การลดต้นทุนของเสียในกระบวนการผลิตไดออกทิลพทาเลตโดยประยุกต์ใช้วิธีการซิกซ์ ซิกมา

นาย รณภพ กิตติอัครเสถียร

สถาบนวิทยบริการ

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FAILURE COST REDUCTION IN DIOCTYL PHTHALATE MANUFACTURING PROCESS USING SIX SIGMA APPROACH

Mr. Ronnapop Kittiakrastien

สถาบนวทยบรุการ

A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Engineering Program in Engineering Management The Regional Centre for Manufacturing Systems Engineering Faculty of Engineering Chulalongkorn University Academic Year 2007 Copyright of Chulalongkorn University

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วิทยานิพนธ์ฉบับนี้มีวัตถุประสงค์เพื่อศึกษาวิธีการลดต้นทุนของเสียที่เกิดขึ้นใน กระบวนการผลิตไดออกทิลพทาเลต โดยประยุกตีใช้วิธีการซิกซ์ ซิกมา ซึ่งสามารถแบ่งออกได้เป็น 5 ชั้นตอน คือ ขั้นตอนการกำหนดปัญหา ขั้นตอนการวัด ชั้นตอนการวิเคราะห์ ขั้นตอนการปรับปรุง และขั้นตอนการควบคุม ในขั้นตอนแรกได้ศึกษาสภาพปัญหา กำหนดเป้าหมายและขอบเขตของ การปรับปรุง ซึ่งมุ่งเน้นไปที่การลดต้นทุนของเสียอันเนื่องมาจากคุณภาพด้านสี และความ ด้านทานกระแลไฟฟ้า ในขั้นตอนการวัด ได้ทำการหาค่าความสามารถของกระบวนการและศึกษา ความแม่นยำและความถูกต้องของระบบการวัด โดยใช้วิธีการสอบเทียบและประเมินผลด้านรีพีท ทะบิลิตี้และรีโปรดิวซิบิลิตี้ของระบบการวัด จากนั้นวิเคราะห์หาสาเหตุของปัญหาโดยการระคม สมองและใช้แผนภูมิก้างปลา จากนั้นจัดลำดับความสำคัญของสาเหตุโดยประยุกต์วิธีการ วิเคราะห์ข้อบกพร่องและผลกระทบ เมื่อสามารถระบุถึงสาเหตุหลักของปัญหาได้แล้วขั้นตอนต่อไป คือ การทดสอบความมีนัยสำคัญของสาเหตุและปรับปรุงเพื่อลดของเสียจากกระบวนการผลิตโดย การทดลอง เพื่อกำหนดค่าที่เหมาะสมของปัจจัยการผลิตที่มีนัยสำคัญ โดยใช้หลักการออกแบบ การทดลองทางวิศวกรรม ในขั้นตอนการควบคุมได้เก็บข้อมูลเพื่อการยืนยันผลการทดลอง และ จัดทำมาตรการควบคุมและป้องกันปัญหา

จากผลการประยุกต์ใช้วิธีการซิกซ์ ซิกมา พบว่าค่าความสามารถของกระบวนการในเรื่อง ของลี และความต้านทานกระแสไฟฟ้าดีขึ้น จาก 0.92 เป็น 1.48 และ จาก 1.03 เป็น 1.57 ตามลำดับ ซึ่งเป็นผลทำให้ต้นทุนที่เกิดขึ้นจากของเลียในการกระบวนการผลิตเฉลี่ยต่อไดรมาส ลดลงจาก 1,327,368 บาทในปี 2007 เป็น 0 บาทในไตรมาสแรกของปี 2008

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This objective of this research is to reduce the failure cost production in Dioctyl Phthalate (DOP) manufacturing process by using Six Sigma approach. Six Sigma approach consists of 5 phases which are Define phase, Measure phase, Analyse phase, Improve phase, and Control phase. In the Define phase, the problem of failure cost in DOP finished product and objective of the project. were identified with the scope focusing on reducing failure cost regarding two types of defects: high colour and low resistivity. Then, in the Measure phase, process capabilities of DOP finished product were calculated the measurement system evaluation and verification were performed by calibration and Gage R&R method. After that, in the Analyse phase, the causes of the defects were identified by brainstorming and organized by the Cause-and-Effect diagram. Then, the causes of defects were prioritized by applying Failure Mode and Effects Analysis (FMEA). Then, in the Improve phase, Design of Experiment (DOE) was applied to determine significant causes that affect the quality of DOP finished product colour and low resistivity value. Then, optimum factor operating conditions were determined. After implementing the new operating conditions, control charts were used to monitor a process and to inform when to take corrective actions in the Control phase. The results of this research are that the process capability of colour parameter was improved from 0.92 to 1.48 and from 1.03 to 1.57 for resistivity value parameter. Thus, the failure cost of DOP reprocessing was reduced from 1,327, 368 baht quarterly in 2007 to 0 baht in the first quarter of 2008.

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CHAPTER I

INTRODUCTION

1.1 Background of the Research

At the present, production cost, product quality and process improvement in petrochemical industry are more important than the past. Due to high competition in petrochemical industry and large requirement of the customer, improvement of process capability needs to be implemented. The nonconforming product and inconsistency process affect to production cost and reputation of company. The reworking or degrading of nonconforming product leads to benefit and cost in the company. Six Sigma approach is used to reduce process variation, improve process capability, and reduces nonconforming product which reduce the failure cost production. Six Sigma consists 5 phases which are define, measure, analyze, improve, and control phase. Techniques in Six Sigma such as Failure Modes and Effects Analysis (FMEA), Design of Experiment (DOE), Statistical Process Control (SPC) are used in each phase.

1.2 Description of Manufacturing Process

Dioctyl Phthalate (DOP) is the most commonly used plasticizer in the PVC industry. It is a colourless high boiling point liquid, highly stable to light and soluble in most common solvents. DOP is also used in lacquers to improve resistance to abrasion and is compatible with ethyl cellulose to improve its low temperature flexibility. DOP process can be divided into 4 parts as shown in figure 1.1.



Figure 1.1: DOP Manufacturing Process

1. Reaction Part

The first part of DOP manufacturing process is reaction part. Hot oil control valve is opened to control reactor temperature at 160 °C. Phthalic Anhydride (PA) and Octyl Alcohol (OA) are transferred from raw material tank to reactor. After that hot oil control valve is fully opened to increase reactor temperature to 190 °C. Catalyst is added at this temperature. After reactor temperature reaches 215 °C, operator will take sample to check Acid Value (AV) and Colour and control the reactor temperature at this temperature with slightly vacuum pressure about 2 hours. After 2 hours, operator will take sample to check AV until AV is 0.8 mg KOH/g sample. Then, operator starts stripping process about 1 hour and checks AV before crude DOP is transferred to next part. In the study company, 2 reactors are operated and 6 batches are produced a day.

