Application of Near-infrared Spectroscopy in Batch Process Control

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Abstract: While batch processes are gaining ever increasing importance in the manufacturing industries, control of the product quality remains to be a serious challenge. To improve overall process understanding and control, new analytical techniques, such as Near-Infrared (NIR) Spectroscopy, are starting to be employed in industry. Currently, these techniques are primarily used for process monitoring purposes and have not yet been explicitly included in feedback control systems. This paper investigates the ability of three different control systems to adequately control a simulated batch reactor using the NIR spectra as feedback information such that the product meets quality specifications. The particular problem considered in this paper is adequate representation of the NIR spectrum using a single variable that is then controlled by employing Model Predictive Controller (MPC). It is shown that the resulting controller performances are highly variable if the controlled variable is chosen by selecting a single peak in the NIR spectrum to represent that variable. On the other hand, by using Principal Component Analysis (PCA) to extract information from all of the wavenumbers and represent it using a single composite variable, which is then controlled, it is shown that the process can be adequately regulated.

Keywords: Batch Process, Near Infrared Spectroscopy, Model Predictive Control, Chemical Reactor

1. INTRODUCTION

Batch processes are gaining ever increasing importance in the manufacturing industries. They are particularly prevalent in the polymer, pharmaceutical and specialty chemicals industries where the focus is on the production of low-volume, high-value added products. However, a major problem that is faced by those involved in batch processing is the application of reliable control systems. The characteristics associated with batch processes that make them particularly challenging to control include the presence of time-varying and nonlinear dynamics, multitude of unmeasured disturbances such as concentrations of various raw materials, and the presence of irreversible behaviour (Bonvin 1998).

This paper deals with the application of control systems to a chemical batch reactor for which the requirement to manufacture high quality product often translates into the control problem of tracking the reference temperature profile (Cott and Macchietto 1989). This is because the reaction rates involving raw materials, intermediates and products are highly dependent on the temperature. As a result, the composition of the product is also highly dependent on the reactor temperature. The reference profile design consists of characterising, in terms of the reactor temperature, the following three main stages of batch reactor operation: heating up the reactor; controlling the reactor temperature to meet the process requirement and then cooling down the reactor. However, temperature control of batch reactors can be a difficult task due to the process nonlinearities and the absence of the steady-state operation (Shinskey 1996). Aziz et al. (2000) analyzed the performance of different types of controllers in terms of their ability to track a reference profile of reactor temperature.

Even if the adequate temperature control system is in place and the reactor temperature does follow closely its reference profile, there is no guarantee that the final product will meet its specifications. For example, changes in the reaction rates and/or inclusion of a new raw material (as an impurity) can introduce new reaction pathways, which may cause the final composition of the product to change significantly. As a result, product quality can deteriorate even in the presence of a satisfactory temperature control system. Hence, it would be highly useful to construct a control system that would focus on regulating not the reactor temperature but some other variables that are much more directly related to product quality. As a result, such control system should be able to maintain high quality product in the presence of disturbances.

Near-infrared (NIR) spectroscopy represents a set of non-destructive analytical techniques that have been extensively used to extract chemical and physical information from a product sample based on scattered light (Reich 2005). NIR spectroscopy has been widely used in the pharmaceutical industry to test raw materials, control product quality and monitor processes (M. Blanco 1998; Donald A. Burns 2001; Luypaert, Massart 2007). In the food industry there have been several applications of NIR spectroscopy being used for continuous process monitoring and control (Huang 2008).

Since the NIR spectra reflect the composition of the product, they represent excellent feedback information that could be used by control system to ensure the high quality of a product. So far NIR spectroscopy has been widely used for monitoring of manufacturing processes (Reich 2005; Jorgensen 2004; Scarff 2006). However, there is currently no publication proposing a method of explicitly using NIR spectra as feedback information to control the temperature of a reactor in order to ensure that the manufactured product conforms to high quality standards.

One clear problem in using NIR spectra as feedback information is the large number of variables that are needed to replicate information contained within the NIR spectrum. Arguably the number of variables should be equal to the number of spectral channels (wavenumbers) in order to completely characterise a given NIR spectrum. However, if this guideline is followed then the resulting control problem will potentially have several hundred controlled variables which could not be simultaneously controlled using typically only a handful or even just one or two manipulated variables.

