Multivariate statistical real-time monitoring of an industrial fed-batch process for the production of specialty chemicals

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A B S T R A C T

A large number of production processes for the manufacturing of specialty chemicals, pharmaceuticals, foodstuff, and materials for microelectronics are run in batch mode. Batch processes are “simple” in terms of equipment and operation design, but are often quite complicated in terms of product quality monitoring and of production scheduling and organization. In this paper an industrial case study is presented where the challenges related to the real-time estimation of the required time to manufacture a resin and to the instantaneous product quality estimation are addressed using multivariate statistical techniques. The industrial process is poorly automated, subject to several disturbances, and the batches have uneven lengths. It is shown that stage and batch lengths can be estimated in real time with an average error that is not larger than 20% of the inherent batch-to-batch variability, whereas quality estimations can be provided within the accuracy of the hardware instrumentation, but 240 times faster. The industrial benefits deriving from the use of the proposed monitoring system have been a drastic reduction of the number of samples that need to be analyzed by the lab, prompter adjustment of the processing recipe with consequent reduction of the total processing time, and improved capability to plan the production.

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1. Introduction

Specialty chemicals (e.g. coatings, detergents, resins, adhesives, pigments, additives) are typically obtained by batch or semi-batch processing, where a "recipe" is used to coordinate a sequence of elementary operations (e.g. charge, mix, heat up, react, separate, cool down, discharge), which may be repeated several times during a batch.

Much is reported in the literature on the multivariate statistical monitoring of batch processes in the sense of detection of anomalous or faulty conditions by the analysis of process variables trends (Nomikos and MacGregor, 1994; Kosanovich et al., 1996; Neogi and Schlags, 1998; García-Muñoz et al., 2003; Kourti, 2003; Gunther et al., 2007). Information of this kind is extremely valuable to improve process understanding; however, in most cases it can be used only after the completion of the batch, regardless of the fact it has been obtained in real-time or from a retrospective analysis of the batch itself. For example, if a change in the quality of feeds is diagnosed on-line, the supplier would be interviewed before the next batch is started; if an incipient fouling of a heat exchanger is detected, maintenance would be started at the end of the batch; if the end-point quality of the product is anticipated to be out of specification, the product would not be delivered to the customer, and the reasons for this faulty batch would possibly be analyzed by interrogating the monitoring model. Much less attention has been directed towards the development of monitoring strategies able to provide some kind of information that can be used directly within the same batch from which this information is obtained. In this context, two typical challenges that need to be addressed by a monitoring system in the production of specialty chemicals are the real...
time estimation of the length of the batch (or the length of any production stage within the batch), and the real time estimation of the instantaneous quality of the product.

There are several specialty productions for which the total batch length is not known a priori, nor is it the length of any processing stage within the batch. Knowing in advance the processing time is useful for several reasons. In fed-batch processes, for example, new fresh material is charged into the process vessels at a convenient time instant. The ability to estimate in real time this instant (which may change from batch to batch) can result in savings both in the number of quality measurements to be processed by the laboratory and in the required total processing time (Marjanovic et al., 2006). On a different perspective, real-time estimation of the total length of the batch can be very useful for production planning, scheduling of equipment use, as well as to coordinate the operating labor resources.

Real time knowledge of the instantaneous quality of the product is extremely important in the manufacturing of several specialties. In fact, if the instantaneous product quality is not found to track a specified trajectory, the processing recipe must be adjusted in real time (possibly several times during a batch), and the batch is kept running until the end-point quality meets the specification. However, in most cases the product quality is not measured in real time, and to contain the laboratory-related expenses (in terms of: need of dedicated personnel, consumption of chemicals, and use analysis equipment) only few product samples are taken during the course of a batch and sent to the lab for analysis. Even so, in a typical industrial scenario where several productions are run in parallel, as many as 15,000 samples may need to be taken and analyzed each year, which can add up to an important fraction of the total product cost. From an operation perspective, due to the scarce number of measurements available during a batch and to the time delay inherent to the analysis, significant drifts on the quality profiles may be experienced before any intervention can be done on the batch being run. The net result is that the recipe adjustments are delayed, the total length of the batch is increased, and the economic performance of the process is further penalized.

