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SCALE-UP OF BATCH PROCESSES VIA DECENTRALIZED CONTROL

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Abstract: The economic environment in the specialty chemicals industry requires short times to market and thus the ability to develop new products and processes rapidly. This, in turn, calls for large scale-ups from laboratory to production. Due to scale-related differences in operating conditions, direct extrapolation of conditions obtained in the laboratory is often impossible, especially when terminal objectives must be met and path constraints respected. This paper proposes a decentralized control scheme for scaling-up the operation of batch and semi-batch processes. The targets to be reached are either taken directly from laboratory experiments or adjusted to account for production constraints. Some targets are reached on-line within a given run, while others are implemented on a run-to-run basis. The methodology is illustrated in simulation via the scale-up of a semi-batch reactor.

Keywords: Scale-up, Batch processes, Decentralized control, Batch control, Iterative learning control, Run-to-run control

1. INTRODUCTION

The production of a wide variety of specialty chemicals, pharmaceutical products, food and agricultural products, dyes, composites and polymers calls for small production volumes and variable requirements, which has made batch or semi-batch processing the prime mode of operation. The production of specialty chemicals in batch reactors typically involves following conservative recipes that have been developed in the laboratory with the objective to achieve satisfactory productivity and meet safety and quality requirements. The extrapolation of these recipes to large-scale operation is often made difficult by differences in both equipment and scale. Of particular importance are differences in mass and heat transfer characteristics, residence time distributions, surface-to-volume ratios and heat removal, which can lead to considerable performance degradation and constraint violations (Bisio and Kabel, 1985).

Until recently, conservative approaches to scale-up have been used, mainly performing experiments at intermediate scales of operation before going to large-scale production. However, since the production of specialty chemicals is the subject of intense competition, it has become imperative to go as quickly as possible from a limited number of laboratory experiments to batch production campaigns. Dynamic simulation is avoided because it is often difficult and time consuming to develop sufficiently accurate models (Bonvin, 1998). Current industrial practice uses reaction calorimeters to perform screening experiments and obtain a sound chemical knowledge regarding the reac-

tion phases, the solvent and the type of catalyst. Then, statistical experimental design techniques are used to 'optimize' the process. The key factors (e.g., solvent, type of catalyst, temperatures, reactant concentrations, feed rates, pH, reaction time) that affect the main outcome of the process (e.g., conversion, purity, reaction time, safety indicators) are identified (Roberge, 2004). A limited number of experiments is usually performed, leading to some empirical model that describes the cause-effect relationship in the process, e.g. in the form of a quadratic response surface as a function of the key factors.

When the initial investigations reveal the presence of scale-dependent effects, the experimental design study should be carried out at a larger (pilot plant) scale. However, since there is often only limited time and resources for pilot-plant design, construction and operation, batch processes are often scaled-up directly to production scale by setting some of the independent factors to conservative levels for safety reasons.

Ideally, one wishes to obtain similar performance by extrapolating the laboratory recipe to production scale. However, due to the aforementioned scale-dependent uncertainty, different (and often lesser) performance is obtained and part of the operational requirements may be violated. Fortunately, measurements can be used to compensate the effect of uncertainty by adjusting the available manipulated variables consisting of independent factors and manipulated profiles. With the availability of measurements, scale-up can be considered as a control problem, i.e. the setpoints correspond to the desired specifications and the manipulated variables are the free elements of the batch operation.

Batch processes are characterized by two types of outputs, the run-time outputs (quantities that can be measured on-line) and the run-end outputs (quantities that are only available at final time). Hence, the control problem has many facets: It is multivariable and involves meeting objectives (setpoints) both during the batch and at final time. By appropriately defining the manipulated variables (independent factors and input profiles) and assigning them to the different performance objectives, there results a decentralized control scheme.

The paper is organized as follows. Section 2 reviews the available control approaches for batch processes. The scale-up problem and the formulation of a decentralized control problem are presented in Section 3. The methodology is illustrated in simulation via the scale-up of a semi-batch reactor in Section 4, and conclusions are drawn in Section 5.

