Integration of Statistical and Engineering Process Control in a Batch Processes Monitoring: Case of Alkyd Polymerization Reactor

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Abstract

The objective of this paper is to develop a framework that integrates two important concepts: Statistical process control (SPC) and engineering process control (EPC). Most of the literature researches on integrated SPC/EPC systems are focused into continuous process mainly with Algorithmic SPC. The integrated SPC/EPC systems in batch process control have not received the same degree of attention. In particular, there is an only Run-to-Run (RTR) control methodology application, which is mostly focused in semiconductor industry. This paper is a first of its kind in integrated SPC/EPC systems that applied in batch process and it based on a data-driven quality improvement tools. The proposed SPC/EPC integration is performed continually in two successive phases: (1) Active SPC for the batch making advance, and (2) RTR control action between batches. Control limits for critical variables are developed using information from the historical reference distribution of past successful batches. EPC application is based on the development of progressive knowledge-based rules. For a validation purpose, the proposed approach is applied to data collected from an industrial batch alkyd polymerization reactor, which evolution is monitored by measuring the overflow water weight, the acidity index and the viscosity of samples withdrawn from the reactor. This industrial process is poorly automated, subject to several disturbances, and the batches have uneven lengths. The synthesis is stopped at the maximum yield allowed by the gelation point of the cold product. Through this case study application, process engineers at the company are now able to use a valuable decision making tool when the production process is affected by certain disruptions, with obvious consequences on product quality, productivity and competitiveness.

Keywords: *SPC/EPC, Active SPC, Run-to-Run, Batch process control, Alkyd polymerization reactor*

1. Introduction

Improving product quality is the most preoccupation of industrial managers and engineers. This objective becomes more critical in various industries. Essentially because, the lack and the increasing prices of raw materials and energy require today a decrease of production costs for large technical processes. In fact, a continuing processes control in manufacturing systems becomes more and more necessary. Particularly, innovative monitoring and control techniques of the batch process operations are strongly needed in the process control field. Since, batch and semi-batch processes play a significant role in the production and processing

of high-value-added materials and products. Examples include the production of polymers, pharmaceutical and food manufactures, biochemical reactors, batch distillations (for the separation and transformation of materials), semi-conductor industries, the processing of materials by injection molding and etching processes.

Typically, the manufacturing of a batch involves charging ingredients to the vessel, processing them under controlled conditions, and discharging the final product. A batch operation is considered successful if the values of the process variables remain within acceptable limits while following the recipe prescribed for the process, resulting in a uniform, high-quality product. 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.

There are numerous monitoring and control studies in batch-polymerization reactor. It is better to divide them into two research areas: (1) mathematical kinetic models of polymerization systems for the estimation and control schemes implementation such as in [1, 3], and (2) process control of polymerization: The knowledge of processes and the information obtained from them, allow studying the design problems of trajectories, as well as the control scheme design at different levels such as: feedback control, and feedforward-feedback control.

According to [4], it can be see that control processes of polymerization industry is elderly difficult. In fact, high-quality in the batch process usually means that the same thing is consistently produced in each batch run. However, batch processes generally exhibit some batch-to-batch variation arising from such things as deviations of the process variables from their specified trajectories, errors in the charging of the recipe of materials, and disturbances arising from variations in impurities. Abnormal conditions that develop during these batch operations can lead to the production of at least one batch or a whole sequence of batches with poor-quality product if the problem is not detected and remedied. Most industrial batch processes are run without any effective form of real-time, on-line monitoring to ensure that the batch is progressing in a manner that will lead to a high-quality product or to detect and indicate faults that can be corrected prior to completion of the batch or can be corrected in subsequent batches.

Moreover, it is often the case that batch plants are poorly automated, and may require intervention by the operating personnel to provide online adjustments of the operating recipe in order to avoid the production of off-spec products. In fact, with respect to product quality control, most batch processes are run in an open-loop fashion, because information about product quality is not available online, but is obtained offline from laboratory assays of few product samples. Because of the lack of real time information on the product quality, it may be difficult to promptly detect quality shifts and to counteract them by adjusting the operating recipe accordingly. Therefore, a quality control strategy for a batch process often reduces to the online control of the trajectories of some key process variables (which can be measured online), and possibly to some midcourse intervention on the operating recipe to compensate for the shifts detected in the product quality measured offline.

To ensure high-quality product, batch process modeling and monitoring has attracted many research interests. For many years, chemical and process industries have successfully employed statistical process control (SPC) as a tool for monitoring and maintaining the consistency and operation of process systems. Traditional SPC approaches involve plotting trends of important quality parameters and ensuring that these trends do not violate pre-specified control limits. Despite the success of SPC tool, several limitations affect its ability to provide accurate monitoring of batch processes. Recent application studies have indicated that multivariate statistical technology can provide some support in maintaining consistent

operation in complex batch processes [5]. Multivariate statistical process control (MSPC), which only require process history data, have been widely applied in literature such as in [6] and [7]. Among them, multi-way principal component analysis, multi-way partial least squares, and model predictive controllers is the most well-known ones as used in [8, 9]. However, these methods and techniques are either time-consuming or hard to implement in practice. Recent years have witnessed the appearance of several research studies in this field. However, there have been scarce publications using real production data and demonstrating the practical application of the integration. This article tries to fill this gap, as it gives an example of integrated EPC/SPC applied to a real and complex batch-polymerization reactor. In fact, automation of the implementation of the previous powerful statistical tools for process supervision and monitoring provide a valuable decision making asset for process engineers and plant personnel.

This paper fall to three research area: integration of SPC and EPC, batch processes monitoring and control, and alkyd polymerization reactor. The objectives of this study are (a) to detail a literature review of SPC/EPC integration, (b) to establish an integrated SPC/EPC methodology for a batch process, and (c) to illustrate the proposed approach with an application of the analysis and monitoring of an industrial batch alkyd polymerization reactor. To present how we are achieved our goals; the remainder of the article is organized as follows. Section 2 illustrates the theoretical background and the related literature review for integrated SPC/EPC systems. Following this, Section 3 describes the batch-polymerization monitoring and control. Section 4 presents the proposed approach. Section 5 details the case study data and results. Finally, Section 6 presents the concluding remarks and perspective of the study.

2. Integrated SPC/EPC Systems

SPC has been traditionally achieved by successive plotting and comparing a statistical measure of the variable with some user defined control limits. If the plotted statistic exceeds these limits, the process is considered to be out of statistical control. Corrective action is then applied in the form of identification, elimination or compensation for the assignable causes of variation. The most common charts used are the Shewhart, Exponential Moving Average (EWMA), range and Cumulative Sum (CuSum) charts. Theses traditional control charting discipline of monitor then adjust when out of control is not the best strategy for process improvement in processes with some kind of dynamics. This is especially true in processes having a tendency to drift or wander away from the target. In these contexts, another approach called stochastic control or engineering process control (EPC), also known as automatic process control (APC), is needed. This approach is based on process compensation and regulation (feedback and/or feed-forward control), in which some easily manipulable process variables are adjusted following some control rules with the goal of keeping the key characteristic (controlled variable) close to the desired target. This requires good knowledge of the relationship between the controlled variable and the manipulated variables, as well as an understanding of process dynamics. If the cost of making adjustments (control actions) in the manipulable process variables is negligible, then the variability of the controlled variable is minimized by taking control actions in every sample. This is in sharp contrast to SPC.