2. Neutralization Part

Crude DOP from each reactor is cooled down and transferred to neutralization tank (2 neutralization tanks are operated). At this part, crude DOP temperature is controlled at 90-99 °C. Crude DOP is neutralized by adding water and Sodium Hydroxide (NaOH) which is calculated by AV of reaction part. During adding NaOH, agitator in neutralization tank is turned on about 15 minutes. After that crude DOP is

settled about 3 hours and checked quality which is AV, Colour, and Specific Gravity (SG) before crude DOP is transferred to next part.

3. Distillation Part

Crude DOP from both neutralization tanks is transferred to distillation column as continuous process. Residual volatile such as water and OA in crude DOP is removed by steam stripping at vacuum pressure. Operator checks crude DOP quality which is OA content before transferred to next part.

4. Filtration Part

Crude DOP from distillation column is transferred to storage tank. Activated carbon as a decolouring agent and filter aid are added in this tank. DOP is filtered in filter plate and checked quality which is AV, Colour, water content, %OA, %DOP and Resistivity Value (VR) before transferred to DOP finished product storage tank.

1.3 Statement of the Problem

Quality of DOP finished product needs to be checked before filled into drum and delivered to customers. The results of six specifications of DOP finished product which are AV, Colour, water content, %OA, %DOP and Resistivity Value (VR) are collected and determined process capability as shown below:

สถาบันวิทยบริการ จุฬาลงกรณ์มหาวิทยาลัย



Figure 1.2: Process Capability of DOP Finished Product, Acid Value



Figure 1.3: Process Capability of DOP Finished Product, Colour



Figure 1.4: Process Capability of DOP Finished Product, Water Content



Figure 1.5: Process Capability of DOP Finished Product, %OA



Figure 1.6: Process Capability of DOP Finished Product, %DOP



Figure 1.7: Process Capability of DOP Finished Product, Resistivity Value

In figure 1.3 and figure 1.7, they show that process capability (C_{pk}) for DOP finished product colour is 0.92 and process capability (C_{pk}) for DOP finished product resistivity value is 1.03 which are lower than 1.33, while process capability of other DOP specifications is higher than 1.33. Normally, process capability which is lower than 1.33 has high variation in process, therefore, these process capabilities should be improved.

According to process capability above, the company encounters high DOP finished product colour and low resistivity value problems which lead to high production cost on reprocessing of nonconforming product.

Reprocessing of high DOP colour can be done by adding activated carbon, while reprocessing of low resistivity value can be done by adding filter aid and replacing filter paper. It also has loss production opportunity due to reprocessing both high DOP colour and low resistivity value product which can be determined based on numbers of failure batch.

Failure cost production on reprocessing of high DOP colour, low resistivity value and loss opportunity of production can be summarized in table 1.1 as follows:

		100	0		
-2					Total per
0.4	Jan	Feb	Mar	Average	quarter
Number of production, batch	70	67	57	65	194
Production consumption, m ³	2,100	2,010	1,710	1,940	5,820
High colour product consumption, m ³	30	90	150	90	270
Low VR product consumption, m ³	180	240	30	150	450
Lost production opportunity on reprocessing, m ³	120	180	90	130	390
Net Sales, baht	5,806,080	5,557,248	4,727,808	5,363,712	16,091,136
High colour product reprocessing cost, baht	8,560	25,680	42,800	25,680	77,040
Low VR product reprocessing cost, baht	68,822	91,763	11,470	57,352	172,056
Lost production opportunity on reprocessing, baht	331,776	497,664	248,832	359,424	1,078,272
Total of failure cost, baht	409,158	615,107	303,102	442,456	1,327,368
Percentage of failure cost, baht	7.0%	11.1%	6.4%	8.2%	8.2%

Table 1.1 Failure Cost Production on Reprocessing of Colour and VR

Notes:

- 1. Volume of production, high colour product and low VR product per batch is 30 m³
- Lost production opportunity is based on half number of high DOP colour and low VR reprocessing.
- 3. Cost of high DOP colour product reprocessing is based on material cost of activated carbon and high DOP colour product consumption.
- 4. Cost of low VR product reprocessing is based on material cost of filter aid and filter paper and low VR product consumption.
- 5. Labour cost of normal operation and reprocessing are same, therefore, labour cost is not considered.
- 6. Rejected product cost is not considered due to finished product has been checked before selling to customers.
- 7. Percentage of failure cost is determined by net sales and total of failure cost.

In table 1.1, estimated failure cost due to reprocessing of DOP product per quarter is 1,327,368 baht or 8.2% of net sales.

As describe above, there are some problems that affect the DOP process capability.

- 1. Raw material specification is not suitable.
- 2. Operating condition or operating procedure such as temperature, pressure, catalyst consumption or reaction time is not suitable.
- 3. Human factors such as little experience, misunderstand, and low responsibility of process.
- 4. Lack of controlling, monitoring and maintaining consistency of process.

In this research, Six Sigma approach will be used to identify problems of DOP quality and process and solve those problems.

The objective of this research is to reduce failure cost of reprocessing on nonconforming product due to problems regarding colour and resistivity value parameters.

1.5 Scope of the Research

This research will study all related DOP processes that may affect the failure costs due to colour and resistivity value parameters. The processes include reaction, neutralization, distillation, and filtration part. Regarding reaction and neutralization parts, 2 lines of reactors and neutralization tanks are studied.

1.6 Expected Benefits

It is expected that this research will reduce DOP nonconforming product and failure cost such as reprocessing cost of nonconforming product. It also improves DOP process capability.

1.7 Research Procedure

- 1. Review of literatures and related studies of Six Sigma, failure modes and effects analysis, design of experiment and statistical process control.
- 2. Define phase
 - 2.1 Collect process and quality analysis data.
 - 2.2 Define objective of research.
- 3. Measure phase
 - 3.1 Identify current process capability.
 - 3.2 Analyze and verify the accuracy and precision of measurement system.
 - 3.3 Summarize the results and recommendation to the next phase.

4. Analyze phase

- 4.1 Identify possible causes of the two defect types using Fishbone diagrams.
- 4.2 Prioritize the causes of defects by applying failure modes and effects analysis.
- 4.3 Summarize the results and recommendation to the next phase.
- 5. Improve phase
 - 5.1 Set up design of experiment.
 - 5.2 Determine the effect of significant factors on product quality problem
 - 5.3 Define the suitable method to improve product quality.
 - 5.4 Implement into the process.
 - 5.5 Summarize the results.
- 6. Control phase
 - 6.1 Collect data from design of experiment.
 - 6.2 Compare process capability and failure costs before and after improvement.
 - 6.3 Select key parameter and prepare suitable statistical process control chart.
 - 6.4 Implement control plan.
- 7. Summarize the results and recommendation.
- 8. Prepare draft of the thesis report.
- 9. Thesis examination.

1.8 Research Schedule

The research schedule has been proposed and shown in table 1.2.

