Integration of Statistical Process Control (SPC) and Engineering Process Control (EPC) in Batch Process

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Abstract— Batch process is one of a main type of process in the chemical industry alongside continuous process. It is also called as discontinuous process due to the nature of the batch process. The main industry that utilizes batch process is the kind that produce low volume products and also, industry that produce specialty chemicals. The main principle of a batch process operations is by loading the reactants in a vessel and no material can be added or removed during the process. It is a very difficult encounter to production and process engineers to complete the task of achieving optimal performance of industrial batch processes. Batch processes have many major issues such as they are big batch-to-batch variations, highly non-linear dynamics, and difficulty in real-time measurement. It is significant to control the process in real-time, in order to avoid many issues such as off-spec product. The production of off-spec product is a major problem as the batch has to be eliminated and thus incurring loss. While the study of EPC and SPC is garnering attraction in controlling strategy, the implementing of the integration is still rare and few studies and research focus on it compare to continuous process. In this research, first, the real-time monitoring system of the integration of statistical process control (SPC) and engineering process control (EPC) in batch process is simulated. Secondly, the effectiveness of real- time monitoring systems of the integration of statistical process control and engineering process control in batch process is studied. The research revolves around a simulated case study of penicillin production. The type of control strategy deploy is the integration between statistical process control (SPC) and **Engineering Process Control (EPC).**

I. INTRODUCTION

In the chemical industry, there are two main types of process which are continuous process and another one, discontinuous or batch process. The kind of industries that mainly utilizes batch operations are the one that produce low-volume products and also, industries that produce specialty chemicals. The main principle of a batch process operations is by loading the reactants in a vessel and no material can be added or removed during the process. This operation is more flexible than continuous process because it allows the adjustment of the run time and temperature profile. Batch processes are typically produces below 10,000 metric tons per year [1].

Batch processing is important in producing specialty chemicals. Industries such as bio fermenters in the pharmaceutical sector, and crystallizers in many industries, also utilize batch process. Typically, the development of the operation of batch processes is done in the laboratory. This development involves 2 aspects which are reaction and separation recipes. The order of operations is prespecified. The operations are executed in specialized process equipment, to yield a specific quantity of product. The series of steps to be carried out on the specialized equipment are prespecified. The steps are such as cooling, distillation, heating, reaction, crystallization, and drying [1]. The processing sequence is repeated on a prespecified schedule, until the production volume achieved desired amount. In order for the target reactions or process to take place, the limit of temperature, concentrations, and flow rates required is prespecified too. It is a problematic task to adjust pressure, temperature, and feeding profiles by controlling strategies when the development from the laboratories are scaled up to production.

The application of batch processing is because of its economic nature but the objective also can be expressed in terms of technical goals which include productivity, product quality, and safety. The increment of quantity of final product per unit of time indicates the increasing of productivity. It also can represent the increment of number of batches per shift, as it is beneficial to reduce the time of production. Impurities is usually caused mainly by the side reactions and recycled impure solvents. Impurities is undesirable as it can make an acceptable product into off-spec product. This shows that the presence of impurities is crucial and negative. Impurities removal is typically not practical or involve additional separation steps which reduce productivity and costly. In the laboratory, there is usually less time for a complete process investigation. One method to overcome this is by online monitoring of the safety elements such as contamination and thermal runaway. In real application of batch process, the plants are usually nonlinear, and the exact model is unknown [2]. Statistical process control (SPC) is integrated with Engineering Process Control (EPC) to sense faults and reduce adjustment of the processes. This research analyzes the effectiveness of real time monitoring that integrates SPC with EPC especially in fault detection and corrective action.

Batch processes are the often-sought method in high valueadded products manufacturing. In this type of production, the variables display robust within-batch autocorrelation along with batch-to-batch correlations [3]. The batch processes bring many obstacles to the operation of batch processes. The batch processes are often poorly modelled and incomplete. This is caused by wide range of operating conditions that need to be represented by the models. Typically, in industry, the batch product quality is controlled, but the variable is determined can only be determined at the end of the process and it is not available in real-time. Challenges aside, batch processes have also its own opportunities that make it interesting to be discovered [4]. This is because, industrial chemical processes are usually slow when comparing it to mechanical and electrical systems. This can enable bigger sampling periods. Moreover, extensive online computations can be done.