SPC and EPC originated in different industries, the parts industry and the process industry, and have been developed independently with some controversy in the past because of the different meaning of ‘control’ in both approaches: process monitoring (SPC) versus process regulation (EPC). It is true that a dynamic process should not be adjusted using SPC, but it is also true that assignable causes cannot be detected with EPC [10]. In fact, regulation schemes react to process upsets; they do not make any effort to remove the root causes [11]. Therefore,

EPC and SPC should be considered as two complementary (not alternative) strategies for quality improvement. SPC monitoring procedures seek to reduce output variability by detecting and eliminating assignable causes of variation. On the contrary, EPC tries to minimize output variability by making regular adjustments exploiting the process dynamics (common-cause system). Hence, ideas from both fields can be used together in an integrated EPC/SPC system called to secure both optimization and improvement. Based on our literature review, there are three type of integration: algorithmic SPC, Active SPC, and Run-to-Run.

2.1. Algorithmic SPC

Conventional SPC is basically an off-line technique. Whilst there are many reports of successful cases in the parts manufacturing sector, this passive control strategy does not suit continuous systems. Here, in addition to keeping products within specifications, there is a requirement to keep the process operating. Depending on the complexity of the process, the time taken to identify, eliminate and compensate for assignable causes of variation may not be acceptable. Nevertheless, the aim of both EPC and SPC is to increase plant profitability. Thus, it is reasonable to expect that the merger of these two apparently dichotomous methodologies could yield strategies that inherit the benefits associated with the parent approaches.

The first attempts to integrate EPC and SPC appeared long ago, with the work of Barnard in 1959 [12]. Using the machine-tool case study, the author demonstrated that automatic control and statistical control can be used in parallel. Explicitly, MacGregor [13] was the first approach in EP/SPC integration. He suggested the use of control charts for monitoring the behavior of a process under EPC. Box and Kramer [14] mention that the origin of statistical process monitoring was in the parts industry, whereas EPC had its origins in the process industry. The concept of integrating EPC and SPC techniques uses EPC to reduce the effect of predictable quality variations, and uses SPC to monitor the process for detection of assignable causes. References [13, 14, 15, 16, 17] have presented an overview descriptions of this integration concept. Such a strategy is sometimes called 'Algorithmic SPC' (ASPC), referring to the integrated use of algorithmic model based controllers and SPC techniques. ASPC is a proactive approach to quality improvement that reduces predictable variations in quality characteristics using feedback and feed-forward techniques and then monitors the entire system (by plotting deviations from the target, prediction errors, adjustments, etc.) to detect and help remove unpredictable process upsets. It is considered as a marriage of control theory and SPC that aims to reduce both short-term and long-term variability by replacing the traditional control charting discipline of ‘monitor, then adjust when out of control’ with ‘adjust optimally and monitor’.

Inspired by these previous works, several other authors became notorious in the field, leading to different approaches that reveal two great concerns associated with this type of integration: (1) identification of the variables that must be monitored: if only output variables (quality characteristics), input variables (adjustable variables) or both of them, and (2) decision on whether to use automatic or manual controllers, the latter being or not constrained by statistical control; such decision would depend on adjustment costs and type of adjustment. Successful applications of ASPC and thorough research of this methodology can be found in the literature such as [10, 18, 19, 20, 21, 22, 23]. Reference [24] extended the SPC/EPC integration to Multivariate engineering process control using multivariate statistical process control. However, most of the literature research on SPC/EPC integration methodology concentrates on continuous processes. For polymerization industry case, reference [25] proposed the integration of SPC and EPC in a continuous polymerization process. A several

regulation strategies were compared to reduce polymer viscosity deviations from target. Note, though ASPC, that the process is still being controlled by an automatic controller that is the process is being controlled all the time.

2.2. Active SPC

Another way to integrate the two control approaches is to provide on-line SPC. Statistical models are used not only to define control limits, but also to develop control laws that suggest the degree of manipulation to maintain the process under statistical control. Thus, in applications to continuous processes, the need for an algorithmic automatic controller is avoided, leading to a direct or 'active' SPC strategy [26]. Indeed, the technique is designed specifically for continuous systems. In contrast to ASPC, manipulations are made only when necessary, as indicated by detecting violation of control limits. As a result, compared to automatic control and ASPC, savings in the use of raw materials and utilities can be achieved using active SPC.

2.3. Run-To-Run

Run-To-Run (RTR), also known as Run-By-Run, process control techniques that also combine SPC and EPC concepts have been developed and applied, mostly, to semiconductor manufacturing processes. A "run" can be a single wafer, a lot, a batch, or any other grouping of semiconductor products undergoing the same set of process conditions. An RTR process refers to a process, such as a wafer-etching process or auto-body stamping process, in which a control action, e.g., a change in process parameter, can only be implemented between runs (or batches) instead of during a run. SPC act as a supervisor indicating the need for RTR control action. A review of RTR control can be found in [27, 28].

This adjustment can be effectively performed using EPC, which attempts to maintain a product quality measurement at a desired target value by identifying and adjusting recipe (i.e. the inputs or initial conditions) variables to correct for departures from the desired target. Reference [29] described a RTR automatic process controller that detects drifts due to chemical buildup tool and material degradation, or other causes from RTR and suggested changes to the recipe that will maintain conformance of the product quality measurements to the desired target values. Reference [15] discussed controlling product quality measurements through recipe adjustments on a RTR basis. Within the same context, reference [30] used the recipe data with the parameter profiles (measurements for a given process variable over the processing period in real-time) to predict product quality measurements using multi-way, multi-block partial least squares (PLS). Reference [31] used both the recipe and parameter profiles to form summary scores that monitor both characteristics relative to a stable process.

