BATCH PROCESS MONITORING THROUGH THE INTEGRATION OF SPECTRAL AND PROCESS DATA

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Abstract: Most applications of MSPC have tended to focus upon the manufacture of a single product with separate models being developed to monitor individual recipes. With process manufacturing trends being influenced by customer demands there has been an increase in the manufacture of a wide variety of products, there is a real need for process models which allow a range of products, grades or recipes to be monitored using a single process model. With increasing attention now being paid to the FDA Process Analytical Technologies (PAT) initiative, the use of spectro-chemical information for enhanced monitoring of reactions and is now gaining impetus. An application of the performance monitoring of a multi-recipe multi-reactor industrial batch polymer manufacturing is discussed in which NIR spectroscopic data is also integrated with process data to provide enhanced batch monitoring.

Keywords: Multivariate Statistical Process Control; Spectroscopic Data; Multi-recipe, Batch Reactors

1. INTRODUCTION

An area of rapidly growing interest for the monitoring of processes is that of Multivariate Statistical process Control (MSPC). MSPC schemes are typically based on the statistical projection techniques of Principal Component Analysis (PCA) and Projection to Latent Structures (PLS) and their multi-way extensions for batch processes. Reported practical applications of MSPC have focused on the production of a single manufactured product i.e. one grade, one recipe, etc. with separate models being used to monitor different types of products (e.g. Nomikos & MacGregor, 1994; Kosanovich & Piovoso, 1995; Kourti *et al*, 1995; Martin *et al*, 1999, 2002; Weighell *et al*, 2001; Wise & Gallacher, (1996).

Most applications of MSPC, or better termed Multivariate Process Performance Monitoring (MPPM), have focused upon the production of a single manufactured product i.e. one grade, one recipe, etc. with separate models being used to monitor different types of product. With process manufacturing increasingly being driven by market forces and customer needs and perceptions, the necessity for flexible and responsive manufacturing is becoming essential. This is particularly the case in the manufacture of specialty products where new product formulations require to be introduced to the market over a short time scale to ensure competitive advantage and product diversification, as well as, for example, in pharmaceutical manufacturing where new strains are routinely introduced. Thus there is a real need for process monitoring models which allow a range of products, grades or recipes to be monitored using a single process representation. Such process representations will enable the performance monitoring of a number of different product types using a single process representation.

2. THE MULTI-GROUP ALGORITHM

The elimination of between group variation is a prerequisite for statistical process monitoring, so that interest can focus on within process (product) variability. This normally requires constructing separate control charts for each type of product or grade to be monitored. In many process monitoring

situations this may be impractical because of the large number of control charts required to monitor all the products being manufactured and the limited amount of data available from which to develop a process representation. An extension to PCA allows the construction of multi-group pooled covariance matrix models has been proposed (Lane *et al*, 2001) which is based on combining the variance-covariance matrices of each of the individual groups (Krzanowski, 1984; Flury, 1987). The loadings for the latent variables are then calculated from the pooled variance-covariance matrix of the individual groups. The method is based on the assumption that a common eigenvector subspace exists for the individual variance-covariance matrix of the individual product grades. The pooled sample variance-covariance matrix ($\mathbf{S}$) which forms the basis of the generic model approach is defined as a weighted sum of the g individual variance-covariance matrices $\mathbf{s}_1, \mathbf{s}_2, \ldots, \mathbf{s}_g$:-

$$\mathbf{S} = \frac{(n_1 - 1)\mathbf{s}_1 + (n_2 - 1)\mathbf{s}_2 + \ldots + (n_g - 1)\mathbf{s}_g}{(N - g)} \qquad (1)$$

for $i = 1, \ldots, g$, where N is the total number of observations, g is the number of groups and n_i is the number of observations within group i. Through the pooled sample variance-covariance matrix of the individual product grades, the principal component loadings are calculated. More recently a multi-group PLS approach have been proposed (Lane, Martin and Morris, 2003; Martin and Morris, 2003). These novel developments allow the monitoring of several different sets of operating conditions by a single model. The potential of these developments which allow, with some restrictions, the monitoring of different recipes, different unit operations and with different number of measured variables sets of operation his development are far-reaching and provide a major step forward in ensuring the wide application of multivariate process performance monitoring in process manufacturing.