It is a very difficult encounter to production and process

engineers to complete the task of achieving optimal performance of industrial batch processes. It is important to keep the process in its optimum operating range in order to maintain productivity thus, ideal set point trajectory tracking is an important element that needs attention [5]. Batch processes have many major issues such as they are big batch-to-batch variations, highly non-linear dynamics, and difficulty in real-time measurement [6]. These issues are crucial in obtaining good operations. Industries such as the specialty chemical and pharmaceutical industries have this problem in their operation. The process is operated within strict limits, to improve consistency and reduced production costs.

It is significant to control the process in real-time, in order to avoid many issues such as off-spec product. The production of offspec product is a major problem as the batch has to be eliminated and thus incurring loss. While the study of EPC and SPC is garnering attraction in controlling strategy, the implementing of the integration is still rare and few studies and research focus on it compare to continuous process [7]. There are several objectives in this research. First is to integrate the statistical process control (SPC) and engineering process control (EPC) in batch process. Secondly, is to simulate the real-time monitoring system of the integration of statistical process control (SPC) and engineering process control (EPC) in batch process.

Batch process is chosen in this research which mean continuous process is not included. The control strategies used to monitor the batch process are statistical process control (SPC) and engineering process control (EPC). There are several types of statistical process control, and in this research Shewart's Control Chart is used. For engineering process control, the type chosen is cascade system because it is the most reliable advance process control technique used in industry. EPC are used extensively in chemical industry. This is because variation is usually largely autocorrelated [8]. In this research, a semi batch process is modelled and simulated. The semi batch process chosen is penicillin production [9]. The case study is simulated using a web-based simulator known as Pensim Simulator. The simulator is used to generate 10 successful batches. 1 batch is run for 400 hours with 1-hour sampling time. The main parameter of the batch process is the penicillin concentration as the controlled variable. The selected key variable in this semi batch system is the aeration rate, substrate feed flow rate, substrate feed temperature and pH value.

II. METHODOLOGY

A. Proposed Approach

The flowchart of the proposed approach of this research is exhibit in Figure 1. The proposed SPC/EPC integration approach is composed into two phases which are offline monitoring and online monitoring and control.

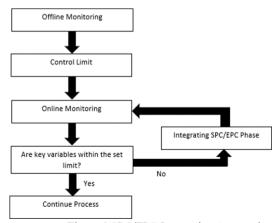


Figure 1 SPC/EPC Integration Approach

B. Flowchart of Research Methodology

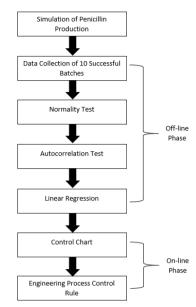


Figure 2 Flowchart of Research Methodology

The flowchart of research methodology that is applied in this research paper is exhibit in Figure 2. The first step of the methodology is to simulate a batch process. In this research, a case study of semi-batch penicillin production is chosen. The penicillin production is simulated using a web-based software develop by Illinois Institute of Technology which is called Pensim. Second step is to collect 10 successful batch processes data. This data is used to be analyzed in off-line monitoring phase. The first step in offline monitoring phase is to do a normality test and autocorrelation test. This is to ensure that the data collected have normal distribution and independent. After that, linear regression of the data is determined. In the on-line monitoring phase, by using control chart, the control limits of the variables are determined. In this research paper, Minitab 18 is used to do all the tests and control charts. Lastly, by referring to the control chart, the EPC rule of the variable is applied to ensure the process within the desired range of control.

C. Simulator

One of the most common industrial sectors that uses batch processing is the pharmaceutical industry, which uses this approach in production of high value added pharmaceutical and biochemical products. The particular type of batch process that is used in this industry is fermentation processing. This type of process therefore was used as a simulation to evaluate the performance of the different control algorithms developed in this thesis. One of the simulations used in this work was penicillin production published in which included built-in PID controllers.