3. Batch Monitoring and Control

Batch reactors are the most common reactor used in polymerization engineering. They may vary in size from a five gallon pilot unit to a 30,000 gallon (or greater) production size [32]. Removal of the heat of polymerization is accomplished by circulating coolant through a jacket or by refluxing monomer and solvent. The main advantage of batch reactors versus continuous processes is the flexibility to accommodate multiple products. They are well suited for low-volume products and for products for which there are numerous grades (as in specialty polymers), because each batch can be made according to its own recipe and operating conditions without the costs incurred when a continuous reactor is shut down and restarted. Process control of batch reactors must address the main disadvantage of batch

reactors versus continuous ones, namely variability within a batch and/or variability from batch to batch. This variability is particularly important in batch free radical polymerization, where the time of formation of a single chain is only a very small fraction of the batch time and therefore in homogeneity results from the fact that polymer chains can be formed under very different conditions during the course of the batch [33]. This is especially significant for composition control in a free radical batch copolymerization reactor where, unless special control strategies are deployed, polymer chains formed early in the reaction may contain a higher fraction of the more reactive monomer than the chains formed later in the reaction (i.e. compositional drift). On the contrary, in step growth polymerization (e.g. polyamides and polyesters), where the growth time of an individual chain is approximately the batch time, the effects of the changing reaction environment and hence within batch in homogeneities are much less of an issue, since all chains will see the same changing environment [32].

In many cases of batch-polymerization control there are no online measurements of polymer quality (e.g. polymer composition and molecular weight) during the batch and these measures of end use properties are only available at the end of the batch. In this case recipe modifications from one run to the next are common. The minimal information needed to carry out this type of batch-to-batch control is a static model relating the manipulated variable to the quality variables at the end of the batch. As pointed out in [34], this model can be as simple as a steady state (constant) gain relationship or a nonlinear model that includes the effects of different initial conditions and the batch time. The philosophy of SPC can be very useful in this case, since the polymer quality variable (for example the Mooney viscosity in elastomers manufacture) can be plotted for each successive batch on a Shewhart (x-bar) chart with the upper and lower control limits placed at three standard deviations above and below the target. The likelihood of a point outside the control limits means that the batch is out-of-control and the batch recipe and possibly the sequence logic must be adjusted for the next batch. If the quality variable for the batch is within the control limits, no control action is taken to prevent manipulations of the batch process based on stochastic variations within it. As a consequence, we can conclude that this procedure of batch control can be fall into Run-to-Run methodology.

Within EPC reasoning, it is possible to implement sophisticated control strategies during the batch by establishing operating trajectories for initiator addition, monomer addition, and/or reactor temperature to achieve desired polymer properties in minimum time, maximize productivity, or tailor the polymer molecular weight distribution. This is typically accomplished by solving off line an optimization problem using a kinetic model of the process as shown for example in [35, 36]. These essentially open loop trajectories constitute a form of feedforward control and are then implemented as part of the batch sequential logic and recipe management system using ladder logic and binary logic diagrams.

The application of SPC charts to batch processes in literature has been increased. At the beginning, most SPC methods use only the product quality measurements obtained at the end of each batch such as in [15] and therefore monitor only the batch-to-batch variation as detailed in RTR integration methodology. Reference [37] recognized that the process variable measurements taken during a batch run, although transient in nature, do follow a certain dynamic pattern, and they proposed a simple SPC technique for monitoring a single measurement variable. Afterward, references [9, 38, 39], and many others, proposed multivariate SPC (MSPC) methods for the analysis and on-line monitoring of batch processes. They assumed that the only information needed to develop these methods is a historical data base on measured process variable trajectories from past successful batches. In batch polymer process reactors the primary process variables such as pressure, temperature, level and flow are recorded during the batch as well as the quality variables at the end of the batch. However,

it may be very difficult to obtain a kinetic model of the polymerization process due to the complexity of the reaction mechanism, which is frequently encountered in the batch manufacture of specialty polymers.

Recently, many authors such as in [6, 40, 41, 42, 43, 44, 45] have used advanced statistical techniques such as multi-way principal component analysis, multi-way partial least squares, and model predictive controllers along with an historical database of past successful batches to construct an empirical model of the batch. This empirical model is used to monitor the evolution of future batch runs. Subsequent future unusual events can be detected during the course of the batch by referencing the measured process behavior against this "in control" model and its statistical properties. It may be therefore possible to detect a potential bad batch before the run is over and take corrective action during the batch in order to bring it on aim.

It should be noted that some authors, such as [46, 47], employ the name "data-driven quality improvement" (DDQI), referring to the advanced statistical tools application. In the related previous literature, DDQI constructs a statistical model from operation data, analyzes the cause of inferior quality and low yield, selects manipulated variables, and optimizes the operating conditions that can achieve the desired quality. Reference [48] proposed a product design method based on linear/nonlinear multivariate analysis. DDQI uses such conventional methods for modeling processes and optimizing operating conditions, but DDQI has several additional important functions as described in [47]. The advanced features of DDQI include: (1) handling qualitative variables as well as quantitative variables in a unified framework and (2) modeling batch processes and optimizing operation profiles through wavelet analysis and multivariate analysis. Multiple regression analysis is the simplest method for building a quality model, but it cannot be used if a co-linearity problem occurs. To cope with this problem, principal component regression and partial least squares can be used.

Nevertheless, when multivariate principal component analysis or other advanced statistical techniques is used for on-line batch monitoring, the future behavior of each new batch must be inferred up to the end of the batch operation at each time and the batch lengths must be equalized. This represents a major shortcoming because predicting the future observations without considering the dynamic relationships may distort the data information, leading to false alarms. In addition, the majority of these methods and techniques are either time-consuming or hard to implement in practice. To overcome the drawbacks of actual approaches, the aim of this work is to propose a statistical batch monitoring approach based on SPC and EPC integration.

4. The Proposed SPC/EPC Integration Approach

Figure 1 exhibits the framework of the proposed SPC/EPC integration approach and detail procedure is given as follows. The performance of statistical process monitoring of batch processes can be enhanced by incorporating external information such as batch-run specific and process specific information as recommended in [49]. The proposed SPC/EPC integration approach is composed into two phases: off-line training and on-line monitoring and control. The off-line training constructs a statistical model from operation data, analyzes the cause of inferior quality and low yield, selects manipulated variables, and optimizes the operating conditions that can achieve the desired quality. The off-line training is composed of four steps: (1) Handle qualitative variables as well as quantitative based on quality improvement tools, (2) Select the process characteristics and the critical monitoring variables, (3) Data collection of all past successful batches, and (4) Modeling batch processes and optimizing operation profiles variable.

