On-line Multivariate Statistical Monitoring of a Fed-Batch Sugar Crystallisation Process

A. Simoglou1, P. Georgieva2, E.B. Martin1, A.J. Morris*, S. Feyo de Azevedo*

1 Center for Process Analytics and Control Tech., Merz Court, University of Newcastle, Newcastle upon Tyne, NE1 7RU, UK Tel: +44 191 222 6231; Fax: +44 191 222 5748
2 Dep. Chemical Engineering, Faculty of Engineering, University of Porto, R. Dr. Roberto Frias, 4200-465 Porto, Portugal Tel: +351 22 508 1694; Fax: +351 22 508 1632

Abstract

The paper describes statistical process control tools that have been applied for the on-line monitoring of an industrial fed-batch sugar crystallisation process. The process is characterised by distinct operating phases during operation and the presence of strong non-linear, dynamic relationships between the variables. Process performance is controlled manually by operators based on their experience. The success of each batch is determined at the end of the batch run through off-line crystal size distribution measurements. The development and application of a monitoring tool based on the on-line frequent process measurements could be of significant benefit, since it could realise early detection of operational changes, process faults and hence a reduction in the number of off-specification batches.

Keywords: Statistical process control, batch sugar crystallization

1. Introduction

Sugar crystallisation occurs through the mechanisms of nucleation, growth and agglomeration. The impact of operating conditions on these activities is not always well-understood. It can be characterised as a non-linear, non-stationary process. Significant research has been carried out into the development of first principles models (Feyo de Azevedo et al., 1994) to describe the process. More recently hybrid models based on both first principles and neural network models have been proposed (Georgieva et al., 2003). However, these models have been developed for process state estimation, the prediction of crystal size distribution (CSD) and for various control objectives including tracking, optimisation and robustness. The issues of the on-line detection of changes in process operation, early warning of process malfunctions and potential production failures have not been directly addressed by the existing models.

In this paper an empirical statistical approach is adopted for the development of a statistical model based on the available process measurements. Techniques investigated include Moving Window Principal Component Analysis (MWPCA, Lennox et al., 2001); Batch Observation (BO, Wold et al., 1998) and Batch Dynamic Principal

* Email: julian.morris@ncl.ac.uk Email: sfeyo@fe.up.pt
Component Analysis (BDPCA, Chen and Liu, 2002). These approaches assume that the available data captures the underlying process operating conditions. Then multivariate statistics such as Hotelling’s T2 control charts are calculated along with their appropriate control limits. These metrics and associated control limits are then used for the on-line monitoring of the process and the classification of a batch as in- or out of specification.

2. Process operation

The batch cycle of white sugar production is divided into several sequential phases. During the first phase the pan is partially filled with a juice containing dissolved sucrose (termed liquor). The liquor is concentrated by evaporation, under vacuum, until supersaturation reaches a predefined value (typically 1.15). At this stage seed crystals are introduced into the pan to induce the production of crystals. This is the beginning of the second (crystallisation) phase. As water is evaporated, the dissolved sugar concentration increases, resulting in crystal growth. As evaporation takes place further liquor or water is added to the pan to maintain the level of supersaturation and increase the volume. For economical reasons, towards the end of this phase, the liquor is replaced by other juices of lower purity (termed syrup). The third phase consists of tightening which is principally controlled by evaporation capacity. At the end of a batch, the pan is filled with a suspension of sugar crystals in heavy syrup, which is dropped into a storage mixer before centrifugation. Present pan control involves the manipulation of the feed flow rate of sugar liquor/syrup and the vacuum pressure. The process objective is to obtain sugar of high quality, which is measured in terms of the purity, shape and CSD at the batch end. CSD is quantified by the mass averaged crystal size (MA) and the coefficient of variation (CV). The desired values for MA are 0.5-0.6 mm and for CV: 28-32 %.

