

Real-time monitoring of an industrial batch process

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Abstract

This paper describes the development of a real-time monitoring system for a batch process operated by Aroma and Fine Chemicals Limited. The process shares many similarities with other batch processes in that cycle times can vary considerably, instrumentation is limited and inefficient laboratory assays are required to determine the end-point of each batch. The aim of the work conducted in this study was to develop a data driven system to accurately identify the end-point of the batch. This information can then be used to reduce the overall cycle time of the process. Novel approaches based upon multivariate statistical techniques are shown to provide a soft sensor that can estimate the product quality throughout the batch and provide a long-term estimate of the likely cycle time. This system has been implemented on-line and initial results indicate that it offers potential to significantly reduce operating costs.

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1. Introduction

The task of achieving optimal performance of industrial batch processes represents a very difficult challenge to production and systems engineers. Highly non-linear dynamics, large batch-to-batch variations and real-time measurement difficulties are problematic for consistent operations. This can be a concern in many industries, particularly the pharmaceutical and specialty chemical industries. For many applications, it is imperative that operation be maintained within strict limits, as improved consistency will result in reduced production costs.

In recent years, a wide variety of techniques have been applied to provide improved monitoring and control of industrial batch processes. These studies have involved the application of predictive based strategies (Shi, El-Farra, Mingheng, Mhaskar, & Christofides, 2006), neural networks (Lennox, Hiden, Montague, Kornfeld, & Goulding, 2001), Kalman Filters (Clark-Pringle & MacGregor, 1997) and multivariate statistical process control (MSPC) techniques. For application to general

batch processes, MSPC techniques have proven to be very successful (Martin & Morris, 1996).

MSPC covers a wide variety of algorithms, with the principle techniques applied to batch processes being parallel factor analysis (Bro, 1997), Tucker3 (Smilde, 1992) and multi-way principal component analysis (MPCA) and multi-way partial least squares (MPLS). Although successful applications involving parallel factor analysis, Tucker3, MPCA and MPLS have all been reported, a recent article by Westerhuis, Kourti, & MacGregor (1999) indicates that the multi-way techniques offer significant advantages over parallel factor analysis and Tucker3.

MPCA and MPLS have been used to address a variety of batch control and monitoring issues. In their original paper Nomikos and MacGregor (1994) illustrated how MPCA and MPLS could be used to provide an early indication of whether a batch was performing well or not. Lakshminarayanan, Gudi, Shah, and Nandakumar (1996) extended the application of MPCA and MPLS and showed how the techniques could be used to identify poorly performing batches and provide a real-time estimate of product quality (biomass concentration) when applied to a simulation of a fermentation process. Lennox et al. (2001) showed how the techniques could be used in an industrial fermentation study to detect sensor failure and provide a real-time estimate

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of product quality and also an estimate of the final productivity of the batch. More recently, MPLS techniques have been integrated within advanced control frameworks to provide improved control of batch processes (Flores-Cerrillo & MacGregor, 2002; Zhang & Lennox, 2003).

This paper describes the application of MPLS to an industrial batch process operated by Aroma and Fine Chemicals in the UK. This process is a multi-stage catalytic reaction which converts raw materials into a final product. The purpose of the investigation conducted on this process was to provide a mechanism for estimating the conditions within the batch such that the end-point of the batch could be identified with greater accuracy than is currently available. As with many batch processes that are reliant upon laboratory measurements, it is not clear with the process under investigation when the exact end-point of the batch is reached. It is however expected that substantial savings can be made to operating costs if the precise end-point of the batch could be identified.

In conducting this case study, limitations with standard approaches for applying MPLS and MPCA were identified and novel solutions were developed which enabled the techniques to be applied to the process under investigation. It is believed that the techniques developed in this study provide a generic approach to estimating batch end-point in systems with variable cycle times.

In the following section of this paper, a description of the process is provided. This is followed by a description of partial least squares and its multi-way extension. A novel multivariate statistical formulation, based upon multi-way partial least squares is then described and its application to the case study discussed. The developed system has now been implemented on-line and initial results from this system are presented. Finally, a list of conclusions from this work is provided.

2. Process description

The process considered in this study is part of a multiple-stage plant that produces a specialty chemical. The process operates in batch form and is responsible for the hydrogenation of a raw material, A, into a final product, B.