3. MULTI RECIPE BATCH MANUFACTURING

An application to an industrial semi-batch polymer manufacturing process is discussed. Process data is available every five minutes and comprises temperature corrected viscosity, reactor temperature, vapour temperature, pressure / vacuum, distillation column temperatures, and NIR spectral data throughout the batch. Four different recipes were considered which were run in five different reactors. In order to overcome this combination of multiple recipes and different reaction vessels, common subspace models were developed. The polymerisation reaction is monitored through observation of the polymer end groups. Although chain length of a polymer cannot be measured directly during the reaction, the value of end groups can be used to infer the progress of the reaction. The end group measure acts as a quality variable to monitor batch progress and are often determined using an off-line wet chemical analysis.

Figure 1 shows the PCA plot of the five different reactors used in the polymer production whilst Figure 2 shows the PCA plot of the four different recipes processed. Clearly the between 'group' (between cluster) variability observed in the plots prevents the detection and diagnosis of any subtle variations (within cluster) variability and process malfunctions which might take place during the batch run.

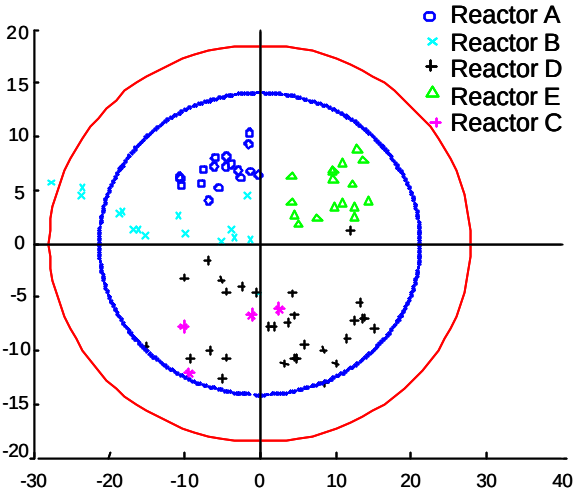

Figure 1: Bivariate Scores plot for five different reactors (PC1 versus PC2)

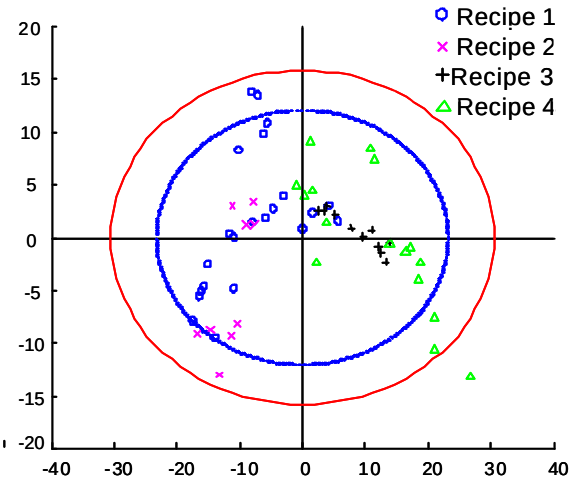

Figure 2: Bivariate Scores plot of for four different recipes (PC1 versus PC2)

An example of the impact of this 'between group' variability is shown in Figure 3 which plots the through batch scores for a combined model for two recipes. The separation of the two recipes into separate regions is clearly observed. Also notable are the varying batch completion times. This results in a monitoring model that exhibits severe 'between recipe' variability and which lacks sensitivity to detect and enable the diagnosis of subtle process malfunctions. In contrast Figure 4 shows a similar

plot for a single recipe model where the random scatter of the scores plots through the batch run can be observed and which is suitable for 'within group' batch monitoring.

