

Trajectory Tracking of Exothermic Batch Reactor using NIR Spectroscopy

Olufemi Osunnuyi* Ognjen Marjanovic* Jian Wan* Barry Lennox*

* School of Electrical and Electronic Engineering, The University of Manchester

Abstract: The control of exothermic chemical batch reactors has received much attention in literature over the years for their increasing importance in manufacturing industries and also the unique quality control challenges that they provide. However, most of the control schemes proposed to deal with these challenges make use of models that implicitly control the product quality. For example it is assumed that a control scheme that successfully regulates the reactor temperature along an *a priori* calculated optimal profile should imply satisfactory quality trajectory control. In this paper it is shown that this assumption is not robust enough to deal with some kinds of disturbances that may occur during the batch. It is also shown that product quality control can be greatly improved by proposing a new control scheme that makes use of NIR spectroscopic measurements as feedback information for a quality control system. The results of two controllers using this scheme are compared with a more widely used implicit control strategy in two test cases with unmeasurable system disturbances.

Keywords: Batch process, Near Infrared Spectroscopy

I. INTRODUCTION

Batch and semi-batch processes are gaining increasing importance in manufacturing industries. Due to the flexibility they offer they are prevalent in the polymer, pharmaceutical and specialty chemicals industries where the focus is on the production of low-volume, high-value added products. An example of a batch process is the chemical batch reactor where the batch operation consists of charging the reactor, controlling the reactor temperature to meet some processing criterion, shutting down and then emptying the reactor. There is a steady increase of heat required during the initial phase of the batch to obtain the desired reaction temperature after which appropriate cooling is required to maintain the required temperature for the remainder of the batch [1, 2]. Previously not much attention was paid into how the reactor temperature reached the desired set points. However, due to work done on obtaining optimal reactor profiles so as to increase the yield of the desired product, research work subsequently focused on trajectory tracking control of batch reactors [3-5].

However, challenges occur when trying to implement reliable trajectory tracking control systems in batch processes due to the absence of steady state operation which requires the controller to track a highly transient profile within a short operating regime [6]. Some other unavoidable inherent

characteristics include presence of time-varying and nonlinear dynamics, multitude of unmeasured disturbances such as concentrations of various raw materials, and the presence of irreversible behaviour which all inevitably affect the reliability of overall quality control [7]. There has been quite a bit of literature dedicated to improving the quality of control in this regard. For instance, the capability to track the optimal reference profile of the reactor temperature for various kinds of dual-mode controllers was compared in [1]. Also, a multivariate control strategy based on empirical dynamic PCA models for the trajectory tracking of the optimal profile was developed in [8].

All these publications however, have been based on the assumption that the successful regulation of the reactor temperature should result in good control of the quality of the desired product. However, 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 [9]. For example if a disturbance occurred that altered the dynamic relationship between the product quality and the reactor temperature, the production of the product quality will depreciate despite successful regulation of the reference temperature profile. Hence, the control objective should be focused on controlling variables that are more directly related to the product quality.

In a quite a number of process industries, Near-infrared (NIR) spectroscopic measurements are widely used in providing a more direct quantification for the quality of the product. NIR spectroscopy is based on the absorption of electromagnetic radiation at wavelengths in the range 780–2500 nm [10]. This frequency range covers mainly overtones and combinations of the lower-energy fundamental molecular vibrations making the NIR intensities significantly weaker than the fundamental bands from which they originate [11]. Thus, the low molar absorptivity of NIR bands permits operation in the reflectance mode and hence the non-destructive recording of spectra of solid samples with minimal or no pre-treatment, thereby substantially increasing the throughput [12, 13]. Uses of NIR spectroscopy are widespread in the pharmaceutical industry to test raw materials, control product quality and monitor processes [12-15]. NIR analytical techniques have also found popularity in the food industry being routinely used for the compositional, functional and sensory analysis of food ingredients, process intermediates and final products [10].