Available Measurements

The industrial unit is equipped with 15 sensors. The on-line recorded physical measurements are related with i) feed conditions - flowrate, brix (measured by refractometry), temperature and purity of the fed liquor/syrup and the flowrate and temperature of the water; ii) steam conditions - flowrate, temperature and pressure of the steam; and iii) variables inside the pan - pressure and temperature of the vacuum, the brix and the temperature of the solution, the level in the pan.

3. On-line multivariate statistical process control (SPC) techniques

Batch process modelling and monitoring has been always a challenging problem in chemical engineering due to the presence of non-linear behaviour and serial correlation, correlated and/or collinear data, varying batch lengths and multi-product production. Current empirical techniques include the bi-linear approaches of multi-way Principal Component Analysis (PCA) and multi-way Partial Least Squares (PLS), and the tri-linear methodologies of PARAFAC. Although the above bi-linear and tri-linear techniques have been successfully applied to batch processes they experience a number of limitations. For example they do not incorporate the process dynamics and the duration of the batches is assumed to be constant. Moreover, for on-line monitoring, it
is required that the whole batch trajectory is known. This requirement results in certain
assumptions being made in order to in-fill the unknown future values of the batch
trajectory. To overcome these problems and deal with unequal batches two alternative
approaches have been recently proposed, Batch Observation (BO, Wold, 1998) and
Moving Window Principal Component Analysis (MWPCA, Lennox et al. 2001). These
methodologies do not capture the dynamic behaviour of a batch process. Chen and Liu
(2002) proposed Batch Dynamic Principal Component Analysis (BDPCA), in an
attempt to capture the batch process dynamics. Due to the limited space of this paper, no
theoretical background to the above techniques is given. In the present communication,
the three techniques (BO, MWPCA and BDPCA) are applied in an on-line mode to
monitor the operating performance of the sugar crystallisation process. The monitoring
statistics used are those of Hotelling’s $T^2$ control charts.

4. On-line SPC of sugar crystallisation

Data from 14 industrial runs were available from a sampling period of 4 months.
Batches were discriminated as in-specification or out-of-specification based on the final
CSD properties. Eleven batches were identified as being in-specification whilst three
had MA values out with the specification range.

Data pre-processing

Prior to developing the statistical models the data was pre-processed. Data pre-
processing involves two stages: i) phase selection and ii) variable selection. It was
decided that the phases to be modelled were crystallisation and tightening. The initial
phase before seeding was excluded from the analysis was because no specific faults can
occur during this phase. The variables included in the analysis were vacuum pressure,
steam flowrate, steam pressure, steam temperature, brix of the solution, supersaturation
and temperature of the solution. Supersaturation is not a measured variable but can be
determined from the available measurements. It was included in the data set because it
is a critical parameter in sugar crystallisation.

On-line univariate SPC

The technique of on-line univariate SPC was applied to the three batches identified as out
of specification. Fig.1 shows the variables for one of the bad batches along with
univariate, $±3\sigma$, control limits. Note that the variables lie within the univariate control
limits for most of the batch run. Those observations that lie out with the limits, for
example time point 89 in Fig. 1a, are still close to the limits and thus it is concluded that
they are spurious signals, i.e. on average one in hundred observations will lie outside the
99% action limits. Thus by applying on-line univariate SPC only in one of the three bad
batches periods of out-of-specification operation were identified.

On-line multivariate SPC

The multivariate SPC techniques of MWPCS, BO and BDPCA were applied to the
process data. All three schemes are linear, can deal with unequal batch duration and to a
certain degree can describe the underlying process dynamics. Having built the three
models from the in-specification data, the next step was to test them on the out-of-
specification batches. The results are summarised in Fig. 2 to Fig.4. Due to lack of
space only bad Batch 1 is depicted on the plots. $T^2$ control charts are shown alongside their corresponding contribution plot. The contribution plot shows the contribution of the original seven process variables to the $T^2$ value for the maximum out of control value. For example Fig.2a shows how the $T^2$ statistic would evolve if MWPCA were applied on-line for out-of-specification batch 1. $T^2$ takes its maximum value at time point 89, thus Fig. 2b shows the $T^2$ contribution plot for this particular point. In all the control charts, the values shown were scaled so that the run-time control limits were equal to unity for the whole batch run. This way of presenting the control charts is more friendly for process operators. It also allows the three statistical methods to be compared on a common basis as the degree to which the $T^2$ statistic exceed the limit can be quantified.