The operation of this batch process can be divided into three distinct phases: heating; gassing; cooling. During the heating phase the reactor is filled with raw material and the temperature is raised to a specific level. The duration of this phase varies, as it is dependent upon the characteristics of the raw material, principally its moisture content. Following the heating phase, the gassing phase begins as hydrogen gas is fed to the reactor. The hydrogen flow causes the raw material to be hydrogenated, producing the final product. During this phase the temperature and pressure in the reactor is regulated within upper and lower constraints. The duration of the gassing phase also varies significantly as a result of changes to the properties of the raw material.

Finally, when laboratory samples indicate that the gassing phase is complete, the process enters its final stage, the cooling phase, where the contents of the reactor are cooled and then transferred to downstream processing. The process variables that

are measured continuously through each batch cycle are as follows:

1. Output from the temperature controller (the manipulated variable is the flow of cooling water).
2. Temperature in the reactor.
3. Output from the pressure controller (the manipulated variable is the flow of hydrogen in to the reactor).
4. Pressure in the reactor.
5. Flow rate of hydrogen into the reactor.

In addition to these measurements, a laboratory analysis is performed on the final product. The final product is analysed in the laboratory when experienced operators believe that the gassing phase is complete and the amount of material, A, remaining in the batch is below a specific target value. Depending upon the results of this laboratory analysis, the batch may be cooled. If the amount of material, A, in the sample exceeds a target value, the batch is held at an elevated temperature for another hour, delaying the cooling phase until the target for the concentration of A is attained. Operators can use the measured rate of hydrogen flowing into the reactor to provide an indication of when the gassing phase is complete. Unfortunately, this simple analysis is not always accurate as the conversion of A continues for a varying period of time after the flow of hydrogen into the reactor has ceased. Unfortunately, operations staff have been unable to identify the reason why some batches continue to react for long periods after the hydrogen flow ceases. There is therefore a reliance on laboratory measurements to determine when the reaction is complete.

This reliance on intermittent laboratory measurements means that the process is not achieving optimal operation. Nearly all production batches will have been processed longer than necessary before they enter the cooling phase. Therefore there is the potential to reduce the overall cycle time for this process if the end-point can be clearly identified. The ideal solution would be to provide a real-time measurement of the concentrations of A and B in the reactor. Unfortunately, such an instrument is not available and a key aim of this work was to provide a data driven mechanism able to identify when the desired concentrations of A and B in the reactor have been reached. The successful development of such a mechanism could lead to a substantial reduction in operating costs as the decision to terminate a batch could be made earlier than is currently possible.

3. Partial least squares (PLS)

3.1. Basic algorithm

PLS is a tool that can be applied whenever plant variables can be divided into cause (**X**) and effect (**Y**) values. The method is typically used in preference to alternative identification algorithms, such as multiple linear regression (MLR) when developing data-based models. Its advantage is that unlike certain other identification algorithms it is able to produce accurate and robust models in situations where high levels of correlations exist between the cause variables.

PLS works by selecting factors of cause variables in a sequence which successively maximises the explained covariance between the cause and effect variables. Given a matrix of cause data, \mathbf{X} (of size $m \times nx$, where m is the number of observations and nx is the number of cause variables), and effect data, \mathbf{Y} (of size $m \times ny$, where ny is the number of effect variables), a factor of the cause data, \mathbf{t}_k (length m), and effect data, \mathbf{u}_k (length m), is evaluated, such that

$$\mathbf{X} = \sum_{k=1}^{np < nx} \mathbf{t}_k \mathbf{p}_k^T + E \quad \text{and} \quad \mathbf{Y} = \sum_{k=1}^{np < ny} \mathbf{u}_k \mathbf{q}_k^T + F \quad (1)$$

These equations are referred to as the *outer relationships*. The vectors \mathbf{t}_k are mutually orthogonal. These vectors and the \mathbf{u}_k vectors are selected so as to maximise the covariance between each pair $(\mathbf{t}_k, \mathbf{u}_k)$. E and F are errors and \mathbf{p}_k and \mathbf{u}_k are referred to as loading vectors.

Linear regression is performed between the \mathbf{t}_k and the \mathbf{u}_k vectors, to produce the *inner relationship*, such that:

$$\mathbf{u}_k = b_k \mathbf{t}_k + \varepsilon_k \quad (2)$$

where b_k is a regression coefficient, and ε_k refers to the prediction error. The PLS method provides the potential for a regularised model through selecting an appropriate number of scores, or latent variables, \mathbf{u}_k in the model (np). Furthermore, it is often found that a relatively small number of the low-index latent variables can explain the greater part of the variation in both the cause and effect variables. Cross validation can be used to select the necessary number of latent variables. For further details of the PLS algorithm, the reader is referred to Geladi and Kowalski (1986).