In this paper, an industrial case study is presented where the challenges related to the real time estimation of the required processing time and to the instantaneous product quality estimation are addressed using multivariate statistical techniques based on the projection onto latent structures (Wold et al., 2001). Reference is made to a fed-batch process where a resin is produced. It is shown that, by appropriately using existing techniques, information can be extracted from available process measurements that can significantly improve the overall performance of the process. Although the results presented are tailored to a specific case study, we nevertheless believe that the approach we have taken is quite general, and can be useful both to practitioners and to academics for successful design and implementation of data-based monitoring techniques.

2. The process and related challenges

A high molecular weight polyester resin is produced by catalytic poly-condensation of carboxylic acids and alcohols in an industrial facility where several different specialty chemicals are manufactured. The product quality is determined by the combined values of two indicators, namely the resin acidity number ($N_A$) and the resin viscosity ($\nu$). A schematic of the production process is shown in Fig. 1.

Two distinct production stages are present. Raw materials, catalyst and additives are initially charged to the reactor. When the charge is completed, production Stage 1 is started. The objective of this stage is to react partially the fresh materials until a pre-polymer with loosely specified characteristics is obtained. As soon as Stage 1 is completed, new ingredients are loaded into the reactor, and the pre-polymer is further processed until the quality indicators reach a target set of values. Then, production Stage 2 begins, a catalyst is charged, and the material is processed until it reaches the quality specifications. At that point Stage 2 terminates, the final product is discharged, and the processing equipment is ready for a new batch (cleaning of the equipment may be necessary).

The most important pieces of equipment within which the process is carried out are a stirred tank reactor provided with external and internal coils for heating/cooling the reaction mass, and a tray distillation column that separates the reaction products and partially recycles them to the reactor. Ancillary equipment is also present, including a vacuum pump, a scrubber and a conditioning system for the heating oil.

Several process variables are measured online every 30 s all over the plant. These variables include temperatures on different locations of the reactor, reactor pressure, temperatures on different trays of the distillation column, inlet/outlet temperatures of the heating/cooling media, agitator speed, and some additional process measurements that (due to the proprietary nature of the data) we are not listing here. The time profiles of three process variables are reported for a typical batch in Fig. 2, where the lengths of Stage 1 and Stage 2 for the batch are also indicated. Note that the process is poorly automated: for
example, the reactor temperature is controlled in automatic by manipulating the flows of the heating/cooling media, but the temperature setpoint profile is assigned manually by the operators on a case-by-case basis. Furthermore, each batch is subject to several disturbances. For these reasons, the total duration of a batch is very variable (from 50 to 80 h) and seemingly unpredictable, as are the lengths of Stage 1 and Stage 2. Therefore, it is hard for the management to appropriately schedule the use of equipment when several batches are to be processed in series or in parallel.

Real-time measurement of product quality is not available. Quality measurements are obtained offline and suffer from two drawbacks. First, they are expensive. In fact, when quality assessment is needed, a sample is taken (manually) and sent to the laboratory for analysis. Therefore, specific personnel must be dedicated to sample collection and sample analysis. Secondly, quality measurements are delayed: the analysis results are available at best 0.5 h after a sample has been taken.

Typically, a company would try to save on the personnel-related expenses by reducing the number of samples to be analyzed. For example, during Stage 1 the main concern is knowing the time at which new fresh materials should be loaded into the reactor (end of Stage 1). Only very few samples (on the order of 3–5) are normally taken during this stage, starting at the time when “presumably” the pre-polymer is close to specification. Therefore, detection of the end of Stage 1 may be significantly delayed.

Also during Stage 2 a very limited number of samples is taken (about one sample every 2 h of operation) to contain measurement-related costs. A typical empirically derived chart for Stage 2 monitoring looks like the one shown in Fig. 3.

If the sample quality is found to lie outside the broken bounds, the operators must adjust the production recipe according to a given procedure. Note that only 6 samples were taken to monitor Stage 2 in the case of Fig. 3, despite the fact that this stage lasted as long as 32.5 h. More timely information on product quality evolution would be highly desirable, because the production recipe could be adjusted more promptly. Delays on recipe adjustments may result in significant increase of the processing time and in potential loss of the end-point quality.

Summing up, to improve the process operation and to reduce the measurement-related costs, a real-time monitoring system is sought that allows: (i) to estimate the duration of Stage 1, in such a way as to reduce the number of quality measurements that are required to assess online the termination of the stage; (ii) to estimate the instantaneous values of the quality indicators during Stage 2, in such a way as to promptly counteract any deviation from the desired quality profile by adjusting in real time the processing recipe; (iii) to estimate the total duration of the batch, in such a way as to allow to schedule the use of the different pieces of equipment for different productions in the same facility.

