Robust Dynamic Optimization of a
Semi-Batch Emulsion Polymerization
Process with Parametric Uncertainties
- A Heuristic Approach -

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Abstract:
Optimized exothermic semi-batch emulsion polymerization typically exhibits active arcs of the constraints for the reactor temperature. Given parametric uncertainties in the process model, this might lead to constraint violations which are a safety concern. Therefore, an approach is investigated, such that constraint violations are less likely and is hence robust feasible. The two-model approach is presented as an approximate solution. Therein, two models, the nominal and a worst-case model are optimized simultaneously. Because of the challenges in defining the worst-case, a heuristic method is presented to define the worst-case parameter and its parameter value. The results of the process optimization with the two-model approach are compared with the results of the optimization with the nominal model solely. In addition the feasibility of both optimization strategies is compared by simulating hundred different scenarios with random parameter values from the uncertainty set. The presented approximation does not guarantee robust feasibility, but path constraint violations are less likely due to the introduced conservatism compared to the original optimization.

Keywords: Robust dynamic optimization, parameter uncertainty, uncertain dynamic systems, optimal control problem

1. INTRODUCTION

Polymerization and other batch processes are difficult to control, because of their unsteady states and dynamics which need to be considered. Optimization and control of semi-batch and batch processes are widely studied, but the application to industrial processes is due to different obstacles only rarely the case (Schild et al., 2014). Optimization of the processes could reduce production costs, improve product quality still satisfying the safety requirements (Srinivasan et al., 2003). But as Bonvin (1998) stated, one of the biggest challenges for the industry is safety, which cannot be ensured, if there are uncertainties in the process model. The field of robust control is an active research topic (Lucia et al., 2012; Streif et al., 2014).

Considering models with parametric uncertainties, there are mainly three different approaches. A short overview of them is given by Ruppen et al. (1995):

- Nominal deterministic control, which is only suitable, when the optimal control profiles are insensitive to the uncertain parameters.
- Considering a probability distribution of the uncertain parameter value.
- A min-max approach that yields the best result for the worst expected realization of the possible parameter values. However it gives poor results for the representative values (Lepore et al., 2007).

Most of these approaches consider the influence of the uncertainty to the objective function and to the constraints equivalently. An exception is given by Loeblein et al. (1999), who sets his focus to robustly satisfying the path constraints with a back-off strategy of the path constraints. Herein, the two-model approach is presented, which also has the aim of robustly satisfying constraints. The two-model approach is a combination of the min-max approach and the sampled multi-model approach of Terwiesch and Agarwal (1992), with only considering two models instead of multiple. This has the advantage that one is able to solve the problem in real-time and only with the focus to robustly satisfying the constraints.

2. DYNAMIC OPTIMIZATION

2.1 Nominal Case

In this section the dynamic optimization problem (optimal control problem) is introduced. This problem without accounting uncertainty in the parameter values $p$ is called the nominal case in the following. For better readability in the equations the time interval $T := [t_0, t_f]$ is introduced, which starts at the time $t_0$ and ends with the final batch
time $t_f$. Furthermore the variables $z(t) := (x(t), y(t))$ is introduced, combining the differential variables $x$ and the algebraic variables $y$. The nominal case of the dynamic optimization problem is given by

$$\begin{align*}
\min_{u,z,t_f} & \quad \Phi(x(t_f)) \\
\text{s.t.} & \quad \dot{x}(t) = f(t, z(t), u(t), p^{\text{nom}}), \quad \forall t \in T \\
& \quad 0 = g(t, z(t), u(t), p^{\text{nom}}), \quad \forall t \in T \\
& \quad x(t_0) = x_0, \\
& \quad 0 \geq h(t, z(t), u(t), p^{\text{nom}}), \quad \forall t \in T \\
& \quad 0 \geq e(t_f, z(t_f), p^{\text{nom}}),
\end{align*}$$

(1a)

where $\Phi$ is the objective function and $u$ are the degrees of freedom (time-dependent controls). The optimal control problem constraints contain the dynamic model equations for the process, which consists of the algebraic equations (1c) and the differential equations (1b) with the initial conditions (1d). The additional constraints are subdivided into path constraints (1e), which have to be satisfied over the whole time horizon, and endpoint constraints (1f), which have to be fulfilled at the final time $t_f$.