In this paper, the problem of incorporating NIR spectrum as feedback information is addressed by using two different approaches. Both approaches utilise Model Predictive Control (MPC) framework but with a different definition of a controlled variable. The first approach is based on an idea of selecting wavenumber corresponding to one of the spectral peaks as a controlled variable. However, there are currently no clear guidelines regarding the selection of the peak to be considered as a controlled variable. The second approach is to use multivariate statistical analysis tools, namely Principal Component Analysis (PCA), in order to extract the information from NIR spectrum and represent it in a format of a single composite variable. This composite variable can then be regulated by means of a control system. Assessment of the controllers' performances is conducted using a simulated chemical batch reactor. The NIR spectrum is simulated by assuming that it is a linear combination of pure spectra related to individual compounds.

2. PRELIMINARIES

In this section the general concepts of Model Predictive Control (MPC) and Principal Component Analysis (PCA) are briefly introduced in order to facilitate the understanding of the control methodologies employed in the paper.

2.1 Model Predictive Control (MPC)

MPC (Maciejowski 2002) refers to a class of control algorithms that utilise an explicit process model to predict the future response of a plant. At each sampling instant, the MPC algorithm attempts to optimise future process behaviour by computing a sequence of adjustments that should be made to the manipulated variables. The first input in the optimal sequence is then implemented, and the entire calculation is repeated at the next sampling instant.

The key ingredient of the MPC controller is a prediction model used to forecast future process behaviour. In this paper the ARX structure (auto regressive with exogenous inputs) is chosen as the prediction model, and it is given as follows:

$$y(k) = -\sum_{i=1}^{n_y} a_i y(k-i) + \sum_{j=1}^{n_u} b_j u(k-j) + e(k) \quad (1)$$

where $y(k)$ and $u(k)$ are the controlled and manipulated variable, respectively, at a sampling instant k. The model error is represented by $e(k)$. The order of the ARX model is determined by the values of n_y and n_u.

This cost function for the selection of the appropriate control action is given in (2).

$$J = \sum_{i=1}^{p} \alpha\left(y_r(k+i/k) - \hat{y}(k+i/k)\right)^2 + \sum_{j=1}^{m} \beta\Delta u(k+j-1/k)^2 \quad (2)$$

Where J is the cost function to be minimized, p and m are the prediction and control horizons, respectively. y_r and $\hat{y}$ are the reference (set-point) values and estimated future output values, respectively, α and β are the weighting parameters for the controlled and manipulated variables, respectively. Finally, Δu is the change in manipulated variable (incremental control move) that is to be computed by the MPC algorithm.

The target of the cost function in (2) is to force the future output to track the reference trajectory over the specified prediction window p, while taking into account the balance between error energy and incremental control energy.

2.2 Principal Component Analysis (PCA)

The primary objective of Principal Component Analysis (PCA) is to capture the majority of variation present in data using a minimal number of composite variables, named principal components (PCs) (Johansson 2001; Berrar 2003). This dimensionality reduction is performed by exploiting the inter-dependence between measured process variables, such as individual wavenumbers in the NIR spectra.

For the analysis of spectroscopic data, such as that obtained from the NIR instruments, the power of PCA lies in its ability to condense the correlated information from hundreds of wavenumbers into a small number of mutually orthogonal principal components (PCs). Formally, PCA performs the following matrix decomposition:

$$\mathbf{X} = \mathbf{T}\mathbf{P}^{\mathbf{T}} + \mathbf{E} \quad (3)$$

where $\mathbf{X}$ represents measured process data organised in n rows and m columns. PCA decomposes this data matrix into the product of two matrices **T** and **P**, as shown in (3). **T** and **P** matrices contain as columns the so-called PCA scores and PCA loadings, respectively. **E** matrix represents the information contained within the matrix **X** that is not represented in the first nc principal components. Normally, each column of the data matrix **X** corresponds to a particular process variable, while the particular row is related to a specific sampling instant in time. In the context of NIR spectra, the columns of **X** represent specific spectral channels or wavenumbers while the rows contain data related to the whole NIR spectrum measured at a particular instance in time.