3.1.1. Multi-way PLS

PLS is a linear tool, which unfortunately limits its effectiveness when applied to batch processes which are typically non-linear in nature. For such processes, the multi-way data unfolding techniques proposed by Nomikos and MacGregor (1994) tend to be used. Multi-way principal component analysis and multi-way partial least squares have been applied successfully to a variety of processes, including fed-batch fermentation systems (Lakshminarayanan et al., 1996; Lennox et al., 2001) and nuclear waste storage tanks (Wise & Gallagher, 1996).

The unfolding of process data is best explained using Fig. 1 as an illustration. The data collected from the process can be thought of as a three-dimensional matrix of size $I \times J \times K$, where I is the number of batches for which data is available, J the number of variables that are measured and K is the number of samples collected during the batch. This matrix is transformed, or unfolded, into a two-dimensional matrix, with all measurements collected from each batch occupying a single row of the resulting two-dimensional matrix. The resulting matrix is then of size $I \times J \times K$. This approach to unfolding assumes that there are an equal number of observations made during each batch. This assumption is quite restrictive and alternative approaches are discussed later.

For many batch processes, only a single measurement of quality is available for each batch. This measurement being made at

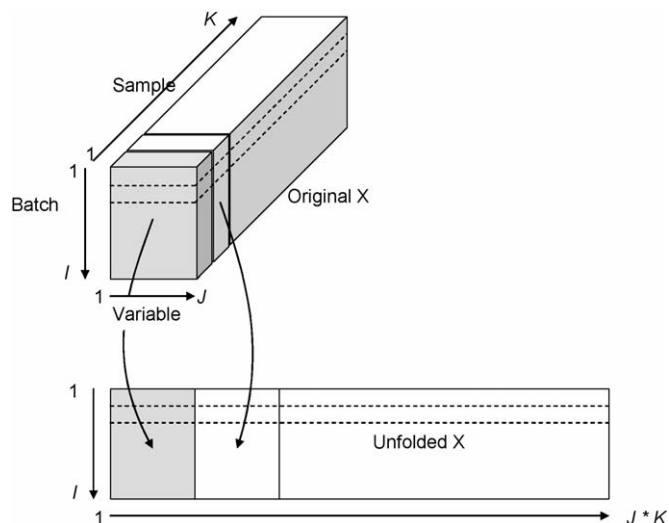


Fig. 1. Unfolding process.

the end of the batch. This data forms a vector \mathbf{y} of size $I \times 1$. The columns of the unfolded matrix, \mathbf{X} , and vector \mathbf{y} are then normalised. This scaling serves to remove the trajectories that the process variables tend to follow. After scaling the data, PLS can be applied to the unfolded data to generate a regression model capable of estimating the value of the quality variable given the values of the measurements during the entire batch. The size and nature of the unfolded matrices means that alternative regression techniques, such as ordinary least squares would not be suitable and therefore PLS is applied.

3.2. Model development

3.2.1. Data collection

The first stage in the development of the soft-sensor was to collect data from the process. Although a substantial amount of historical data was available, the data for each batch only contained a laboratory measurement at the end of the cycle. In those batches where this laboratory measurement indicated that the reaction was not complete a second and even third laboratory measurements were made. However, the purpose of this study was to develop a model able to estimate the concentration of A and B in the batch throughout the gassing phase. To develop such a model it is necessary to have measurements of the A and B concentrations during the entire batch.

Following consultation with plant engineers and operators, measurements of the concentrations of A and B in the batch were sampled at approximately 2-h intervals during a series of test batches. The procedure, which was then followed to develop the model meant that data from both the batches containing multiple samples and the historical batches containing only one or two samples could be utilised.

3.2.2. Data preprocessing

Process engineers familiar with the process are able to make a reasonable estimate of the likely duration of each batch by considering how exothermic the reaction is in the early stages of the heating phase. This knowledge is important as it contains

implicit information of the properties of the raw materials and consequently the progression of the batch. To capture this information in the model it was found that it was necessary to create two artificial variables: the cumulative sum of the output from the temperature controller and the cumulative sum of hydrogen flow into the reactor. The temperature within the reactor is maintained within a fixed upper and lower limit during the batch cycle. The cumulative sum of the output of the temperature control system provides an indication of the rate at which heat is released from the reaction. This property provides a measure of the properties of the raw material used in the process. These properties make an impact to the batch cycle time.

