# Measurement-based Optimization in the Batch Chemical Industry

D. Bonvin and B. Srinivasan
Institut d'Automatique, École Polytechnique Fédérale de Lausanne
CH-1015 Lausanne, Switzerland

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**Abstract**: The optimal operation of batch processes has attracted more attention in recent years since, in the face of growing competition, it is a natural choice for reducing production costs, improving product quality, and meeting safety requirements and environmental regulations. Since the models currently available in industry are poor and carry a large amount of uncertainty, standard model-based optimization techniques are by and large ineffective, and optimization methods need to rely more on measurements.

This paper presents a new framework, whereby important characteristics of the optimal solution are identified and serve as references to a feedback control scheme. Thus, optimality is achieved by tracking, with no numerical optimization being required on-line. When only batch-end measurements are available, the proposed method leads naturally to an efficient batch-to-batch optimization scheme. The approach is illustrated via simulation of a semi-batch reactor in the presence of uncertainty.

**Keywords:** Dynamic optimization, Optimal control, Batch chemical industry, On-line optimization, Batch-to-batch optimization, Run-to-run optimization.

## 1 Introduction

Batch and semi-batch processes are of considerable importance in the chemical industry. A wide variety of specialty chemicals, pharmaceutical products, and certain types of polymers are manufactured in batch operations. Batch processes are typically used when the production volumes are low, when isolation is required for reasons of sterility or safety, and when frequent changeovers are necessary. With the recent trend in building small flexible plants that are close to the markets of consumption, there has been a renewed interest in batch processing (Macchietto 1998).

This paper considers batch and semi-batch processes in the same manner and, thus herein, the term 'batch processes' includes semi-batch processes as well. The operation of batch processes typically involves following recipes that have been developed in the laboratory in such a way that they can be implemented safely in production. However, owing to differences in both equipment and scale, industrial production almost invariably necessitates modifications of these recipes in order to ensure productivity, safety, quality, and satisfaction of operational constraints (Wiederkehr 1988). The operators use heuristics gained from experience to adjust the process periodically (whenever this is allowed), which leads to slight improvements from batch to batch (Verwater-Lukszo 1998). A certain conservatism is necessary to guarantee feasibility despite process disturbances.

To shorten the time to market (by bypassing an elaborate scale-up process) and to reduce operational costs (by reducing the conservatism), an optimization approach is called for, especially one that can handle

uncertainty explicitly. Operational decisions such as temperature or feed rate profiles are then determined as the solution to an optimization problem, where the objective is of economic nature and the various technical and operational constraints are considered explicitly. Furthermore, due to the repetitive nature of batch processes, these problems can also be addressed on a batch-to-batch basis.

The objectives of this paper are twofold: i) address the industrial practice prevailing in the batch chemical industry and the resulting optimization challenges, and ii) present a novel scheme that uses a few process measurements directly (i.e., without the often difficult step of model refinement) towards the goal of optimization.

The paper is organized as follows. The industrial practice in batch processing is presented in Section 2. Section 3 develops the measurement-based optimization framework, which is then illustrated via a simulated example in Section 4. Finally, conclusions are drawn in Section 5.

# 2 Industrial Practice in Batch Processing

It is difficult to address in generic terms the perspectives prevailing in the batch chemical industry since the processing environments and constraints differ considerably over the various activities (specialty chemicals, pharmaceuticals, agro and bio products, etc.). Thus, the situation specific to the production of intermediates in the specialty chemical industry will be emphasized in this section.

## 2.1 Operational Objectives

The fundamental objective is of economic nature. The investment (in time, personnel, capital, etc.) should pay off, as the invested capital has to compare favorably with other possible investments. This fundamental objective can in turn be expressed in terms of technical objectives and constraints, which are presented next.

- *Productivity:* This is the key word nowadays. However, high productivity requires stable production so as to reduce the amount of corrective manual operations that are costly in terms of production time and personnel. Reducing the time necessary for a given production is particularly interesting when the number of batches per shift can be increased. In multi-product plants, however, equipment constraints (bottlenecks) and logistic issues often limit productivity.
- *Product quality:* Quality is often impaired by the appearance of small amounts of undesired by-products. The presence of impurities (also due to recycled solvents) is very critical since it can turn an acceptable product into waste. Removing impurities is often not possible or can significantly reduce throughput. When the separation of an undesirable by-product is difficult, the selectivity objective may be quite important. Reproducibility of final product composition despite disturbances and batch-to-batch variations is important when the process has to work closely to some quality limit (for example, when the quality limits are tight).
- *Safety aspects:* The safety aspects (runaway, contamination, etc.) are of course very important. Safety requirements can lead to highly conservative operation, especially for slow and exothermic reactions. Here, the real obstacle is the lack of on-line information. If information about the state of the process were available, the process engineer would know how to guarantee safety or react in the case of a latent problem.
- *Time-to-market:* The economic performance is strongly tied to the speed at which a new product/process can be developed. The product lifetime of specialty chemicals is typically shorter than for bulk chemicals. Since the production in campaigns reduces the time to learn, it is necessary to

learn quickly and improve the productivity right away. Also, there is a trend in the specialty chemical industry to skip pilot plant investigations unless the process is difficult to scale up.