Due to the fact that the columns of the loadings matrix P are orthogonal, the expression for the calculation of scores is given as:

$$\mathbf{T} = \mathbf{XP} \tag{4}$$

It is the expression in equation (4) that will be utilised in this paper in order to condense information from hundreds of wavenumbers present in **X** into a single composite variable, namely the score associated with the first principal component.

3. CONTROL METHODOLOGY

3.1 Temperature Cascade Control (TCC)

A standard control problem in chemical reactor operation is that of controlling reactor temperature such that it follows a certain pre-computed reference trajectory, which should in turn ensure that the product quality will be satisfactory. Ultimately, reactor temperature is controlled by manipulating the flow of coolant or steam into the reactor's jacket. However, due to the presence of numerous disturbances, such as the feed temperature and the temperature of the incoming coolant, this control problem is addressed by employing two controllers in master-slave configuration, as shown in Fig.1.

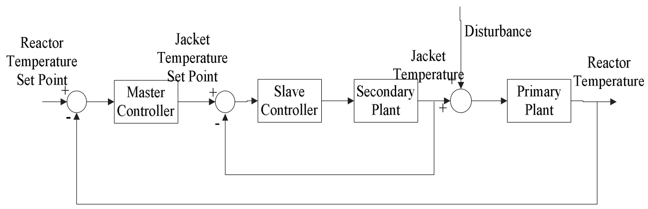

Fig. 1. Control of Reactor Temperature Using TCC System

The primary control loop, also known as the Master control loop, controls reactor temperature by adjusting the inlet jacket temperature set-point. The secondary control loop, known as the slave control loop, regulates jacket temperature by manipulating the flow of either coolant or steam into the jacket. Hence, the manipulating variable of the master control loop is the set-point for the slave control loop. This method of cascading controllers is very popular in the process industries and is particularly useful when there are disturbances associated with the slave controller's manipulated variable (Seborg 2004).

In this paper, a PI controller is used in the primary (slave) control loop while the PID controller is employed in the master (primary) control loop.

Note that the TCC system controls product quality implicitly, through the regulation of reactor temperature. The main problem with such implicit control arises with the occurrence of specific disturbances and process dynamics' changes, which adversely affect the underlying relationship between the reactor temperature and the product quality. As a result, optimal temperature profile will change. However, unless the optimal profile is calculated in real-time, TCC system will typically not have access to it. Instead, TCC will use existing reference trajectory, which is sub-optimal and may result in unsatisfactory product quality as demonstrated in the results section of this paper.

3.2 Wavenumber-Based MPC Control (Wn-MPC)

Spectroscopic instrumentation is being increasingly used to provide measurements, such as NIR spectra, that are in some way closely related to the product quality. By incorporating these measurements as feedback information into the control system the product quality control is addressed more explicitly when compared to the TCC scheme. One possible control system structure that incorporates NIR spectra as feedback information is shown in Fig. 2.

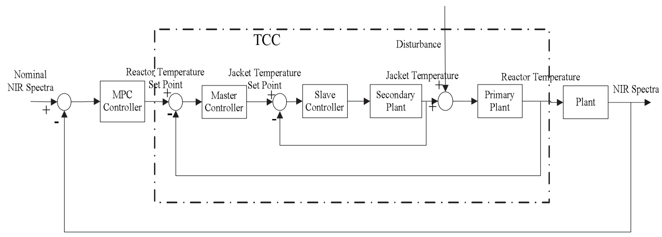

Fig. 2. Basic Structure of Wn-MPC Control

This new control structure incorporates the TCC system from Fig. 1 and augments it with the additional outer control loop, namely MPC control loop. The manipulated variable of the MPC controller is the reactor temperature set-point while controlled variables are the intensities of NIR spectra at a particular set of wavenumbers. Hence, within this control system structure, the TCC system can be viewed as a slave controller while MPC can be viewed as a master controller. This control system structure will be referred to as Wn-MPC.

The reference profile for the wavenumber is obtained by collecting NIR spectra from a 'nominal' batch, during whose progression no major disturbances were present and the standard TCC control scheme was used.