In developing a multi-way partial least squares model it is typically necessary for the cycle time for each batch to be equal. This is necessary for the construction of the matrices described earlier. Unfortunately, as with many batch processes, the process investigated in this study has cycle times that can vary by an order of magnitude. In such situations there exist three possible solutions:

1. Data collected from each batch is only considered up to the time of the shortest batch. Thus data collected during the later stages of the longer batches are not considered.
2. A secondary progression variable is identified and the process data is synchronised around this variable.
3. Dynamic time-warping techniques are employed to equalise the length of each of the batches.

Unfortunately, for the process investigated in this study it is the data collected at the end of the batch that is of greatest importance as these measurements provide an indication of whether or not the reaction is complete and the cycle has finished. Therefore, the first option is unsuitable. Furthermore, no secondary progression variables were found to be suitable for this process. The technique for creating a progression signal discussed in for example Eriksson, Johansson, Kettaneh-Wold, and Wold (2001) and Rothwell, Martin, and Morris (1998) was applied in this work. However the results were not satisfactory. Dynamic time warping (Kassidas, MacGregor, & Taylor, 1998) is a relatively complex method for coping with variable cycle times, which has difficulties in its application in real time (Westerhuis et al., 1999). In this work the decision was made to use a novel and simpler approach.

The approach that was adopted in this work for catering for the variable cycle times was a two-stage process. In the first stage a series of *pseudo batches* were created. This is explained in Fig. 2. This figure shows data collected from two batches, batch 1 and batch 2. The data from each of these batches is unfolded into a vector, as described earlier. During batch 1, four measurements of product quality are collected at various stages during the batch. These samples are labelled $y_{1,1}$, $y_{1,2}$, $y_{1,3}$ and $y_{1,4}$. Only a single quality measurement is made during batch 2 and this measurement was made at the end of the batch, labelled y_2 . As there are four quality measurements taken during batch 1, this batch is divided into four pseudo batches. The unfolded data collected up to the first quality measurement during batch 1 is labelled batch 1a, the data collected between the first and

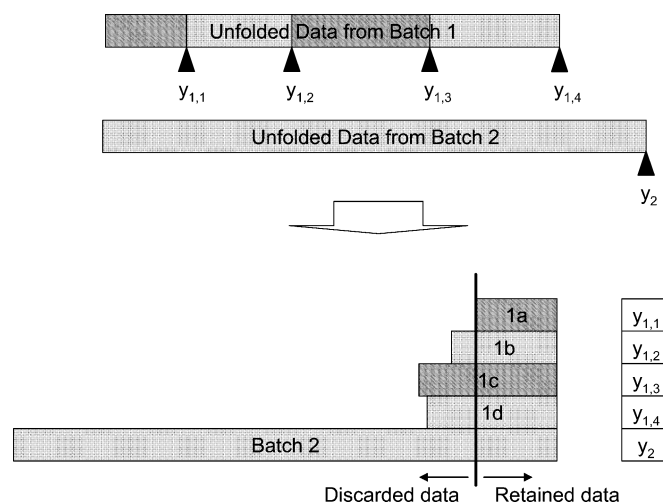


Fig. 2. Synchronisation of data.

second quality measurements is labelled batch 1b and so on. In performing this operation it is clear that there will be large variations in the time for each of the pseudo batches and full batches. To equalise the length of the unfolded vectors for each batch, the vectors are synchronised to the end-point, as shown in Fig. 2 and the batch with the minimum length is identified. In this study a minimum batch length of 2 h was used and the cause matrix was created from this data. All the earlier measurements were discarded.

Following the generation of the unfolded cause and effect matrices, the coefficients of the model were identified using the standard PLS algorithm described earlier.

In real-time, a vector of cause data, collected over a moving window of 2 h, will be applied to the model. This will mean that no estimate of product quality will be available during the first 2 h of the batch. However, after this time a continuous estimate of product quality will be available.

