PROCESS IMPROVEMENT OF REACTIVE DYE SYNTHESIS USING SIX SIGMA CONCEPT

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Abstract. This research focuses on the problem occurred in the reactive dye synthesis process of a global manufacturer in Thailand which producing various chemicals for reactive dye products to supply global industries such as chemicals, textiles and garments. The product named “Reactive Blue Base” is selected in this study because it has highest demand and the current chemical yield shows a high variation, i.e. yield variation of 90.4% – 99.1% (S.D. = 2.405 and Cpk = -0.08) and average yield is 94.5% (lower than the 95% standard set by the company). The Six Sigma concept is applied aiming at increasing yield and reducing variation of this process. This approach is suitable since it provides a systematic guideline with five improvement phases (DMAIC) to effectively tackle the problem and find the appropriate parameter settings of the process. Under the new parameter settings, the process yield variation is reduced to range between 96.5% – 98.5% (S.D. = 0.525 and Cpk = 1.83) and the average yield is increased to 97.5% (higher than the 95% standard set by the company).

1. INTRODUCTION
Reactive dye is the synthesis via the chemical reaction of colour and reactive components. The differences between reactive and natural dyes in terms of chemical properties are the chemical resistance of water, reactivity used in dyeing application process, and variety of colours. The reactive dye is popular for dyeing on cellulose fibres because the shade and brightness are depended on the nature or structure of the reactive dye [1]. Recently, the reactive dye has developed the chemical properties that increase the reactivity to cellulose fibres and solubility in water. Most reactive dyes after completing the synthesis process are sold in a powder form. The synthesis process of the reactive dye has to be controlled for all parameters to match with the chemical property of the raw material. The control of the process parameter and condition depends on the reactive dye structure, synthesis operation and chemical property. The special property of the reactive dye is the key component. All textile industries need to concentrate on the process improvement to optimize the synthesis condition and parameters to be matched with the types of reactive dyes. The objective of the improvement process is to gain high chemical yield and low variation [2]. Currently, the variation of the chemical yield from the reactive dye synthesis process of the company is high and the yield is lower than the company’s standard (95%). In this paper, the Six Sigma method is used to find the significant factors affecting the chemical yield and optimize the conditions suitable for controlling the parameters in each
step of the reaction process. The Six Sigma DMAIC procedure comprises five key phases, i.e. Define, Measurement, Analyze, Improve and Control phases [3-5].

2. DEFINE PHASE
In this case study, the product named Reactive Blue Base is selected because it has highest demand and shown a high variation in the chemical yield, 90.4 – 99.1\% (S.D. = 2.405, Cpk = -0.08). In addition, its average chemical yield is only 94.5\% which is lower than 95\% (company’s standard). This product is synthesized via 3 process reactions, i.e. 1\textsuperscript{st} Condensation, 2\textsuperscript{nd} Condensation and Vinylation processes. 1\textsuperscript{st} Condensation and 2\textsuperscript{nd} Condensation involve the addition of the reactive substances and Vinylation is the process that converts the structure of in-reactive dye to reactive dye via the transformation process of chemical structure. In each process step, different conditions and controlled parameters are used depending on the type and chemical property of the raw material. The factors to control in each step of the process are temperature, additional time of chemicals, maintaining time and pH. The historical data of the chemical yield show S.D. = 2.40 and average = 94.5\%. The chemical yield by production batch and descriptive statistics are shown in Fig.1 and Fig.2, respectively.

![Time Series Plot of Yield](image1)

**Figure 1.** Chemical yield by production batch

![Boxplot of Yield](image2)

**Figure 2.** Descriptive statistics of yield

3. MEASUREMENT PHASE
The measurement phase includes three steps, i.e. variation analysis of raw material usage from different suppliers, measurement system analysis and problem identification analysis.