Hence, the integrity of the quality of information provided by the NIR spectra can be exploited as essential feedback information used by the control system to enable enhanced quality production.

In this paper a quality control scheme that uses NIR spectroscopic measurements for the control of an exothermic batch reactor is proposed. In addressing the evident problem of dealing with the enormous amount of NIR spectra data two different controllers were designed and both compared with a conventional implicit reactor temperature controller using two different case studies.

II. METHODOLOGY

A. Introducing NIR Measuring Point

The most widely used method of controlling exothermic batch reactors involves regulating the reactor temperature along an optimal profile which has been calculated offline. The intention being that this optimal profile will help yield the product quality trajectory that maximises the conversion of the desired product. One of the most common forms of this implicit control scheme is the Temperature Cascaded Control (TCC) shown in Fig 1. TCC uses cascade control for the control of the reactor temperature. The slave controller controls the jacket temperature, whose setpoint is determined by the reactor temperature controller.

It can be observed in Fig 1 that the control loop is closed with the reactor temperature and hence will only reject disturbances that occur within the loop (m_{d1}) i.e. on G_1 . An example of this disturbance could be a change in the heat transfer coefficient. However, when dealing with disturbances occurring outside this loop (m_{d2}), i.e. on G_2 , the regulation of the reactor temperature proves incapable of tracking the desired trajectory for product quality. The reason being that the disturbance m_{d2} changes the underlying relationship between T_R and M_{ABCD} ; hence the reactor temperature trajectory ought to change to yield the desired conversion of Mc . The best way of solving this problem is to introduce a new measuring point in the reactor that measures a variable that is directly related to the product quality, thus enabling the incorporation of another control loop using this new variable as feedback as shown in Fig 2.

This relationship ensures that the setpoint to the reactor temperature is dynamic, as opposed to the TCC scheme. In this paper the NIR spectroscopic measurements are used as this feedback information.

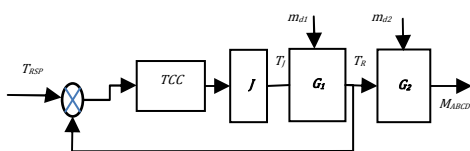


Figure 1: Block diagram of Temperature Cascaded Control on Batch Process

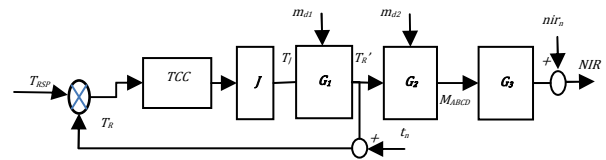


Figure 2: Block Diagram of Batch Process producing NIR Spectra

B. Creation of Artificial NIR Spectra

The measured NIR Spectra is constructed under the assumption of Beer-Lambert Law for low concentration absorption media. This law enables us to obtain the NIR Spectra as the weighted sum of the individual linear mappings of the concentrations through their respective ‘pure NIR spectra’ [16]. These individual linear spectral mappings represent the NIR spectra of each component. And their sum is the NIR spectra of the entire batch run.

Assuming a matrix $M \in \mathbb{R}^{N \times m}$ which contains m different compounds for N number of samples in a batch run and $\theta \in \mathbb{R}^{A \times m}$ as the pure spectra matrix for these compounds with A number of wave channels, then a complete batch of NIR Spectra ($NIR \in \mathbb{R}^{N \times A}$) can be calculated according to:

$$NIR(i,:) = \frac{M(i,:)}{(\sum_j^m M(i,j))} * \theta^T \quad (1)$$

In order to obtain the pure spectra, an artificial spectra generator was designed to create pure spectra not related to any particular compound but sufficiently shaped to resemble forms of common pure spectra used in industry.

It was observed with non-artificial pure spectra that quite often the ‘spectral peaks’ were very similar in shape to standard Gaussian distribution. This basic assumption was essential in developing the code that would produce reasonable pure NIR Spectra for the purposes of the case studies considered in this paper.