In addition to identifying a soft sensor to estimate the concentration of A and B in the reactor, a second model was identified. This model was developed to give operators and engineers an early prediction of the likely cycle time for the batch. This model was created by using PLS to identify the relationship between an alternative cause matrix and effect vector. The cause matrix was an unfolded matrix that contained all the process measurements recorded in the first 2 h of the batch only and the effect vector contained the cycle times for each of the batches. The purpose of this model was to provide an early estimate of the likely cycle time of a batch. This estimate being made after the batch had been in operation for 2 h. The availability of such a model offered the potential to provide process operators and engineers with the ability to plan and schedule future operation of the plant with greater accuracy.

4. Results

4.1. Off-line results for A/B concentration estimation

Fig. 3 shows a comparison of the actual and estimated B concentrations in all the evaluation batches (i.e. those batches

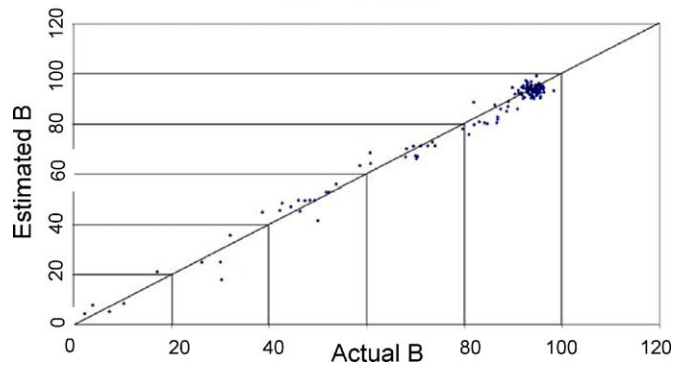


Fig. 3. Comparison of estimated and actual B concentrations.

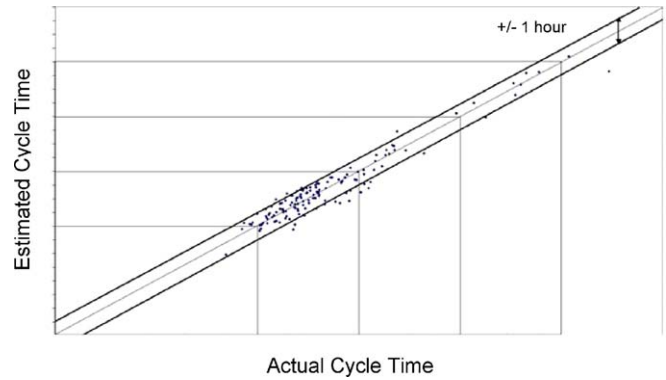


Fig. 5. Comparison of estimated and actual cycle time.

not used in the identification of the model). This figure shows that the accuracy of the estimates is good with 95% of estimates accurate to within $\pm 3\%$. The R^2 value for these data was 0.97. Although this accuracy is considered to be sufficient during the early stages of the batch, it will be necessary for this accuracy to be improved towards the end of the batch. The estimate at the end of the batch is critical as this is when the decision needs to be made as to whether the gassing phase is complete or not.

Fig. 4 shows the continuous estimates made of the concentrations of A and B in the reactor for an evaluation batch. The squares and circles in this figure show the actual measurements recorded in the laboratory during the batch, with the solid lines the continuous estimates of A and B concentration provided by the developed model. The results from this batch are representative of the results for other batches and show that good estimates of A and B concentrations are achieved through the duration of the batch.

4.2. Off-line results for batch time estimation

Fig. 5 compares the estimated with the actual cycle times for each evaluation batch. The solid diagonal lines indicate that for the vast majority of the batches, approximately 85% of them, the estimated cycle time is correct to within ± 1 h. The R^2 value

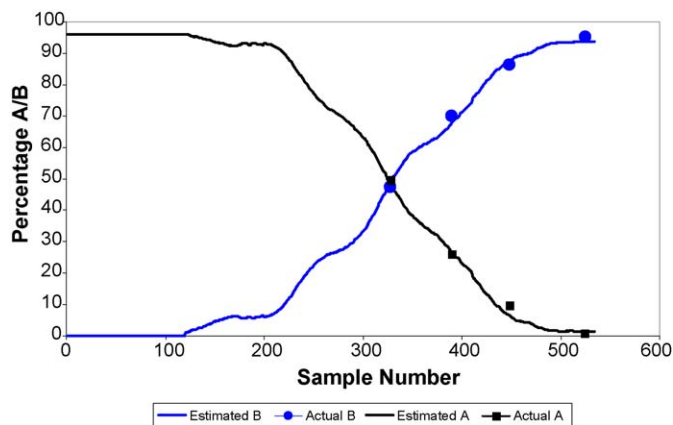


Fig. 4. Estimates of A and B concentrations.

for these data was 0.92. These results were considered by plant engineers to be an accurate and useful estimate of the cycle time, particularly as the estimate was made only 2 h after the start of the batch. The accuracy of the model is a result of the fact that the data collected during the initial heating up phase of the reaction provides an indication of how exothermic the reaction is, which is closely related to the cycle time.