2. CONTROL OF BATCH PROCESSES

The control of batch processes differs from that typically found in continuous processes for two main reasons: (i) Batch processes have no steady-state operating point, and (ii) they are characterized by two 'time' variables, the run time t and the run index k. The first reason implies the presence of time-varying profiles for the setpoints and the manipulated variables. The second reason provides additional degrees of freedom for meeting the control objectives since the work does not necessarily have to be completed in a single batch but can be distributed over several batches. However, this brings into picture an additional type of outputs that need to be controlled, i.e. the run-end outputs z. The main difficulty is that these outputs are only available at the end of the run.

The model of a batch process, including the two time variables t and k and the run-end outputs z, can be written generically as:

$$\dot{x}_k(t) = F(x_k(t), u_k(t)), \qquad x_k(0) = x_{0,k} \quad (1)$$

$$y_k(t) = H(x_k(t), u_k(t)) \quad (2)$$

$$z_k = Z(x_k[0, t_f], u_k[0, t_f]) \quad (3)$$

where t refers to the run time and the subscript $(.)_k$ to the run index. x represents the state vector, u the input vector, and t_f the final time. F and H are vector functions and Z is a vector operator. There are two types of outputs, the run-time outputs $y(t)$ –or, equivalently, the complete profiles $y[0, t_f]$– that are measured on-line, and the run-end outputs z evaluated at the end of the batch. The corresponding setpoints express the control objectives, i.e. the desired run-time output profiles $y_{\rm sp}(t)$ or $y_{\rm sp}[0, t_f]$ and the desired run-end output values $z_{\rm sp}$.

Accordingly, there are two types of control objectives, and also different ways of reaching them as illustrated in Figure 1 and discussed next (Bonvin *et al.*, 2005).

Implementation aspect	*Control objectives*	
	Run-time setpoints $y_{sp}(t)$ or $y_{sp}[0,t_f]$	Run-end setpoints z_{sp}
On-line	1 On-line control $u(t) \rightarrow y(t) \rightarrow y[0,t_f]$ (PID)	2 Predictive control $u[t,t_f] \rightarrow z_{pred}(t)$ (MPC)
Run-to-run	3 Iterative learning control $u_k[0,t_f] \rightarrow y_k[0,t_f]$ (ILC)	4 Run-to-run control $\mathcal{U}(\pi_k) = u_k[0,t_f] \rightarrow z_k$ (R2R)

Fig. 1. Control approaches to meet run-time and run-end objectives.

2.1 On-line control of run-time outputs

The approach is similar to that used in continuous processing. However, though certain controlled variables such as temperature may be constant, the key process characteristics such as process gain and time constants can vary considerably. Hence, the need to provide adaptation or efficient feedforward control to handle the main part of the expected variations. Control is typically done using PID techniques or more sophisticated alternatives whenever necessary. Formally, this controller can be written as:

$$u_k(t) = \mathcal{K}(y_k(t), y_{\mathrm{sp}}(t)) \tag{4}$$

where $\mathcal{K}$ is the on-line controller run-time outputs and $y_{\mathrm{sp}}(t)$ the setpoint at time instant t.

2.2 On-line control of run-end outputs

With this approach, it is necessary to predict the run-end outputs based on measurement of the run-time outputs. Model predictive control (MPC) is well suited to that task (Nagy and Braatz, 2003). Note, however, that the (mechanistic) process models needed for prediction are often very simplified and thus of limited accuracy in batch processes. The controller can be written as:

$$u_k(t) = \mathcal{P}(z_{\mathrm{pred},k}(t), z_{\mathrm{sp}}) \tag{5}$$

where $\mathcal{P}$ is the on-line controller for run-end outputs and $z_{\mathrm{pred},k}(t)$ the prediction of z available at time instant t.

2.3 Run-to-run control of run-time outputs

The manipulated variable profiles can be generated using Iterative Learning Control (ILC) that exploits information from previous runs (Moore, 1993). Clearly, this strategy exhibits the limitations of open-loop control for run-time operation, in particular the fact that there is no feedback correction for run-time disturbances. Yet, this scheme is highly efficient for generating a feedforward input term. The controller has the following structure:

$$u_{k+1}[0, t_f] = \mathcal{I}(y_k[0, t_f], y_{\mathrm{sp}}[0, t_f]) \tag{6}$$

where $\mathcal{I}$ is the iterative learning controller for the run-time outputs. It processes the entire profile of the current run to generate the entire manipulated profile for the next run.