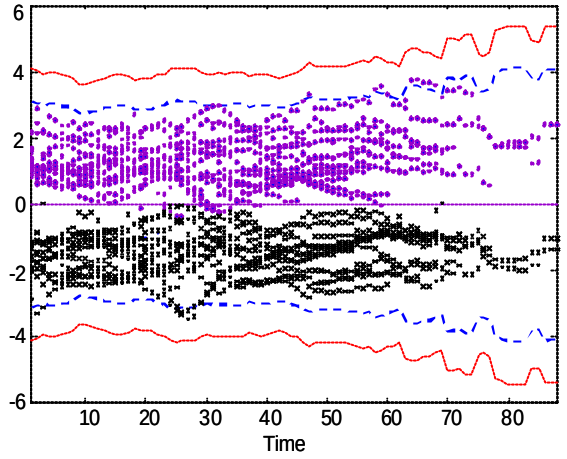

Figure 3: Scores Plot for PC1 for two recipes (Recipe 1 – Black; Recipe 2 – Purple)

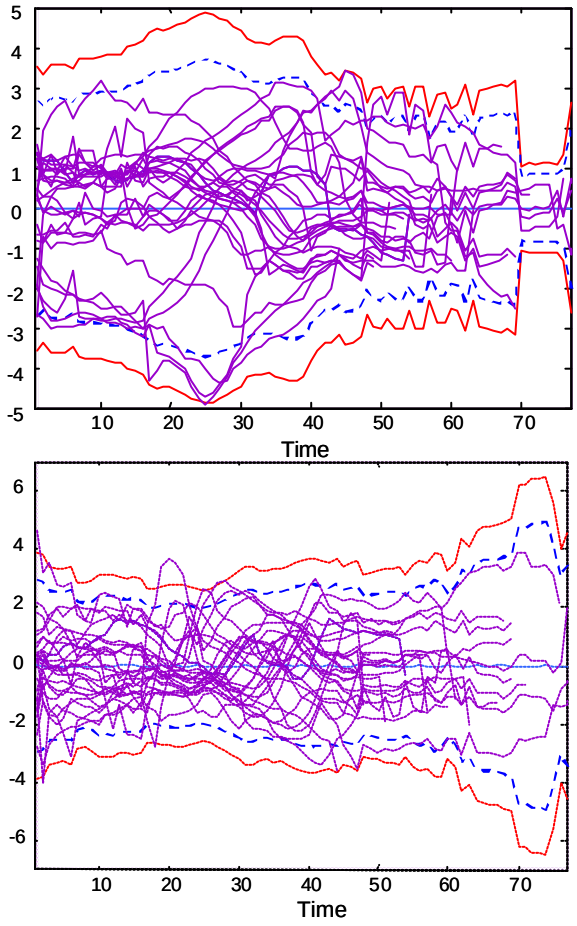

Figure 4: Scores Plot for a single recipe model PC1 (upper plot) and PC2 (lower plot)

Figure 5 shows schematically the off-line laboratory monitoring of the progress of the polymerisation reaction where the quality of the batch is assessed by considering the hydroxyl number (OH#) and acid value (COOH#) of the reactor contents. Accurate knowledge of these values is required to fix the point at which the batch meets the desired reaction curve. The relationship between the hydroxyl number and viscosity is controlled to follow the path of the reaction curve, with upper and lower limits used to account for allowed process variability. When the trajectory enters a pre-defined end zone the batch is terminated.

Data from thirty two 'acceptable' batches which operationally appeared to exhibit nominal operations were available and included measurements of seven process variables - Temperature Corrected Viscosity; Reactor Temperature, Reactor Vapour Temperature, Bottom Column Temperature, Middle Column Temperature; Top Column Temperature, and Reactor Pressure / Vacuum