3.1. Variation analysis of raw material usage from different suppliers and lots
In the chemical reaction process, the quality of raw material is important to the acceptability of the final product. This research aims to reduce the variation of chemical yield in the reactive dye synthesis process. Therefore, variation analysis of raw material usage from different suppliers and lots are importance because the quality of raw material usage in this research is identified as a controllable factor (supplier selection). The quality of raw materials supplied by both suppliers (Supplier A and Supplier B) are analyzed in terms of chemical properties on lot-to-lot for each suppliers (total 30 lots each) by following the company specification (specification setup for the main component should be over 96\%). The analysis results of the materials from Supplier A and Supplier B are shown in Fig.3 and Fig.4, respectively.
Figure 3. Raw material analysis from Supplier A

Figure 4. Raw material analysis from Supplier B

The results indicated that the quality of lot-to-lot raw materials supplied by Supplier A is not significantly different (P-value = 0.278). This result is the same for Supplier B (P-value = 0.338). As a result, the raw materials from both suppliers can be used to synthesize the dye. In addition, this confirms that the quality of raw material usage is the controllable factor in this research.

3.2. Measurement System Analysis

The chemical instrument analysis called “high performance liquid chromatography (HPLC)” is used to analyze the percentage of the chemical composition during the reactive dye synthesis process. Another automatic measuring instrument, such as pH maintaining machine, is calculated before being used and tested to assess their precision and accuracy agents of the pH standard solution. For lab instruments, we can conclude that all measuring instruments are acceptable and ready to use for analyzing the accuracy and precision of the HPLC machine of chemical composition during the reactive dye synthesis. Three lab analysts are performed to test the chemical composition of the reactive dye synthesis process for 10 batches in total with two replicates [6]. The conclusion is shown in Table 1 which can be stated that the part-to-part variability is 99.41% and total gage R&R variability is 0.59%. Therefore, the measurement system is acceptable in terms of accuracy and variability.

| Source          | Variance Components | Contribution rate |
|-----------------|---------------------|-------------------|
| Total Gage R&R  | 0.01936             | 0.59%             |
| Repeatability   | 0.01924             | 0.58%             |
| Reproducibility | 0.00011             | 0.00%             |
| Operators       | 0.00011             | 0.00%             |
| Part-To-Part    | 3.27067             | 99.41%            |
| Total Variation | 3.2900            | 100.00%           |

3.3. Problem Identification Analysis

The team comprising several experts in the reactive dye synthesis process, especially for the Reactive Blue Base, brainstorms about the relevant factors that can affect the chemical yield. The cause-and-effect diagram with 5M1E methodology is applied in this study process. It is revealed that the number of tentative causes is 24 factors. These factors are used to analyze the relationship of causes and effects using cause-and-effect matrix by scoring these relationships with 0, 1, 3 and 9, respectively.
The factors with significant impacts on the chemical yield are selected and shown in Fig. 5 (Pareto chart).

The results from the cause-and-effect matrix analysis indicates that 8 factors have high scores. These factors are further screened by using Failure Mode and Effects Analysis (FMEA) as shown in Fig. 6. Four factors with high rating are selected for conducting the design of experiment (DOE) in the analyze phase, which is 66.17% of the total score.

**4. ANALYSIS PHASE**

This phase involves the root cause analysis of the problem. From the Pareto chart analysis, 4 factors are selected to study in this analysis phase including time maintaining, addition time of chemical, pH and temperature. The Box Behnken design of experiments [7] with 4 factors and 3 levels are adopted. The experiments for reactive dye synthesis are separated into 2 steps, i.e. process study and optimize the factor. For 1\textsuperscript{st} Condensation process, 3 factors to be experimented include \( \text{pH} \), temperature and addition time of chemical (Table 2). In addition, 3 factors are tested on 2\textsuperscript{nd} Condensation process including \( \text{pH} \), temperature and time maintaining (Table 3). The response variable for each step of the processes is the Main component result is analysed by the HPLC machine and it must pass the specification in each process (Main component>90\% for 1\textsuperscript{st} Condensation process and Main component>75\% for 2\textsuperscript{nd} Condensation process).