Therefore in order to use this code in constructing a particular pure spectrum the following would need to be specified: the desired number of spectral channels (or wave numbers), the number of Gaussian distributions and the maximum and minimum widths (i.e. the width range) of these ‘bell-shaped’ Gaussian distributions. With randomly selected wave channels acting as mean centred positions (μ) and with standard deviations (σ) also randomly chosen from the Gaussian width range, each Gaussian was generated on single a spectrum using the Gaussian function:

$$f(x; \mu, \sigma^2) = \frac{1}{\sigma\sqrt{2\pi}} e^{-\frac{(x-\mu)^2}{2\sigma^2}} \quad (2)$$

where x represents the individual wave numbers.

The normalised sum of all these spectra incorporates all the individual Gaussians and thus forms a single pure NIR Spectrum. This procedure is repeated for as many compounds are present in each specific simulation. An example of artificially generated pure spectra for four compounds is shown in Fig 3.

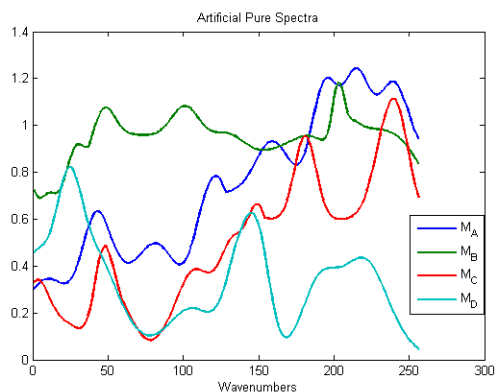


Figure 3: Sample of Artificially generated pure NIR Spectra

C. Quality Control Methodology

1) NIR Data Reduction for SISO Control

The control scheme used to regulate the product quality is achieved by closing the loop at the new NIR spectra measuring point as shown in Fig 4.

When closing the new control loop a new block Y is included because of the large amount of feedback information that is typical of NIR measurements. In this scheme block Y is used to reduce the number of variables that is fed back to the new quality controller C_q . In this paper, in which a SISO simulation is considered for the case study example, block Y represents two different data reduction techniques.

The first method of data reduction is a *wavenumber-selector* in which the most appropriate wavenumber for control is used. The selection of the appropriate wavenumber is very important because a wrong selection will lead to very poor control. One way of selecting the most appropriate wavenumber to control is by selecting the peak amplitude occurring in an NIR sample. However, this approach is based on trial and error. In this paper, the wave number from the NIR reference spectra with the largest variance was used. The reason for this choice is that the highest varying wavenumber will contain the best qualitative information of G_2 and also the highest amplitude does not necessarily imply the highest varying wavenumber as shown in Fig 5.

The second data reduction technique used was making block Y a Principal Component Analysis (PCA) model. This reduces each fed back NIR spectrum into a specified number of latent PCA variables based on the extraction of the largest possible variance in the data set.

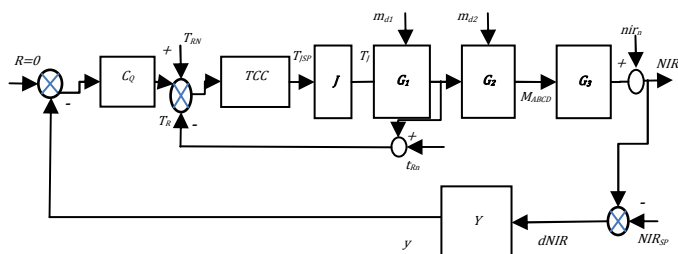


Figure 4: Quality Controller implemented on Batch Process using NIR Spectral measurements