The simulation is known as Pensim was provided by the Illinois Institute of Technology and modified to obtain the final penicillin concentration as the quality variable instead of the biomass concentration.

A major reason for selecting this simulation was that penicillin is one of the most useful groups of antibiotics used in modern medicine. Furthermore, the Pensim simulation has been used as a benchmarking tool in a range of publications describing new batch monitoring and control designs.

III. RESULTS AND DISCUSSION

A. Simulation Study: Penicillin Production

A simulation study from a pharmaceutical industry has been studied. In this section, before the result of the simulation study is discussed, the application of the proposed approach to data collected from this process will be discussed first. This is to develop a better understanding regarding the process. In this simulator, the mechanistic model of Bajpai and Reuss [9] is extended. Details of this simulation are well explained in the research of Birol et. al [10]. The mechanistic model has been substantially augmented by the inclusion of aeration rate, agitation power, feed flow rates of substrate and oxygen, carbon dioxide concentration, feed coolant and bioreactor temperatures, generated heat and the medium pH. Figure 2 is the schematic representation of the process. In a typical penicillin production process, the bioreactor is switched to the fed-batch mode of operation after 40 hours of batch growth phase when the cells enter their stationary phase [9].

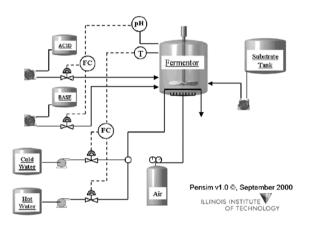


Figure 3 Process Flow Diagram of Penicillin Production [9]

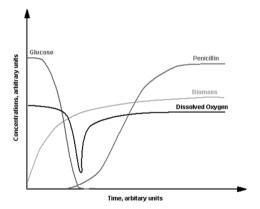


Figure 4 Time Profiles of Glucose, Penicillin, Biomass Concentrations and Dissolved Oxygen [9] Result of Simulation

The penicillin production is simulated 10 times using the Pensim Simulator. This is to obtain essential data for 10 successful batches. One batch is simulated for 400 hours with 1-hour sampling time. The input variables of this case study simulation are aeration rate, agitator power, glucose feed temperature, pH setpoints and temperature set-points. The measured variables are glucose concentration, dissolved oxygen concentration, culture volume, carbon dioxide concentration, pH, culture temperature and generated heat. The manipulated variable is glucose feed rate. These are the variable trajectories for one batch of the simulated penicillin production. The output variable are biomass concentration and penicillin concentration.

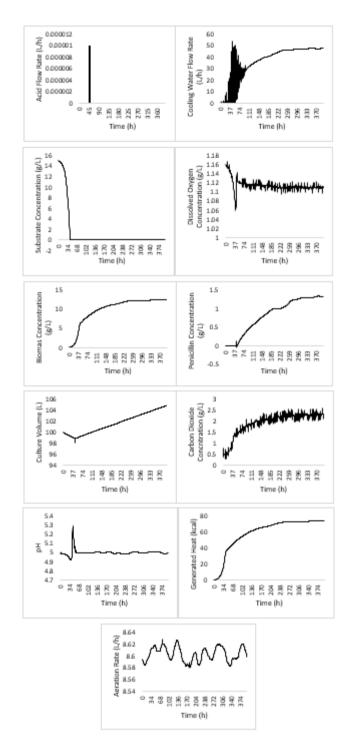


Figure 5 Variable Trajectories of the Penicillin Simulator

B. Off-line Monitoring Phase

Off-line monitoring phase is a process analysis step which must be done to kick off the case study analysis. Process analysis is done to fulfill several objectives. Among of the objectives are first, to obtain a full understanding regarding the entire process, this includes the relationship between the quality requirement and the performance metrics of both inputs and output conditions. Second objective of the process is to detect and select the key variable of the process that can reduce the inefficiencies of the process.