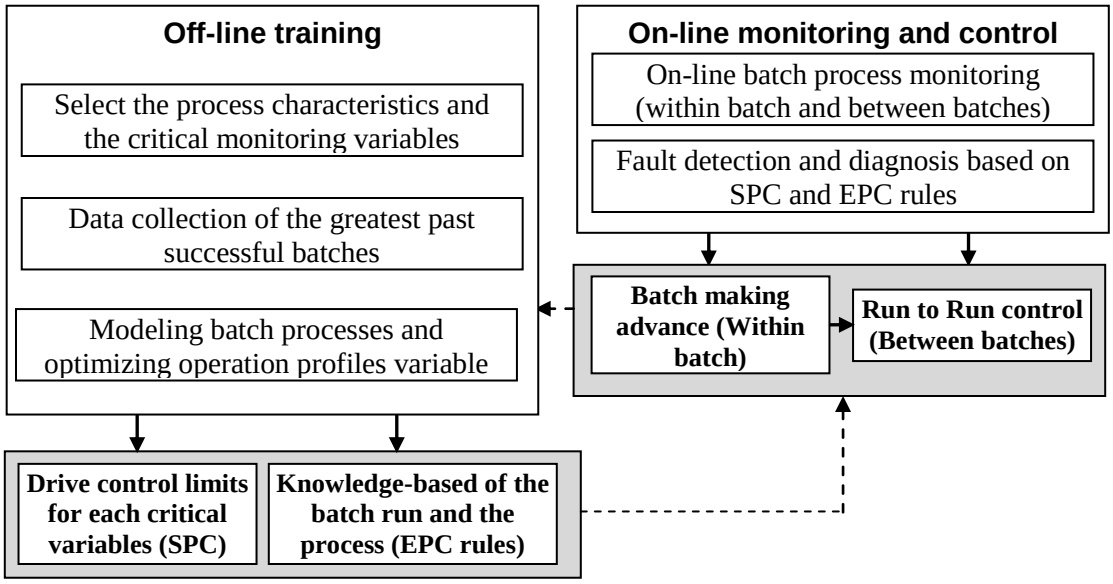


Figure 1. The Architecture of the Proposed SPC/EPC Integration Approach

After that, the on-line monitoring and control is performed continually in two successive phases: (1) Active SPC for the batch making advance, and (2) RTR control action between batches. Active SPC is applied to Batch making advance control. RTR control updates operating conditions or operation profiles for the next batch and it gives set-points to local controllers on the basis of information provided by the off-line training, i.e., the quality model, the manipulated variables or control structure, and the optimal operating conditions. In addition, control charts detect and diagnose faults on the basis of the statistical model built in the off-line training.

5. The Case Study

5.1. The Alkyd Reactor Description

To check the performance of the proposed SPC/EPC integration approach, an experimental alkyd polymerization is considered. In fact, oil-modified polyesters, commonly known as alkyd resins, have a high demand as coating resins due to their low production cost and variety of properties (durability, color retention, brightness, etc.). Commonly, an industrial batch alkyd cook is monitored, controlled and stopped on the basis of discrete-delayed measurements of Acidity Index (AI) and C (cold)-viscosity as described in [50]. The acidity measures the conversion of monomers into polymer (i.e. conversion of the acid functional group), and the C-viscosity reflects the complex branched molecular architecture of the polymer.

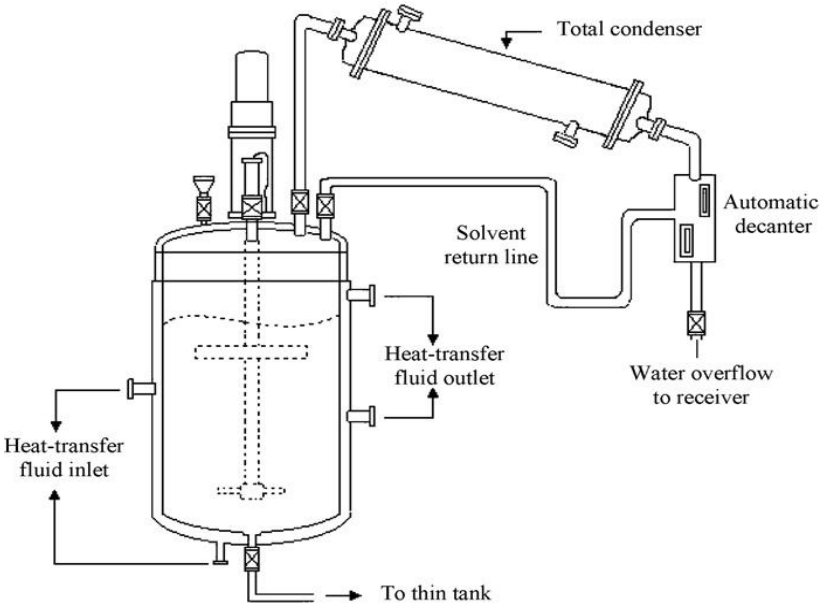


Figure 2. The Alkyd Reactor under Study

In an alkyd reactor (Figure 2), either synthetic or natural fatty acids, polybasic acids, and polyols are polymerized via endothermic reversible complex polyesterification reactions. The reactor is equipped with a condensing-decanting system to remove produced water in order to avoid equilibrium and favor the forward reaction to produce the polymer. The reactor load has a low level of solvent to assist the reaction water withdrawal by dragging the water in an azeotropic solvent–water vapor through the condensing-decanting system, where the water is dropped and the solvent is refluxed to the reactor. The temperature is maintained constant by means of a conventional controller.

5.2. Off-line Training

The first step in the real case study is process analysis which is called in this paper as off-line training. The results of this study are showed progressively as presented in the proposed SPC/EPC integration approach. The purpose of the process analysis is to understand the entire process, including the critical relations between the quality requirements and the performance metrics of both input and output conditions. The initial understanding and selection of factors in the order of their importance aims to reduce inefficiencies in the process.

5.2.1. Select the process characteristics and the critical monitoring variables: In the case study, three key variables were used to determine the reaction advance and the polymer product quality are Overflow Water Weight (OWW), AI and C-viscosity. Their monitoring is done on the basis of laboratory analysis of cold-diluted samples, usually taken out at periodic sampling times. The reacting mixture sample is cooled down and diluted because the hot reacting mixture viscosity barely decreases with conversion, and is excessively large for a standard industrial viscometer. In this way, the obtained measurements of OWW, AI and C-viscosity are discrete-delayed. For instance, Table 1 gives the measurements of every 30 min a reacting mixture sample which was taken out, cooled, diluted and analyzed to obtain OWW, AI and C-viscosity. The process is monitored online through these large number of process

measurements. However, these measurements are noisy, auto-correlated and cross-correlated. Quality measurements are only available off-line, and they are scarce, delayed and unevenly spaced in time. The operating procedure for a batch evolves through a nominal recipe, which is subject to several online adjustments made by the plant personnel depending on the actual evolution of the batch, as it is monitored by the quality measurements. The batch length (BL) exhibits a large variability. All of these features make each batch hardly reproducible and the online quality estimation a challenge.