The nominal parameter values $p^{\text{nom}}$ are often the estimated parameter values based on experimental data. In addition to the estimated value there is an uncertainty range in each estimated parameter value. These uncertainty ranges span a (polyhedral) uncertainty set $P$. If the constraints should be satisfied for all parameters of the uncertainty set $P$, the dynamic optimization results in a semi-infinite program (Polak, 1987; López and Still, 2007), but with additional dynamic behavior. Even for the nominal case, the path constraints can be interpreted as a semi-infinite program (Sachs, 1998; Fu et al., 2015), due to the infinite time points of the time horizon, where the constraints have to be satisfied. In this work it is assumed that a well-adapted time grid is an appropriate approximation for the path constraints of the semi-infinite dynamic optimization problem in time. The semi-infinite constraints due to the uncertainty set $P$ are in this work approximated by the two-model approach (Hartwich, 2013).

### 2.2 Two-Model Approach

The focus of the two-model approach is to robustly satisfy the constraints, which might be safety related. For this robust feasibility not only the nominal model is optimized, but rather the nominal model together with a worst-case model simultaneously. The influence of the uncertain parameters to the endpoint constraints and the objective function value is not critical for the process operation. Thus, only the nominal model is used for these functions.

In the two-model approximation the variables are doubled. There is one set of equations with the estimated parameters and is denoted with $\text{nom}$. The other set presents a worst case at a critical time point with respect to a certain path constraint and is indicated with $\text{wc}$. The objective is only affected by the nominal set of equations, whereas the path constraints have to be satisfied from each. For better readability in the following equations, it is $z^i(t) := (x^i(t), y^i(t))$ with $i \in \{\text{nom}, \text{wc}\}$. The two-model approximation of the robust feasible optimal control problem can be described as:

$$\begin{align*}
\min_{u,z^0,z^{wc},t_f} & \quad \Phi(x^{\text{nom}}(t_f)) \\
\text{s.t.} & \quad \dot{x}^{\text{nom}}(t) = f(t, z^{\text{nom}}(t), u(t), p^{\text{nom}}), \quad \forall t \in T \\
& \quad 0 = g(t, z^{\text{nom}}(t), u(t), p^{\text{nom}}), \quad \forall t \in T \\
& \quad x^{\text{nom}}(t_0) = x_0(p^{\text{nom}}), \\
& \quad 0 \geq c(t_f, z^{\text{nom}}(t_f), p^{\text{nom}}), \\
& \quad z^{\text{wc}}(t) = f(t, z^{\text{wc}}(t), u(t), p^{\text{wc}}), \quad \forall t \in T \\
& \quad 0 = g(t, z^{\text{wc}}(t), u(t), p^{\text{wc}}), \quad \forall t \in T \\
& \quad x^{\text{wc}}(t_0) = x_0(p^{\text{wc}}), \\
& \quad 0 \geq h(t, z^{\text{wc}}(t), u(t), p^{\text{wc}}), \quad \forall t \in T,
\end{align*}$$

(2a)

where $p^{\text{nom}}$ are the estimated values with the uncertainty set $P$ and $p^{\text{wc}}$ the worst-case parameter values. The challenge is to define these worst-case parameter values. In general the worst case does not necessarily lie at the bounds of the uncertainty range $P$ and is state and time-dependent. Therefore in this work a value is heuristically selected and is used to represent the worst case. This heuristic approach is based on a sensitivity analysis at its optimum and presented in Section 4.2 after the optimization of the nominal case.

### 3. CASE STUDY: TWO MONOMER SEMI-BATCH EMULSION POLYMERIZATION PROCESS

The importance of robust control for polymerization processes is increasing due to the high industrial demands. An overview of polymerization processes is given in Asua (2007). The semi-batch emulsion polymerization for a two monomer co-polymerization is considered as a case study:

**Reaction:** The seeded polymerization is characterized by complex reaction kinetics, consisting of the following reaction steps: initialization, highly exothermic propagation, back biting, tertiary propagation, transformation and termination. In emulsion polymerization three phases form, namely aqueous phase, polymer particles and monomer droplets. The monomers are assumed to react with the polymer in the particle phase only, and the reaction in the aqueous phase is neglected. As soon as the aqueous phase is saturated with monomers monomer droplets form. This happens if there is more monomer fed into the reactor, than it can be absorbed in the particle phase to react. Until now, most of the production is recipe based and the isothermal reactor temperate $T_R$ is realized with a PID control. The emulsifier, the initiator and the monomers are fed for a certain fixed time horizon to the reactor. Thereafter the feed is stopped and the reactor temperature $T_R$ is held constant to achieve the desired product properties. This feeding period is embraced by a heating and a holding period, resulting in the overall polymerization process. In this work the focus is the feeding period, because it represents the most challenging control part (Lucia et al., 2014).