## 2.2 Industrial Situation and Needs

In order to attain the aforementioned operational objectives, the industrial situation must be analyzed from both the technical and organizational perspectives. The technical aspects are discussed in detail below. For a discussion on the organizational aspects, the reader is referred to (Bonvin *et al.* 2001).

- *Performance improvement:* From the previous subsection, there is an immediate need to improve the performance of batch processes. Though process improvement could be met effectively via optimization, there have been only a few attempts in industry to optimize operations through mathematical modeling and optimization techniques.

  One of the reasons is that the chemists in the laboratories and operators in the plants were used to thinking in terms of constant values (experimental planning results in static maps between design variables and process performance). New sensors (e.g. spectroscopic measurements) provide time-dependent insights, while increasing computing power (e.g. modern DCS systems) make on-line time-varying decisions possible. As a result, the chemists in the laboratories start to vary process inputs as a function of time.

  There are situations where variable input profiles can be of considerable interest. For example, in batch crystallization, gains of up to 500% can be obtained by adjusting the temperature, the removal of solvent or the addition of a precipitation solvent as functions of time. Large gains are also possible in reactive semi-batch distillation. Furthermore, it is more and more common to adjust the feed rate in semi-batch reactors so as to force the heat generation to match the cooling capacity of the jacket.

- *Constraints:* When optimization is used to improve performance, constraints play an important role since optimal operation is often on the boundary of the feasible region. Industrial processing is naturally characterized by soft and hard constraints related to equipment and operational limitations and to safety aspects. In batch processing, there is the additional effect of terminal constraints (selectivity in reaction systems, purity in separation systems, admissible levels of impurities, etc.). Furthermore, in multi-product batch production, the process has to fit in an existing plant. Thus, ensuring feasible operation comes before the issue of optimality, and process designers normally introduce sufficient conservatism in their design so as to guarantee feasibility even in the worst of conditions.

- *Modeling:* The main bottleneck in using optimization in the batch industry is the fact that standard optimization methods rely heavily on a dynamic model of the process. It is illusory to expect constructing detailed kinetic models since the molecules treated in the batch industry are typically more complex than in the commodity industry and often result in complicated reaction pathways. Thus, the development of dynamic models may exceed one man-year, which is incompatible with the objectives of batch processing.

  Modern software tools such as Aspen Plus, PRO/II, or gPROMs have found wide application to model continuous chemical processes (Marquardt 1996, Pantelides and Britt 1994). The situation is somewhat different in the batch specialty chemistry. Though batch-specific packages such as Batch Plus, BATCHFRAC, CHEMCAD, BatchCAD, or BaSYS are available, they are not generally applicable. Especially the two important unit operations, reaction and crystallization, represent a considerable challenge to model at the industrial level.

  What is often sought in batch processing is simply the ability to predict the batch outcome from knowledge of its initial phase. Modeling is often done empirically using input/output static models on the basis of statistical experimental designs. Sometimes the model is a set of simple linguistic rules based on experience. Often, the model consists of a simple energy balance, or the main dynamics are expressed via a few ordinary differential equations. The modeling objective is not accuracy but

rather the ability to describe the major tradeoffs (e.g. conversion, separation, selectivity) present in the process.

Thus, a large amount of uncertainty stems from modeling (errors in model structure and parameters) since, according to the philosophy of batch processing, little time is available for the modeling task. In addition, uncertainty enters in the reactant quality (changes in feedstock), which represents the main source of batch-to-batch variations. Also, process disturbances and measurement noise contribute to the uncertainty in process evolution (e.g. undetected failure of dosing systems, change in the 'quality' of utilities such as brine temperature, variation in the 'quality' of manual operations such as solid charge).

The problem of scale-up can also be viewed as one of (model) uncertainty. The data available from laboratory studies do not quite extrapolate to the production level. Thus, when the strategies developed in the laboratory are used at the production level, they do carry a fair amount of uncertainty. Furthermore, the pressure to reduce costs and speed up process development calls for large scale-ups with a considerable amount of extrapolation.

