Chapter 2

APPLICATION AND CASE STUDIES

Outline

- SBR Semi-Batch Reactor System: Monitoring
- Batch Pulp Digester: Inferential Kappa Number Control
- Nylon 6,6 Autoclave: Monitoring & Inferential Control of Quality Variables
- Continuous Pulp Digester: Inferential Kappa Number Control
2.1 PCA MONITORING OF AN SBR SEMI-BATCH REACTOR SYSTEM

2.1.1 INTRODUCTION

Background

- In operating batch reaction systems, certain abnormalities (e.g., increased feed impurity level, catalyst poisoning, instrumentation malfunctioning) develop that eventually throw the quality completely off spec.

- It is desirable to catch these incipient faults quickly so that the problem can be rectified.

- It is desirable not to rely on lab measurements for this purpose since this will introduce significant delays.

Key Idea

- Use more easily measured process variable trends to classify between normal batches and abnormal batches.

- The key problem is to extract out the key identifying features (*finger prints*) from trajectories of large amount of variables.

Application

- An SBR Polymerization Reactor.
2.1.2 PROBLEM DESCRIPTION

Process / Problem Characteristics

- Reaction:

  \[ \text{Styrene} + \text{Butadiene} \rightarrow_{\text{polymerization}} \text{Latex Rubber} \]

- Emulsion Polymerization

- The reactor is initially charged with seed SBR particles, initiator, chain-transfer agent, emulsifier, a small amount of styrene and butadiene monomers.

- Batch duration is 1000 minutes.

- The following measurements are available with 5 minute interval:
  - flow rates of styrene
  - flow rates of butadiene
  - temperature of feed
  - temperature of reactor
  - temperature of cooling water
  - temperature of reactor jacket
  - density of latex in the reactor
  - total conversion (an estimate)
  - instantaneous rate of energy release (an estimate)

Available Data

- 50 batch runs with typical random variations in base case conditions (such as initial charge of seed latex, amount of chain transfer agent and level of impurities).
• Two additional batches with “unusual disturbances.”

  – impurity of 30% above that of the base case was introduced in the butadiene feed at the beginning of the batch.

  – impurity of 50% above that of the base case was introduced in the butadiene feed at the halfway mark.

2.1.3 RESULTS

End-Of-Batch Principal Component Analysis
• Establish the mean trajectory for each variable and compute the deviation trajectory.

![Diagram showing average trajectory and actual measurement over time]

• Normalize each variable with its variance.

• Perform "lifting", that is, stack all the trajectories into a common vector to obtain a single vector \( \mathbf{Y} \) for each batch. Then, form a matrix \( \mathbf{Y} \) by aligning \( \mathbf{Y} \) for the entire 50 batches. Note the dimension of \( \mathbf{Y} \) is \( 9 \times 200 \). Clearly there are only a few modes of variations in this vector.

• Determine the principal component directions (eigenvectors of \( \mathbf{Y} \) with significant eigenvalues). Three components were judged to be sufficient.

\[
\mathbf{Y} = \begin{bmatrix}
v_1 & v_2 & v_3 & v_4 & \cdots & v_{1800}
\end{bmatrix}
\begin{bmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3 \\
\sigma_4 \\
\vdots \\
\sigma_{1800}
\end{bmatrix}
\begin{bmatrix}
v_1^T \\
v_2^T \\
v_3^T \\
v_4^T \\
\vdots \\
v_{1800}^T
\end{bmatrix}
\]
- Compute the principal component score variables for each batch:

\[ t_i(j) = v_i^T Y(j), \quad i = 1, \cdots, 3 \quad j = 1, \cdots, 50 \]

The first two P.C. scores for the 50 batches and the two bad batches are plotted below:

Compute the covariance matrix (diagonal) \( R_t \) for the P.C.’s. Establish the 95% and 99% confidence limits (ellipses) for the P.C.’s.
One can also use Hotelling Statistics:

\[ D = t^T R_i^{-1} t \frac{N(N - m)}{m(N^2 - 1)} \sim F_{m,N-m} \]

Here \( t = [t_1, t_2, t_3]^T \) and \( N = 50 \) and \( m = 3 \).

- Compute the residuals and establish the 95% and 99% confidence limits for the square sum (assuming normality of the underlying distribution). The SPE (sum of the squares of the residuals) for each batch is plotted against the confidence limits:

**During-Batch Principal Component Analysis**

- The main issue in applying the PC monitoring during a batch is what to do with the missing future data.
Options are:

- Assume for all the variables that the future deviation will be zero.
- Assume for each variable that the current level of deviation will continue until the end of batch.
- Use statistical correlation to estimate the future deviation.

We will denote the lifted vector at time $t$ with missing future measurements filled in as $\hat{Y}_t(j)$, where $t$ and $j$ denote the time and batch index.

- For each time step, the confidence limits for the SPE and P.C.’s can be established.
- Now, at each time step for each batch, compute the P.C.s and SPE and compare against the confidence intervals.
Good Batch
Bad Batch I
Bad Batch II