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Online batch recipe adjustments for product quality control using empirical models: Application to a nylon-6,6 process

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Abstract

The application of batch profile characterization tools to enhance process understanding by uncovering the signature of the primary disturbances on the profiles and its effect on the product quality is illustrated on a nylon-6,6 process. The historical profile data for the fixed recipe operation are systematically studied to understand the primary disturbances affecting the process, and it is shown that good online predictions of the final product quality are possible much before the completion of the batch from the available measurement profiles. A simple online recipe adjustment strategy based on the predicted quality deviation from the target is proposed. Results show that the recipe adjustments significantly reduce the variation in the final product quality. Issues in the use of empirical prediction models from recipe-based data are discussed.

Keywords: Quality control; Within-batch control; Empirical modeling; Batch recipe adjustment

Introduction

One of the primary objectives of batch manufacturing in the chemical industry is the consistent production of on-target quality batches. This is due to the premium on the quality of the value-added chemicals that are typically manufactured using batch processes. Even though the final product quality is a major concern, online quality measurements are seldom available. The batch operation is consequently based on a fixed recipe, which may represent years of process experience, with variables considered important, such as reactor temperature, being controlled to a prespecified trajectory. A completed batch is characterized as either on-spec or poor quality from laborious analytical measurements on a sample of the product.

The fixed-recipe-based operation with tight profile trajectory control helps in rejecting many disturbances that can potentially affect the final product quality. However, some significant common cause sources of variation, such as impurities in the raw material, are not compensated for, resulting in high quality variation. The occurrence of these disturbances is reflected, although indirectly, in the various online measurements such as temperatures, pressures, and flows. Measurement profiles for several past batches are typically stored in a historical database. The database is a rich source of information, which can be systematically studied to understand the sources of variation affecting the process and to suggest strategies for improving the process, especially for tighter quality control. The development of batch data mining tools has thus received much attention in recent years. These profile characterization methodologies essentially quantify the systematic variability along

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the time axis and the measurement axis. Tools such as dynamic time warping [1] and template matching [2] characterize the former, while multivariate methods such as principal component analysis [3] and partial least squares [4] are used for characterizing the latter.

The reported applications use profile characterization methodologies towards various ends. Process monitoring with the past successful operation as the baseline for comparison is the primary goal in several articles [5–10]. Some applications focus on the development of quality prediction models from the profiles [11–13]. Quality predictions are central to the implementation of online recipe adjustment strategies, also referred to as inferential control. Russell *et al.* demonstrate recursive data-based prediction and control of product quality for a nylon-6,6 process [14]. Midcourse recipe correction using empirical models has been demonstrated on a semibatch process [15]. Recent reports use the empirical predictions in model predictive quality control [16,17].

A major criticism of the quality control applications in the reported literature is the use of empirical models that are not based on a fixed-recipe operation but include batches spanning the control moves in the training set. Such rich data sets are seldom available, especially in the very conservative industrial environment, limiting the applicability of the methodologies. Also, the strategy for quality control is implicitly assumed. The strategies may not always be very obvious for a process. Indeed, the real challenge for process improvement lies in deciphering from the historical data, the signature of the primary sources of variation on the measurement profiles, and their effect on the product quality. In cases when the occurrence of the disturbances or significant quality deviations from target can be inferred from the measurement profiles well before the end of the batch, corrective action can be taken to reduce the variability in the product quality. This is referred to as within-batch control and its demonstration using empirical models forms the thrust of the work reported here.

Data mining tools are applied to profiles from a recipe-based nylon-6,6 process, and opportunities for online recipe adjustments for tighter control of the polymer molecular weight (MW), the primary quality variable, are identified. A simple online recipe adjustment scheme based on final product quality prediction is suggested. Results from implementation of the within-batch control scheme show significant improvement in the product quality variability. Emphasis is laid on the physical relevance of the various empirical model parameters to understand the cause and effect relationships governing the process. Such an understanding is essential in proposing an effective control strategy.

The article is organized as follows. The recipe-based nylon-6,6 process is briefly described in the next section. The database of profiles generated from the simulation is then subjected to data mining to obtain time and magnitude scale parameters that characterize the profiles. The correlation structure of the scale parameters is studied to uncover the signature of the primary disturbances affecting the process. The scale parameters are also used to build online product quality prediction models. It is shown that good predictions are possible much before the end of the batch opening the possibility of online recipe adjustments. Within-batch control schemes involving the addition of amine salt and reducing the jacket pressure are proposed. The improvement in quality control due to the implementation of the online recipe adjustments is quantified. A discussion of the various engineering issues and conclusions that can be drawn from the work complete the article.

The Nylon-6,6 process

Nylon-6,6 is produced by the polycondensation of hexamethlene diamine (HMD) with adipic acid. The main polymerization reaction is described as amine end groups (A) and carboxylic end groups (C) on polymer chain ends or monomer, reacting reversibly to form polymer links (L) and water (W) as

$$A + C \leftrightarrows L + W. \quad (1)$$

In order to achieve high molecular weight and conversion, the water must be vaporized to shift the equilibrium towards the products. An autoclave reactor with a heating jacket is ideal for this purpose. A schematic of the process is shown in Fig. 1. The measurements available are the reactor temperature, reactor pressure, vent vapor flow rate, and the jacket pressure. Dowtherm is used as the heating fluid in the jacket. The dynamic material and energy balances are modeled using the