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# Industrial experiences with product quality control in semi-batch processes

Yasuaki Yabuki [a,*], Tooru Nagasawa [a], John F. MacGregor [b]

[a] *Mitsubishi Chemical Corporation, 1-Toho-cho, Yokkaichi Plant, Mie, Japan*

[b] *Department of Chemical Engineering, McMaster University Hamilton, Hamilton, Ont., Canada L8S 4L7*

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**Abstract**

Two practical approaches to the control of final product quality in semi-batch reactors are illustrated with industrial experiences. The first is an orthodox approach to the control of final quality that is achieved through a high degree of automation of all the reactor charging operations, good temperature and pressure control, and the precise sequencing of steps implemented throughout the course of the batch. The second approach to the control of final quality is to use mid-course correction policies. Both approaches to the control of final quality are illustrated with an industrial example. Through our experience, the first approach, although very basic, is shown to be very important to reduce the variability in the final product quality. The second approach is useful to compensate for the new disturbances that are affecting the batch being run at the current time. Good practice is that mid-point control policies should be used after the basic automation step has been implemented as efficiently as possible. 

*Keywords:* Batch processes; Semi-batch processes; Quality control; Industrial practice

## 1. Introduction

The control of final product quality in an industrial semi-batch reactor is a difficult problem for various reasons. Foremost among them is the fact that robust on-line sensors for monitoring the progress of product quality development during the batch are almost never available. Rather, quality variables are usually measured only on the final product after the batch is completed. Furthermore, batch reactors exhibit highly nonlinear dynamic behavior, and the complex nonlinear kinetic and dynamic models needed for model-based inferential and nonlinear control are rarely available for industrial processes. As a result, most batch and semi-batch reactors are operated in an open loop manner with respect to product quality.

The basic approach to control final quality is achieved through a high degree of automation of all the reactor charging operations, the start-up sequence, and sequence of steps that must be implemented throughout the course of the batch. This automation eliminates the variability that might otherwise arise from the batch-to-batch variations in the implementation of these steps. Good temperature and pressure controls are used to minimize any effect of variations in these variables on final quality.

In spite of using such advanced automation, some batch-to-batch variations in quality will still be present. If this quality variability exhibits predictable trends (i.e. autocorrelation) due to persistent trends in raw material properties, impurities, catalyst activities, etc., then batch-to-batch feedback control schemes can be used to eliminate it. Such schemes (e.g. Box & Jenkins, 1970, Chapter 11; Vander Wiel, Tucker, Faltin & Doganaksoy, 1992) are usually based on the ideas of minimum variance control. They involve making small recipe adjustments to each new batch based on the recent history of quality measurements from past batches.

Although this batch-to-batch feedback control can eliminate the predictable component of quality varia-

* Corresponding author.

*E-mail address:* 2704158@cc.m-kagaku.co.jp (Y. Yabuki).

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tion between successive batches, it does nothing to compensate for the new disturbances that are affecting the batch being run at the current time. If adequate on-line sensors are available to detect the presence of disturbances arising from sources such as raw materials, impurity and catalyst variations or from charging or start-up variations, and if models can be developed to predict the effects of these detected disturbances on the final product quality, then model-based within-batch feedback control is possible. This within-batch control will lead to a further reduction in the product quality deviations from target for each batch.

Various approaches to this within-batch control problem can be taken depending upon the type of on-line information available and upon the complexity of models available. Kozub and MacGregor (1992) present an approach to the within-batch control of multiple product quality variables in the semi-batch emulsion polymerization of styrene–butadiene rubber (SBR) using nonlinear control and state estimation. However, this approach requires a good fundamental dynamic model of the SBR emulsion polymerization process, and a set of good on-line sensors (e.g. for residual monomer concentrations and particle size) to ensure observability of the state variables throughout the time history of the batch. Neither of these is usually available in an industrial setting.

Data-based quality prediction and control approaches to the within-batch control problems have been proposed. These approaches explore a more realistic approach to within-batch product quality control that is based on simple and readily available on-line measurement and some off-line analyses.

Multivariate statistical process control (MSPC) has been introduced for batch and semi-batch processes based on the concept of multiway PCA or multiway PLS (Nomikos & MacGregor, 1994, 1995). This statistical monitoring is done using on-line process measurements rather than off-line quality measurements. With this technique, an abnormal batch can be detected quickly, providing an opportunity to stop the batch or make compensatory adjustments.

Mid-course control adjustment policies with empirical models (based on PLS or neural network models) have been proposed for reducing predictable deviations of final product quality variables from their target values. Tsen, Jamg, Wong and Joseph (1996) used artificial neural network models to develop a one-shot control policy based on using only a single intermediate measurement.

Yabuki and MacGregor (1997) proposed a related strategy using PLS models. If the predicted quality deviations were outside a statistically defined 'no control' deadzone, a model was then used to calculate a semibatch control move designed to bring the batch back under statistical control.

Russel, Kesavan, Lee and Ogunaike (1998) proposed recursive data-based prediction and control of batch product quality. The proposed control approach can be viewed as shrinking-horizon model-predictive control based on empirical models.

In this paper, two practical approaches to the control of final product quality in semi-batch reactors are illustrated with industrial experiences. The first is an orthodox approach to the control of final quality, achieved through the implementation of a high degree of automation of all the reactor charging operations, good temperature and pressure control, and the precise sequencing of steps implemented throughout the course of the batch. The second approach to the control of final quality is to use mid-course correction policies. Both approaches to the control of final quality are illustrated with an industrial example.

## 2. An orthodox approach

Eliminating variations in initial conditions and automating the sequence of steps in batch processes is a very important approach to reduce the variability in the final product quality. If the cause of variability in the final product quality is detected and eliminated, it will be the best remedy for the reduction of variation in the final product quality.

### *2.1. Process description; polymer plant A*

In a solution polymerization, a solvent is initially charged and heated up to at the desired initial temperature by use of the steam jacket. Then, monomer and initiator are fed at the constant flow rate to the reactor till the batch end. One batch takes approximately 70 min to complete the cycle from the charge of monomer and initiator through discharge of the polymer.

### *2.2. Improving final product quality*

The final product quality variables for polymer plant A showed significant batch-to-batch variations before improvements were taken by an orthodox approach. In an attempt to identify important sources of variability that might be affecting the final product quality, several steps were taken. First, a fish-bone style diagram (Ishikawa, 1976) was drawn to clarify the cause and effect relationships and to select candidates for assignable causes. The data-base of batch polymerization histories was then analyzed using multi-way PCA and PLS methods (Nomikos & MacGregor, 1994, 1995) to identify process variables that were correlated with the final quality, and to identify the time periods during the batch where these correlations were high. In this way several variables at various stages in the process