So, for an optimization approach to be useful in the batch industry, it should: i) not rely too heavily on an accurate model of the process or, equivalently, be able to handle the uncertainty present in the model, and ii) use the qualitative information that is available with the chemists and operators.

- *Measurements:* Quality measurements are typically available at the end of the batch *via*, for example, off-line chromatographic methods (GC, HPLC, DC, IC). In addition, physical measurements such as temperature, flow, pressure, or pH may be available on-line during the course of the batch. However, they are rather unspecific with respect to the key variables (concentrations) of the chemical process. Other on-line measurements such as conductivity, viscosity, refractive index, torque, spectroscopy, and calorimetry are readily available in the laboratory, but rarely in production. On-line spectroscopy (FTIR, NIR, Raman) (McLennan and Kowalski 1995, Nichols 1988) relies on multivariate calibration for accurate results, i.e., the spectral measurements need to be calibrated with respect to known samples containing all the absorbing species. Pseudo on-line GC and HPLC are less effective in batch processing than with continuous processes due to relatively longer measurement delays.

  When quality measurements are not directly available, state estimation (or soft sensing) is typically utilized. However, physical on-line measurements are often too unspecific for on-line state estimation in batch processes (e.g. heat balance models are too unspecific with respect to the chemical transformations of interest). Current practice indicates that there are very few applications of state estimation in the specialty chemistry. However, state estimation works well in fermentation processes due to the availability of additional physical measurements and the possibility to reconstruct concentrations without the use of kinetic models (Bastin and Dochain 1990).

  As will be described in detail later, the idea used in this paper is to go as close to the constraints as possible with the use of measurements. This way, the conservatism that is needed to guarantee feasibility can be considerably reduced, leading to improved performance.

An interesting feature of batch processing is the fact that batch processes are repeated over time. Thus, the operation of the current batch can be improved by using the off-line measurements available from previous batches. The objective is then to reach the optimum over as few batches as possible. Also, with the tendency to skip pilot plant investigations whenever possible, this type of process improvement is of considerable interest for the initial batches of a new production campaign.

The technical aspects specific to batch processing and the corresponding requirements with respect to process operation improvement are summarized in Table 1. The main conclusion is that a framework that relies on measurements rather than on an accurate model of the process for calculating the optimal inputs is indeed needed. Such a framework is developed and analyzed in the next section.

| Industrial practice | Needs |
|---|---|
| Need to improve performance | Optimization approach |
| Operational and safety constraints | Conservatism to guarantee feasibility |
| Inaccurate model and disturbances | Measurements to compensate the effect of uncertainty |
| On-line and off-line measurements | Tracking of constraints with reduced conservatism |

Table 1: Industrial practice and corresponding needs regarding process improvement

# 3 Measurement-based Optimization

Uncertainty in the form of disturbances and modeling errors is always present in industrial settings. The idea of Measurement-Based Optimization (MBO) is to compensate the effect of uncertainty using measurements. Among the various methodologies possible, the scheme presented here will choose appropriate references, and optimality will be achieved by *tracking* these references. This way, on-line numerical optimization is avoided.

The reference signals should ideally be invariant under uncertainty. The choice of these reference signals is based on the necessary conditions of optimality and constitutes the novelty in the proposed MBO methodology. Thus, the optimization problem is expressed as the satisfaction of selected constraints and the regulation of certain sensitivities around zero.

## 3.1 Description of the Scheme

The following steps are involved in the proposed MBO scheme:

- *Determination of the structure of the optimal solution:* The dynamic optimization problem considered has two types of constraints: i) the path constraints impose bounds on the inputs and the states *during the batch*, and ii) the terminal constraints limit the outcome of the batch *at final time*. For such a problem, the optimal solution can be shown to have the following properties (Bonvin *et al.* 2001):

  1. The inputs are in general discontinuous. The time at which an input switches from one interval to another is called a *switching time*.
  2. Two types of arcs (constraint-seeking and compromise-seeking) are possible between switching instants. In a constraint-seeking arc, the input is determined by a path constraint, while in the other type of interval, the input lies in the interior of the feasible region.
  3. The switching instants can also be constraint-seeking or compromise-seeking, depending on whether they are determined by terminal constraints or not.

  The structure of the optimal solution is described by the type and sequence of arcs, and the set of active terminal constraints. These can be obtained in two ways: (i) educated guess by an experienced operator, or (ii) inspection of the solution obtained from numerical optimization using a simplified model. Quite often, experience dictates the qualitative shape of the inputs. Otherwise, a simplified (tendency) model of the process can be used to compute a numerical solution in which the various arcs are identified.