	Year 2007	JUN	JUL	AUG	SEP	OCT	NOV	DEC	JAN	FEB
1.	Review of literatures	19	19,8		9/1	61		61		
2.	Define phase	66					101			
	2.1 Collect process data									
	2.2 Define objective of research									
3.	Measure phase									
	3.1 Identify process capability									
	3.2 Verify measurement system									
	3.3 Summarize and recommend to the									
	next phase									

		Year 2007	JUN	JUL	AUG	SEP	OCT	NOV	DEC	JAN	FEB
4.	Anal	yze phase									
	4.1	Identify possible causes of the two									
		defect types using Fishbone									
		diagrams.									
	4.2	Prioritize the causes of defects by									
		applying failure modes and effects									
		analysis	0.11								
	4.3	Summarize and recommend to the									
		next phase									
5.	Impr	ove phase	0								
	5.1	Set up DOE									
	5.2	Determine the effect of factors on									
		process capability									
	5.3	Define the suitable method to	600								
		improve process capability									
	5.4	Implement into the process									
	5.5	Summarize the results									
6.	Cont	trol phase		2.8 .4							
	6.1	Collect data from design of									
		experiment	COR								
	6.2	Compare process capability and	1.20	11							
		failure cost before and after					5				
		improvement					7				
	6.3	Select key parameter and prepare					1				
		suitable statistical process control									
		chart									
	6.4	Implement control plan									
7.	Sum	marize the results and		۲Ľ							
	reco	mmendation	~					0			
8.	Prep	pare draft of report	9	19.2	7	197	01	າລ	0		
9.	Thes	sis examination	60					161			

Table 1.2 Research Schedule (Cont.)

CHAPTER II

RELATED THEORETICAL STUDIES AND REVIEW OF LITERATURE

2.1 Related Theoretical Studies

Six Sigma can be described as a business process improvement approach to find and eliminate causes of defects and errors, reduce cost of operations, improve productivity, better meet customer expectations, and achieve higher asset utilization and returns on investment in manufacturing and processes. Six Sigma is focused on improving each of these basic metrics: quality, productivity, cost and profitability. The DMAIC process model in figure 2.1 can provide a bridge for improvement of existing business processes that will help to realize the performance goals of improved quality, productivity, cost and profitability. (Evans and Lindsay, 2005).



Figure 2.1: Six Sigma and Process Improvement (Evans and Lindsay, 2005: 3)

The five steps which are define, measure, analyse, improve, and control in the DMAIC methodology in Six Sigma that can be described as follows: (Evans and Lindsay, 2005)

2.1.1 Define Phase

The first step is to clearly define the problem after Six Sigma project is selected. It must describe the entails selecting the problem to address, clearly defining the improvement opportunity, building commitment among all stakeholders, and understanding the process and customer requirements.

The problem statement should identify customers and the critical to quality (CTQ) characteristics that are important to customer satisfaction. These are the most impact on product or service performance which describes the current level of performance or the nature of errors or customer complaints, identifies the relevant performance metrics, calculates the cost or revenue implications of the project, and quantifies the expected level of performance from successful Six Sigma effort.

2.1.1.1 Organising for Six Sigma Projects

Six Sigma improvements are generally carried out by project. A project is a temporary work structure that starts, produces an output or outcome, and then shuts down. Projects are implemented by teams. A team-based project approach provides for board participation and ensures including the right mix of skills.

2.1.1.2 People Skills

People are key process improvement and Six Sigma effort. Good people discover opportunities, develop innovative solutions, and find ways to make team work. Technical skills are important for a successful Six Sigma project. Six Sigma team members need to have the technical skills to perform the analyses which are required for the DMAIC process. People skills can be learned but often take more time than is available for a single project; therefore, they should be a routine part for employee's educational programme.

Some of the essential elements for effective process improvement are shared vision and behavioral skills. A shared vision can unify a team and provide the motivation for successfully implementing the project. People who are technically oriented may neglect behavioral skills, thinking that such skills are unnecessary in order to solve technical problems. Behavioral skills require both knowledge and practice.

2.1.1.3 Project Definition

A Six Sigma project can be defined as a problem scheduled to set project goals and monitor progress. Project definition should include formal project mission statement which called a charter to define the project, objectives, and deliverable. Six Sigma project charters should clearly define the problem, the internal or external customer requirements, existing measures and performance benchmarks, the expected benefits of the project in terms of performance measures and financial justification, a project timeline, and the resources and data needed to carry out the project. Two important parts of a project definition are high level process map and a clear identification and validation of customer requirements.

2.1.1.3.1 High Level Process Map: SIPOC

A high level process map defines the boundaries of the Six Sigma project by identifying the process, its inputs and outputs, and its suppliers and customers. A SIPOC process map deriving from suppliers, inputs, process, outputs, and customers is shown in figure 2.2.



Figure 2.2 General Structure of SIPOC Process Map (Evans and Lindsay, 2005: 78)

SIPOC maps provide an overview of the key elements in the process and help to explain that who is the process owner, how inputs are acquired, who the process serves, and how it adds value. Inputs are goods and services required

by a process to generate outputs. Outputs may be physical items, documentation, and electronic information. Inputs are provided by suppliers, who may be external or internal to the organisation. Customers are the people, departments or organisations that receive outputs, and which also can be external or internal to the organisation.

2.1.1.3.2 Identifying Customer CTQs

Many characteristics of the products or services are critical to quality (CTQ) from customer's perspectives. CTQs maybe classified into 3 categories, as suggested by a Japanese professor, Noriaki Kano:

- Dissatisfiers: Requirements that are expected in a product or service.
- 2. Satisfiers: Requirements that customer say they want.
- Exciters/delighters: New or innovative features that customer do not expect.

Meeting customer expectations that is providing satisfiers is often considered the minimum required to stay in business. To be competitive, companies must surprise and delight customers by going beyond the expectation.

2.1.2 Measure Phase

The measure phase focuses on understanding the current performance of the improvement of selected process, and collecting any necessary data needed for analysis. This approach helps to communicate the most important factors that can be controlled or changed to improve CTQs and to define the experiments to confirm how input variables affect response variables. It also sets the stage for the control phase by defining those factors that require monitoring and controlling. It also includes assessment of the measurement system to ensure validity of measurements and evaluating the process capability.

Measurement is the act of quantifying the performance dimensions of products, services, processes, and other business activities. Measures and indicators refer to the

numerical information. The term of indicator is often used for measurements that are not a direct measure of performance. Measurement and indicators provide critical information about business performance and are fundamental to Six Sigma.

Product and service quality indicators focus on the outcomes of manufacturing and service processes. A common indicator of manufacturing quality is the number of nonconformities per unit or defect per unit. Nonconformities per unit are often reported as rates per thousand or million which common used as defects per million opportunities (dpmo).

2.1.2.1 Data Collection

Six Sigma relies on good data for understanding process performance and tracking improvements from design changes. Therefore, it must be made to collect accurate data. The first step in any data collection is to develop operational definitions for all measures that will be used. Clearly, any data are meaningless unless they are well defined and understood without ambiguity.

Data required for Six Sigma projects may be collected in a variety of ways. Data sheet use simple columnar or tabular form to record data. Many types of automated systems are used to improve both the accuracy and speed of data collection. However, much data collection is done manually such as check sheets which provide a convenient means of recording.

Sampling forms the basis for most data collection. In choosing the appropriate type of sampling method, it must consider what sample is designed to do. Several approaches to sampling can be used as follows:

- Simple random sampling: Every item in the population gas equal probability of being selected.
- Stratified sampling: The population is partitioned into groups and a sample is selected from each group.
- 3. Systematic sampling: Every *n*th (4th, 5th, etc.) item is selected.