Table 1. OWW, AI and C-viscosity Measurements within Batch

Time (Hr)	OWW (g)	IA	C-viscosity (s)
0	0.0	-	-
0.25	22.0	-	-
0.50	43.0		
0.75	61.0	-	3.00
1.25	88.5	70.0	4.75
1.75	108.0	52.0	5.00
2.25	118.5	42.0	6.02
2.75	127.0	37.0	6.75
3.25	133.0	33.0	7.20
3.75	136.5	29.5	8.04
4.25	140.0	25.0	11.03
4.75	142.0	22.0	14.00
5.25	144.0	19.5	18.04
5.75	145.0	17.6	25.03
6.25	145.5	15.0	32.01
6.75	146.0	12.5	41.00
7.25	146.2	10.0	50.00
7.75	146.4	9.0	61.50

Discrete-delayed measurements are employed to correct the operation by adding reactants, and to decide when to stop the batch in order to obtain a product within specifications (i.e., conversion and viscosity). In particular, the key decision on the batch termination is taken on the basis of a time-ahead prediction of viscosity, using table guides for monitoring reaction advance in conjunction with the operators' experience. Even though this monitoring-control scheme has been employed in industrial reactors, a more efficient and better reactor operation requires, among other aspects: (i) the improvement of existing monitoring and control schemes and their automation, (ii) the development and the actualization of knowledge rules which can be used to correlated to reaction advance, and (iii) the development of significant control charts for batch control.

The batch must be stopped at a certain conversion below the gelation point of the cold product. A drift from the prescribed C-viscosity trajectory as shown in Fig. 3, signifies that the polymer structure is drifting from its nominal value, and an abrupt change means that the cook is running away from its nominal motion.

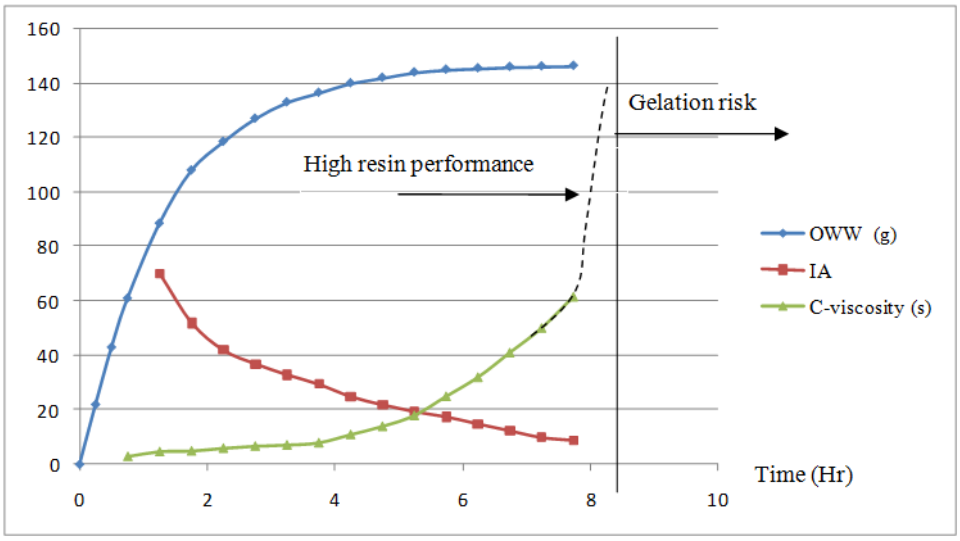


Figure 3. The Evolution of OWW, AI and C-viscosity within Batch

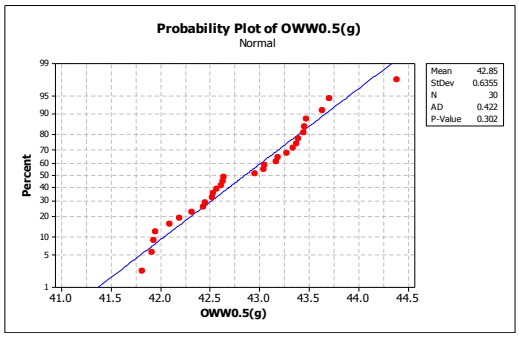
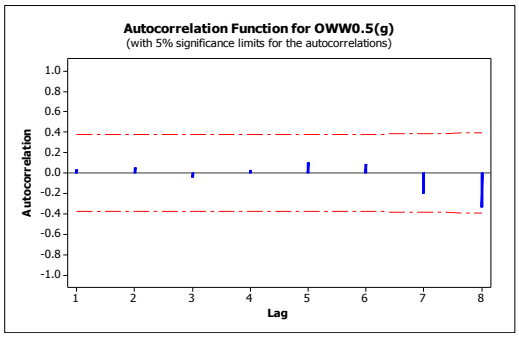
5.2.2. Data collection of the greatest past successful batches: Based on interviews with three technical experts of the company, four critical variables were selected as critical to monitor the reactor. This selection of variables is based on historical production reports. These variables are: the value of OWW measured after 0.5 hours of the start of the reaction denoted by "OWW0.5", the value of IA measured after 3 hours of the start of the reaction denoted by "IA3", the value of C-viscosity measured also after 3 hours of the start of the reaction denoted by "CV3", and the value of C-viscosity measured after 6.25 hours of the start of the reaction denoted by "CV6.25". Each one of theses critical variables are measured for thirty successful batches. These measures and each batch length which denoted by "BH" were presented in Table 1.

5.2.3. Modeling batch processes and optimizing operation profiles variable: The analysis of Table 1 shows that the four variables selected and BL are random fluctuations. In this phase, these variables will be modeled and this in order to apply SPC. Shewhart control charts make assumptions about the plotted statistic, namely (1) it is normally distributed, i.e. the data has a normal probability density function, and (2) it is independent, i.e. a value is not influenced by its past value and will not affect future values. Each test is applied for each selected variable.

Table 2. Measure of Critical Variables for Thirty Successful Batches

Exp	OWW0.5(g)	IA3	CV3(s)	CV6.25 (s)	BL (Hr)
1	43.18	32.7	7.00	32.00	7.97
2	41.91	36.1	6.60	32.35	7.95
3	43.04	35.7	7.09	32.23	7.62
4	42.95	35.6	6.94	32.10	7.65
5	41.95	35.6	6.96	32.02	7.80

6	43.45	35.0	7.36	32.13	7.52
7	42.53	34.1	7.02	32.05	7.90
8	43.34	34.8	7.09	32.20	7.70
9	43.27	35.0	6.93	31.86	7.62
10	42.57	34.6	7.14	32.20	7.80
11	41.93	35.1	7.07	32.19	7.87
12	42.61	37.2	7.09	32.04	7.43
13	42.63	35.7	6.74	32.15	7.80
14	43.05	34.9	6.95	32.03	7.72
15	42.32	37.4	7.22	32.12	7.42
16	42.19	34.1	7.46	32.11	7.80
17	42.09	33.6	7.00	32.10	8.05
18	43.17	33.2	6.91	32.20	8.01
19	43.47	36.3	6.80	31.90	7.58
20	43.37	36.3	7.31	32.23	7.72
21	42.45	33.7	6.67	32.23	8.15
22	43.39	35.4	7.06	32.08	7.58
23	43.44	34.0	7.04	32.29	7.85
24	43.63	32.8	6.58	32.00	8.03
25	44.38	35.3	7.06	32.09	7.55
26	42.64	34.3	7.02	32.17	7.85
27	42.52	33.9	6.58	32.11	8.10
28	42.43	37.3	7.08	32.08	7.55
29	43.70	35.1	6.94	32.00	7.58
30	41.82	33.2	6.80	32.11	8.20