- *Choice of invariant references for adaptation under uncertainty:* In the presence of uncertainty, the numerical values of the inputs in the various arcs and the switching times might change considerably. However, it is fair to assume that the *type and sequence* of arcs remain unchanged. Thus, even in the presence of uncertainty, the active constraints (both path and terminal) must remain active and the cost sensitivities must remain at zero. This way, optimality is approached by working close to the

active constraints and by regulating the sensitivities around zero. The quantities that remain invariant under uncertainty are referred to as *invariants*. The path constraints and sensitivities will be labeled $I^\eta$ and the terminal constraints and sensitivities $I^\pi$.

It is important to identify the variables that need to be adapted in the presence of uncertainty to meet path and terminal constraints. On the one hand, specific arcs $\eta(t)$ are adjusted to meet path constraints. On the other hand, the parameters $\pi$, which typically consists of switching times between the various arcs and approximations of inputs in compromise-seeking arcs, are adapted to meet terminal objectives.

- *Tracking of invariants using measurements:* The structure given in Figure 1 is proposed to track the invariants by use of feedback. The invariants $I^\eta_{ref} = 0$ and $I^\pi_{ref} = 0$ are tracked with the help of path and terminal feedback controllers, respectively. The trajectory generator computes the current inputs $u(t)$ as a function of $\eta(t)$ and $\pi$ that are generated by the path and terminal controllers. Note that the implementation is *model-free* and *measurement-based*, though a model might be necessary to set up the scheme.

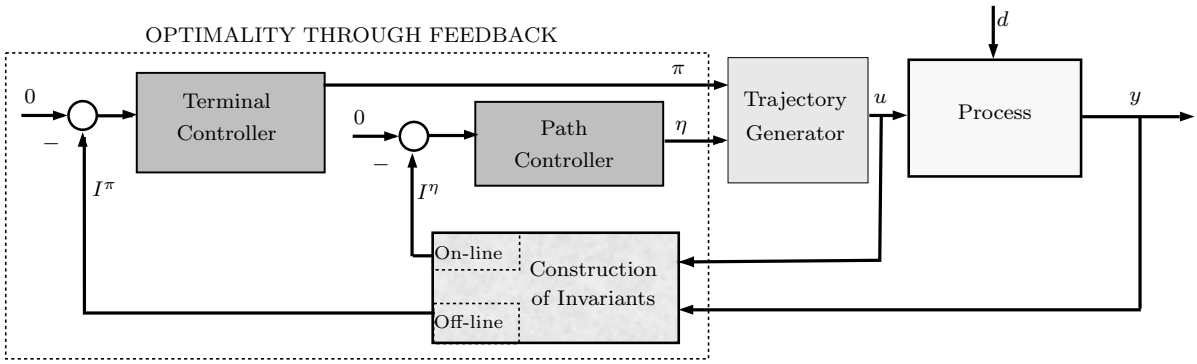


Figure 1: Invariant-based optimization scheme

## 3.2 Practical Applicability of MBO

If the structure of the optimal solution (type and sequence of arcs, active terminal constraints) of the true (unknown) system coincides with that proposed in Step 1 of the procedure, MBO will be capable of optimizing the true system. Thus, the applicability of MBO in practice will depend on (i) the robustness of the proposed input structure with respect to uncertainty (modeling errors and disturbances), and (ii) the ability to measure the path and terminal constraints. These issues are briefly discussed next.

- *Role of the model:* If the structure of the optimal input cannot be obtained from experience or educated guess, then a process model is needed to determine the structure of the optimal input numerically. Thus, the goal of the model is only to provide the correct structure for the optimal input. So, in contrast to model-based optimization approaches or what is sought for simulation purposes, there is no need for a detailed model or for accurate parameter values. The model simply needs to reflect the major tradeoffs specific to the optimization problem at hand. The parts of the model that do not address these effects can be discarded.

- *Construction of invariants from measurements:* If $I^\eta$ and $I^\pi$ are not measured directly, they need to be reconstructed from the available measurements. In the case of constraint-seeking arcs and parameters, the invariants correspond to physical quantities (path or terminal constraints). Off-line measurements of terminal quantities are in general available. In most cases, a path constraint involves a variable that can be measured, or the constraint can be rewritten in terms of a quantity that can be measured.

For example, the path constraint corresponds to a bound on temperature or pressure, or a constraint on heat removal can be rewritten as a constraint on the cooling temperature. In such cases, on-line measurement of the path constraint is directly available. On the other hand, if the path constraint cannot be measured directly, some type of inference or state estimation is necessary.

For computation of sensitivities, either a model of the process or multiple process runs are required, which is typically more difficult. However, since tracking path and terminal constraints is usually much more important than regulating sensitivities, regulating the sensitivities can often be neglected. In case this becomes an issue, the reader is referred to (Bonvin *et al.* 2001).