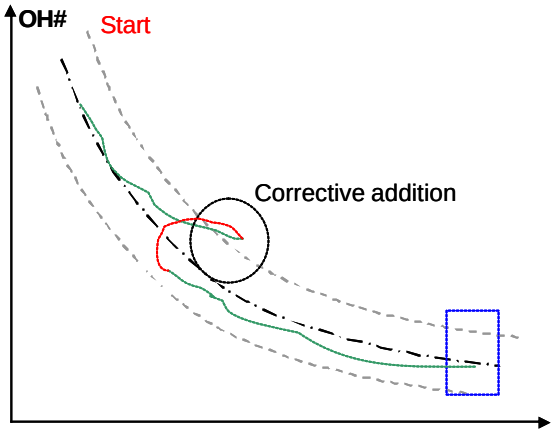

Figure 5: Reaction Monitoring – OH# versus Viscosity

In order to assess the ability of the multi-group pooled covariance modelling to detect subtle process events, a monitoring model was initially built by simply combining the different recipes resulting in a monitoring model typical of that shown in Figure 3. One particular batch exhibited non-nominal operation due to increase in the bottom column temperature, from the start of the batch. This increase in column bottom temperature rose through the column and towards the end of the batch non-nominal operation was experienced in the temperature at the top of the column. This batch was projected onto the combined monitoring model. Figure 6 shows the through-batch plot of the first and second principal component scores respectively. It can be observed that there is no substantial out-of-statistical control signal indicating a potential processing problem on which the operational staff could act although the PC2 sores plot does show some deviation from what might be expected at the beginning of the batch.

With a pooled covariance multi-group model the sensitivity of the model to more subtle process faults becomes apparent as shown by the random scatter of the scores in Figure 7. The performance of the multi group monitoring model, Figure 8, where it can be seen that the PC1 score plot picks up an out-of-

control signal due to the column temperature problem at around 26 mins whilst in PC2 it is picked up at around 40 mins. This study comparing the combined recipe modelling approach with the multi-group model approach demonstrates a major issue with conventional MSPC monitoring approaches that simply combining different recipes or product grades can lead to poor monitoring performance with a loss of model sensitivity.

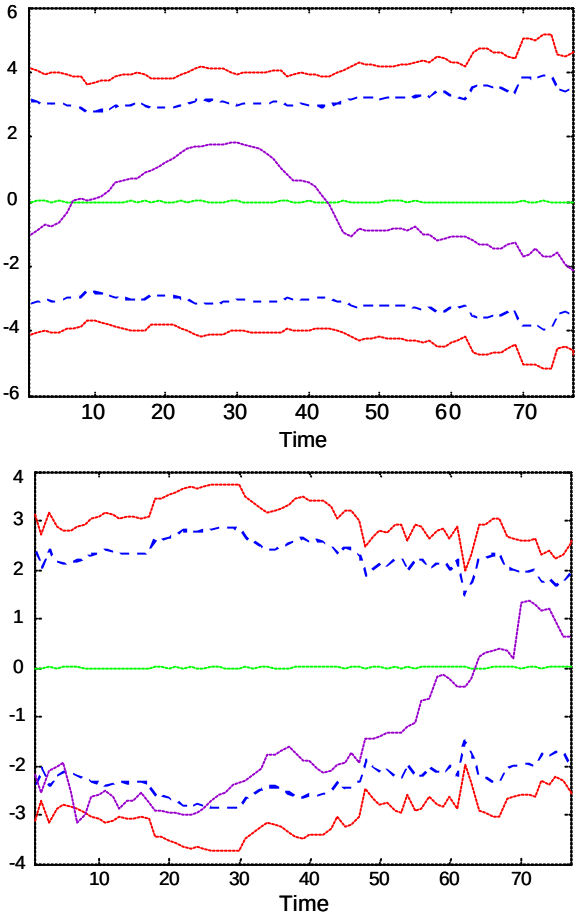

Figure 6 Scores Plot against time for PC1 (upper plot) and PC2 (lower plot) - Abnormal Batch

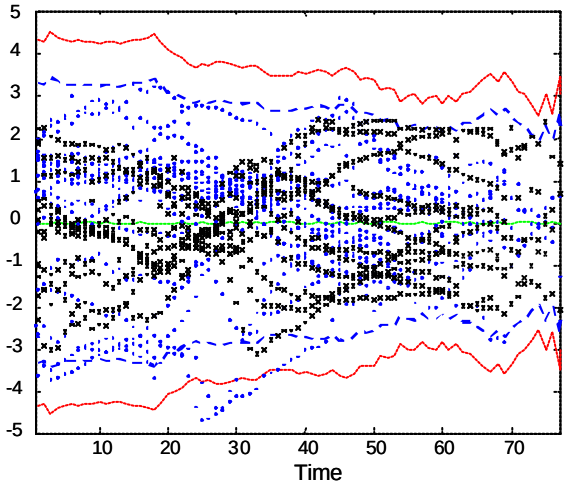

Figure 7: Scores Plot for PC1 Multi-Group Model (Recipe 1 – Black; Recipe 2 - Blue)