Normality test	Autocorrelation test (independent)
	

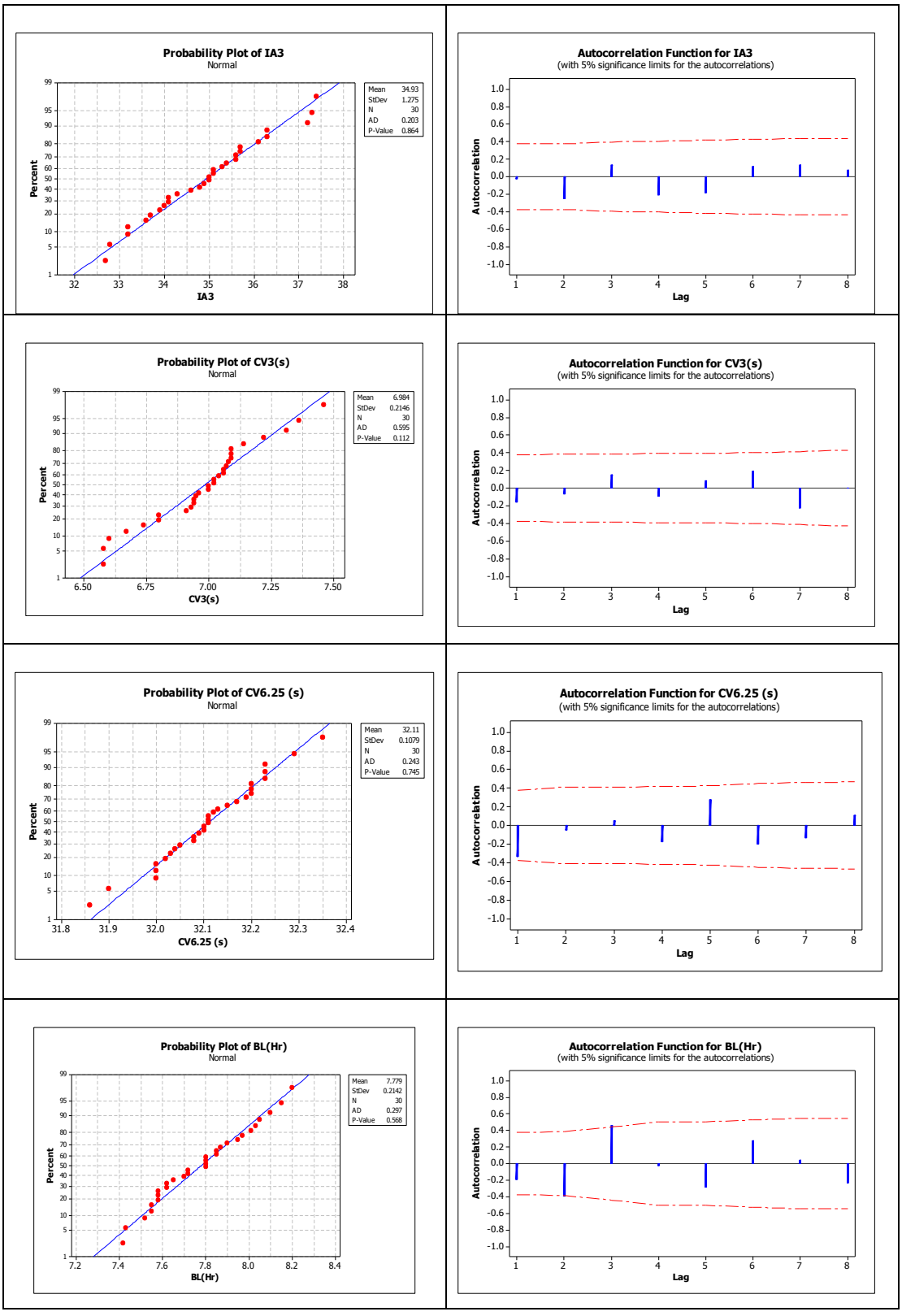


Figure 4. Shewhart's Hypothesis Tests Applied to each Selected Variables

In addition, to predict BL for a new batch, we try to model BL as a linear regression with the four selected variables. The use of Minitab 14 has confirmed the equation (1) with a R^2 coefficient equal to 93.5 %. The table shows a P-value less than 0.05 for each variable.

$$BL(i) = 6.58 - 0.118*OWW0.5(i) - 0.119*IA3(i) - 0.330*CV3(i) + 0.396*CV6.25(i) \quad (1)$$

Table 3. Statistical Tests of the Regression of the BL

Predictor	Coef	SE Coef	T	P
Constant	6.584	3.660	1.80	0.084
OWW0.5(g)	-0.118	0.0179	-6.58	0.000
IA3	-0.119	0.0089	-13.24	0.000
CV3(s)	-0.330	0.0537	-6.14	0.000
CV6.25 (s)	0.396	0.1054	3.76	0.001

5.3. On-line Monitoring and Control

5.3.1. On-line batch process monitoring (within batch and between batches): After verifying the test of normality and the test of independence of Shewhart, the control chart of each selected variable is applied as shown in Fig. 4. To monitoring the new batch advance, it is necessary to measure (OWW measured after 0.5 hour, IA measured after 3 hours, C-viscosity measured also after 3 hours, and C-viscosity measured after 6.25 hours) and place the point in the corresponding control chart. These values are also used to predict the BL as indicated in equation (1).