- *Difference in time scale – on-line vs. off-line measurements:* In general, there is a difference in time scale between the path controller and the terminal controller. The path controller works within a batch using on-line measurements (running index is the batch time $t$). The terminal controller operates on a batch-to-batch basis using off-line measurements (running index is the batch number $k$). If on-line measurements are not available, the path controller is inactive.

- *Disturbance rejection and backoffs from constraints:* The presence of disturbances influences both $\eta(t)$ and $\pi$ and thus $u(t)$. Disturbances affecting $\eta(t)$ within the batch are rejected by the path controller. However, the effect of any disturbance within the batch on $\pi$ cannot be rejected since the terminal controller only works on a batch-to-batch basis. Constant disturbances (e.g. raw material variations) can be rejected from batch-to-batch by the terminal controller.

  In the presence of disturbances and parametric uncertainty that cannot be compensated by feedback, the use of conservative margins, called backoffs, is inevitable to ensure feasibility of the optimization problem (Visser *et al.* 2000). The presence of measurement errors also necessitates backoffs.

# 4 Example - Semi-batch Reactor with a Safety Constraint

## 4.1 Description of the Reaction System

- *Reaction:* $A + B \rightarrow C$.
- *Conditions:* Semi-batch, isothermal.
- *Objective:* Minimize the time needed to produce a given amount of $C$.
- *Manipulated variable:* Feed rate of $B$.
- *Constraints:* Input bounds, constraint on the maximum temperature reached under cooling failure, constraint on the maximum volume.
- *Comments:* This reaction system is adapted from Ubrich *et al.* (1999). In the case of a cooling failure, the system becomes adiabatic. The best strategy is to immediately stop the feed. Yet, due to the presence of unreacted components in the reactor, the reaction goes on. Thus, chemical heat will be released, which causes an increase in temperature. The maximum attainable temperature is given by $T_{cf}$, the temperature reached following a cooling failure:

$$T_{cf}(t) = T(t) + \min(c_A(t), c_B(t)) \frac{(-\Delta H)}{\rho \, c_p} \tag{1}$$

  where the parameters are described in the next subsection, and the term $\min(c_A, c_B)$ serves to calculate the maximum extent of reaction that could occur following the failure.

  Without any constraints, optimal operation would simply consist of adding all the available $B$ at initial time (i.e., batch operation). However, because of the safety constraint, the optimal solution corresponds to feeding $B$ in such a manner that the safety constraint is not violated. Once the volume constraint is attained, the feed rate is set to zero.

## 4.2 Problem Formulation

*Variables and parameters:* $c_X$: Concentration of species X, $n_X$: Number of moles of species X, $V$: Reactor volume, $u$: Feed rate of B, $c_{Bin}$: Inlet concentration of B, $k$: Kinetic parameter, $T$: Reactor temperature, $T_{cf}$: Temperature under cooling failure, $\Delta H$: Reaction enthalpy, $\rho$: Density, and $c_p$: Heat capacity.

*Model equations:*

$$\dot{c_A} = -k\, c_A\, c_B - \frac{u}{V} c_A \qquad c_A(0) = c_{Ao} \tag{2}$$

$$\dot{c_B} = -k\, c_A\, c_B + \frac{u}{V}(c_{Bin} - c_B) \qquad c_B(0) = c_{Bo} \tag{3}$$

$$\dot{V} = u \qquad V(0) = V_o \tag{4}$$

The concentration of $C$ is given by

$$c_C = \frac{c_{Ao} V_o + c_{Co} V_o - c_A V}{V}. \tag{5}$$

The numerical values are given in Table 2.

| | | |
|---|---|---|
| $k$ | 0.0482 | $\frac{\text{l}}{\text{mol h}}$ |
| $T$ | 70 | °C |
| $\Delta H$ | −60000 | $\frac{\text{J}}{\text{mol}}$ |
| $\rho$ | 900 | $\frac{\text{g}}{\text{l}}$ |
| $c_p$ | 4.2 | $\frac{\text{J}}{\text{gK}}$ |
| $c_{Bin}$ | 2 | $\frac{\text{mol}}{\text{l}}$ |

| | | |
|---|---|---|
| $u_{min}$ | 0 | $\frac{\text{l}}{\text{h}}$ |
| $u_{max}$ | 0.1 | $\frac{\text{l}}{\text{h}}$ |
| $T_{cf_{max}}$ | 80 | °C |
| $V_{max}$ | 1 | l |
| $n_{C\,des}$ | 0.6 | mol |

| | | |
|---|---|---|
| $c_{Ao}$ | 2 | $\frac{\text{mol}}{\text{l}}$ |
| $c_{Bo}$ | 0.63 | $\frac{\text{mol}}{\text{l}}$ |
| $V_o$ | 0.7 | l |