2.4 Run-to-run control of run-end outputs

The input profiles are parameterized using the input parameters π_k, i.e. $u_k[0, t_f] = \mathcal{U}(\pi_k)$. This way, the batch process can be seen as a static map between the input parameters π_k, which are set before the run starts, and the run-end outputs z_k. Control can then be implemented using discrete integral control laws, i.e. $\pi_{k+1} = \pi_k + K(z_{\mathrm{sp}} - z_k)$ (François *et al.*, 2005). Formally, the controller can be written as:

$$u_{k+1}[0, t_f] = \mathcal{U}(\pi_{k+1}), \;\; \pi_{k+1} = \mathcal{R}(z_k, z_{\mathrm{sp}}) \tag{7}$$

where $\mathcal{R}$ is the run-to-run controller for the run-end outputs and $\mathcal{U}$ the input parametrization.

Of course, it is possible to combine on-line (feedback) and run-to-run (feedforward) control for either of the outputs. Except for predictive control that involves prediction, all the other control approaches use measurements only, i.e. they do not necessitate a process model for implementation – a very nice feature for the scale-up of batch processes.

3. SCALE-UP PROBLEM

3.1 Operational requirements

Because of differences in equipment and scale, the bounds on time-dependent quantities (also called path constraints) are generally different in production and in the laboratory. Examples of such path constraints include the maximum pressure in a pump, the heat removal capacity of a reactor and the maximum flowrate through a valve. Other constraints are inherent to the chemical system and do not depend on the equipment scale, such as the maximum temperature to prevent decomposition of a chemical compound. Beside path constraints, the operation of batch processes requires satisfying terminal requirements such as quality specifications or some economic perfomance. The path constraints and the terminal requirements constitute the *operational requirements* that have to be respected in production.

3.2 Recipe from the laboratory

The chemist investigates the possible synthesis routes in the laboratory and selects the key chemical parameters such as the solvent and the catalyst. The study of reaction systems in reaction calorimeters or automated laboratory reactors gives valuable information for the selection of a recipe that satisfies the operational requirements. A recipe is characterized by a set of parameters ρ_{lab} and time-varying input variables $u_{lab}(t)$. ρ_{lab} typically includes the feed concentration, the initial conditions and the amount of catalyst, while $u_{lab}(t)$ corresponds to the feed-rate policy and/or the reactor temperature profile.

So far as possible, the laboratory experiments are run in such a way that they respect operational requirements equivalent to those of production via

a *scale-down approach*. Conservatism is typically introduced in recipes so that these requirements can be met also in the presence of (slightly) different conditions. In practice, this is not always possible due to the presence of large uncertainties. The control objective is then to stir the system to the specified operational requirements.

3.3 Control problem

The formulation of the control problem involves the selection of the manipulated and controlled variables. All the elements of the recipe not being chosen as manipulated variables constitute the fixed part of the recipe and are applied without feedback from the process.

The manipulated variable profiles $u(t)$ are parameterized by time-varying arcs and the switching times between the various arcs. The resulting manipulated variables in the control problem consist of a number of arcs $\eta(t)$, and parameters π (including some of the ρ-parameters). The controlled variables include the run-time outputs $y(t)$ and the run-end ouputs z. An objective of the controlled system is to reach the corresponding setpoints, $y_{\rm sp}(t)$ and $z_{\rm sp}$, after as few batch runs as possible.

The number of manipulated variables in the control problem is chosen equal to the number of controlled variables, thus leading to a square multivariable control problem:

- Manipulated variables (MV): $\eta(t)$, π
- Controlled variables (CV): $y(t)$, z
- Control setpoints (SP): $y_{\rm sp}(t)$, $z_{\rm sp}$

A control scheme is proposed in Figure 2, where $y(t)$ is controlled on-line with the on-line controller $\mathcal{K}$ (feedback) and run-to-run with the ILC-controller $\mathcal{I}$ (feedforward). Furthermore, z is controlled on a run-to-run basis using the R2R-controller $\mathcal{R}$. Note that this control scheme does not require a mechanistic process model.