**Conditions:** The reactor considered is a semi-batch, ideally mixed 10l reactor and is operated non-isothermally with a jacket for heating and cooling. The according flow-sheet is sketched in Figure 1. In large industrial reactors the cooling capacity is the limiting factor, hence the feed is cooled to handle the exothermic reaction. In a 10l reactor this problem does not occur. Therefore the feed is heated to 350K to simulate this limited cooling capacity.
Model Equations: The used mass and energy balances as well as kinetics, closure equations and equations related to phase equilibrium can be found in Krämer (2005). In addition, moment equations have been used to describe polymer properties. The full dynamic model of the polymerization process consists of 117 algebraic equations and 22 differential equations and is similar to the model from Zubov et al. (2012).

Objective: Maximizing the space-time-yield (mass of product per batch time and reactor volume).

Solution Strategy: The optimal control problem is solved with DyOS (Schlegel et al., 2005). The problem is iteratively integrated with LIMEX and optimized with SNOPT. The time-variant controls are approximated as piecewise constant parameters.

Controls: There are two controls with each 20 equidistant, piecewise constant degrees of freedom in time: the mass inlet flow of monomer \( \dot{m}_{\text{in}} \) and the jacket inlet temperature \( T_{\text{jin}} \). The controls are bounded. The monomer mass inlet flow can vary between \( \dot{m}_{\text{in}} \in [0 \frac{\text{kg}}{\text{s}}, 1 \frac{\text{kg}}{\text{s}}] \) and the jacket inlet temperature has a range of \( T_{\text{jin}} \in [300 \text{K}, 380 \text{K}] \).

Constraints: The reactor temperature \( T_R \) is a safety path constraint and it has to be below an upper value \( T_{\text{Rmax}} = 365 \text{K} \) during the whole process. The endpoint polymer quality constraints consists of the number average molecular weight \( M_N \in [40 \frac{\text{kg}}{\text{mol}}, 60 \frac{\text{kg}}{\text{mol}}] \), the fraction of polymerized monomer 1 in the polymer \( y_1 \in [0.5, 0.7] \) and the overall conversion \( X_{\text{tot}} \in [0.98, 1] \). In addition the reactor volume \( V_R < 10 \text{l} \) is limited and hence another endpoint constraint.

Parameters related to safety path constraint \( T_{\text{Rmax}} \): The reaction heat transfer coefficient between the jacket and the reactor content \( kA \) and the propagation rate constants of both monomers \( k_{p1} \) are difficult to measure and are therefore assumed to have an uncertainty interval of \( \pm 50\% \). In contrast the heat of reaction for each monomer \( \Delta H_1 \) and \( \Delta H_2 \) can measured well such that their uncertainty interval is assumed to have a range of \( \pm 10\% \). Herein these values are based on prior studies. Typically together with parameter estimation, an estimate of the uncertainty range is obtained.

4. RESULTS

4.1 Optimization Results - Nominal Case

The optimization of the nominal case fulfills the constraints and gives an optimized operation, which takes only 7129s and the objective function value of the space-time-yield is \( 0.057 \frac{\text{kg}}{\text{m}^3 \cdot \text{s}} \).

Figure 2 shows that the reactor temperature \( T_R \) increases rapidly to its upper limit \( T_{\text{Rmax}} \) and exhibits active path constraint until 4500s. Towards the end of the batch process the temperature decreases.

Figure 3 gives the optimal trajectories of the controls. The monomer mass inlet flow \( \dot{m}_{\text{in}} \) is in the upper figure and the temperature of the jacket inlet stream \( T_{\text{jin}} \) compared to the reactor temperature \( T_R \) (dotted line) in the lower figure.

In the first part of the process before the reactor temperature reaches its upper bound, the reactor is heated. This can be seen in Figure 3, where the jacket inlet temperature...
If the reactor temperature $T_{Jin}$ is higher than the reactor temperature $T_R$. Thereafter the reactor is cooled until the end of the process. At the time, where the temperature constraint is active, the jacket inlet temperature $T_{Jin}$ is at its lower bound and hence at maximum cooling. With the last feeding peak, the process is cooled a bit less to convert the remaining monomers.