- 4. Cluster sampling: A typical group (ex. division of the company) is selected, and a random sample is taken from within the group.
- 5. Judgment sampling: Expert opinion is used to determine the location and characteristics of a definable sample group.

Errors in sampling generally cause from sampling error and systematic error. Sampling error occurs naturally and results from the fact that a sample may not always be representative of the population. The only way to reduce sampling error is to take a larger sample from the population. Sources of systematic error include bias, noncomparable data, uncritical projection of trends, causation, and improper sampling. Systematic errors can be reduced or eliminated by design experiment.

2.1.2.2 Data Summarisation

Raw data needs to be organised, summarised, and visualised to turn them into information. Summarising data calculates means, standard deviations, and other statistical measures. Graphs and chart provide a convenient way of visualising and communicating information

2.1.2.3 Measurement System Evaluation

Accurate Six Sigma performance depends on reliable measurement systems. Measuring quality characteristics generally requires the use of the human senses which are seeing, hearing, feeling, tasting, and smelling and the use of some type of instrument or gauge to measure the magnitude of the characteristic.

2.1.2.3.1 Metrology

Gauges and instruments used to measure quality characteristics must provide correct information, which is assured through metrology – the science of measurement. Metrology is defined as the collection of people, equipment, facilities, methods, and procedures used to assure the correctness or adequacy of measurements. The need for metrology causes from every measurement is subject to error. Whenever variation is observed in measurements, some portion is due to measurement system error. Some errors are systematic (called bias); others are random. The size of the errors can affect the quality of the data and resulting decisions. The evaluation of data obtained from measurement is not meaningful unless the measurement instruments are accurate, precise, and reproducible.

Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value or standard. The lack of accuracy reflects a systematic bias in the measurement such as gauge out of calibration, worn, or used improperly by operator. Accuracy is measured as the amount of error in a measurement in proportion to the total size of the measurement. One measurement is more accurate than another if it has a smaller relative error.

Precision is defined as the closeness of agreement between randomly selected individual measurements or results. Therefore, precision related to the variance of repeated measurement. A measuring instrument with low variance is more precise than another having a higher variance.

Repeatability or equipment variation is the variation in multiple measurements by individual using the same instrument. Reproducibility or operation variation is the same measuring instrument when different individuals use it to measure the same parts, and indicates how robust the measuring process is to the operators and environmental conditions. Cause of poor reproducibility might be poor training of the operators in the use in the use of the instrument or unclear calibrations on the gauge dial.

2.1.2.3.2 Measurement System Evaluation and Verification

The accuracy, repeatability, and reproducibility of any measurement system must be quantified and evaluated. Calibration is the most important functions of metrology to compare a measurement device or system having a known relationship to national standards against another device or system whose relationship to national standards is unknown. Gage repeatability and reproducibility or Gage R&R studies determine how much of observed process variation is due to measurement system variation. There are two types of Gage R&R studies which are crossed and nested. Crossed Gage R&R study is used when each part is measured multiple times by each operator while nested Gage R&R study is used when each part is measured by only one operator. Two methods for assessing repeatability and reproducibility in Gage R&R study are X and R, and ANOVA. The X and R method breaks down the overall variation into three categories which are part-to-part, repeatability, and reproducibility while ANOVA method goes one step further and breaks down reproducibility into its operator, and operator-by-part, components. Therefore, the ANOVA method is more accurate than the X and R method due to it considers the operator by part interaction.

Acceptability of measurement system can be determined by using the following guideline (AIAG, 2002).

- 1. Total Gage R&R contribution in the %Study Variance column
 - a. Less than 10% the measurement system is acceptable.
 - Between 10% and 30% the measurement system is acceptable depending on the application, the cost of measuring device, cost of repair, or other factors.
 - c. Greater than 30% the measurement system is unacceptable and should be improved.
 - 2. Total Gage R&R contribution in the %Contribution column
 - a. Less than 1% the measurement system is acceptable.
 - b. Between 1% and 9% the measurement system is acceptable depending on the application, the cost of measuring device, cost of repair, or other factors.
 - c. Greater than 9% the measurement system is unacceptable and should be improved.

The number of categories (AIAG, 2002) represents the number of non-overlapping confidence intervals that will span the range of product variation or the

number of groups within process data that measurement system can discern. This number is calculated by dividing the standard deviation for parts by the standard deviation for gage, then multiplying by 1.41 and truncating this value.

It is suggested that when the number of categories is less than 2, the measurement system is of no value for controlling the process, since one part cannot be distinguished from another. When the number of categories is 2, the data can be divided into two groups, say high and low. When the number of categories is 3, the data can be divided into 3 groups, say low, middle and high. A value of 5 or more denotes an acceptable measurement system.

2.1.2.4 Process Capability Evaluation

Process Capability is the range over which the natural variation of the process occurs as determined by the system of common causes what the process can achieve under stable conditions. Process capability is important to both product designers and manufacturing engineers, and is critical to achieving Six Sigma performance. Knowing process capability allows one to predict how well a process will meet specifications and to specify equipment requirements and the level of control necessary.

2.1.2.4.1 Process Capability Studies

A process capability study is a planned study designed to yield specific information about the performance of a process under specified operating conditions. Typical questions that are asked in a process capability study include the follows:

- 2. Where is the process centre?
- 3. How much variability exists in the process?
- 4. Is the performance relative to specifications acceptable?
- 5. What proportion of output will be expected to meet specifications?
- 6. What factors contribute to variability?

Many reasons exist for conducting a capability study. Manufacturing may wish to determine a performance baseline for process, to prioritize projects for quality improvement, or to provide statistical evidence of quality for customers. Three types of studies are often conducted.

- 1. A peak performance study determines how a process performs under ideal conditions.
- 2. A process characterisation study is designed to determine how a process performs under actual operating conditions.
- 3. A component variability study assesses the relative contribution of different sources of total variation.

To obtain useful information, the sample size should be fairly large, generally at least 100. Process capability only makes sense if all special causes of variation have been eliminated and the process is in a state of statistical control.

One of the properties of a normal distribution is that 99.73 percent of the observations will fall within three standard deviations from the mean. Thus, a process that is in control can be expected to produce a large percentage of output between μ - 3 σ and μ + 3 σ , where μ is the process average. Therefore, the natural tolerance limits of the process are $\mu \pm 3\sigma$. A six standard deviation spread in commonly used as measure of process capability.