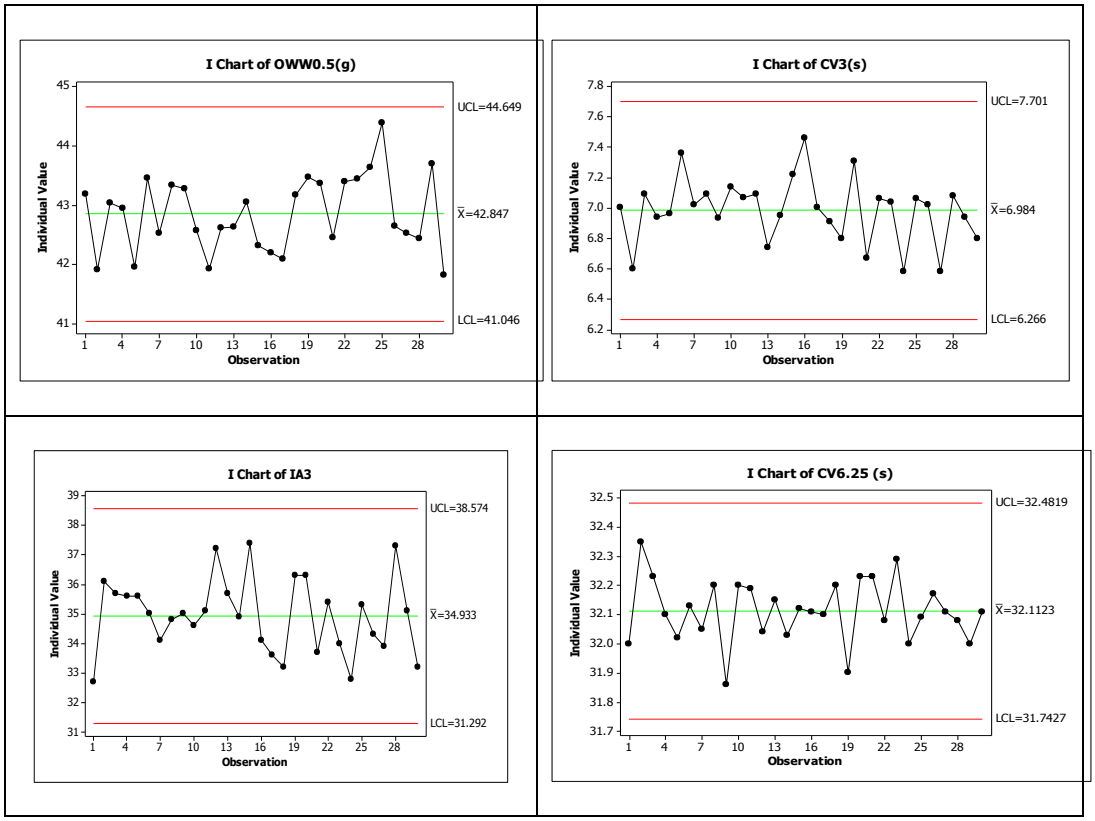


Figure 5. Control Chart for each Selected Variable

5.3.2. Fault detection and diagnosis based on SPC and EPC: For reasons of confidentiality, only a few rules of EPC were presented in Table 4. For each control chart, there are two cases that arise: Exceeding the Upper Limit of Control (ULC) up or exceeding the Lower Control Limit (LCL) down.

Table 4. Extract from the EPC Rules According Control Charts Limits

Variables	Current value is less than LCL	Current value is greater than UCL
OWW0.5	Elevate the temperature of heating or Add catalyst.	Reduce the heating temperature
IA3	Add acid and verify the result after 0.5 hour	Add polyol and verify the result after 0.5 hour
CV3	Elevate the heating temperature	Care must be taken. The C-Viscosity variable should be measured every 30 min
CV6.25	Elevate the heating temperature	Care must be taken. The C-Viscosity variable should be measured every 15 min

The production control procedure of a new batch is as follows. First, the operator measures the amount of water after 30 minutes of the progress of the reaction denoted by OWW0.5. If this amount of water is between 41.046 g and 44.649 g (as indicated in the control-chart located in the upper right of Figure 5), then the reaction proceeds without any assignable cause and the process is stable. Otherwise, the operator must refer to Table 4 to determine the best corrective action. Also this operation is repeated in the same way for the other variables, which are IA3, CV3, and CV6.25. Second, the operator provides the breakpoint of the reaction by using Equation 1. Finally, all data recorded during this monitored batch must be kept in the database.

6. Conclusion

This paper proposes a new integrated SPC/EPC system that applied in batch process. The proposed SPC/EPC integration is performed continually in two successive phases: (1) Active SPC for the batch making advance, and (2) RTR control action between batches. Control limits for critical variables are developed using information from the historical-data reference distribution of past successful batches. EPC application is based on the development of progressive knowledge-based rules. For a validation purpose, the proposed approach is applied to data collected from an industrial batch alkyd polymerization reactor which evolution is monitored by measuring the overflow water weight, the acidity index and the viscosity of samples withdrawn from the reactor. Through this case study application, process engineers at the company are now able to use a valuable decision making tool when the production process is affected by certain disruptions, with obvious consequences on product quality, productivity and competitiveness. For better performance, proposing a real time monitoring and control system is our perspective direction.

References

[1] J. Alvarez, T. López and E. Hernández, "Robust estimation of free-radical homopolymer reactors", Journal of Process Control, vol. 10, no. 5, **(2000)**.

[2] H. Hernández and J. Alvarez, "Robust estimation of continuous nonlinear plants with discrete measurements", Journal of Process Control, vol. 13, no. 1, **(2003)**.

[3] H. Hernández, T. Lopez and J. Alvarez, "Estimation of alkyd reactors with discrete-delayed measurements", Chemical Engineering Journal, vol. 160, **(2010)**.

[4] H. Amrehn, "Computer Control in the Polymerization Industry", Automatica, vol. 13, **(1977)**, pp. 533-545.

[5] B. Lennox and O. Marjanovic, "Monitoring and Control of Batch Processes", Control Engineering, vol. 52, no. 5, **(2005)**.

[6] S. Rännar, J. F. MacGregor, and S. Wold, "Adaptive batch monitoring suing hierarchical PCA", Chemometrics and Intelligent Laboratory Systems, vol. 41, no. 1, **(1998)**.

[7] N. Lu, Y. Yao and F. Gao, "Two-dimensional dynamic PCA for batch process monitoring", AIChE Journal, vol. 51, **(2005)**.

[8] P. Nomikos and J. F. MacGregor, "Monitoring batch processes using multiway principal component analysis", AIChE Journal, vol. 40, no. 8, **(1994)**.

[9] P. Nomikos and J. F. MacGregor, "Multivariate SPC Charts for Monitoring Batch Processes", Technometrics, vol. 37, no. 1, **(1995)**.

[10] G. E. P. Box and A. Luceno, "Statistical Control by Monitoring and Feedback Adjustment", Wiley: New York, **(1997)**.

[11] M. J. Willis and M. T. Tham, "Advanced process control", School of Chemical Engineering and Advanced Materials, unpublished paper, Newcastle University, **(1994)**. http://lorien.ncl.ac.uk/ming/advcontrl/apc.htm.

[12] G. A. Barnard, "Control Charts and Stochastic Processes", Journal of the Royal Statistical Society, vol. 21, no. 2, **(1959)**.

[13] J. F. MacGregor, "On-Line Statistical Process Control", Chemical Engineering Process, vol. 84, no. 10, **(1988)**.

[14] G. E. P. Box and T. Kramer, "Statistical process monitoring and feedback adjustment: a discussion", Technometrics, vol. 34, **(1992)**.

[15] S. A. Vander Wiel, W.T. Tucker, F.W. Faltin and N. Doganaksoy, "Algorithmic Statistical Process Control: Concepts and an Application", Technometrics, vol. 34, **(1992)**.

[16] W.T. Tucker, F.W. Faltin and S. A. Vander Wiel, "Algorithmic Statistical Process Control: An Elaboration", Technometrics, vol. 35, no. 4, **(1993)**.

[17] D. C. Montgomery, J. B. Keats, G. C. Runger and W. S. Messina, "Integrating Statistical process control and engineering process control", Journal of Quality Technology, vol. 26, no. 2, **(1994)**, pp. 79-87.