3. **Projection to latent structures**

In order to design the monitoring models, a multivariate statistical technique is used, namely projection to latent structures (PLS), also known as partial least squares regression (Geladi and Kowalski, 1986; Kourti and MacGregor, 1995; Wise and Gallagher, 1996). PLS is a regression technique that allows one to deal with correlated process variable measurements.
Data signals. Usually, a small number of LVs is sufficient to explain the variance of the process data ($X$) that is most predictive for the quality data ($Y$). The $X$ and $Y$ matrices are decomposed as

$$X = TP^T + E = \sum_{a=1}^{A} t_a p_a^T + E \quad (1)$$

and

$$Y = UQ^T + F = \sum_{a=1}^{A} u_a q_a^T + F \quad (2)$$

A linear inner relationship is enforced between the vectors $t_a$ and $u_a$:

$$u_a = b_a t_a \quad (3)$$

where $A \leq J$ is the number of selected LVs, $t_a$ and $u_a$ are the score vectors, $p_a$ and $q_a$ are the loading vectors, and $E$ and $F$ are the residual matrices, which are minimized in a least-squares sense. The score vectors $t_a$ and $u_a$ contain information on how the samples relate to each other. The loading matrix $P$ and $Q$ are composed by orthonormal vectors that include information about the relation between the process variables. The residual matrices include the non-systematic part of the data signals. Usually, a small number of LVs is sufficient to extract the information contained in $X$ and $Y$, no matter how large the dimension of these matrices is. The cross-validation technique (Wold, 1978) can be used to determine the optimal number of LVs, although it may be unreliable when non-linearity and autocorrelation are present (Ku et al., 1995); furthermore, it considers only the reference set and not the validation one.

When batch processes are considered, the estimation problem is complicated by the fact that a third dimension (i.e. time) must be considered. Therefore, the data matrices take the form of three-way arrays. To process a three-way matrix through a standard PLS technique, matrix “unfolding” is needed, i.e. the three-dimensional dataset must be rearranged in a bi-dimensional matrix. Basically, two methods can achieve the unfolding of a three-way matrix (Fig. 4): batch-wise unfolding (Nomikos and MacGregor, 1994) and variable-wise unfolding (Wold et al., 1998). These methods consider different categories of variability (Kourti, 2003; Camacho et al., 2008) and determine the auto-scaling direction, giving dissimilar interpretation to the LVs. To be implemented, the batch-wise unfolding method requires that all batches have the same time length, where the time variable may be either the “actual” time or a fictitious time-synchronizing variable. Some methods for batch alignment/synchronization are discussed by Kourti (2003) and by Üney et al. (2003).

The total number of batches available for this study was 36. Of these batches, 27 were designated as the calibration dataset; the remaining 9 batches constituted the validation dataset.

### 4. Stage 1 monitoring: estimation of the stage length

The number of quality measurements within this stage is too small to allow designing a PLS model for the real-time estimation of the product quality. Therefore, to monitor the evolution of the batch, the stage length $r$ was estimated instead. The possibility of knowing the value of $r$ in advance would allow the operators to further reduce the number of samples that need taking for analysis, because samples would only be taken starting from the time when the stage

![Fig. 3 – A typical monitoring chart used industrially for the resin production during Stage 2. Acidity number is reported as the abscissa (decreasing values from left to right), and viscosity as the ordinate. Non-standard units are used for both quality indicators. Circles indicate a sample for which quality measurements are available from the lab. The measured values of acidity number and viscosity should always fall within the bounds (broken lines). Real-time recipe adjustments are needed when a sample falls outside the bounds. Time increases (non-linearly) from the lower-left corner to the upper-right one.](image)
is expected to terminate. Thus, if the performance of the estimator is adequate, only one or two samples may be sufficient to detect the stage termination time.

The length of Stage 1 in the available dataset ranged between 14.2 and 26.1 h, i.e. the length variability (~12h) is about 1.5 times the length of an operator’s shift window. By checking the process variable profiles, it was noted that during this stage the profile of each process variable displays similar trends in all batches, and only the stage length seems to discriminate one batch from another one (a PCA analysis could also be used to ascertain this similarity). This is not surprising, because most of the time is spent in Stage 1 to heat up the ingredients inside the reactor, and the heating procedure is similar for all batches (although subject to the operators’ manual intervention, because the setpoint of the temperature ramp is adjusted manually). Therefore, because a certain degree of similarity was apparent among the batches, a multi-way PLS model (Nomikos and MacGregor, 1994) using batch-wise unfolding (Fig. 4) was developed to provide real-time estimation of the stage length.