Due to the fact that the active temperature constraint is related to safety concerns, the operation at this optimum is dangerous for application with the likely associated uncertainties in the parameter values. As an example a worst case is simulated to show the possible constraint violation of the reactor temperature $T_R^{wc}$ in Figure 2. This is derived by simulating the model with the optimal inputs from the nominal case optimization, but increasing the heat transfer coefficients $k_{p0i}$ by 50%, the reaction enthalpies $\Delta H_i$ by 15% and decrease the heat transfer coefficient by 20%. To avoid this behavior, the two-model approach is applied to the process after finding the worst-case parameter values based on the sensitivity analysis in the following section.

4.2 Sensitivity Analysis

To decide which parameter values should be chosen for the worst-case model the relative sensitivities

$$T_R(p)(\tau) = \frac{p}{T_{R}^{\text{nom}}(\tau)} \frac{\partial T_{R}^{\text{max}}}{\partial p} \bigg|_{\tau=\tau, p=p^{\text{nom}}, u=u^{\text{nom}}}$$

are analyzed, where $u = u^{\text{nom}}$ indicates the optimal solution of the nominal case optimization. The sensitivities give a hint about the influence strength to the temperature (path constraint) by changing the parameter value $p$.

In Figure 4 the sensitivity of the heat transfer coefficient $kA$ with respect to the reactor temperature $T_R$ has a sign change at that point in time where the heating of the process stops and the cooling phase starts. When the process is heated, the reactor temperature $T_R$ increases if the heat transfer coefficient $kA$ is higher. In contrast, when the process is cooled, an increase in the reactor temperature $T_R$ is induced if the heat transfer coefficient $kA$ is lower.

The sensitivities of the reactor temperature $T_R$ with respect to the propagation rate constants $k_{p0i}$ for both monomers in Figure 5 are positive until approximately 5800s. After that the sensitivity related to the monomer 1 becomes negative.

The sensitivity of the reactor temperature $T_R$ with respect to the reaction enthalpies $\Delta H_i$ for both monomers in Figure 6 have solely positive values over the whole time horizon.

4.3 Selection of the Worst-Case Parameter Values

The results of the sensitivity analysis give the direction in which the parameter value creates a worst case, but it does not provide any information where the worst case lies in the uncertainty interval. We assume it to be at the boundary of the interval if the sensitivity has no sign change. The worst-case parameter values are chosen based on the analysis combined with process knowledge as explained in the following.

The heat transfer coefficient $kA$ has a large uncertainty range, because it is difficult to measure and it is approximated with a constant value, even though it actually changes over time. The sensitivity for the heat transfer coefficient $kA$ has a sign change. That means for a maximal reactor temperature $T_R$, the value of the heat transfer coefficient $kA$ should be maximal in the beginning and afterwards minimal ($kA_{\text{wc}} = 0.5kA_{\text{nom}}$). Consequently it is problematic to create a worst case with the minimal or maximal heat transfer coefficient $kA$. In the following we choose $kA_{\text{wc}} = 0.8kA_{\text{nom}}$, such that the inverted influence in the heating phase is not maximal and the worst-case influence in the cooling phase is not neglected.

The propagation rate constants $k_{p0i}$ have parameter values with a large uncertainty range, because they are difficult to identify. Until 5800s a higher reactor temperature $T_R$ would be achieved with a higher propagation rate constant $k_{p0i}$. The negative sensitivity after that is not critical, because the reactor temperature is not close to its upper bound anymore. Therefore this influence is neglected and the worst-case parameter value is chosen to be at the upper bound with $k_{p0i}^{\text{wc}} = 1.5k_{p0i}^{\text{nom}}$.

The reaction enthalpies $\Delta H_i$ have in general well measurable and estimated parameter values. Therefore, their uncertainty range is small and not necessarily causes problems in the application of the model for optimization.
However the sensitivity is solely positive over the whole time horizon, this makes the enthalpies suitable parameters for creating a worst-case scenario for the temperature and may also cover the uncertainty in the heat transfer coefficient $kA$. Therefore the worst-case parameter value is overestimated with $\Delta H_{\text{wc}} = 1.15\Delta H_{\text{nom}}$. That means that the chosen set of worst-case values lies outside of the uncertainty range $P$ to cover the uncertainty of the process model due to other possible uncertain parameter values of the set $P$.

### 4.4 Optimization Results - Two-Model Approach

The optimization with the two-model approach results in an objective function value of the space time yield of $0.044 \pm 0.002$ and is hence 22% lower compared to the optimization of the nominal case. The batch time $t_f$ with the two-model approach takes 8338s and is compared to the optimization in the nominal case 17% longer, which can be seen in Table 1. In addition, the constraints are all fulfilled.