The control problem must be robust towards uncertainty in the sense that the setpoints must remain reachable in the presence of perturbed operating conditions. In practice, flexibility can be introduced by choosing conservative setpoint values for a number of extensive controlled variables such as the batch duration or the productivity (e.g., by backing-off the values obtained from the laboratory experiments). Only those set-points corresponding to controlled variables not dictated by 'hard' operational requirements can be relaxed though. This is illustrated through a semi-batch reactor example in the following section.

4. APPLICATION TO A SEMI-BATCH REACTOR

4.1 Reaction system and operational requirements

Consider the following parallel reaction scheme:

$$A + B \longrightarrow C, \qquad 2\,B \longrightarrow D. \tag{8}$$

The desired product is C, while D is undesired. The reactions are exothermic. A jacketed reactor of 7.5 $\rm m^3$ will be used in production. This reaction scheme represents one step of a rather long synthesis route, and the reactor assigned to this step is part of a multi-purpose plant. Allocation of the different operations in the plant requires that the reaction duration does not exceed 240 min. The final selectivity of D, $y_D(t_f)$ defined below, should not exceed $y_{D,max}$=18%. The lowest achievable cooling jacket temperature is $T_{j,min} = 10$°C. The manipulated input profiles are the feed rate F, and the flowrate of coolant through the jacket F_j. With this, the operational requirements are:

$$T_j(t) \geq 10^{\circ}\mathrm{C} \tag{9}$$

$$y_D(t_f) = \frac{2\ n_D(t_f)}{n_C(t_f) + 2\ n_D(t_f)} \leq 0.18 \tag{10}$$

where n_C and n_D denote the number of moles of C and D in the reactor, respectively.

4.2 Recipe from the laboratory

In the laboratory, a 1.5 l reaction calorimeter is used. The reactor is initially filled with A, and B is added at the constant feed rate $\bar{F}$. The reaction is performed isothermally at $T_r = 40$°C. Though the cooling rate q_c is not a limitation in the laboratory, a scale-down of the production path constraint (9) is proposed as:

$$[q_{c,max}]_{\rm lab} = [(T_r - T_{j,min})UA]_{\rm prod}\,/r, \tag{11}$$

where $r = 5000$ is the scale-up factor and $UA = 3.7 \times 10^4$ J/mol °C the estimated heat transfer capacity of the production reactor. With $(T_r - T_{j,min}) = 30$°C, the maximum cooling rate, $q_{c,max}$, is 222 J/min. Note that $q_{c,max}$ restricts the value of $\bar{F}$.

The fixed part of the recipe includes the initial concentrations of A and B, c_{A_o} and c_{B_o}, the feed concentration of B, $c_{B_{in}}$, the reactor temperature T_r, and the initial volume V_o. The numerical values of the recipe are given in Table 1. The results obtained for a batch run in the laboratory are given in Table 2. Notice the conservatism in the recipe with respect to the operational requirements $y_{D,max}$ and $q_{c,max}$.

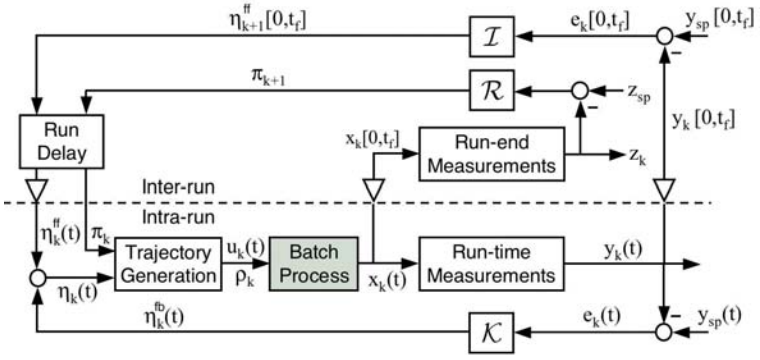

Fig. 2. Control scheme for scale-up implementation. Notice the distinction between intra-run and inter-run activities. The symbol $\bigtriangledown$ is used to indicate a change in viewing the time argument, e.g. from a trajectory to an instantaneous value when going downward and conversely when going upward.

Table 1. Recipe for the laboratory.