Table 2: Model parameters, operating bounds and initial conditions

*Optimization problem:*

$$\begin{aligned} \min_{u(t),t_f} \quad & J = t_f \\ s.t. \quad & (2) - (5) \\ & T_{cf}(t) \le T_{cf_{max}} \\ & V(t_f) \le V_{max} \\ & n_C(t_f) \ge n_{C\,des} \\ & u_{min} \le u \le u_{max} \end{aligned} \tag{6}$$

## 4.3 Determination of the Structure of the Optimal Solution

*Types of arcs:* The possible options for the optimal solution are the input bounds and the path constraint that corresponds to meeting the safety constraint $T_{cf} = T_{cf_{max}}$: (i) $u = u_{min}$, (ii) $u = u_{max}$, and (iii) $u = u_{path}$.

*Specific choice of experimental conditions:* The number and sequence of arcs effectively present in the optimal solution depend on the experimental conditions. Let the experimental conditions be chosen such that the number of moles of $B$ that can be added is less than the initial number of moles of $A$, then $c_B(t) \le c_A(t)$. Thus, since isothermal conditions are chosen, the condition $T_{cf}(t) \le T_{cf_{max}}$ implies $c_B(t) \le c_{B\,max}$ with $c_{B\,max} = \frac{\rho\, c_p (T_{cf_{max}} - T)}{(-\Delta H)}$. Furthermore, the initial condition is so chosen that as much $B$ as possible is present, i.e., $c_{Bo} = c_{B\,max}$.

*Sequence of arcs (Figure 2):*

- Since the initial conditions verify $c_{Bo} = c_{Bmax}$, $u = u_{path}$ is directly applied to keep $c_B = c_{Bmax}$, i.e., $T_{cf} = T_{cf_{max}}$.
- Once $V = V_{max}$ is attained, the input is set to $u = u_{min} = 0$.
- Once $n_C = n_{C\,des}$ is attained, the batch is stopped.

The optimal input and the corresponding evolution of the concentrations of $A$, $B$, and $C$ are given in Figure 2. For the numerical values provided in Table 2, $t_s = 11.44\ h$ and $t_f = 19.80\ h$. Notice that $c_B = c_{Bmax} = 0.63\frac{\text{mol}}{\text{l}}$ in the first interval, which corresponds to $T_{cf} = T_{cf_{max}}$.

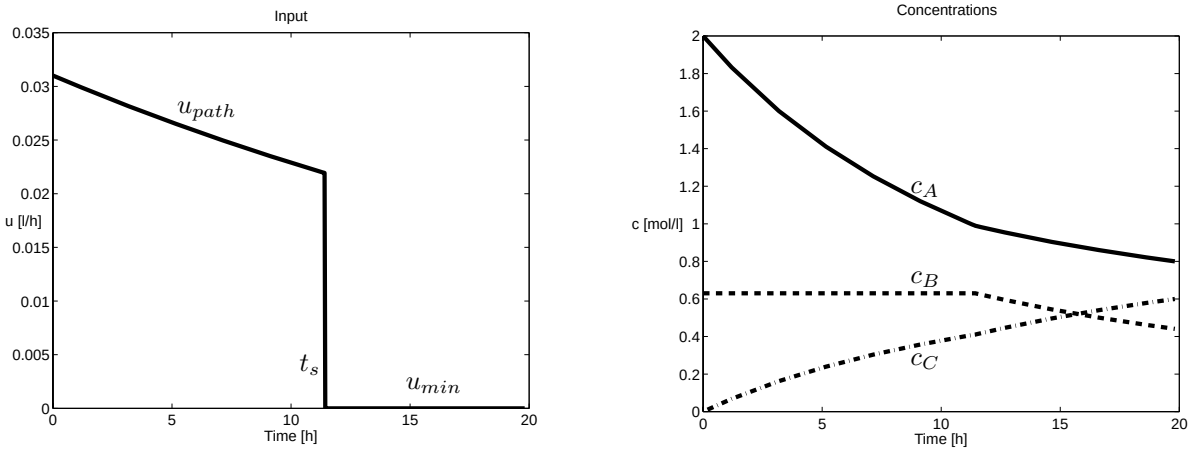


Figure 2: Optimal input and evolution of the concentrations

*Effect of different experimental conditions:*

1. If $c_{Bo} < c_{Bmax}$, the optimal input has an additional arc. Initially, the input is at the upper bound, $u = u_{max}$, in order to attain the path constraint as quickly as possible. Once $c_B$ reaches $c_{Bmax}$, the two arcs presented in Figure 2 form the optimal solution.
2. If the number of moles of $B$ that can be added is larger than the initial number of moles of $A$, the optimal input has an additional arc. Once $c_B(t) = c_A(t)$ is attained, the input switches to its maximum value since it does not affect $T_{cf}$ any longer. Then, when the volume reaches $V = V_{max}$, the input is set to $u = u_{min}$.