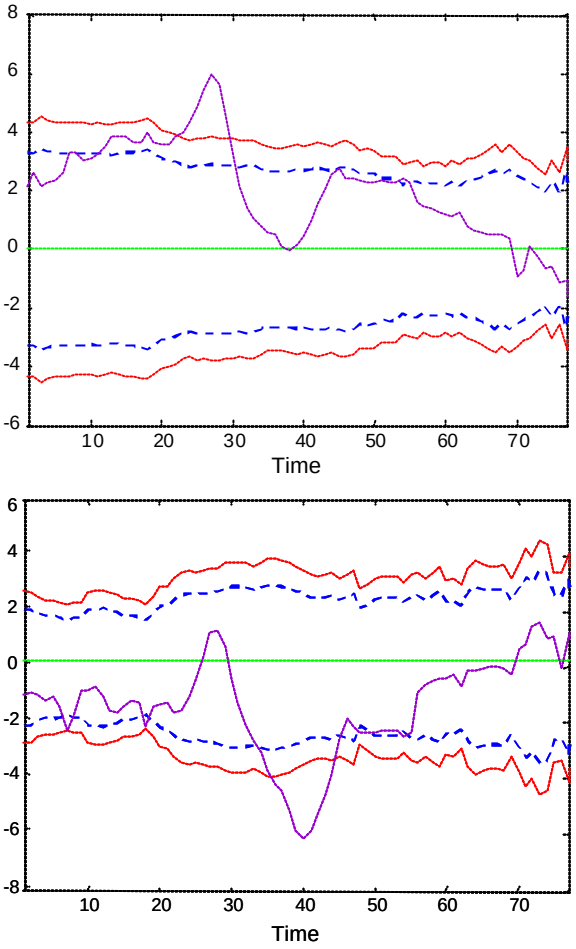

Figure 8: Multi-group Model Monitoring plot PC1 (upper plot) and PC2 (lower plot)

5. SPECTRAL AND PROCESS DATA

Most recently, and in particular driven by the FDA initiative in Process Analytical Technologies (PAT) in the bio-processing sector, the use of spectro-chemical information, such as NIR, for the on-line real-time monitoring of processes is receiving increasing attention, especially from the pharmaceutical and food manufacturing industries. Thus the integration of spectroscopic data with process data for enhanced process performance monitoring becomes attractive. There are a number of approaches to handing spectroscopic data such as multiplicative scatter correction, standard normal variate transformation, orthogonal signal correction, second derivative and wavelet transformation which have been investigated.

PCA applied to the raw un-scaled spectral data was used to identify the more significant wavelengths prior to applying a wavelet transformation using a Symmlet #8 mother wavelet. Figure 9 (upper plot) shows the wavelengths of bond frequencies identified by the process chemists which are characteristic of the chemistry that dominate the end quality aspects of the polymerisation reaction whilst the lower plot shows the wavelet transformation.