T_r	40	°C	$c_{B_{in}}$	5	mol/l
c_{A_o}	0.5	mol/l	c_{B_o}	0	mol/l
V_o	1	l	t_f	240	min
$\bar{F}$	0.0004	l/min			

Table 2. Laboratory results.

$n_C(t_f) = 0.346$ mol	$\max_t(q_c(t)) = 182.6$ J/min
$y_D(t_f) = 0.1706$	

4.3 Control problem for production

Temperature control is achieved via a cascade control scheme, with a slave and a master loop as shown in Figure 3. The master loop receives the reactor temperature setpoint $T_{r,\mathrm{sp}}$, and computes the feedback part of the jacket temperature setpoint $T^{\mathrm{fb}}_{j,\mathrm{sp}}$, while the slave loop adjusts F_j so as to track the jacket temperature setpoint $T_{j,\mathrm{sp}}(t) = T^{\mathrm{ff}}_{j,\mathrm{sp}}(t) + T^{\mathrm{fb}}_{j,\mathrm{sp}}(t)$. The feedforward term for the jacket temperature setpoint $T^{\mathrm{ff}}_{j,\mathrm{sp}}$, is constant at 20°C. Alternatively, an ILC controller could be used to adjust $T^{\mathrm{ff}}_{j,\mathrm{sp}}(t)$. This does not seem necessary here as the feedback action alone allows satisfactory control of the reactor temperature (see Figure 4 below).

The goal of the scale-up is to reproduce in production the final selectivity obtained in the laboratory, while guaranteeing a given productivity of C. Therefore, $n_C(t_f)$ and $y_D(t_f)$ are chosen as the controlled variables. If the laboratory results were directly extrapolated to production, the final productivity of C would be: $[n_C(t_f)]_{\mathrm{prod}} = r \times [n_C(t_f)]_{\mathrm{lab}} = 1730$ mol. However, due to scale-related uncertainty, a certain backoff is introduced on the productivity setpoint to make the control problem more flexible. Two manipulated parameters are needed to control these two run-end outputs. Hence, the feed rate profile $F[0, t_f]$ is parameterized using a 2-stage piecewise-constant approximation (with parameters F_1 and F_2). Overall, the control problem reads:

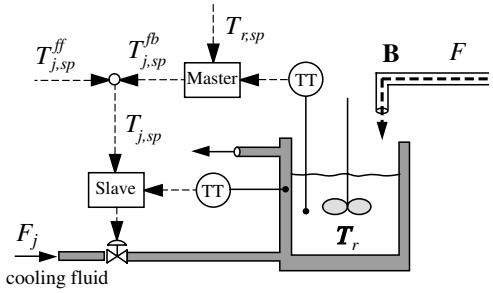

Fig. 3. Cascade structure for temperature control.

- MV: $\eta(t) = T_{j,\mathrm{sp}}(t), \quad \pi = \left[\, F_1,\ F_2 \,\right]^T$
- CV: $y(t) = T_r(t),\ z = \left[\, n_C(t_f),\ y_D(t_f) \,\right]^T$
- SP: $y_{\mathrm{sp}}(t) = 40°\mathrm{C},\ z_{\mathrm{sp}} = \left[\, 1530 \text{ mol},\ 0.17 \,\right]^T$

The proposed control scheme includes the following elements: (i) A cascade scheme, with master controller $\mathcal{K}$, that controls the reactor temperature in real time , and (ii) an integral run-to-run controller $\mathcal{R}$ that controls z by adjusting π:

$$\text{(i)}\ T^{\mathrm{fb}}_{j,\mathrm{sp}}(t) = k_{\mathcal{K}} \left(e(t) + \frac{1}{\tau_{I,\mathcal{K}}} \int_0^t e(\tau) d\tau \right) \quad (12)$$

$$\text{(ii)}\quad \pi_{k+1} = \pi_k + G^{-1} K_{\mathcal{R}} [z_{\mathrm{sp}} - z_k] \quad (13)$$

with $e(t) = T_{r,\mathrm{sp}}(t) - T_r(t)$, $k_{\mathcal{K}}$ the scalar proportional gain, and $\tau_{I,\mathcal{K}}$ the integral time constant of the PI master controller. The integral term of the run-to-run controller is based on the sensitivities of the run-end output errors with respect to π, i.e. $G = \partial(z_{sp} - z)/\partial\pi$, which are estimated experimentally from laboratory or process data (François *et al.*, 2005). Notice that G is a 2×2 matrix, and $K_{\mathcal{R}}$ is the diagonal controller gain matrix.