Since each wavenumber in the NIR spectra represents a candidate variable to be used as feedback information, there may be hundreds of potential controlled variables. Therefore, serious practical problem that arises when attempting to implement Wn-MPC is to decide on the set of wavenumbers that will be used as controlled variables. Currently, there are no clear guidelines as to which wavenumber should be selected for control purposes. In this paper a range of wavenumbers was selected and their suitability was evaluated by incorporating them into Wn-MPC as controlled variables.

3.3 PCA Score-Based MPC Control (Sc-MPC)

In order to incorporate information from all of the wavenumbers into a feedback signal, a modified control system structure is used, as shown in Fig. 3.

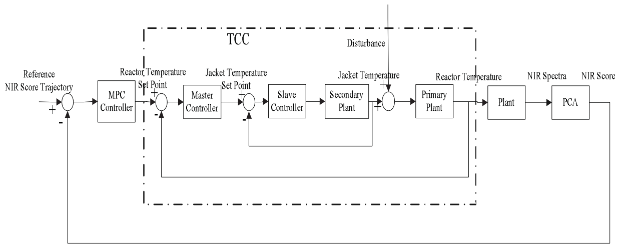

Fig. 3. Basic Structure of the Sc-MPC Control

This control scheme differs from Wn-MPC in that it includes a block containing the PCA model that pre-processes feedback information, namely NIR spectra. The result of PCA processing is a small set of variables, called scores, that contain information related to all of the measured wavenumbers. This is in contrast to Wn-MPC where the feedback information relates to only a few wavenumbers.

In this paper it is assumed that a PCA model is constructed using NIR spectra collected from a nominal batch. This nominal batch is run in the absence of any major disturbances using TCC control scheme. Hence, the resulting NIR spectra are assumed to represent reference profile that is to be replicated by Sc-MPC. In order to extract the main features from the highly multivariate NIR spectral data into a single variable, PCA model is applied. The resulting score trajectory is used as a reference profile that Sc-MPC is required to follow.

4. CASE STUDY

4.1 Chemical Reactor Simulation

This paper documents the application of three different control systems to a simulated chemical batch reactor taken from Cott (Cott and Macchietto 1989). The reactions taking place are given as follows:

$$A + B \xrightarrow{k_1} C;\; A + C \xrightarrow{k_2} D \tag{5}$$

where *A*, *B* are the raw material, *C* is the desired product and *D* is the waste product, while k_1 and k_2 are the rates of the two reactions.

The control objective is to track the reactor temperature T_r reference trajectory by adjusting the jacket temperature T_{jsp}.

4.2 Disturbance Description

Three different control systems, described in section 3, were evaluated by injecting large disturbance and observing the control system response. Disturbance was chosen to be a reduction in a value of a reaction rate constant k_1 by 8%.

4.3 Prediction Model Identification

Training data for the Recursive Least Squares (RLS) algorithm was obtained using the TCC system structure, shown in Fig. 1. To excite the process dynamics, reference temperature trajectory was perturbed for three batches by adding a PRBS signal of amplitude 0.1 degrees C and switching time of 60 seconds.

In this particular case study ARX based prediction models were developed with $n_y = 2$ and $n_u = 80$. The data-driven identification method of RLS was used to develop dynamic models for both Wn-MPC and Sc-MPC controllers.

The output signal considered during the prediction model identification is the deviation of a controlled variable from its nominal trajectory. This controlled variable may be spectral intensity at the particular wavenumber (in the case of Wn-MPC control) or the value of the PCA score (in the case of Sc-MPC control).

4.4 Wavenumber Selection

In the case of Wn-MPC, candidate controlled variables were taken to be those wavenumbers that corresponded to a local peak of the measured NIR spectrum. In this particular case study the wavenumbers corresponding to the local peaks in the NIR spectra and, therefore, representing the candidate controlled variables were 2, 77, 98, 127, 161 and 232, as illustrated in Fig. 4.