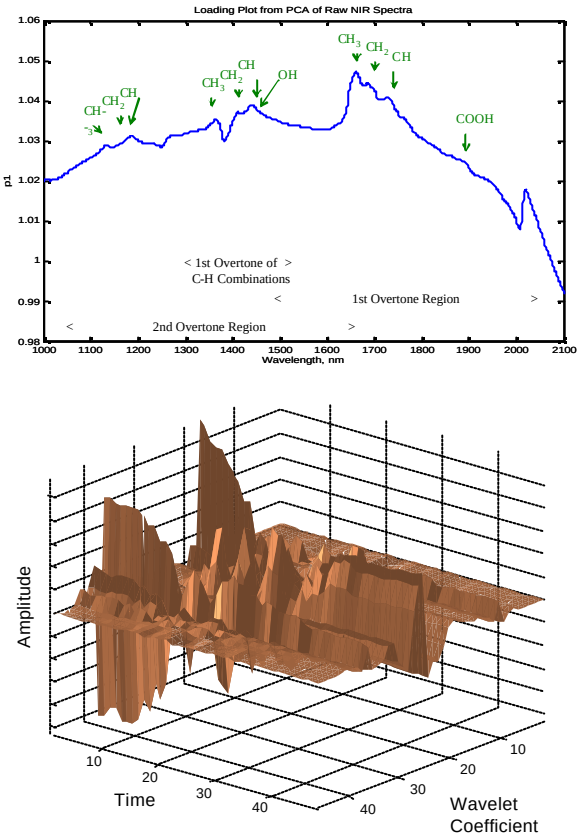

Figure 9: PCA of raw un-scaled NIR spectra (upper plot); Wavelet detail coefficients (lower plot)

The approximation and detail coefficients were investigated at each level, with the most suitable group of coefficients being identified as the detail coefficients at level 5 where each detail coefficient summarised information for approximately 22 wavelengths which is around the size of the broad band of each bond frequency. The two areas of interest in the NIR data sets lie around the 1450nm and 1900nm wavelengths. To capture all of the information from the transformed spectra, the two detail coefficients from either side of these important wavelengths were also extracted for inclusion in the model. This leads to the addition of four extra variables to the monitoring model. As each detail coefficient summarises information from a broad band of wavelengths, this approach is not as sensitive to chemical shifts as other transformation techniques such as the second derivative technique.

A batch which exhibited an extended duration due to mid batch corrections and which was monitored using only the measured process variables is shown in Figure 10. No out-of-control signal was given with no indication that the batch was anything but normal. The batch profiles were then re-examined with the inclusion of a set of variables from the wavelet transformed NIR data. The batch was known to have a longer than average duration.

Comparison of the PC1 scores monitoring chart in Figure 2 with the PC1 monitoring chart shown in Figure 10, reveals that the enhanced model has detected the occurrence of a process malfunction between observations thirty-four to thirty-six. The Scores and SPE contribution plots are illustrated in Figure 12 showing the variables that are contributing to the out-of-control event at observation number thirty-four. These reveal that variable numbers seven and nine lie outside their 99% confidence limits. These variables represent the wavelet detail coefficients of the OH and COOH bond frequencies. It is also noted that the process variables (one to six) lie well inside their respective confidence limits.

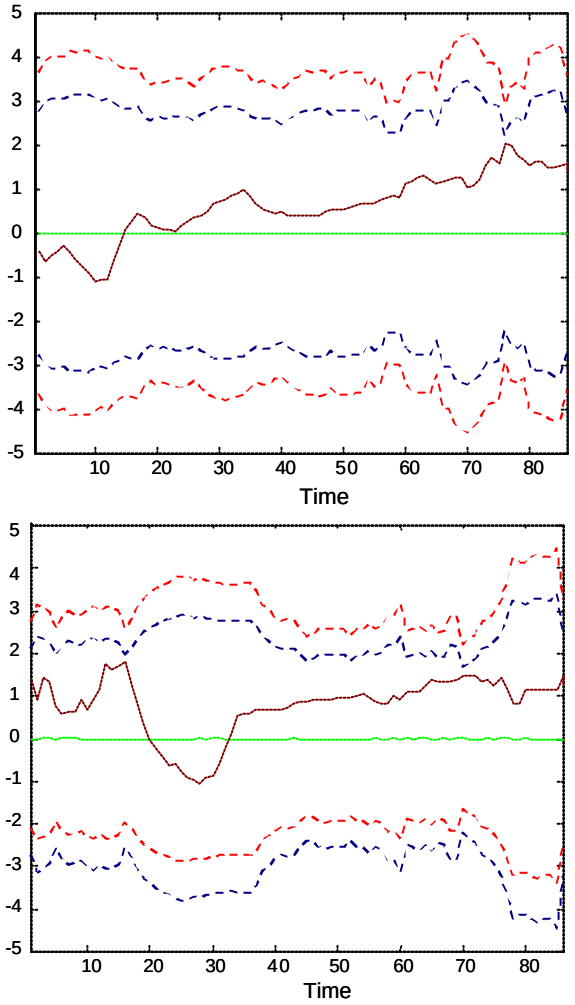

Figure 10: Scores Monitoring plots of process data only PC1 (upper plot) PC2 (lower plot)