Alignment techniques based on the indicator variable approach (Kourtì, 2003; Ündey et al., 2003) proved unsuccessful for synchronizing the process variables trajectories. We conjecture that this is due to the fact that the process variable trajectories are correlated to time in a highly non-linear way. Therefore, a simpler approach was taken: the values of process measurements that had been collected at a time exceeding a threshold value \( t^* \) were simply disregarded. The threshold length was set to be as the shortest Stage 1 length among all the available batches. Namely, \( t^* = 14.2 \) h (i.e. \( t^* = 1700 \) time instants) was set. As for the number of process measurements to be included into the \( X \) matrix, an engineering analysis suggested to discard a small subset of the available measurements, i.e. those measurements that either were expected to provide no contribution to the batch dynamics or had a markedly non-smooth profile throughout

Fig. 4 – Three-way process and quality data matrices and their unfolding according to the variable-wise procedure and the batch-wise procedure.
the batch. Following these indications, constant setpoints and on-off variables were eliminated from the dataset. This resulted in $X$ being unfolded to a $(27 \times J_k)$ two-way matrix $X$, where $J=19$ is the number of process measurements that were eventually retained, and $k$ is the number of time instants used to estimate $r$. The response matrix $Y$ reduced to a $(27 \times 1)$ column vector. Before further processing, the $X$ and $Y$ matrices were auto-scaled.

The real-time estimation of $r$ was accomplished by designing a set of time evolving PLS models to be used within each batch (Ramaker et al., 2005). Each model refers to the time instant $k \leq r$ at which a process measurement becomes available, and uses the process variable values from time instant 1 up to time instant $k$ to estimate $r$. Therefore, the dimension of $X$ increases as time progresses. However, note that this has a negligible effect on the calculation time.

The time-averaged absolute error $\text{TAAE}_i$ of Stage 1 length estimation in batch $i$ over the whole estimation window $t$ is defined as

$$\text{TAAE}_i = \frac{1}{T^*} \int_0^{T^*} |\epsilon_i(t)| dt.$$  

where

$$\epsilon_i(t) = \hat{r}_i(t) - r_i$$

is the instantaneous error of estimation of Stage 1 length in batch $i$, $\hat{r}_i(t)$ is the value of Stage 1 length in the same batch as estimated at time $t$ (i.e. at time instant $k$), and $r_i$ is the actual length of Stage 1 in the same batch. Eq. (4) was evaluated with a finite difference approximation. Averaging the value of $\text{TAAE}_i$ over the $I=9$ validation batches provides the value of the overall average absolute error $\bar{\text{TAAE}}$ for the whole validation dataset.

The number of latent variables to be retained in the model was determined by minimizing the value of $\bar{\text{TAAE}}$. As a result, one latent variable was retained. This single latent variable was not able to capture much of the variability in the $X$ space of the calibration dataset. In fact, for any batch of the calibration dataset only 23–26% of the $X$ variance was explained by the latent variable, which indicates that only a small fraction of the information embedded in the measured process variable profiles is actually correlated with the length of the stage. Correspondingly, about 50–70% of the variance in $Y$ was explained. Nevertheless, the length prediction was quite satisfactory, because the value of $\bar{\text{TAAE}}$ was calculated as 196 time instants ($\sim 1.6$ h), i.e. $\sim 8\%$ of the average length of Stage 1, and $\sim 14\%$ of the variability in the length.

Fig. 5 shows that, with reference to the nine validation batches, the batch-averaged instantaneous absolute error of estimation is moderate ($\sim 2.1$ h) at the very beginning of the operation; it soon decreases down to $\sim 1.8$ h, then, starting from $k \geq 600$ time instants, it further decreases steadily and reaches a minimum of $\sim 1.3$ h at the end of the estimation window. This is an indication that the estimation is progressively collected during the evolution of the batch that proves useful for the estimation of $r$. This issue is further clarified in Fig. 6, which refers to a single validation batch. When incremental information is used to build matrix $X$ (i.e. when the column dimension of $X$ grows with time; evolving model), the estimation of $r$ is smooth and steadily improves after 600 time instants. However, if only instantaneous information is used to build $X$ (i.e. at time $k$, $X$ is only made with measurements taken at $k$; local model), the estimation of $r$ is much more erratic; it would be hard to have the process operators trust such an estimation (note that the actual length, which is also indicated in Fig. 6, is obviously not known when the batch is being run). The information of Fig. 6 can be complemented online with the plots of the Hotelling $T^2$ and squared prediction error statistics, which would provide an indication on whether the estimation is reliable or not.