*Effect of absence of constraints:*

1. If the safety constraint were not there, it would be optimal to operate in batch mode, where all the $B$ is given at the beginning, leading to $t_f = 17.3$ h. So, the "price" to pay for safety is a longer time (19.8 h) to attain the same conversion.
2. Without the volume constraint, the optimal solution would correspond to feeding $B$ along $u_{path}$ until the desired amount of $C$ is produced. Since more $B$ could be added this way, the final time would reduce to $t_f = 18.4$ h.

## 4.4 Choice of Invariant References for Adaptation under Uncertainty

In practice, there can be considerable uncertainty in the stoichiometric and kinetic models. This is reflected here as uncertainty in the kinetic parameter $k$ in the range $0.03 \leq k \leq 0.06$ (the nominal value $k = 0.0482$ used in the simulation is assumed to be unknown).

| | Optimization Scenario | Terminal Constraint $n_C(t_f)$ mol ($n_{C\,des} = 0.6$ mol) | Path Constraint $\max_t c_B(t)$ mol/l ($c_{B\,max} = 0.63$ $\frac{\text{mol}}{\text{l}}$) | Cost (h) | Loss |
|---|---|---|---|---|---|
| 1 | Open-loop application of optimal conservative input | 0.73 | 0.51 | 31.83 | 60% |
| 2 | Adaptation of $t_f$ using off-line measurements of $n_C(t_f)$ (with 5% noise) | 0.62 | 0.51 | 23.18 | 17% |
| | Adaptation of $t_f$ using off-line measurements of $n_C(t_f)$ (no noise) | 0.60 | 0.51 | 21.94 | 11% |
| 3 | Adaptation of $u_{path}(t)$ and $t_f$ using on-line and off-line measurements (with 5% noise) | 0.62 | 0.61 | 21.42 | 8% |
| | Adaptation of $u_{path}(t)$ and $t_f$ using on-line and off-line measurements (no noise) | 0.60 | 0.63 | 19.80 | 0% |

Table 3: Invariant-based optimization. Results averaged over 100 noise realizations, each consisting of run-to-run adaptation over 50 batches.

During the batch, $u_{path}$ could be adjusted via feedback to compensate for the uncertainty and, with the terminology introduced in Section 3, $\eta(t) = u_{path}(t)$ . The switching time $t_s$ is determined upon reaching $V_{max}$. Since $V_o$ is known, the volume that can be added, $V_{added}$, is simply $V_{max} - V_o$. Thus, starting with a feed tank of volume $V_{added}$, the reactant $B$ is fed at the rate $u_{path}$ until the feed tank is empty, which determines $t_s$. The only parameter that needs to be adjusted amidst uncertainty is the terminal time $t_f$ and, thus, $\pi = t_f$. The invariants in this example correspond to the path and terminal constraints of the optimization problem, i.e., $I^\eta = c_B(t) - c_{B\,max}$ and $I^\pi = n_C(t_f) - n_{C\,des}$. In other words, $u_{path}(t)$ could be adjusted using on-line measurement of $c_B(t)$ to satisfy the path constraint $c_B(t) = c_{B\,max}$, and $t_f$ could be adjusted in a run-to-run manner using off-line measurement of $n_C(t_f)$ in order to meet the terminal constraint $n_C(t_f) = n_{C\,des}$.

## 4.5 Tracking of Invariants using Measurements

With respect to the measurements available, different optimization scenarios are considered:

1. *No measurements:* In order not to violate the constraints, a conservative feed profile is designed corresponding to the optimal solution for $k = k_{min}$. The conservative input has the same two arcs $u_{path}$ and $u_{min}$ as the optimal input in Figure 2 with the difference that $u_{path}^{cons}$ is lower and longer (in the range 0.019 l/h – 0.014 l/h, $t_s = 18.33$ h and $t_f = 31.83$ h). This conservative feed profile is applied open loop to the simulated nominal plant.
2. *Batch-end measurements:* Only the measurement of $n_C(t_f)$ is available and, thus, the final time $t_f$ is updated in a batch-to-batch manner. For the first interval, $u_{path} = u_{path}^{cons}$ is applied.
3. *On-line and batch-end measurements:* On-line measurement of $c_B(t)$ is available (in practice, $c_B$ can be inferred from estimation of thermal conversion). The path constraint is kept active using the feedback $u_{path}(t) = u_{path}^{cons} + k_p\,(c_{B\,max} - c_B(t)) + k_i \int_0^t (c_{B\,max} - c_B(t))dt$, where $k_p$ and $k_i$ are the parameters of a PI controller. In addition, the final time $t_f$ is updated in a batch-to-batch manner.