It is difficult to improve the accuracy of the cycle time estimator with the data that is currently available. The reason for this is that the operating procedures on this process are such that the exact time at which the reaction is complete is unknown and only an approximate timing of the end-point is available. This estimate being when operations staff believe the reaction is complete and have ordered a sample to be analysed in the laboratory, which is subsequently found to reveal that the batch is complete. The next stage of this work will investigate whether performing more laboratory tests around the estimated end-point will allow a model with greater accuracy to be realised.

In an attempt to identify the likely improvements that could be made to the cycle time estimates, with an improved knowledge of the precise end-point of the batch a model was developed to estimate hydrogen time. The flow of hydrogen into the reactor is a purely process driven parameter. As hydrogenation takes place, hydrogen is drawn into the reactor. As the hydrogenation reduces, so does the flow of hydrogen into the reactor until the flow of hydrogen stops completely. The time between the hydrogen flow starting, the beginning of the gassing phase, and the point at which the flow stops is termed the hydrogen time. Fig. 6 compares the actual and estimated hydrogen time obtained from a MPLS model.

Fig. 6 shows that compared with the estimates of overall cycle time, the accuracy of the hydrogen time estimate is much improved. In fact for 85% of batches the estimate is accurate to within ± 30 min. The R^2 value for these data was 0.94. The reason for the improved results is that unlike the cycle time there is no uncertainty in the hydrogen time as it is clear when the hydrogen flow has stopped. Although being able to estimate the hydrogen time has little significance for process operations, it does indicate the likely improvements that could be made to the cycle time estimate if the end-point of the batch was known with greater accuracy.

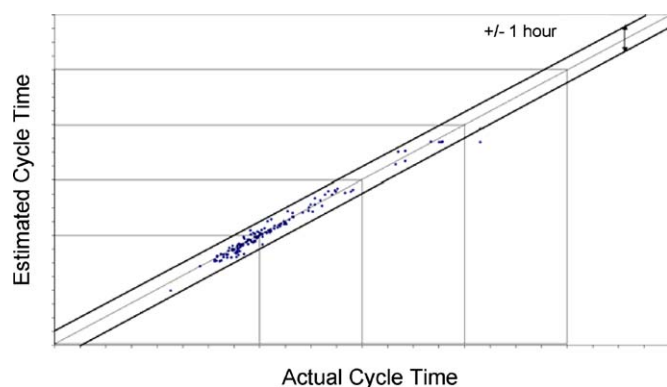


Fig. 6. Estimated vs. actual hydrogen time.

4.3. On-line results

The system described above was installed on the process plant and for a period of 2 months providing real-time advice to process engineers. These trials produced estimates with similar accuracy to those obtained in the off-line studies. In the next stage of the trials, further laboratory tests were to be conducted during the later stages of the batch so that a more accurate measure of batch cycle time could be obtained. However, despite the success of the preliminary trials, the development of the system was suspended after the trial period. The reason for this was because, following a process review, Aroma and Fine Chemicals made changes to its operating practices which meant that the developed system became redundant. Studies are still ongoing at the site to see if the developed technology can be applied to other batch processes.

5. Conclusions

This paper has described how multivariate statistics can be applied to a batch process as a means of extracting information from the data that is routinely collected and logged. Existing multivariate statistical tools were found to have limitations in the application to the process under investigation and as a result of this, novel techniques were developed to enable multi-way PLS to be applied to a batch process which exhibited significant variation in cycle time. The resulting system has been shown to provide an accurate estimate of conditions within the reactor and is also able to provide a long-term prediction of the likely cycle time of the batch. This information can help operators and engineers manage the process better and identify the end-point of the batch with improved accuracy. Following a successful off-line study, the developed system was installed and tested in real-time. During these trials the system was shown to provide benefits to process operations.

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