[18] F. Tsung and J. Shi, "Integrated design of run-to-run PID controller and SPC monitoring for process disturbance rejection", IIE Transaction, vol. 3, **(1999)**.

[19] D. C. Montgomery, M. Yatskievitch and W. S. Messina, "Integrating Statistical Process Monitoring with Feedforward Control", Quality and Reliability Engineering International, vol. 16, no. 6, **(2000)**.

[20] F. Tsung, "Statistical monitoring and diagnosis of automatic controlled processes using dynamic PCA", International Journal of Production Research, vol. 38, **(2000)**.

[21] B. Huang and Y. L. Lin, "Decision Rule of Assignable Causes Removal under an SPC-EPC Integration System", International Journal of Systems Science, vol. 33, no. 10, **(2002)**.

[22] R. Pan and E. del Castillo, "Integration of sequential process adjustment and process monitoring techniques", Quality and Reliability Engineering International, vol. 19, **(2003)**.

[23] A. S. Matos, J. G. Requeijo and Z. L. Pereira, "Integration of Engineering Process Control and Statistical Control in Pulp and Paper Industry", Proceedings of the 18th European Symposium on Computer Aided Process Engineering, **(2008)**.

[24] L. Yang and S. H. Sheu, "Integrating multivariate engineering process control and multivariate statistical process control", International Journal of Advanced Manufacturing Technology, vol. 29, **(2006)**.

[25] C. Capilla, A. Ferrer, R. Romero and A. Hualda, "Integration of Statistical and Engineering Process Control in a Continuous Polymerization Process", Technometrics, vol. 41, no. 1, **(1999)**.

[26] I. Efthimiadu, M. T. Tham, and M. J. Willis, "Engineering control and product quality assurance in the product industries", Proceedings of the International Conference on Quality and It's Applications, **(1993)**, Uni. Newcastle upon Tyne.

[27] E. Del Castillo and A. M. Hurwitz, "Run to run process control: review and extensions", Journal of Quality Technology, vol. 29, **(1997)**.

[28] T. F. Edgar, S. W. Butler, W. J. Campbell, C. Pfeiffer, C. Bode, S. B. Hwang, K. S. Balakrishnan, J. Hahn, "Automatic control in microelectronics manufacturing: Practices, challenges, and possibilities", Automatica, vol. 36, **(2000)**.

[29] E. Sachs, A. Hu and A. Ingolfsson, "Run by Run Process Control: Combining SPC and Feedback Control", IEEE Transactions on Semiconductor Manufacturing, vol. 8, **(1991)**.

[30] T. Kourti, P. Nomikos and J. F. MacGregor, "Analysis, Monitoring and Fault Diagnosis of Batch Processes using Multiblock and Multiway PLS", Journal of Process Control, vol. 5, **(1995)**.

[31] S. D. Grimshaw, S. D. Shellman and A. M. Hurwitz, "Real-Time Process Monitoring for Changing Inputs", Technometrics, vol. 40, no. 4, **(1998)**.

[32] J. R. Richards and J. P. Congalidis, "Measurement and control of polymerization reactors", Computers and Chemical Engineering, vol. 30, **(2006)**.

[33] F. J. Schork, "Reactor operation and control. Polymer reaction engineering", Dordrecht: Kluwer Academic Publishers, **(1993)**.

[34] D. E. Seborg, T. F. Edgar and D.A. Mellichamp, "Process dynamics and control", New York: Wiley & Sons. **(2004)**.

[35] T. J. Crowley and K.Y. Choi, "On-line monitoring and control of a batch polymerization reactor", Journal Processes Control, vol. 6, no. 2/3, **(1996)**.

[36] D. Tyner, M. Soroush, M. C. Grady, J. R. Richards and J. P. Congalidis, "Mathematical modeling and optimization of a semi-batch polymerization reactor", Proceedings of the IFAC Symposium on Advanced Control of Chemical Processes **(2000)**, 3, 983, New York: Elsevier.

[37] C. E. Marsh and T. W. Tucker, "Application of SPC Techniques to Batch Units", ISA Transactions, vol. 30, **(1991)**.

[38] B. Lennox, H. G. Hiden, G. A. Montague, G. Kornfeld and P. R. Goulding, "Application of multivariate statistical process control to batch operations", Computers and Chemical Engineering, vol. 24, **(2000)**.

[39] C. Undey, E. Tatara and A. Cinar, "Real-time batch process supervision by integrated knowledge-based systems and multivariate statistical methods", Engineering Applications of Artificial Intelligence, vol. 16, **(2003)**.

[40] J. Chen and K. C. Liu, "On-line batch process monitoring using dynamic PCA and dynamic PLS models", Chemical Engineering Science, vol. 57, **(2002)**.

[41] J. M. Lee, C. K. Yoo and I. Lee, "Enhanced process monitoring of fed-batch penicillin cultivation using time-varying and multivariate statistical analysis", Journal of Biotechnology, vol. 110, **(2004)**.

[42] P. Facco, F. Doplicher, F. Bezzo and M. Barol, "Moving average PLS soft sensor for online product quality estimation in an industrial batch polymerization process", Journal of Process Control, vol. 19, **(2009)**.

[43] A. Faggian, P. Facco, F. Doplicher, F. Bezzo and M. Barolo, "Multivariate statistical real-time monitoring of an industrial fed-batch process for the production of specialty chemicals", Chemical engineering research and design, vol. 87, **(2009)**.

[44] Y. Yao and F. Gao, "Multivariate statistical monitoring of multiphase two-dimensional dynamic batch processes", Journal of Process Control, vol. 19, **(2009)**.

[45] L. Dobos and J. Abonyl, "Application of non-linear dynamic optimization in advanced process control of product grade transitions of polymerization processes", proceeding of the 20th European Symposium on Computer Aided Process Engineering, **(2010)**.

[46] M. Kano, S. Hasebe, I. Hashimoto and H., Ohno, "Evolution of multivariate statistical process control: Application of independent component analysis and external analysis", Computers and Chemical Engineering, vol. 28, **(2004)**.

[47] M. Kanoa and Y. Nakagawa, "Data-based process monitoring, process control and quality improvement: Recent developments and applications in steel industry", Computers and Chemical Engineering, vol. 32, **(2008)**.

[48] C. M. Jaeckle and J. F. MacGregor, "Product design through multivariate statistical analysis of process data", AIChE Journal, vol. 44, **(1998)**.

[49] H. J. Ramaker, E. N. M. Van Sprang, S. P. Gurden, J. A. Westerhuis and A.K. Smilde, "Improved monitoring of batch processes by incorporating external information", Journal of Process Control, vol. 12, **(2002)**.

[50] F. N. Jones, "Alkyd Resins", Ullmann's Encyclopedia of Industrial Chemistry, Ed. Wiley VCH, **(2003)**.

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