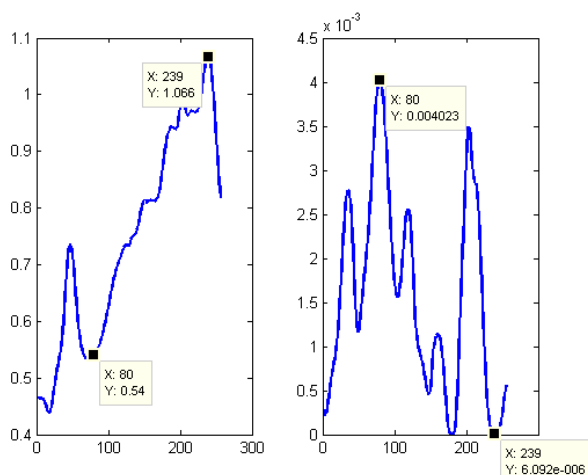


Figure 5: Single NIR spectrum and variation spectrum of entire batch spectra

In the case study of this paper, the results of controllers using these two choices of Y were compared with that of TCC.

2) Deviation Control

Finally, rather than tracking the selected NIR reference wavenumber or the corresponding PCA score trajectory the control requirement in this quality control loop is to minimise the deviation from the quality variable trajectory. This takes into account the advantage of possessing the nominal reactor temperature trajectory which is subsequently added to the setpoint of the quality controller. Quite often the Achilles heel of trajectory tracking control is the wide operating range that the process variables cover during the batch.

By subtracting this dominant transient from the NIR spectrum before feedback the control requirement is in effect reduced to a less complex setpoint tracking and causes the speed of the controller's response to increase. In other words the batch control problem becomes similar to a continuous control problem.

III. CASE STUDY

A. Specification of Model

As a benchmark case to test the control scheme shown in Fig 4 on, modifications were made to the nonlinear exothermic batch reactor model used by Cott and Macchietto [2] (schematically shown in Fig 6) to incorporate NIR Spectral measurements. This model has also been used by Aziz [1] and all of the model parameter values are the same as used in the latter paper except for the time constant of the jacket temperature T_J (τ_J) which is now 5mins as opposed to 3mins and the rate constant 1 for reaction 1 (k_1^1) was changed from 20.9057 to 20.8057. The sample time is 30s.

The control objective in the initial papers that have used this model is to control the reactor temperature by adjusting the setpoint to the inlet jacket temperature.

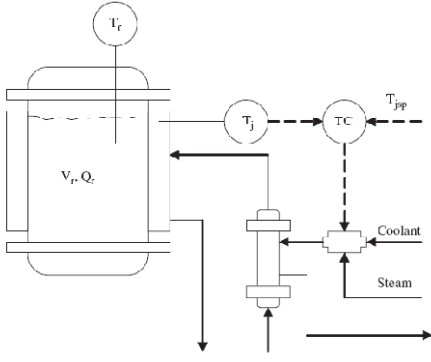


Fig 6: Schematic of a chemical batch reactor [17]

The reactions taking place are as follows:



Where A and B are the raw materials, C is the desired product, D is the waste product and R_i is the reaction rate for reaction i . The dynamic equations of the process that form block G_2 are:

$$\frac{dM_A}{dt} = -R_1 - R_2 \quad (5)$$

$$\frac{dM_B}{dt} = -R_1 \quad (6)$$

$$\frac{dM_C}{dt} = +R_1 - R_2 \quad (7)$$

$$\frac{dM_D}{dt} = +R_2 \quad (8)$$

$$R_1 = k_1 M_A M_B \quad (9)$$

$$R_2 = k_2 M_A M_C \quad (10)$$

$$k_1 = e^{\left(k_1^1 - \frac{k_1^2}{(T_R + 273.15)}\right)} \quad (11)$$

$$k_2 = e^{\left(k_2^1 - \frac{k_2^2}{(T_R + 273.15)}\right)} \quad (12)$$

Where M_i (kmol) is the number of moles for component i , k_i^j is rate constant j for reaction i and block G_3 which converts raw materials and products into a combined NIR Spectra is as given in equation (1).