**Table 2. Factors level in Box Behnken design (1\textsuperscript{st} Condensation process)**

| No. | Input factors         | Units       | Symbols   | Levels of factors |
|-----|-----------------------|-------------|-----------|-------------------|
| 1   | Temperature           | Degree celsius | Temp     | -1 0 1            |
| 2   | \( \text{pH} \)       | -           | \( \text{pH} \) | 5.0 5.5 6.0      |
| 3   | Addition time of chemical | Minute     | Time dosing | 5 10 15          |

**Table 3. Factors level in Box Behnken design (2\textsuperscript{nd} Condensation process)**

| No. | Input factors         | Units       | Symbols   | Levels of factors |
|-----|-----------------------|-------------|-----------|-------------------|
| 1   | Temperature           | Degree celsius | Temp     | 10 20 30         |
| 2   | \( \text{pH} \)       | -           | \( \text{pH} \) | 7.0 8.0 9.0      |
| 3   | Time maintaining      | Minute      | Time maintain | 30 45 60        |
The experimental data are analysed by using the Minitab software and the results in each process step (1st Condensation and 2nd Condensation) are shown in Fig.7 and Fig.8, respectively. The statistical analysis of the results [8] shows that the main effect Temp, pH and Time dosing are significant factors for 1st Condensation process. For 2nd Condensation process, the statistical analysis shows that the main effects, i.e. Temp and pH, are significant factor, whereas the factor of Time maintain is not significant. In addition, two-way interactions of Temp*pH and Temp*Time dosing are also significant for 1st Condensation process.

**Figure 7. ANOVA result of 1st Condensation**

**Figure 8. ANOVA result of 2nd Condensation**
5. IMPROVEMENT PHASE

In this phase, the significant factors in 1st Condensation and 2nd Condensation are further analyzed to find the optimal conditions by using the Response Optimizer in the Minitab software (Fig.9 and Fig.10). All parameters from the Response Optimizer are used as parameter settings for 20 batches of the pilot lab trial to observe the chemical yield in terms of variation and average yield. The chemical yields of the lab trials are shown in Fig.11 and the Descriptive Statistics is shown in Fig.12. The result demonstrates that the chemical yield from the synthesis process is in the range of 96.9% – 98.4% (S.D. = 0.436 and Cpk = 2.02) and the average of chemical yield is 97.6%.

![Figure 9. Parameters setting for 1st Condensation](image1)

![Figure 10. Parameters setting for 2nd Condensation](image2)

![Figure 11. Chemical yield by lab trials](image3)

![Figure 12. Descriptive Statistics of yield](image4)

6. CONTROL PHASE

All levels of the significant factors in each process step and parameter settings are implemented in the production unit. The data are collected from the synthesis process for 34 batches. It is found that the chemical yields from the synthesis process is in the range of 96.5% – 98.5% (S.D. = 0.525 and Cpk = 1.83) and the average chemical yield is 97.5%. Clearly, the chemical yield of the Reactive Blue Base is improved as expected. To maintain the quality of the chemical yield after improvement process, the operating instruction for operational control is documented and training of operators is provided. In addition, the concerned persons are trained to recognize and understand the importance of all parameters control for the reactive dye synthesis process to ensure that the performance improvements are maintained. The comparison of the chemical yield before and after the improvement process are shown in Fig.13.
7. CONCLUSIONS
The Six Sigma concept is applied to improve the process of the reactive dye synthesis. The DMAIC methodology which incorporating the Box Behnken experimental design are employed to find the root causes that can affect the chemical yield of the Reactive Blue Base synthesis process and also indicates how to set the relevant process parameters. The results show clearly that after completing the process improvement each process step can reduce the variation of the chemical yield and increase the average yield to over 95% as expected. In addition, the results and knowledge gained from this case study can also be extended to another products of the company.

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