To appreciate how variable the validation results are, Table 1 provides the time-averaged results for each of the validation batches. Note that the overall $\bar{\text{TAAE}}$ of the validation dataset can be calculated as the time-average of the curve shown in Fig. 5, or as the batch-average of the data reported in Table 1.

For a practical perspective, the results shown in Fig. 6 would be implemented in a slightly different way: the projected time of the day at which Stage 1 is expected to terminate would be shown onto the operators’ display at selected time instants. About 1.6 h before the stage is expected to terminate, the operators can take one product sample and send it to the lab for...
Table 1 – Time-averaged absolute estimation error (TAAE) of Stage 1 length for each of the validation batches

<table>
<thead>
<tr>
<th>Batch</th>
<th>TAAE&lt;sub&gt;i&lt;/sub&gt;</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>2.2</td>
</tr>
<tr>
<td>2</td>
<td>1.1</td>
</tr>
<tr>
<td>3</td>
<td>0.3</td>
</tr>
<tr>
<td>4</td>
<td>0.8</td>
</tr>
<tr>
<td>5</td>
<td>0.6</td>
</tr>
<tr>
<td>6</td>
<td>0.6</td>
</tr>
<tr>
<td>7</td>
<td>4.8</td>
</tr>
<tr>
<td>8</td>
<td>3.2</td>
</tr>
<tr>
<td>9</td>
<td>1.1</td>
</tr>
</tbody>
</table>

Therefore, the number of product samples that need to be analyzed can be minimized, which reduces the operator-related costs. Furthermore, it is possible to know in advance whether the sample should be taken during the current shift or during the next one, which has a favorable impact on the workload organization.

It is interesting to note that only a subset of the variables included in the X matrix provides a significant contribution to the estimation of \( r \). To appreciate this, the index of variable importance in the projection method (VIP; Chong and Jun, 2005) can be calculated for each process variable at each time instant. The results of this “dynamic” VIP analysis are reported in Fig. 7. Process variables with VIP > 1 are considered “important” for the estimation of \( r \). Five process variables show a value of VIP consistently larger than 1 throughout the whole estimation window. These variables are the reactor temperature (variable #16), the outlet temperature of the heating oil (variable #5), the setpoint for the inlet temperature of the heating oil (#15), the inlet temperature of the heating oil (#14), and the setpoint for the reactor temperature (#17). This suggests that the most important variables for the estimation of \( r \) are those associated to the thermal behavior of the reactor, which is consistent with what one would expect from engineering judgment. Furthermore, starting from \( k \cong 400 \) time instants (i.e. 3.3 h), the VIP index keeps increasing for all of these variables, indicating that “thermal behavior” and stage length get more and more correlated after that time. Then, after \( k \cong 1500 \) time instants (~12.5 h), VIP decreases for these variables, meaning that the correlation between thermal behavior and stage length starts vanishing after that time. This indicates that the “temperature footprint” of the process is almost completely traced about 12 h after the process has been started, which is consistent with the fact that the profiles of most process variables start flattening after ~12 h from the beginning of the batch.

5. Stage 2 monitoring

During Stage 2 the pre-polymer is transformed into the end product. Interventions on the recipe are carried out by the operators during this stage in response to the quality measurements coming from the lab.

To monitor this production stage two tools were designed: a soft sensor estimating in real time the product quality from process measurements, and a soft sensor estimating in real time the total length of the stage.