The reader is referred to the original publications for a more complete description of the model which include the description of block G_1 .

B. Test Case Description

The evaluation of the performance of the three controllers will be carried out on two different test cases. The controllers to be compared are:

a) TCC (PID)

b) Wn-PID (i.e. C_q being a PID controlling a single wave number)

c) Sc-PID (i.e. C_q being a PID controlling the first PCA score only).

The two simulated test cases are to evaluate the controllers' ability to reject disturbances occurring at G_1 (m_{a1}) and G_2 (m_{a2}) respectively:

TEST 1: A drop in the heat transfer coefficient, which occurs due to deterioration of the thermal jacket over a period of time and causes a change in the operating conditions. This will test the robustness of the controllers in reacting to a change in an unmeasured parameter.

TEST 2: A 2% increase in the rate constant 1 for reaction 1 (k_1^1). This causes a disturbance in the reaction rate of reaction 1 and therefore fundamentally changes the model relationship between the reactor temperature and the reactants (and hence products) for which the controllers were nominally designed for.

C. Controller Assessment

Performance Index (PI): Under most industrial applications constructing a PI using the measurement of M_C is not feasible due to the extremely high cost of purchasing quality analysers. However, because a simulation example is being used it was convenient assessing the performance of the controllers using M_C and at the same time easily adapting this PI to the NIR spectra as well.

The MAE (mean absolute error) of the M_C of each controller from the nominal trajectory was calculated as follows:

$$MAE_C = \sum_i^N \frac{|M_C(i) - \bar{M}_C(i)|}{N} \quad (13)$$

The same was calculated for all the wavenumbers of the NIR spectra. And to combine these metrics into a singular measure (MAE_{NIR}) the square root of the sum of the squared scaled mean absolute errors was used.

D. Results and discussion

TEST 1: Fig 7 and Fig 8 show the responses of all three controllers to a change in the heat transfer coefficient. When the heat transfer coefficient decreases there is less flow of heat from the inlet jacket to the reactor which results in a rapid rise in the reactor temperature. The controllers are required to respond quickly to this change to avoid a large overshoot of the reactor temperature which would cause the off-specification production of the desired product. In both Fig 7 and Fig 8 it is observed that the quality controllers responded quicker to this change than the TCC. The extra control loop enables additional product quality-related feedback information to the jacket temperature and a quicker adjustment than that of the TCC.

Also, of the two quality controllers Fig 7 and Fig 8 show that the Sc-PID reacts more rapidly to the disturbance when compared to the Wn-PID. This is due to Sc-PID using

information that more comprehensively represents the system dynamics of the reactor than the Wn-PID. This advantage is clearly represented in Fig 7b where the output of Sc-PID signals a further and quicker lowering of the jacket temperature than the Wn-PID. The effect of this as seen in Fig 8a is a lower jacket temperature drop particularly at the end of the heating phase of the reactor, resulting in a lower reactor temperature overshoot and better tracking of the optimal profile. Overall, the effect of tracking the desired product trajectory is shown in Fig 8b to be significantly better when using Sc-PID and the performance indices in Table 1 show considerably lower values for the quality controllers –with Sc-PID being the lowest –than that of the implicit TCC.

TEST 2: Fig 9 and Fig 10 give the responses of all three controllers to a disturbance in the reaction rate due to an increase k_1' responsible for producing C. With this disturbance occurring at G_2 in Fig 4, the TCC lacks any essential feedback data informing it to adjust the reactor temperature to suit the new dynamic relationship. Therefore as shown in Fig 9a the TCC successfully tracks the now sub-optimal profile which results in very poor tracking of the desired product trajectory and a selected wavenumber (80) as show in Fig 10a and 10b. Using an implicit product quality control scheme (such as TCC) essentially loses the batch in such conditions. Whereas the product quality-related information supplied by the NIR spectroscopic measurements enables the quality controllers to create a new optimal trajectory for the reactor temperature as seen in Fig 9a. By reducing the overall temperature of the reactor in the presence of the disturbance there is a closer tracking of the quality variables as seen in Fig 10a and Fig 10b.