In general, the three methods identify periods of out-of-specification operation during the run in all three batches for $T^2$ control charts. Thus by applying multivariate SPC as opposed to univariate SPC, periods of out-of-specification operation can be detected. In Fig. 2a, the MWPCA based $T^2$ control chart for batch 1, there is a period of out-of-specification operation around time point 89. The univariate control charts indicated out-of-specification operation for the time period 88-90. More specifically the univariate control charts of vacuum pressure (Fig. 1a) and solution temperature (Fig. 1d) move outside the limits for these time points. In the multivariate $T^2$ control chart, out-of-specification operation starts earlier and takes its maximum value at time point 89. The $T^2$ contribution plot for time point 89 (Fig.2b) indicates that variable 1 (vacuum pressure) is responsible for the out-of-control signal. This conclusion is in accordance with the univariate SPC control charts.

$T^2$ control charts for the other two methods for batch 1,BDPCA (Fig. 3a) and BO (Fig. 4a) provided maximum $T^2$ out-of-control signals for the time period around point 345. Univariate SPC provides an out-of-control signal for the brix of the solution for this time period Fig. 1b. However the fault is not detected as early as for the multivariate control charts and the level of excursion outside the limits is smaller for the univariate control charts. The contribution plots for BDPCA (Fig. 3b) and BO (Fig. 4b) indicate that the brix of the solution is mainly responsible for the out-of-control signal. Similar conclusions can be drawn for the monitoring of batch 3. The multivariate SPC techniques provide earlier warning of the process faults than univariate SPC with the $T^2$ statistic clearly exceeding the control limits.

For batch 2, both the univariate and multivariate SPC control charts detect that process operation was not similar to that represented in the reference data set of the eleven good batches.

The three multivariate monitoring techniques of MWPCS, BO and BDPCA exhibit similar performance and capability in terms of distinguishing between in and out-of-specification behaviour. However BDPCA captures the underlying dynamic relationship between the process variables more clearly than the other two approaches. For example according to the control charts in Fig.3a an abnormal situation arises around time point 270. Examining the time trajectories of the measured variables for batch 1, it is observed that a bit earlier, around time point 250, the steam pressure (Fig.1f) and flowrate (Fig.1e) that have to be kept around 1.9-2.2. bars and 7-9 ton/hr, respectively, for safety reasons, go outside of this limit and exhibit rather erratic behaviour. This causes a rapid decrease in the brix (Fig.1b) and subsequently supersaturation (Fig.1c)
goes out of the metastable zone (decreases), which results in the dissolution of the existing crystals. These variations are captured by the contribution plots (Fig. 3b) and the alarm signal generated by the monitoring scheme is justified.
5. Conclusions

In this paper three on-line multivariate process performance monitoring schemes for an industrial sugar crystallisation process were applied and compared with univariate SPC techniques. The process itself is challenging since it is carried out in multiple phases and there exists strong non-linear and dynamic effects between the variables. The methods proposed to develop the on-line monitoring scheme were Moving Window Principal Component Analysis (MWPCA), Batch Observation (BO) and Batch Dynamic Principal Component Analysis (BDPCA). The monitoring schemes were applied in an on-line mode for three batches whose final product was out-of-spec. It was found that all methods could identify clearly periods of bad operation for all three batches and perform better than the traditional univariate SPC. However a certain priority can be given to BDPCA model because it has quality variables closely correlated and easily interpreted by the process measurements.

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References