2.1.2.4.2 Process Capability Indexes

The relationship between the natural variation and specifications is often quantified by a measure known as the process capability index. The process capability index, C_p , is defined as the ratio of the specification width to the natural tolerance of the process. C_p relates the natural variation of the process with the design specifications in a single, quantitative measure. In numerical terms, the formula is

$$C_p = \frac{UTL - LTL}{6\sigma}$$

where		
UTL	=	upper tolerance limit
LTL	=	lower tolerance limit
σ	=	standard deviation of the process

Two important facts about the C_p index should be pointed out which relate to process conditions and to interpretation of the values that have been calculated. First, the calculation of the C_p has no meaning if the process is not under statistical control. The natural spread (6 σ) should be calculated using a sufficiently large sample to get a meaningful estimate of the population standard deviation (σ). Second, a C_p of 1.00 would require that the process be perfectly centred on the mean of the tolerance spread to prevent some units from being produced outside the limits. The goal of all units being produced within specifications with a C_p of 1.33 is much easier to achieve, and still easier with a C_p of 2.00. Based on the experience of the practitioners, the C_p of 1.5 have suggested as a safe lower limits. A value above this level will practically guarantee that all units produced by a controlled process will be within specifications.

To include information on process centring, one-sided process capability indexes are as follows:

 $C_{pu} = \frac{UTL - \mu}{3\sigma}$ (called the upper one-sided index) $C_{pl} = \frac{\mu - LTL}{3\sigma}$ (called the lower one-sided index) $C_{pk} = \min(C_{pl}, C_{pu})$

The low value of C_{pk} indicates that the worst case is unacceptable. Process capability indexes depend on the assumption that the distribution of output is normal. Process capability may be affected by measurement error. If the measurement error is large, the process capability indexes must be reviewed with caution.

2.1.3 Analyse Phase

The Analyse phase focuses on why defects, errors, or excessive variation occur. After potential variables are identified and measured, experiments are conducted to verify the hypothesized relationships by formulating some hypothesis to investigate, analysing the data and reaching reasonable and statistically supportable conclusions as to the root cause of the problem.

Statistical methods have applications in many areas of Six Sigma, including product and market analysis, product and process design, process control, testing and inspection, identification and verification of process improvements, and reliability analysis.

2.1.3.1 Basic Statistical Method

The discipline of statistics encompasses a wide variety of tools and techniques.

2.1.3.1.1 Statistical Inference

Statistical inference is concerned with conclusions about populations based on sample data. To be able to make probability statements about the relationship between sample statistics and population parameters and inferences, it needs to understand sampling distributions.

1. Sampling distributions: Different samples will produce different estimates of the population parameters. Therefore, sample statistics such as \bar{x} , s, and p are random variables that have their own probability distribution, mean, and variance. These probability distributions are called sampling distributions. When using sample random sampling, the expected value of \bar{x} is the population mean μ , or $E(\bar{x}) = \mu$.

2. Confidence Intervals: A confidence interval (CI) is an interval estimate of population parameter that also specified the likelihood that the interval contains the true population parameter. This probability is called the level of confidence, donated by 1 - α , and is usually expressed as a percentage.

2.1.3.1.2 Hypothesis Testing

Hypothesis testing involves inferences about two contrasting propositions (hypotheses) relating to the value of a population parameter, one of which is assumed to be true in the absence of contradictory data. The hypothesis is usually called the null hypothesis. The symbol H₀ is used to indicate the null hypothesis, where null refers to the hypothesis of no difference. For example, the null hypothesis would be that there is no difference between the two methods (the estimated population means of the throughput for the two production processes, μ_1 and μ_2 , are identical) which can be given as follows: H₀: $\mu_1 = \mu_2$ or H_a: $\mu_1 \neq \mu_2$. The alternative hypothesis (H_a) is simply that the mean throughput of the proposed method (μ_1) is higher than that of the current production process (μ_2), that is, H_a: $\mu_1 > \mu_2$

2.1.3.1.3 Enumerative and Analytic Studies

One of the biggest mistakes in using statistical methods is confusing data that are sampled from a static population (cross-sectional data) with data sampled from a dynamic process (time series data). A static population can be analysed to estimate population parameter such as the mean, variance, or proportion. Confidence intervals and hypothesis tests can be applied. However, the purpose of sampling from a process is generally to predict the future. The characteristics of the population may change over time as a plot of sample means or variances. In such cases, confidence intervals and hypothesis tests are not appropriate unless the time series can be shown to be stationary or constant mean and variance over time. Deming called the analysis of a static population an enumerative study, and the analysis of a dynamic time series an analytic study. Applying classical statistical inferences to an analytic study is not appropriate because they provide no basis for prediction. Thus, it is important to understand how to apply statistical tools properly.

2.1.3.1.4 Regression and Correlation

Regression analysis is a tool for building statistical models that characterise relationships between a dependent variable and one or more independent variables. The relationship may be linear or no relationship at all. To develop a
regression model, the first specify the type of function that best describes the data. A visual indication of the type of relationship between two variables can usually be seen in a scatter diagram. Typically, the variables in question represent possible cause and effect relationship.

Correlation is a measure of a linear relationship between two variables as the correlation coefficient. Correlation coefficients will range from -1 to +1. A correlation of 0 indicates that the two variables have no linear relationship to each other. A correlation coefficient of +1 indicates a perfect positive linear relationship; as one variable increases, the other will also increase. A correlation coefficient of -1 also shows a perfect linear relationship, except that as one variable increases, the other decrease.

2.1.3.2 Tools for Process Analysis

2.1.3.2.1 Process Mapping

A process map or flowchart identifies the sequence of activities or the flow of materials and information in a process. Process maps help the people involved in the process understand it much better and more objective by providing a picture of the steps needed to accomplish a task.

Process maps help team members understand how a process operates and who the key supplier and customers are. Once the flowchart is constructed, it can be side to identify sources of errors or defects, unwanted variation, and opportunity for improvement.

2.1.3.2.2 Value Stream Mapping

The value stream refers to all activities involved in designing, producing, and delivery goods and services to customer. These activities include the flow of materials throughout the supply chain, transformation activities in the delivery process, and the flow of information needed to support these activities. A value stream map shows the process flows similarly to an ordinary process map; however, the differences lies in that value stream maps highlight value-added versus non value-added activities, and include times that activities take. Value stream maps might include other information such as machine uptime and reliability, process capacity, and size of batches moving through the process.

2.1.3.2.3 Statistical Thinking

Statistical thinking is a philosophy of learning and action based on these principles:

1. All work occurs in a system of interconnected processes.

2. Variation exists in all processes.

3. Understanding and reducing variation are keys to success.

2.1.3.2.4 Root Cause Analysis

A root cause is defined as that condition having allowed or caused a defect to occur permanently prevents recurrence of the defect in the same, or subsequent, product or service generated by the process.

A useful approach for identifying the root cause is the 5 why technique. This approach forces one to redefine a problem statement as a chain of causes and effects to identify the source of the symptoms by asking why, ideally five times.

2.1.3.2.5 Cause and Effect Diagrams

A cause and effect diagram is a sample graphical method for hypothesising a chain of causes and effects and for sorting out potential causes and organizing relationships between variables. Cause and effect diagrams are constructed in a brainstorm. Everyone can get involved and feel they are an important part of the problem solving process. Usually, small groups drawn from operations areas or management work with a trained and experienced facilitator. The facilitator guides attention to discussion of the problem and its causes. As a group technique, the cause and effect method requires significant interaction between group members. The facilitator who listens carefully to the participants can capture the important ideas. A group can often be more effective by thinking of the problem broadly and considering environmental factors, political factors, and employee issues.