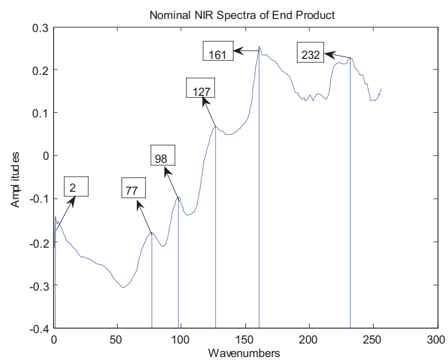

Fig. 4. Selection of spectral peaks as controlled variables

For each of these wavenumbers prediction model was identified and the corresponding MPC controller was constructed and evaluated. The corresponding controllers are designated with a chosen wavenumber written within brackets following a label Wn-MPC. For example, Wn-

MPC(127) designates Wn-MPC controller that utilises wavenumber 127 as the controlled variable.

4.5 PCA Model Development

A PCA model was developed using NIR spectra collected from a single nominal batch. The first PCA score captured 93.8% of the variation present in the NIR spectra and was used as a reference trajectory in the subsequent implementation of Sc-MPC controller. The loadings vector associated with the first PCA score was then used in real-time to compute score value from the measured NIR spectra according to equation (4).

4.6 Results and Discussion

For each controller (TCC, Wn-MPC and Sc-MPC) the process was perturbed using the identical large disturbance described in section 4.2. The resulting NIR spectra that corresponded to particular controllers along with the reference spectrum are plotted in Figures 5 and 6.

Fig. 5 shows the NIR spectra obtained when the controllers used to regulate the batch reactor were TCC, Sc-MPC and Wn-MPC(77). Sc-MPC can be seen to outperform both TCC and Wn-MPC(77). In fact, the NIR spectrum obtained when using Sc-MPC controller was found to be very similar to the reference spectrum, as shown in Fig. 5. On the other hand, both TCC and Wn-MPC(77) clearly failed to reject the disturbance as evidenced by considerable deviation of their respective NIR spectra from the reference spectrum.

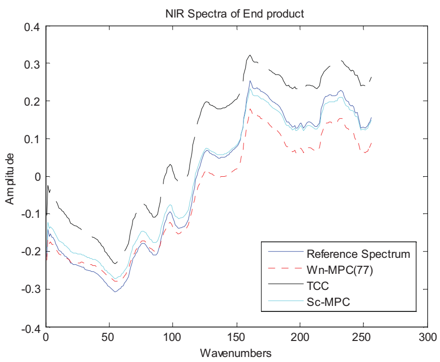

Fig. 5. NIR spectra of end product obtained when using TCC, Wn-MPC(77) and Sc-MPC

The reason for the discrepancy in performance between the TCC and Sc-MPC lies in the fact that the TCC control system does not consider NIR spectra as its feedback information and, furthermore, its reference temperature profile is not adjusted to account for the presence of the large disturbance, which has modified the underlying relationship between temperature and product quality. On the other hand, Sc-MPC explicitly considers regulation of the NIR spectra by using the composite of spectral measurements as its feedback information. Wn-MPC(77) also delivered sub-optimal performance because the spectral data contained in wavenumber 77 appeared not to be sufficient to characterise the majority of information contained in the entire NIR spectrum. Wn-MPC(77) is an example of Wn-MPC controller with its controlled variable obtained by randomly selecting one of the prominent peaks in the NIR spectrum, which is not an unlikely scenario in real applications.

The performances obtained by controlling NIR trajectories at different wavenumbers (77 127 161) using Wn-MPC controllers change largely, as demonstrated in Fig. 6.

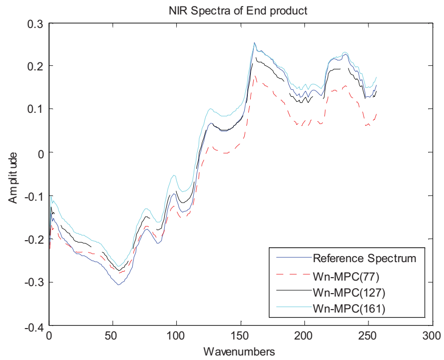

Fig. 6. NIR spectra of end product obtained when using Wn-MPC(127), Wn-MPC(161) and Wn-MPC(77)

The sum of square errors of the NIR spectra and its nominal values by Wn-MPC at every wavenumber are calculated and showed in Fig. 7. This figure shows a large variation in performance achieved by Wn-MPC controllers that utilise different wavenumbers (2, 77, 98, 127, 161, 232) as their controlled variables.