The multiple group process monitoring techniques were also demonstrated to have good fault detection and diagnostic capabilities. It has been demonstrated that the application of a multi-group performance monitoring provides sensitivity to subtle process malfunctions not provided by the standard MSPC approaches. The potential for enhanced fault detection and diagnosis capabilities of the multi group model with spectroscopic information, in this case using wavelet decomposition of NIR spectra, has also been demonstrated.

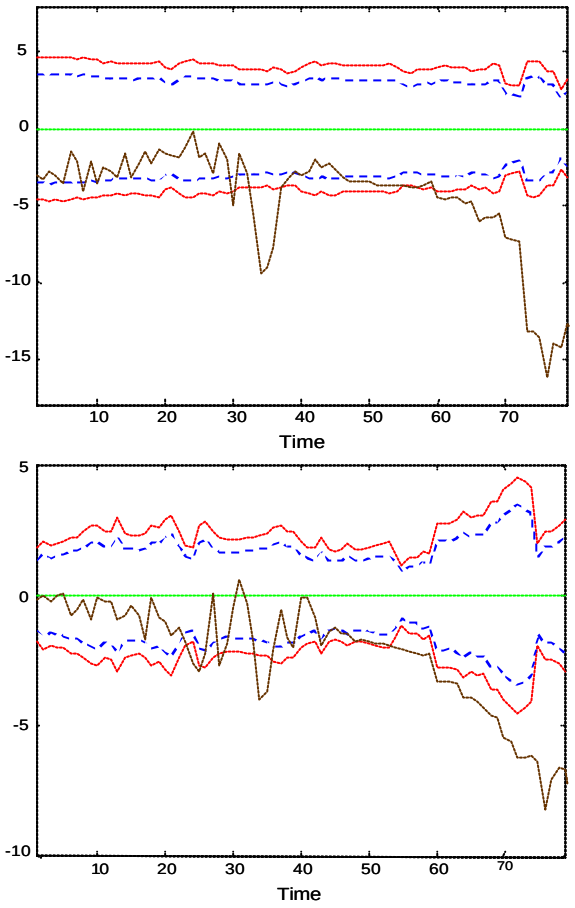

Figure11: Through-batch Scores Monitoring Charts PC1 (upper plot) and PC5 (lower plot)

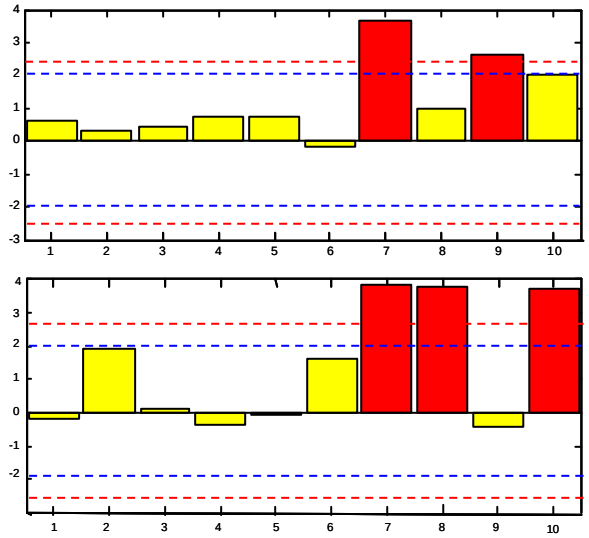

Figure 12: Scores (upper plot) and SPE (lower plot) contribution plots for PC1 observation number 34

5. CONCLUSIONS

The application presented in the paper is from a manufacturing process where the amount of data from each distinct set of unit processes and product recipes were different and limited. By applying the multi-group pooled correlation approach, all the products being manufactured could be monitored using a small number of monitoring charts. In this way the cost and time required to update the models can be significantly reduced.

6. AKNOWLEDGMENTS

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