatz, 2002; Gunawan et al., 2002). The zeroth-order moment cannot be measured accurately since it depends strongly on the number of very small crystals, and the very small crystals cannot be seen, even under an optical microscope. The higher order moments cannot be measured as accurately due to their strong dependence on the statistics of the larger particles in solution. Hence, at the jth sampling time the measurements are:

$$\boldsymbol{y}_j = \begin{bmatrix} \mu_{10} \\ \mu_{01} \\ \mu_{11} \\ \mu_{20} \\ \mu_{02} \\ C \end{bmatrix}_j \tag{18}$$

Each measurement is assumed to have normally-distributed noise. The variance for each measurement was estimated from experimental data. The standard deviation for the measurement noise for each moment at jth sampling time was set to 10% of the measurement. The standard deviation of the solute concentration measurement noise was set to 0.005 g solute/g solvent, which is reasonable for ATR-FTIR spectroscopy (Togkalidou, Tung, Sun, Andrews & Braatz, 2002).

Table 2 lists the kinetic parameters for KH_2PO_4 in water which were obtained experimentally in a set of experiments similar to (Gunawan et al., 2002), and are treated as the true values for this study. The cross-moment equations were integrated for a batch production run of 160 min using the LSODES solver (Hindmarsh, 1983).

For all optimization problems, the cooling profile $T(t)$ was parameterized by a linear spline (Wylie & Barrett, 1995) to reduce the infinite-dimensional nonlinear program to a finite-dimensional nonlinear program, which was solved using sequential quadratic programming (Zhou, Tits & Lawrence, 1998).

4.6. Results and discussion

The estimates of model parameters and their uncertainties produced by applying D-optimal experimental design are reported in Table 3. The covariance matrix V_θ that quantifies the size of the confidence ellipsoid was computed by integrating the sensitivity equations along with the model equations (Beck & Arnold, 1977; Caracotsios & Stewart, 1985). Although each step in the model identification procedure quantifies a confidence ellipsoid around the nominal parameters and it is this confidence ellipsoid that is used in all robustness analysis calculations, the confidence ellipsoid for a 6-dimensional space is difficult to interpret visually, so the confidence intervals for each parameter are reported instead. Each iteration of the model identification procedure reduces the confidence intervals on each parameter.

The seed characteristics for each experimental design are reported in Table 4, and the corresponding temperature profiles are reported in Fig. 3. The small amount of seed in Runs 1 and 2 agrees with past experimental design studies for one-dimensional crystallization (Chung, Ma & Braatz, 2000). Large seed size and small seed mass provides less surface area for solute to be incorporated into crystal surfaces, which results in a higher average supersaturation throughout the batch runs. The temperature profiles drop rather quickly for most of the batch runs, which also causes relatively large supersaturation. This large supersaturation is needed to excite the nucleation and growth processes (see Eqs. (12)–(14)) so that accurate kinetic parameters can be obtained when parameter estimation is applied. The

Table 2
Kinetic parameters determined from laboratory data

Parameters	Values	Units
g_1	1.478	Dimensionless
$\ln k_{g1}$	6.596	ln (μm/min)
g_2	1.741	Dimensionless
$\ln k_{g2}$	8.707	ln (μm/s)
b	2.045	Dimensionless
$\ln k_b$	15.318	ln (particles per cm^3 min)

Table 3
Kinetic parameters and their confidence intervals (for $\alpha = 0.95$) produced by parameter estimation

Parameters	Initial guess	Run 1	Runs 1 and 2	Runs 1, 2, and 3
g_1	1.00	1.53 ± 0.40	1.60 ± 0.18	1.61 ± 0.12
$\ln k_{g1}$	2.00	7.29 ± 2.31	6.94 ± 1.02	6.97 ± 0.69
g_2	1.30	1.67 ± 0.40	1.88 ± 0.18	1.90 ± 0.12
$\ln k_{g2}$	5.00	8.55 ± 2.32	9.11 ± 1.04	9.17 ± 0.70
b	2.00	2.80 ± 0.64	2.21 ± 0.28	2.23 ± 0.21
$\ln k_b$	10.0	20.0 ± 3.73	15.7 ± 1.63	15.8 ± 1.21