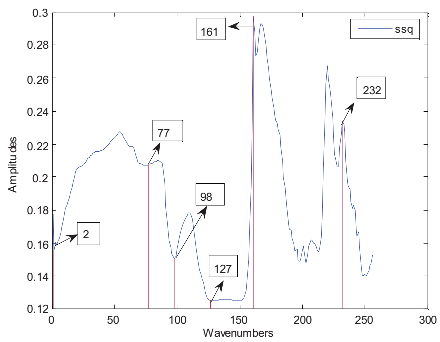

Fig. 7. The sum of square errors (ssq) by Wn-MPC at different wavenumbers

Even if the Wn-MPC is used with an optimally selected wavenumber, which is wavenumber 127 in this particular case study, the resulting control performance was found to be very similar to the performance of the Sc-MPC controller. This is demonstrated in Fig. 8 where the NIR spectra shown were obtained when the process was being controlled using Sc-MPC and Wn-MPC(127).

Hence, the improvement in performance delivered by Wn-MPC(127) is not considerable while the trial-and-error procedure involved in selection of the wavenumber to be controlled may be prohibitively time-consuming and expensive. On the other hand, Sc-MPC delivered satisfactory

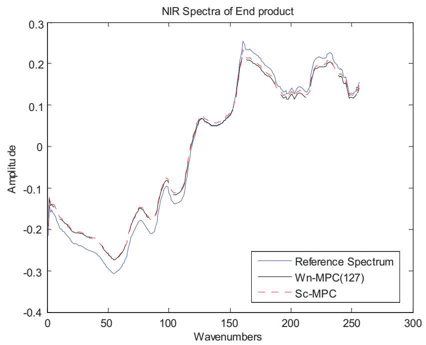

Fig. 8. NIR spectra of end product obtained when using Wn-MPC(127) and Sc-MPC

performance that was similar to that of the Wn-MPC(127) controller. In addition, the controlled variable is automatically selected requiring no trial and error in the case of Sc-MPC. Hence, Sc-MPC controller was found to require minimal user interaction when selecting appropriate controlled variable while also delivering a highly satisfactory performance.

Observed variability in performance can be explained by the fact that all of the considered Wn-MPC controllers focus on the feedback information contained within a single wavenumber. Hence, there may be cases where a chosen wavenumber conveys little information related to other segments of the overall NIR spectrum, such as the wavenumber 77. In these cases the resulting Wn-MPC will not deliver satisfactory performance, as is the case with Wn-MPC(77). Similarly, there may be cases where a single wavenumber does reflect many of the features of the entire NIR spectrum, such as the wavenumber 127. Resulting controller, namely Wn-MPC(127), will then deliver a satisfactory performance.

5. CONCLUSIONS

This paper investigated the ability of three different control systems to adequately control a simulated batch reactor using the NIR spectra as feedback information such that the product meets quality specifications. The first of the three controllers ignored the presence of NIR spectra and was solely concerned with the regulation of reactor temperature such that it follows pre-specified reference trajectory. This controller was found to be inadequate when the large disturbances altered the underlying relationship between reactor temperature and product quality. The other two controllers utilised aspects of the measured NIR spectrum in their formulations. One of these two controllers used spectral intensities at specific wavenumbers (spectral channels) that corresponded to local peaks in NIR spectra as feedback information and was referred to as Wn-MPC. The other controller used multivariate statistical tool, namely Principal Component Analysis (PCA) in order to extract the main features present in all of the wavenumbers and condense this information into a single composite variable that was controlled. This controller was referred to as Sc-MPC. Results of implementing these three controllers on a simulated batch reactor reveal that the Sc-MPC achieved satisfactory control while also requiring no user interaction when deciding on the variable to be controlled. On the other hand, performance achieved by Wn-MPC was found to be highly dependent on the choice of the wavenumber that is to be controlled. However, due to the lack of rigorous guidelines when selecting appropriate wavenumber and the resulting trial and error necessary to determine optimal wavenumber, it is questionable whether Wn-MPC can be used as a practical solution in industrial process control area.

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