**Table 1. Comparison of the results from both optimization approaches.**

| operation     | space-time-yield $[\text{kg s}]$ | final batch time $t_f$ |
|---------------|----------------------------------|------------------------|
| nominal case  | 0.057 $\pm 0.002$                | 7127s                  |
| two-model approach | 0.044 $\pm 0.002$                | 8338s                  |

This optimization fulfills all property endpoint constraints and has in addition no active path constraints for the reactor temperature of the nominal model $T_{R\text{nom}}$ in the two-model approach. In contrast, the worst-case reactor temperature $T_{R\text{wc}}$ exhibits active constraints at some points in time as shown in Figure 7 similar as the reactor temperature $T_R$ in the nominal case.

The optimal trajectories of the controls from the optimization in the two-model approach are compared with the input trajectories from the nominal case in the next section.

### 4.5 Comparison of Control Trajectories

The upper figure in Figure 8 shows the comparison of the monomer mass inlet flows $\dot{m}_{\text{in}}$ of both approaches. The results show that in the two-model approach, there are only a few monomers fed in the beginning of the process. Thereafter the monomers are fed with an almost constant feed rate. An exception is at 3000s, where the flow rate is reduced a little. Compared to the nominal case the average flow rate is lower, but the time horizon of feeding is longer.

The comparison of the jacket inlet temperatures $T_{J\text{in}}$ in the lower Figure 8 shows that in the beginning of the process the heating and cooling phases are similar. The first difference appears at 2000s, where the monomer feed rate of the two-model approach drops a little. At this time, the reactor is heated less than in the nominal approach. Thereafter the reactor is cooled again with its maximum cooling capacity and in the end of the process, where the monomer feed rate stops, the reactor is heated to fulfill the according endpoint constraints.

### 4.6 Discussion

Finally, since the two-model approach gives

- a lower monomer mass inlet flow rate,
- less cooling in the middle of the batch time,
- 17% longer batch time,
- 22% lower space-time-yield,

it is considered to be more conservative compared to the optimization in the nominal case. For the comparison of the robust feasibility of the optimization approaches, the optimal input trajectories are used for simulating hundred different scenarios with random parameter values from the uncertainty set $P$ consisting of $kA \text{range} = \pm 0.5kA_{\text{nom}}$, $k_{\text{range}} = \pm 0.5 k_{\text{nom}}$ and $\Delta H_{\text{range}} = \pm 0.1 \Delta H_{\text{nom}}$. The results in Figure 9 show that there are only a few constraint violations using the two-model approach. In Table 2 the percentage of violations are listed with only 6% in the two-model approach and 82% in the nominal case.

Furthermore one can observe that in the first half of the batch time the bandwidth of the simulated scenarios is narrower. The generated worst case is with a set of parameter values outside the uncertainty set $P$ appropriately chosen.
Table 2. Comparing the amount of violation and the conservatism regarding the averaged space-time-yield for both approaches.

| operation            | violations | $\otimes$ space-time-yield |
|----------------------|------------|---------------------------|
| nominal case         | 8%         | 0.0570 $\frac{kg}{s}$    |
| two-model approach   | 6%         | 0.0429 $\frac{kg}{s}$    |

Fig. 9. Constraint violations of the reactor temperature $T_R$ with simulating the process with 100 random parameter values from the uncertainty set $P$ and the optimal inputs from both approaches.

to cover most of constraint violations caused by parametric uncertainties of the set $P$. This method does not guarantee robust feasibility, but it is an easy generated approach and constraint violations are less likely.

The two-model approach is a promising alternative compared to recipe-based production. It is a kind of a physical back off (Loeblein et al., 1999) with the advantage that the constraint might be active, if it is safe enough dependent on the sensitivity of the path constraints with respect to the worst-case parameters.

5. CONCLUSION

A worst-case model is heuristically developed based on sensitivity analysis defined by heat of reactions $\Delta H_i$ higher than the upper bounds of the uncertainty range, propagation rates $k_{\text{pol}}$ at their upper bounds and a lower heat transfer coefficient $kA$. The lower heat transfer coefficient $kA$ is not chosen to be at its lower bound, because the sensitivity had a sign change and is thus critical to choose at all. The presented two-model approach is more conservative and more robust feasible. The presented approximation does not guarantee robust feasibility, but path constraint violation is less likely compared to the original optimization. The conservatism could be overcome with a closed-loop application of the two-model optimization, which should be applicable, because of the low computational effort.

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