2.1.3.2.6 Failure Mode and Effects Analysis

Failure mode and effects analysis (FMEA) is a disciplined procedure that recognizes and evaluates the potential and actual effects of failure of a product or a process and identifies actions that reduce the chance of a potential failure occurring (Basem and David, 2005). It helps the design for six sigma (DFSS) team members improve their design and its delivery processes by asking "what can go wrong?" and "where can variation come from?" Service design and production, delivery, and other process are then revised to prevent occurrence of failure modes and to reduce variation. Inputs to FMEA include past warranty or process experience, customer wants, performance requirements, specifications and process mappings. For each service functional requirement and process, the team needs to determine possible design and process failure modes and sources of potential variation in all service processes under consideration.

The team should modify the service design and processes to prevent wrong things and develop strategies to deal with different situations, the redesign of processes to reduce variation, and mistake-proofing of services and processes. Effort to anticipate failure modes and sources of variation are iterative. This action continues as the term strives to future improve their service design and its processes.

FMEA is a team activity with representation from project personnel involved with quality, reliability, and operations, and suppliers and customers if possible. There are several types of FMEA as follows: 1. Concept FMEA. Used to analyse systems and subsystems in the early concept and design stages. Focuses on potential failure modes associated with the functions of a system caused by the design.

Design FMEA is used to analyse product or service designs before they are released to production. Design FMEA should always be completed well in advance of prototype or pilot build. It focuses on design functional requirement. It has a hierarchy that parallels the modular structure in terms of systems, subsystems, and components.

Process FMEA is used to analyse processing, assembly, or any other processes. The focus is on process inputs. It has a hierarchy that parallels the modular structure in terms of systems, subsystems, and components.

2. Project FMEA. Documents and addresses failures that could happen during a major program.

3. Software FMEA. Documents and addresses failure modes associated with software functions.

a. FMEA Fundamentals

FMEA can be described as being complementary to the process of defining what a design or process must do to satisfy the customer. The fundamentals of FMEA inputs are depicted in figure 2.3 and the listed below.

1. Define scope, service functional requirements, and design parameters and process steps. For example, in design FMEA, potential failure modes include the delivery of "No" functional requirement (FR), partial and degraded FR delivery over time, intermittent FR delivery, and unintended FR.

2. Identify potential failure modes. Failure modes indicate the loss of at least one FR. The DFSS team should identify all potential failure modes by asking, "In what way will the design fail to delivery its FRs?" as identified in the mapping. Failure modes are generally classified as material, environment, people, equipment, methods, and so on. A potential failure mode can be cause or effect in a higher level subsystem causing failure in its FRs. A failure mode may occur but not necessarily must

occur. Potential failure modes may be studied from the baseline of past and current data, test, and current baseline FMEAs.

3. Potential failure effect. A potential effect is the consequence of the failure of other physical entities as experienced by the customer.

4. Severity. Severity is a subjective measure of serious is the effect of the failure mode. Usually, severity is rated on a scale from 1 (no effect) to 10 (hazardous effect). Severity ratings of 9 or higher indicate a potential special effect that needs more attention and this typically is a safety or government regulation issue as shown in table 2.1. Severe effects are usually classified as critical, significant or control. A critical effect is usually a safety issue and requires more deep study for all causes down to the lowest level. Significant effects are important for the design itself. Control effects are regulated by the government for any public concern. A control plan is needed to mitigate the risks for the significant and critical effects. The team needs to develop proactive design recommendations.





Figure 2.3: FMEA Worksheet (Basem and David, 2005: 244)

Effect	Severity of Effect Defined	Rating
None	No effect.	1
Very Minor	Very minor effect on product quality and/or reduced level of	2
	process performance. Product may have to be reworked.	
Minor	Minor effect on product quality and/or reduced level of process	3
	performance. Product may have to be reworked.	
Very Low	Very low effect on product quality and/or reduced level of process	4
	performance. Product may have to be reworked.	
Low	Low effect on product quality and/or reduced level of process	5
	performance. Product may have to be reworked.	
Moderate	Moderate effect on product quality and/or reduced level of process	6
	performance. Product may have to be reworked.	
High	High effect on product quality and/or reduced level of process	7
	performance. Product may have to be reworked.	
Very High	Very high effect on product quality and/or equipment damaged.	8
	Product can not achieve their specification. They are treated as	
	waste.	
Serious	Potential Hazardous effect. Able to stop production without mishap;	9
	safety related. Disruption to subsequent process operation. Failure	
6	mode involves non-compliance with government regulation.	
Hazardous	Hazardous effect. Safety related – sudden failure in process	10
	production. Failure mode involves non-compliance with	
	government regulation.	

Table 2.1 FMEA Severity Ratings (AIAG, 2002)

5. Potential causes. These are the set of noise factors and the deficiencies are designed in due to the violation of design principles, axioms, and best practices. The study of the effect of noise factors helps the DFSS team identify the mechanism of failure. The analysis conducted by the DFSS team with the help of process mapping allows for the identification of the interactions and coupling of their scoped project with the environment and with customer, and within the processes and subprocesses themselves. For each potential failure mode identified in step 2, the DFSS team needs to enter a cause in this column. There are two basic reasons for these

cases: (1) the design is manufactured and assembled within specifications, (2) the design may include a deficiency that may cause unacceptable variation, or both.

6. Occurrence. Occurrence is the assessed cumulative subjective rating of the process entity failures that could occur over the intended life of the design. FMEA usually assumes that if the cause occurs, it is the failure mode. Based on this assumption, occurrence is also the likelihood of the failure mode. Occurrence is rated on a scale of 1 (almost never) to 10 (almost certain) based on failure likelihood or probability, usually given in parts per million defective (PPM). See table 2.2 for linkage to process capability. The occurrence rating is a ranking scale and does not reflect the actual likelihood. The actual likelihood or probability is based on the failure rate extracted from historical service or warranty data with the same parts. See table 2.3.

7. Current controls. The objective of design controls is to identify and detect the design deficiencies as early as possible. Design controls are usually applied for first-level failures.

Numerical Ranking	Occurrence Likelihood
1	1 in 10 ⁶ (C _{pk} > 1.67)
2	1 in 20,000 (C _{pk} = 1.33)
3	1 in 5,000 (C _{pk} ~ 1.00)
4	1 in 2,000 (C _{pk} < 1.00)
5	1 in 500
6	1 in 100
7	1 in 50
8	1 in 20
9	1 in 10
10	1 in 2

Table 2.2 FMEA Occurrence Linkage to Capability (Basem and David, 2005)

Probability of Failure	Occurrence	Rating
Almost Certain	Failure almost certain. It is inevitable. History of failures exists	10
	from previous or similar design. (1 in 2 or 50%)	
Very High	Very high number of failure likely. (1 in 10 or 10%)	9
High	High number of failure likely. (1 in 20 or 5%)	8
Moderately High	Moderately high number of failure likely. (1 in 50 or 2%)	7
Moderate	Moderate number of failure likely. (1 in 100 or 1%)	6
Low	Low number of failure likely. (1 in 500 or 0.2%)	5
Very Low	Very low number of failure likely. (1 in 2,000 or 0.05%)	4
Remote	Remote number of failure likely. (1 in 5,000 or 0.02%)	3
Very Remote	Very remote number of failure likely. (1 in 20,000 or 0.005%)	2
Absolute Uncertainty	Failure unlikely. History shows no failures. (1 in 10 ⁶ or 0.001%)	1

Table 2.3 FMEA Occurrence Ratings

8. Detection. Detection is a subjective rating corresponding to the likelihood that the detection method will defect the first-level failure of a potential failure mode. This rating is based on the effectiveness of control system. The DFSS team should assess the capability of each detection method and how early in the DFSS effort each method will be used. Team should review all detection methods in step 8 and achieve consensus on a detection rating. Finally, team should rate the method and select the lowest detection rating in case of methods tie. See table 2.4 for example.