Selection of the Process Critical Monitoring Variables

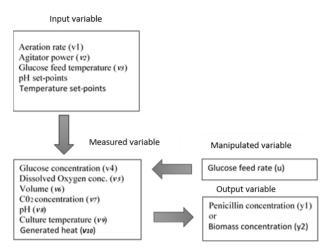


Figure 6 Variable of Penicillin Production

Four inputs key variables were selected as critical to monitor the process and one final output. The key variables are selected based on the analysis of historical database using Minitab 18 program and from the scientific journal of penicillin production. The variables selection is based on historical simulation data. The selected key variables are: the value of aeration rate, substrate feed flow rate, substrate feed temperature and pH value.

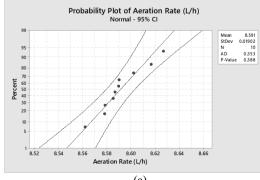
The key variables data that are used for the analysis are at 100th hour of the simulation. The reason is the process is switch to fedbatch mode during 45th hour of the simulation, then the process is stabilized at 100th hour. The variables value should be noted at the earlier phase of the batch process as changes made later to correct the process will be meaningful to influence the final result. Each one of these critical variables is selected from past ten successful processes. The data of key variables and final product output of penicillin concentration were recorded in Table 1.

 Table 1

 Simulated Data of Penicillin Production for 10 Successful Batches

| Batch | Aeration Rate (L/h) | Substrate Feed Rate (L/h) | Substrate Feed Temperature (K) | рН | Penicillin Concentration (g/L) |
|-------|------------------------|---------------------------------|---|---------|--------------------------------------|
| 1 | 8.6020 | 0.04210 | 296.025 | 4.99328 | 1.31704 |
| 2 | 8.5865 | 0.04162 | 296.230 | 5.00636 | 1.29849 |
| 3 | 8.6265 | 0.04315 | 296.040 | 5.00304 | 1.32079 |
| 4 | 8.5850 | 0.04170 | 295.993 | 5.01193 | 1.32963 |
| 5 | 8.6165 | 0.04132 | 296.045 | 5.00548 | 1.33037 |
| б | 8.5895 | 0.04305 | 296.015 | 5.01077 | 1.33407 |
| 7 | 8.5785 | 0.04315 | 296.060 | 5.00666 | 1.32375 |
| 8 | 8.5620 | 0.04302 | 295.965 | 5.00297 | 1.33479 |
| 9 | 8.5780 | 0.04333 | 295.990 | 4.99786 | 1.31727 |
| 10 | 8.5900 | 0.04088 | 295.887 | 4.99689 | 1.32175 |

Probability Plot



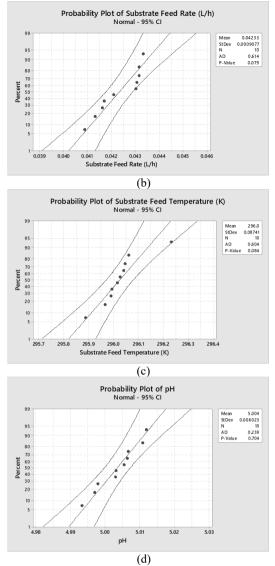
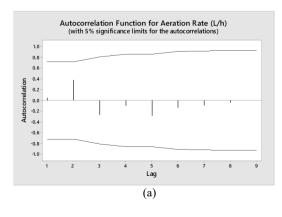


Figure 7 Probability Plot of Selected Key Variable (a) Aeration Rate (b) Substrate Feed Flowrate (c) Substrate Feed Temperature (d) pH

Autocorrelation Test



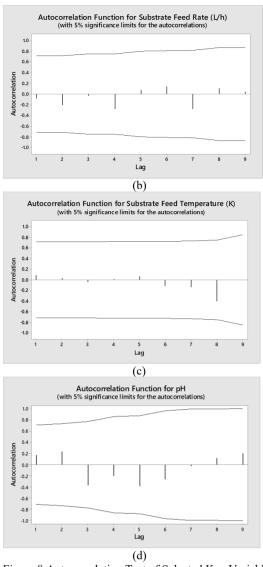


Figure 8 Autocorrelation Test of Selected Key Variables (a) Aeration Rate (b) Substrate Feed Flowrate (c) Substrate Feed Temperature (d) pH

In this phase, the selected key variables are modelled in order to apply SPC tools. The standard assumptions that are usually cited in justifying the use of control charts are that the data generated by the process when in control are normal, which mean the data have a normal probability density function and independent of observations, which mean the value is not influenced by its past value and will not affect future values, distributed with mean μ and standard deviation σ . The test of normality is indicated by producing probability and the test of independence is done by the autocorrelation function. These tests are done by using Minitab 18 program.