The cases of both noise-free and noisy measurements (5% relative Gaussian measurement noise) are considered. The results are given in Table 3. If the measurements are noisy, conservative margins (backoffs) need

to be incorporated so as to guarantee feasibility. The backoffs used are 0.02 mol for $n_{Cdes}$ and 0.02 mol/l for $c_{Bmax}$.

It is seen that with only off-line (or batch-end) measurements, the terminal constraint can be satisfied by adapting the final time $t_f$. The evolution of the final time (which also represents the cost) for batch-to-batch optimization is shown in Figure 3. It can be seen that the solution gets close to the optimum within a few batches.

If, in addition, on-line measurements are available, the path constraint can be kept active as well. Thus, it is possible to get quite close to the optimum by using measurements.

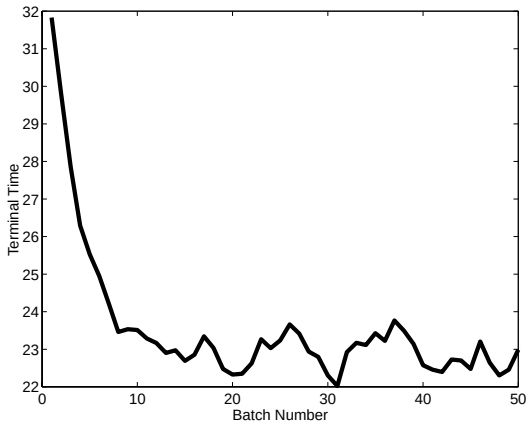


Figure 3: Evolution of final time for one realization of the batch-to-batch optimization with only batch-end measurements of $n_C(t_f)$ (5% measurement noise)

## 4.6 Discussion

The MBO scheme requires no model as long as it is accepted that the optimal solution qualitatively involves two arcs, first $u_{path}$ and then $u_{min}$. As far as the implementation is concerned, $u_{path}$ is determined by a PI-controller upon tracking $c_{Bmax}$, and $u_{min} = 0$. The switching time between the two arcs is determined upon reaching $V_{max}$ and, thus, is determined implicitly. The final time $t_f$ is adjusted in a run-to-run manner by a PI-controller that forces $n_C(t_f)$ to meet $n_{Cdes}$.

Assume that, in addition to the two modeled reactions, the true system also includes $C + B \rightarrow D$, $C \rightarrow E$. This would not affect the type and sequence of arcs ($u_{path}$ followed by $u_{min}$) since the two additional reactions do not introduce the possibility of competition for the reactant $B$ (Srinivasan *et al.* 2001). Thus, the proposed scheme would be equally applicable even in the presence of the two additional reactions.

As a final remark, it is important to stress that the model parameters given in Table 2 are not used for calculating the optimal feed rate. Only the off-line measurement of $n_C(t_f)$ is necessary for implementing the batch-to-batch optimization (Scenario 2). The complete MBO scheme requires in addition the on-line measurement of $c_B(t)$.

# 5 Conclusions

The lack of reliable models and the presence of uncertainty have favored the investigation of process improvement *via* utilization of measurements (sometimes on-line, most often off-line). The major contribution towards process improvement of a constrained batch process is through operation on active constraints. Thus, a feedback-based framework has been proposed to keep the system 'close' to the active constraints. If only off-line measurements are available, this framework results in a batch-to batch optimization scheme with the objective to meet the terminal constraints within a few batches. If on-line measurements are available, the path constraints can also be kept active.

The proposed invariant-based optimization scheme addresses most of the industrial requirements listed in Table 1. More specifically,

- it is aimed at process improvement via the use of time-dependent inputs,
- it guarantees feasibility since the constraints are approached from the safe side,
- it is model-independent as far as the implementation is concerned and is robust against uncertainty since signals that are invariant under uncertainty are tracked,
- if necessary, it uses off-line measurements only.

It is possible to perceive the proposed feedback-based optimization strategy from an industrial perspective. Classical PID control is the most popular technique used currently in industry, and trading it to attain optimality is unacceptable industrially. Therefore, in contrast to most model-based optimization studies, this work attempts to use feedback control for the sake of optimality. In this sense, the approach has great industrial potential and should help take optimization to the batch chemical industry.

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