9. Risk priority number (RPN). This is the product of the severity, occurrence and detection ratings. The range is between 1 and 1000. RPN numbers are used to prioritize the potential failures.

10. Actions recommended. The DFSS team should select and manage recommended subsequent actions where the risk of potential failures is high. After that, an immediate control plan should be performed to control the situation.

Detection	Likelihood of Detection	Rating
Almost Certain	Design control will almost certainly detect a potential	1
	cause/mechanism and subsequent failure mode.	
Very High	Very high chance the design control will detect a potential	2
	cause/mechanism and subsequent failure mode.	
High	High chance the design control will detect a potential	3
	cause/mechanism and subsequent failure mode.	
Moderately High	Moderately high chance the design control will detect a	4
	potential cause/mechanism and subsequent failure mode.	
Moderate	Moderate chance the design control will detect a potential	5
	cause/mechanism and subsequent failure mode.	
Low	Low chance the design control will detect a potential	6
_	cause/mechanism and subsequent failure mode.	
Very Low	Very low chance the design control will detect a potential	7
	cause/mechanism and subsequent failure mode.	
Remote	Remote chance the design control will detect a potential	8
	cause/mechanism and subsequent failure mode.	
Very Remote	Very remote chance the design control will detect a potential	9
	cause/mechanism and subsequent failure mode.	
Absolute	Design control will not and/or can not detect a potential	10
Uncertainty	cause/mechanism and subsequent failure mode; or there is no	
	design control.	

Table 2.4 FMEA Detection Ratings (AIAG, 2002)

2.1.4 Improve Phase

Once the root cause of the problem is understood, team needs to generate ideas for removing or resolving the problem and improves the performance measure of the variable and the CTQs. This gathering idea phase is a highly creative activity because many solutions are not obvious. It is necessary to evaluate ideas and select the most promising including confirming that the proposed solution will positively impact the key process variables and the CTQs.

Problem solutions often require technical or organisational changes. Some sort of decision is used to assess possible solutions against important criteria such as cost, time, quality improvement potential, resources required, effects on supervisors and workers, and barriers to implementation such as resistance to change or organisational culture. To implement a solution effectively, responsibility must be assigned to a suitable person or a group who will follow through on what must be done, where it be done, when it be done, and how it be done.

2.1.4.1 Principles of Process Improvement

2.1.4.1.1 Flexibility and Cycle Time Reduction

Flexibility refers to the ability to adapt quickly and effectively to changing requirement. It might mean rapid changeover from one product to another, rapid response to changing demands, or the ability to produce a wide range of services. Success in globally competitive markets requires a capacity for rapid change and flexibility.

Cycle time refers to the time it takes to accomplish one cycle of a process. Reductions in cycle time serve two purposes. First, speed up work processes so that customer response is improved. Second, reductions in cycles can only be accomplished by stream lining and simplifying processes to eliminate non value-added steps such as rework. This approach forces improvements in quality by reducing the potential for mistakes and errors. By reducing non value-added steps, costs are reduced as well.

2.1.4.1.2 Breakthrough Improvement

Breakthrough improvement refers to discontinuous change. Continuous improvement philosophy is more reflective of traditional quality management approaches. Breakthrough improvements result from innovative and creative thinking. One approach for breakthrough improvement that helps companies achieve stretch goals is known as reengineering, which has been defined as the rethinking and radical redesign of business process to achieve improvements in critical, contemporary measures of performance, such as cost, quality, service and speed. Successful reengineering requires understanding of processes, creative thinking to break away from old traditions and assumptions, and effective use information technology.

2.1.4.2 Tools for Process Improvement

Effective implementation of Six Sigma improvement strategies requires a disciplined application of statistical principles and various tools for implementing the DMAIC process. Many different tools and techniques exist to facilitate Six Sigma projects. Statistical techniques such as design of experiments and Taguchi methods are also important.

2.1.4.2.1 Kaizen Blitz

A kaizen blitz is an intense and rapid improvement process. Blitz teams are generally comprised of employees from all areas involved in the process who understand it and can implement changes on the spot. Improvement is immediate, exciting, and satisfying for all those involved in the process.

2.1.4.2.2 Poka-Yoke (Mistake-Proofing)

Human beings tend to make mistakes inadvertently. Such errors can arise from the following factors:

- 1. Forgetfulness due to lack of concentration
- 2. Misunderstanding because of the lack of familiarity with a process or procedures.
- 3. Poor identification associated with lack of proper attention.

Typical mistakes in production are omitted processing, processing errors, set up errors, missing parts, wrong parts, and adjustment errors. Once mistakes are identified, one might use a cause and effect diagram or other analysis tools to identify the reasons for their occurrence. Poka-yoke is an approach for mistake-proofing process by using automatic devices or methods to avoid simple human error. Poka-yoke is focused on two aspects:

- Prediction or recognising that a defect is about to occur and providing a warning.
- Detection or recognizing that a defect has occurred and stopping the process.

Mistake-proofing a service process requires identifying when and where failures generally occur. Once a failure is identified, the source must be found. The final step is to prevent the mistake from occurring through source inspection, selfinspection, or sequential checks.

2.1.4.2.3 Creative Thinking

All improvement approaches require a high degree of creativity. Many tools and techniques for enhancing creative thinking have been developed such as brainstorming which is a useful group problem solving procedure for generating ideas that can be used in developing a solution to a problem.