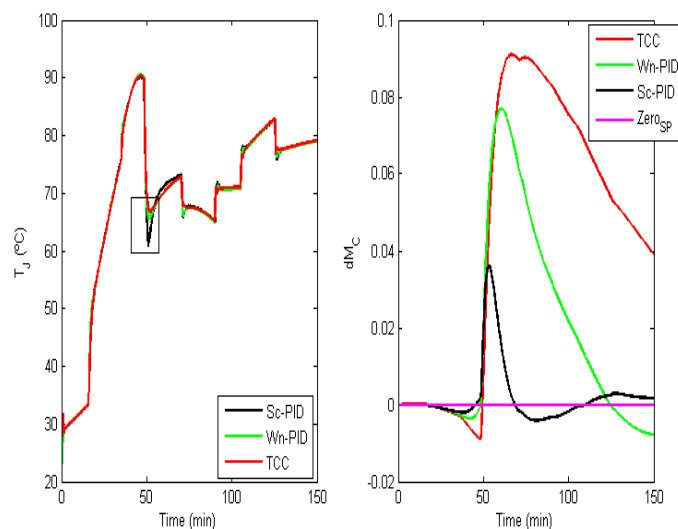


Figure 8: a) Responses to change in heat transfer coefficient in Jacket Temperature and b) Deviation from nominal trajectory in M_C

However, a much better performance of the Sc-PID in comparison to the Wn-PID is again noticed. There is a much quicker and settled response to the presence of this disturbance from the Sc-PID when observing the quality controller outputs of Fig 9b. The reason for this once again is that the first PCA score, which reflects the entire NIR Spectra, contains a lot more system information than any single wave number even when it is the highest varying wavenumber used.

These performances are again reflected in the MAE values of Table 1. The quality controllers far outperform the TCC, with the Sc-PID also proving to be better than the Wn-PID. It is worth noting that when the same test was carried out with Wn-PID to control the peak wavenumber (i.e. Wn_{239}) as opposed to the highest varying wavenumber (i.e. Wn_{80}), the performance index values returned were $MAE_C = 0.7216$ and $MAE_{NIR} = 0.4809$ for Wn_{239} . This very poor performance highlights the need to ensure the appropriate selection of a wavenumber.

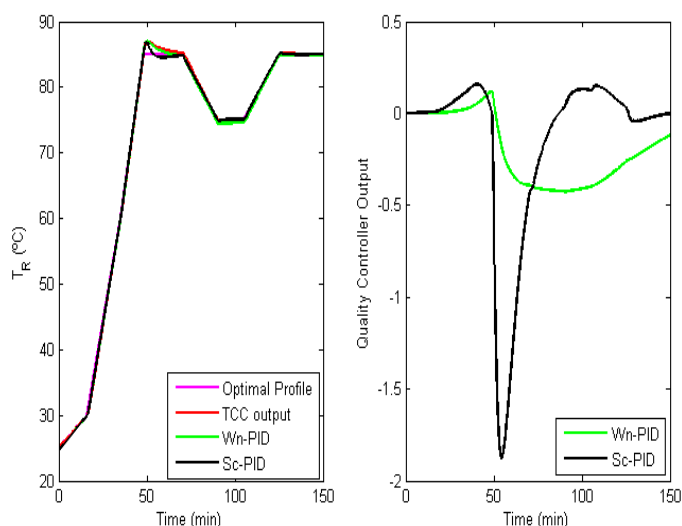


Figure 7: a) Responses to change in heat transfer coefficient in Reactor Temperature and b) Quality controller outputs