The test of normality and the test of independence of each selected key variable are applied as shown in this section. The figure approved standard assumptions, that the data generated by the process are in control, are normally distributed and independent. In the next step, the linear regression of the penicillin concentration and the key variables is model using Minitab 18. This is done in order to predict penicillin concentration for new process.

Linear Regression

Table 2 Analysis of Variance

| Source | | Adj SS | Adj MS | F-Value | P-Value |
|--------------------------------|---|----------|----------|---------|---------|
| Regression | | 0.000807 | 0.000202 | 4.42 | 0.067 |
| Aeration Rate (L/h) | | 0.000017 | 0.000017 | 0.37 | 0.571 |
| Substrate Feed Rate (L/h) | | 0.000040 | 0.000040 | 0.89 | 0.390 |
| Substrate Feed Temperature (K) | | 0.000673 | 0.000673 | 14.73 | 0.012 |
| pH Error | | 0.000308 | 0.000308 | 6.74 | 0.048 |
| | | 0.000228 | 0.000046 | | |
| Total | 9 | 0.001036 | | | |
| | | | | | |

Regression Equation:

Penicillin Concentration (g/L) = 26.89+ 0.075 Aeration Rate (L/h) + 2.40 Substrate Feed Rate (L/h) - 0.1065 Substrate Feed Temperature (K) + 1.039 pH

To predict the final value of penicillin concentration for a new batch, the final penicillin concentration for the 10 successful batches are model as a linear regression with the four selected variables which are aeration rate, substrate feed flow rate, substrate feed temperature and pH value. The linear regression is found by utilizing Minitab 18. By using the Minitab 18, it is confirmed the equation and with a R^2 coefficient equal to 77.94 %. Statisticians explained that R-Sq must be at least 0.70 for the regression line to be considered as meaningful [2].

C. On-line Measuring and Detecting Phase

The achieve or to obtain a successful desired operation is to have an efficient on-line process monitoring. To create an efficient on-line process monitoring, early warning of process disturbances, process malfunctions or faults must be enable. Early detection of process disturbances will lead to the following steps which are locating the source of disturbance. Thus, this enable the efficiency and consistency of production to be significantly improved. Schemes for process monitoring, fault detection and diagnosis can then be used as intelligent supervisory process systems, which can support process operators and engineers in dealing with process deviations and identifying the root cause of these deviations [2]. Process models built from process data are used as the fundamental of these schemes. In this section, the control chart which is an important element in on-line process monitoring is discussed.

On-line Process Measuring

In the previous section the test of normality and the test of independence are verified. The next crucial step is to have the control charts of each selected variable. To monitor the new process, it is necessary to measure the four selected key variables and place the point in the corresponding control chart. These values of the selected key variables are used to predict the penicillin concentration for next process as indicated in the regression equation.

Control Chart

Control charts, or it other name, Shewhart's Charts or processbehavior charts are the most common instrument used in statistical process control. The control chart is one of the most powerful techniques in statistical process control (SPC) to monitor processes and ensure quality [11]. Standard control charts are produced by calculating an average result for a time series of data, plotting this as the central line, and then calculating control limits either side of this mean. These control limits are usually set at plus and minus three standard deviations from the central line. This range will account for approximately 99.7% of all natural, 'common cause', variation. Control charts for aeration rate, substrate feed rate, substrate feed temperature and pH are obtained and for each selected key variable, the control limits are determined.