4.4 Production results

The recipe given in Table 1 is applied in production by scaling-up the extensive variables V_o and $\bar{F}$. Here, $V_o = 5$ m^3 (for all runs), and $F_1 = F_2 = \bar{F}\, r = 2$ l/min for $k = 1$ (subsequently adapted

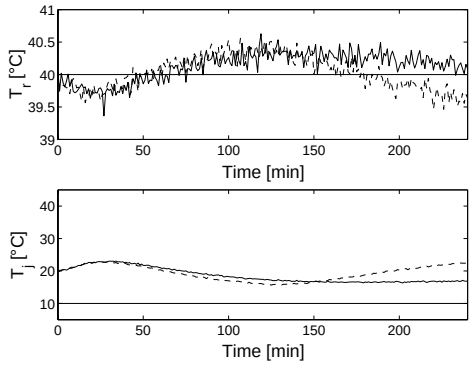

Fig. 4. Temperature profiles for the first and last batches. Top: T_r; bottom: T_j. Solid: k=1; dashed: k=20.

according to (13) for $k > 1$). For the simulation of the production reactor, the kinetic rate constants of the first and second reactions are reduced by 25% and 20%, respectively. Also, Gaussian noise with standard deviation of 0.001mol/l is introduced for the measurement of the final concentrations $c_C(t_f)$ and $c_D(t_f)$, and with standard deviation of 0.1°C for the measurement of $T_r(t)$.

On applying the recipe directly to production, one gets $n_C(t_f) = 1637$ mol and $y_D(t_f) = 0.198$. Therefore, the final selectivity of D violates the operational requirements in the first batch. The batch-to-batch evolution of the run-end outputs $n_C(t_f)$ and $y_D(t_f)$ is shown in Figure 5. It is seen that the requirement on $y_D(t_f)$ is fulfilled after about 5 batches, and the region where the adaptation is within the noise level is attained after 8-10 batches. During the adaptation, parameter F_2 takes a lower value than F_1.

On the other hand, the profiles of T_r and T_j during runs 1 and 20 are shown in Figure 4. Note in particular that the minimum jacket temperature is not reached during the operation, i.e. the operational requirement on $T_{j,min}$ is satisfied.

Clearly, the batch operation is suboptimal in this example, since the productivity is regulated at a somewhat conservative value. A higher production of C could be obtained, e.g., by pushing the operation towards the active operational constraints (9) and (10). However, this self-optimizing alternative should be studied in the context of NCO-tracking (tracking the Necessary Conditions of Optimality) (Srinivasan *et al.*, 2003) and falls beyond the scope of the present paper.

5. CONCLUSIONS

The formulation of a multivariable control problem that is implemented in a two-'time' space has been presented for meeting run-time and run-end objectives for batch processes. This work shows how to construct and operate such a decentralized

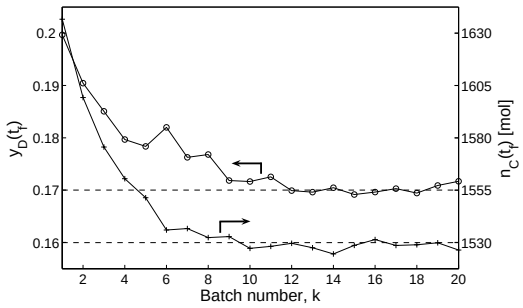

Fig. 5. Approaching the run-end setpoints in a run-to-run manner. (+): $n_C(t_f)$; (∘): $y_D(t_f)$; dashed lines: setpoints.

control approach for scaling-up the operation of batch processes. A control scheme that includes an on-line controller, an ILC controller and a R2R-controller has been proposed. It is illustrated through a simple scale-up example of a semi-batch reactor.

Future work will address the interaction between on-line and run-to-run control activities, as well as the use of self-optimizing decentralized control for the scale-up of batch processes. For this last point, the NCO-tracking methodology will be implemented.

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