Table 4
Seed characteristics computed by model-based experimental design

Parameters	Runs 1 and 2	Run 3
M_{seed} (g)	5.0	36.44
$\bar{r}_1$ (μm)	200.0	200.0
$\bar{r}_2$ (μm)	200.0	200.0
W_1	0.95	0.95
W_2	0.5	0.5

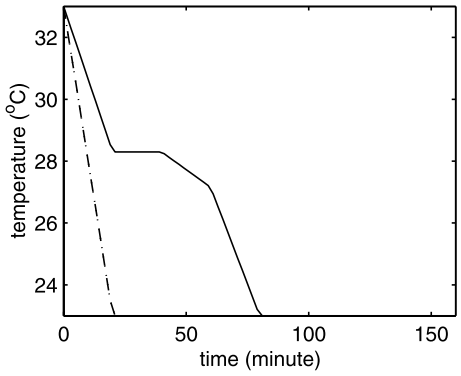

Fig. 3. Temperature profiles computed by model-based experimental design: Run 1 (—), Runs 2 and 3 (- · -).

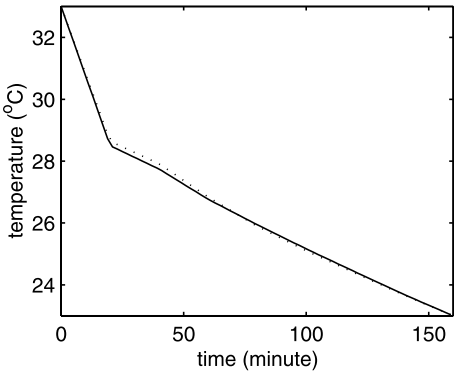

Fig. 4. Optimal temperature profiles for the nominal (—) and worst-case (···) objectives based on a model with kinetic parameters and uncertainty descriptions obtained by applying parameter estimation to simulation data from Runs 1, 2, and 3.

seed characteristics are the same for Runs 1 and 2, and the temperature profiles are the same for Runs 2 and 3.

Table 5 reports the optimal average length and worst-case deviation for crystals obtained by batch optimal control using the nominal and worst-case objectives. Using the worst-case performance objective as the objective in batch optimal control can produce control policies that are more robust than provided by classical optimal control. The robust optimal control approach gives a worst-case deviation in the product quality that is $(40-34)/40 = 15\%$ smaller for estimates of the parameters and parameter uncertainties computed from Runs 1 and 2, and $(31-28)/31 = 10\%$ smaller for Runs 1, 2, and 3. While the improvement in robustness is not huge for this example, it is conjectured that this benefit will tend to increase for systems with more uncertain model parameters and more manipulated variables (which gives more degrees of freedom for improving robustness of the control policies). If the stopping criterion for the model identification procedure is that the worst-case deviation in the product quality should be less than 30 μm, then Run 3 would be the last batch experiment, provided that the worst-case performance objective is used in the batch optimal control formulation.

Fig. 4 shows the optimal temperature profiles obtained using the nominal and worst-case product quality objectives. The temperature profile for the worst-case objective is somewhat less steep for the first half of the batch run, presumably to reduce the potential for excessive nucleation.

Table 5
The optimal average length and worst-case deviation for crystals obtained by batch optimal control using the nominal and worst-case objectives

	Runs 1 and 2		Runs 1, 2, and 3	
	Nominal	Worst-case	Nominal	Worst-case
$\bar{r}_2$ (μm)	374	371	373	371
$\delta\bar{r}_2$ (μm)	40	34	31	28

5. Conclusions

A procedure was proposed for the robust identification and control of batch and semibatch processes. The procedure incorporates robustness analysis tools developed for finite-time control policies that are applicable to nonlinear lumped and distributed parameter systems. The same parameter uncertainty description is used for identification and control, and the batch control procedure can take control implementation uncertainties into account as well. The robustness analysis also provides a stopping criterion for determining when the iterative model identification procedure should end.

It is well-known in the process control community that the best way to reduce the effect of model uncertainties is by implementing an appropriately designed feedback controller. Recently we have been working on integrating the robustness analysis tools into robust feedback controller design for batch and semibatch processes. A similar mathematical framework can be applied, but with the robust optimal control problem solved at each time instance, i.e. in a shrinking-horizon model predictive control formulation (Eaton & Rawlings, 1990). Alternatively, a robust optimization problem can be formulated that directly computes the parameters of a fixed-structure feedback controller. In either case, the control procedure uses the same uncertainty description that is produced by the model identification procedure.

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