5.1. Estimation of the quality indicators

Two PLS regression models were designed, one for the estimation of the acidity number and one for the estimation of the viscosity of the reacting mass. The same process variables used for the estimation of Stage 1 length were retained. Also in this case batch alignment proved unsuccessful. Therefore, being the process variable trajectories quite dissimilar in this stage, the three-way process data matrices were variable-wise unfolded (Fig. 4). However, this approach preserves the non-linearity between the predictors space and the responses one (Kourti, 2003), and should be preferred when the correlation structure of a process is roughly constant (Camacho et al., 2008). To compensate for these drawbacks, the same approach used by Facco et al. (in press) was used in this study, i.e. the production stage was split into different estimation phases, and distinct PLS submodels were designed for each estimation phase. To determine the number of estimation phases, a simple approach proved satisfactory: plotting \( X \)-scores vs. \( Y \)-scores for the whole calibration dataset (Fig. 8) clearly showed that two clusters are present in the score plane, each cluster representing an estimation phase. Therefore, two submodels were built to estimate \( N_A \) (or \( \nu \)) within Stage 2. Note that cluster analysis (Lu and Gao, 2005; Beaver et al., 2007) could have been used for an automatic detection of the clusters in the scores space. However, it should also be noted that the number of clusters must be kept as small as possible because if too few quality measurements are available within a cluster, it may be impossible to design the relevant PLS submodel.
Fig. 8 – Stage 2: scores plane for the first latent variable and for the calibration dataset (reference is made to the time instants when viscosity measurements are available). The squares indicate how a single batch within the dataset projects onto this plane as time progresses from the beginning (1) to the end (15) of the stage. Dashed lines indicate the approximate locations of the clusters.

We observed that “time” is really not a good indicator to assess phase switching in this process. Run-to-run variability is extremely large (for example, Stage 2 length ranges from 27.5 to 48.9 h), and the switching time shows a large variability too. Therefore, submodel switching was linked not to time, but to events: there are certain processing events that occur in all batches and change the correlation structure between the variables, although they occur at a very different time from one batch to another one (a similar approach has been used recently by Doan and Srinivasan, 2008). The occurrence of these events (which can be easily detected on line) dictates phase switching. We believe that this approach is more general than what could be obtained if time was used to designate submodel switching. We found that the switching event was the same both for the $N_A$-model and for the $\mu$-model, and is related to a change of pressure in the reactor that is part of the production recipe during Stage 2.

To attenuate the effect measurement and process noise, and to provide the PLS model (which is inherently static) with “memory”, a moving-window approach was used (Facco et al., in press). The process measurements included into the $X$ matrix were averaged over a moving time window of 900 past time instants (7.5 h), this width having been determined in such a way as to minimize the mean-squared error of prediction on the validation dataset. Not only did this provide a significant smoothing of the estimated quality profiles, but it also increased the amount of predictive information included in the $X$ matrix, which made the quality estimation more accurate.

As observed also by Ku et al. (1995) in a different context, cross-validation proved ineffective for the determination of the number of latent variables (LVs) to be retained in the submodels. Therefore, this number was determined by minimizing the estimation error in the validation dataset. As far as the estimation of $N_A$ is concerned, 6 and 3 LVs were used for Phase-1 submodel and Phase-2 submodel, respectively; for the estimation of $\mu$, 2 LVs were used during Phase 1, and 3 during Phase 2.

Typical estimation results in a validation batch are shown in Fig. 9, where quality estimations (solid line) are compared to lab measurements (dots) in an “industrial” monitoring chart. It can be seen that the estimations compare well to the actual measurements, and can indeed be used as surrogate measurements to guide the operators throughout the application of the processing recipe. It should be remarked that lab measurements are spaced (roughly) by 2 h, whereas quality estimations are made available at the same frequency as process measurements (two per minute). Therefore, the recipe adjustments can be carried out much more promptly if the soft sensor is employed in real time, the chances that product quality drifts outside the acceptable bounds are minimized, and the length of the batch can be shortened. The actual implementation of this soft sensor also allows to significantly reduce the number of samples to be taken and analyzed during a batch. In fact, product samples can be taken only when the product is deemed to be close to specification (upper-right corner of Fig. 9), which contributes to cut the lab-related

Fig. 9 – Stage 2: comparison between lab measurements (circles with measurement uncertainty) and real-time estimation (solid line) of the resin quality in an industrial monitoring chart (validation batch). Acidity number is reported as the abscissa (decreasing values from left to right), and viscosity as the ordinate. Non-standard units are used. The measured values of acidity number and viscosity should always fall within the bounds (broken lines). Time increases (non-linearly) from the lower-left corner to the upper-right one.
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Fig. 10 – Stage 2: (a) batch-averaged instantaneous absolute error of $\tau$ estimation in the validation dataset as a function of time within the length of the estimation window (evolving model) and (b) profile of the VIP index over the estimation window length (3000 time instants) for all process measurements (the numbers above the curves indicate the process variable number designation).

expenses, and allows to redirect the operators to more qualifying duties.