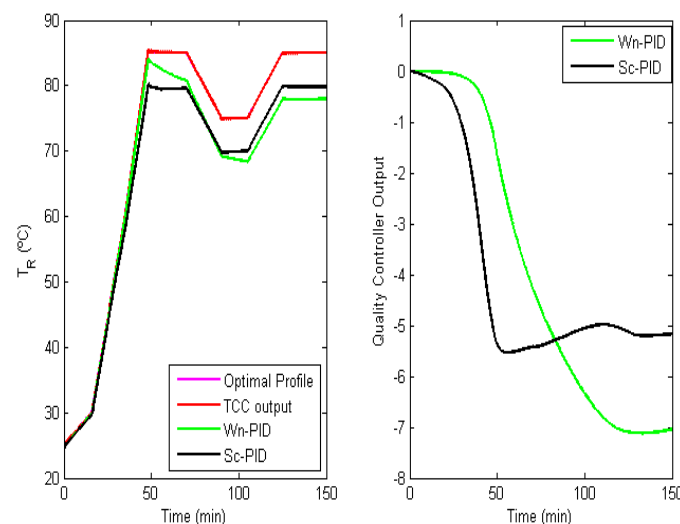


Figure 9: a) Responses to reaction rate disturbance in reactor temperature and b) Quality controller outputs

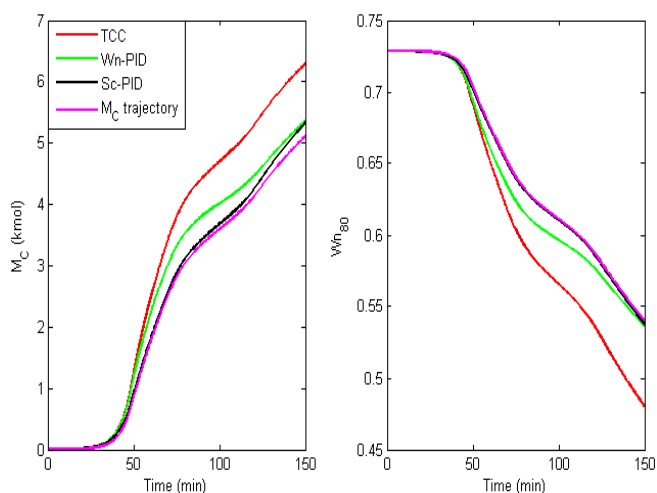


Figure 10: a) Responses to reaction rate disturbance in M_C and b) Wavenumber 80

IV. CONCLUSION

This paper has investigated the use of NIR spectroscopic measurements as feedback for trajectory tracking of the product quality of exothermic batch reactors. This provides a more direct form of tracking the desired product quality as opposed to the implicit form of quality control quite often employed.

Instead of the control objective being the trajectory control of the reactor temperature, the objective was modified to minimising the deviation of a product quality related variable from its nominal trajectory. This scheme not only enables the controller to respond to various disturbances affecting different components of the reactor dynamics, but also greatly increases the speed of the controller's response as well.

These were shown by comparing the results of a standard Temperature cascade control (TCC) scheme with two controllers that utilise NIR spectra as feedback information. The first of these two controllers is Wn-PID, which minimises the deviation of the highest varying wavenumber of the nominal NIR batch from its setpoint trajectory, and the second being Sc-PID, which minimises the deviation of the first PCA score from its nominal trajectory. The results of the two tests carried out reveal that the quality controllers far outperform the TCC. However, the Sc-PID consistently outperformed Wn-PID owing to the fact that the first PCA score contains significantly more information than any single wave number.

Disturbance Type	Controller	MAE_C	MAE_{NIR}
HTCC	TCC	0.0459	0.0256
	Wn-PID	0.0197	0.0097
	Sc-PID	0.0038	0.0020
RR-disturbance	TCC	0.7103	0.4009
	Wn-PID	0.2802	0.1258
	Sc-PID	0.0778	0.0318

Table 1: Table of Mean absolute errors (in M_C and NIR) for the controllers

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