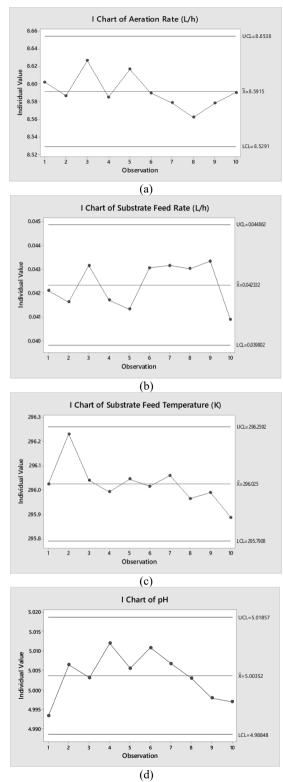


Figure 9 Control Chart of Selected Key Variables (a) Aeration rate (b) Substrate Feed Flowrate (c) Substrate Feed Temperature (d) pH

The control chart in Figure 8 shows a central line, upper and lower control limits. The control limits indicate a boundary which will be used as guidance to the process control engineer, when monitoring the process. The key variable should not exceed the upper control limit up and the lower control limit down in order to get the desired result of the process. When monitoring the process, as the key variable reach the control limit line, the person in charge of the process should be aware and be prepared to execute the engineering process control rules which will be discussed in the next step.

D. Integration of Statistical Process Control (SPC) and Engineering Process Control (EPC)

SPC is important for fault detection and diagnosis and EPC play a major role for corrective mechanism. Two cases arise for each control chart: Exceeding the Upper Limit of Control (UCL) up or exceeding the Lower Control Limit (LCL) down.

Table 3

EPC Rules According Control Charts Limits

| Variable | Current value is less that LCL | Current value is greater than UCL | | |
|-------------------------------|---|---|--|--|
| Aeration Rate | Increase the aeration rate. | Decrease the aeration | | |
| | Check the dissolved | rate. Check the dissolved | | |
| | oxygen concentration. | oxygen concentration. | | |
| Substrate Feed Flow Rate | Elevate the substrate feed | Reduce the substrate feed | | |
| | flow rate. | flow rate. | | |
| Substrate Feed Temperature | Increase the temperature of substrate feed by decreasing the cooling water flow rate. Check the fermenter temperature. | Decrease the temperature of substrate feed by increasing the cooling water flow rate. Check the fermenter temperature. | | |
| рН | Increase the pH value by increasing the base flow rate. | Decrease the pH value by increasing the acid flow rate. | | |

The production control procedure of a new batch is as follows. First, the operator monitors the aeration rate of the progress of the reaction. If this aeration rate 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 3 to determine the best corrective action. Also, this operation is repeated in the same way for the other variables, which are substrate feed flow rate, substrate feed temperature, and pH. The operator must verify whether the process is operating under the desired condition after 1 hour of executing the EPC rules. If the process still exceeding the control limit, the EPC rules must continue to be done. Second, the operator provides the breakpoint of the reaction by using regression equation. The last step is to record all data during this monitored batch in the database. This is important for future data monitoring and analysis.

IV. CONCLUSION

This research paper proposes an integrated system between statistical process control (SPC) and engineering process control (EPC) and its application in batch process. The results proved that by joining the SPC which enable the process engineer for detect the assignable cause at the earliest and regulate the process at the desired perimeter by using EPC. The proposed method is validated by applying the integration to data collected from a simulation case study of penicillin production. The process is monitored by measuring the selected key variable of the process. The case study has helps in demonstrating the effectiveness of the EPC/SPC integration. The process control operator can use this integrated method as a decision-making tool during the production. This is especially when the process disturbance is affecting the production process. Thus, avoiding the any consequences on product quality and productivity earlier. The proposed integrated system in this research does not need continuous regulations on the process and is only implemented when the process is affected by random process disturbance which will affect the whole production. The number of adjustments will be much less and can bring many economic benefits on the business. This control strategy consists of much little computation works and is convenience to be executed on the production ground. For future work, a real time monitoring and control system which will trigger the process automatically when there is a disturbance detected by the SPC method is an interesting subject of study.

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