As for the real time implementation of the soft sensor, online detection of the switching instant from available process measurements was a key issue to guarantee a good performance of the sensor. Standard digital filters were used to protect the soft sensor from measurements noise and spurious events that might disrupt its performance by erroneously triggering a phase switch.

5.2. Estimation of the stage length

Production planning is difficult for this process, because the length of a batch is not known a priori, and changes a lot from batch to batch (e.g. the range of variability of Stage 2 length is as large as three operator’s shift windows). Estimating the stage length in advance is very important to schedule the use of the equipment in the subsequent batches, and to plan the operating labor requirements. For these reasons, a soft sensor was designed to estimate the stage length in real time. The approach was the same used for the real time estimation of Stage 1 length (evolving PLS model), and $r^* = 3000$ time instants was set (all symbols refer now to Stage 2). The results obtained were satisfactory, as shown in Fig. 10a.

On the average, the estimation error on the validation dataset is larger than in Stage 1 ($\text{AAE} = 490$ time instants, i.e. $\sim 4.1$ h). However, this is only $\sim 11\%$ of the average length of Stage 2, and $\sim 20\%$ of the variability in the length, which is well below the length of one operator’s shift. Note that it takes only $\sim 250$ time instants ($\sim 2$ h) to have a satisfactory estimation of the overall length of the stage; after $\sim 1500$ time instants from the beginning of the stage, the average absolute estimation error further decreases by about 1 h. Table 2 provides time-averaged results for each of the validation batches. Fig. 10b shows that no process variable is really “dominant” during Stage 2 as far as the stage length estimation is concerned. Almost all variables provide some kind of contribution to the estimation of $\tau$, and the variables related to the “temperature footprint” of the reactor (e.g. variables #5, 14, 15, 16, and 17) are among the least important.

6. Conclusions

In this paper it was shown through an industrial case study how, by considering a blend of engineering judgment and mathematical modeling, multivariate statistical techniques can be exploited to assist the real-time monitoring of product quality and to deliver helpful information for an effective production planning in the semi-batch processing of specialty chemicals.

An evolving PLS modeling approach was exploited for the estimation of the duration of the batch. Namely, it was shown that, by incrementally using the information gathered during the evolution of the batch, a sound estimation of the length of the batch (or of any processing stage within the batch) can be obtained in real time. Such piece of information is particularly useful in batch processing, as it allows to schedule manual interventions, to optimize the manpower in terms of shifts and roles, to forecast the production time, and to schedule the most convenient utilization of plant equipment. The statistical analysis of the most significant process variables that contribute to determine the length of the batch confirmed that the initial heat-up stage is crucial for the development of the entire batch.

Furthermore, it was shown that the product quality can be estimated in real time from the available process measurements with an accuracy similar to that of the lab instrumentation, but at a much higher frequency, with no delay, and with no need for dedicated personnel. In particular, the definition of different estimation phases, for which different PLS models were tuned up, allowed to counteract the process non-linearity and the changing correlation.

Table 2 – Time-averaged absolute estimation error (TAAEi) of Stage 2 length for each of the validation batches

<table>
<thead>
<tr>
<th>Batch</th>
<th>TAAEi</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.1</td>
</tr>
<tr>
<td>2</td>
<td>6.9</td>
</tr>
<tr>
<td>3</td>
<td>2.3</td>
</tr>
<tr>
<td>4</td>
<td>4.8</td>
</tr>
<tr>
<td>5</td>
<td>9.6</td>
</tr>
<tr>
<td>6</td>
<td>3.3</td>
</tr>
<tr>
<td>7</td>
<td>2.3</td>
</tr>
<tr>
<td>8</td>
<td>3.3</td>
</tr>
<tr>
<td>9</td>
<td>2.2</td>
</tr>
</tbody>
</table>
structure in a simple and effective way. Furthermore, incorporating the soft sensor with a “memory” through a moving window approach was highly beneficial to reduce the process and measurement noise, and to increase the estimation accuracy, without introducing any significant complication in the structure of the soft sensor. Real-time knowledge of product quality can significantly improve the operation of a batch and cut the expenses related to sample handling and analysis.

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References