2.1.4.2.4 Design of Experiments

Design of experiments (DOE), developed by Ronald Fisher in England, dates back to the 1920s. A designed experiment is a test or series of tests that enables the experimenter to compare two or more methods to determine which is better, or determine levels of controllable factors to optimise the yield of a process or minimize the variability of a response variable. Fisher also developed the correct method for analysing designed experiments call analysis of variance (ANOVA). This method breaks up the total variation in the data into components from different sources. DOE provides a powerful tool within the DFSS road map to accomplish breakthrough improvements in products, services or process efficiency and effectiveness by optimising the fulfilment of CTSs, FRs, and DPs. (Basem and David, 2005). DOE is used in research, product and service optimisation setting. The primary objective is usually to extract the maximum amount of unbiased information regarding the factors affecting a process or product from as few observations as possible to minimise cost. DOE techniques are used to uncover the interactive nature of the application that is manifested in higher order interactions which involving three or more factors. To develop an overall DOE, the suggestions are as follows:

- 1. Define the problem and set the objectives
- 2. Select the responses
- 3. Select the factors and levels
- 4. Identify noise variable
- 5. Select the DOE design
- 6. Plan the experiment with regard to resources, supplies, schedule, sample size, and risk assessment.

a. Analysis of Variance (ANOVA)

ANOVA is a methodology to conclude equality of means of multiple populations. The objective of ANOVA is to statistically test the differences between the means of the groups to determine whether they are the same or at least one mean is different. To make this determination, ANOVA partitions the total variability of the data into two parts, the variation between groups and the variation within groups. If the total variation between groups is small compared to the variation within groups, it suggests that the populations are essentially the same. However, a large variation between groups suggests that differences exist in the unknown population means. The variation in the data is computed as a sum of squared (SS) deviations from the appropriate sample mean, and scaled as a variance measure, or mean square (MS). By dividing the mean square between groups by the mean square within groups, an F statistic is computed. If this value is larger than a critical value, F_{crit}, then the data suggest that a difference in means exist. ANOVA steps for a two factor can be determined as follows:

1. Decompose the total variation in the DOE response (y) data to its sources (factor A, factor B, factor A \times factor B interaction and error). The first step of ANOVA is the sum of squares calculation that produces the variation decomposition. The equations are shown as follows:

$$\overline{y}_{i} = \frac{\sum_{j=1}^{b} \sum_{k=1}^{n} y_{ijk}}{bn}$$
 (Row average)

$$\overline{y}_{j} = \frac{\sum_{i=1}^{a} \sum_{k=1}^{n} y_{ijk}}{an}$$
 (Column average)

$$\overline{y}_{ij} = \frac{\sum_{k=1}^{a} y_{ijk}}{n}$$
 (Treatment or cell average)

$$\overline{y} = \frac{\sum_{i=1}^{a} \sum_{j=1}^{b} \sum_{k=1}^{n} y_{ijk}}{abn}$$
 (Overall average)

It can be shown that

$$\sum_{i=1}^{a} \sum_{j=1}^{b} \sum_{k=1}^{n} \left(y_{ijk} - \overline{y} \right)^{2} = bn \sum_{i=1}^{a} \left(\overline{y}_{i} - \overline{y} \right)^{2} + an \sum_{j=1}^{b} \left(\overline{y}_{j} - \overline{y} \right)^{2} + n \sum_{i=1}^{a} \sum_{j=1}^{b} \left(\overline{y}_{ij} - \overline{y}_{i} - \overline{y}_{j} + \overline{y} \right)^{2} + \sum_{i=1}^{a} \sum_{j=1}^{b} \sum_{k=1}^{n} \left(y_{ijk} - \overline{y}y_{ij} \right)^{2}$$

Or simply

 $SS_T = SS_A + SS_B + SS_{AB} + SS_E$

2. Test the null hypothesis with regard to the significance of the factor A mean effect and the factor B mean effect as well as interaction. The actual amount of variability in the response data depends on the data size. A convenient way of expressing this dependence is to say that the sum of squares has degrees of freedom (DF) equal to its corresponding variability source data size reduced by one.

Test for main effect of factor A

H_o: No difference among the mean levels of factor A

H_a: At least two factor A mean levels differ

Test for main effect of factor B

 H_0 : No difference among the mean levels of factor B

H_a: At least two factor B mean levels differ

Test for main effect of factor A \times factor B interaction H₀: Factor A and factor B do not interact in the response mean H_a: Factor A and factor B interact in the response mean

3. Compare the F-test of the mean square of the experimental treatment sources to the error to test the null hypothesis that the treatment means are equal. In the F-test, the F_0 will be compared with F-critical defining the null hypothesis rejection region values with appropriate degree of freedom. If F_0 is larger than the critical value, then the corresponding effect is statistically significant. In ANOVA, a sum of squares is divided by its corresponding degree of freedom to produce mean square that is used in the F-test. An ANOVA is summarized in table 2.5.

Source of	Sum of	Degree of	Mean squares	F _o
variation	squares	freedom		
A	SS _A	a - 1	$MS_A = \frac{SS_A}{a-1}$	$F_0 = \frac{MS_A}{MS_E}$
В	SS _B	b - 1	$MS_{B} = \frac{SS_{B}}{b-1}$	$F_0 = \frac{MS_B}{MS_E}$
AB	SS _{AB}	(a – 1)(b-1)	$MS_{AB} = \frac{SS_{AB}}{(a-1)(b-1)}$	$F_0 = \frac{MS_{AB}}{MS_E}$
Error	SSE	ab(n – 1)	$MS_E = \frac{SS_E}{ab(n-1)}$	2
Total	SS_{T}	abn - 1		

Table 2.5 ANOVA Table

The interaction null hypothesis is tested first by computing the Ftest of the mean square of interaction versus the mean square of error. If the test results in non-rejection of the null hypothesis, then proceed to test the main effects of the factors. If the test results in rejection of the null hypothesis, it can be concluded that the two factors interact in the mean response (y).

Next, test the two null hypothesis that the mean response is the same at each level of factor A and factor B. If one or both tests result in rejection of the null hypothesis, it can be concluded that the factor affects the mean response (y).

b. 2^K Full Factorial Designs

It is sufficient to consider the factors affecting process at two levels. The most intuitive approach to study these factors would be to vary the factors of interest in the full factorial design which is called a 2^{k} with experiment k factors each with two levels, that is, the number of treatment combinations in a two-level full factorial of k factors is $2 \times 2 \dots 2 = 2^{k}$. If there are n replicas of each treatment combination, then the total number of experiment trail is 2^{k} n.

The two-level factorial design is the full factorial design with the least number of runs, an ideal situation for screening experiments. The standard layout for two-level design uses a binary notation with +1 and -1 denoting the high level and the low level, respectively, for each factor. If the experiment has more than two factors, there will be additional in layout matrix. Table 2.6 gives a standard layout for 2^4 factorial experiments. The run number is sequences by standard order, which is featured by the sequence -1 +1 -1 +1 for factor A, -1 -1 +1 +1 for factor B, -1 -1 -1 and +1 +1 +1 for factor C, and so on.

Once layout matrix has been conducted, the next step is contrasts calculation by multiplying the factor column coefficient by the corresponding total and summing them. Next, all effects are computed by following formula:

$$Effect = \frac{Contrast}{2^{K-1}n}$$

where N is the total number of runs

	Factors				Replicas				
Run No.	А	В	С	D	1	2		n	Response Total*
1	-1	-1	-1	-1					(1)
2	1	-1	-1	-1					а
3	-1	1	-1	-1					b
4	1	1	-1	-1					ab
5	-1	-1	1	-1					С
6	1	-1	1	-1					ac
7	-1	1	1	-1					bc
8	1	1	1	-1					abc
9	-1	-1	-1	1					d
10	1	-1	-1	1					ad
11	-1	1	-1	1					bd
12	1	1	-1	1					abd
13	-1	-1	1	1					cd
14	1	-1	1	1					acd
15	-1	1	1	1					bcd
16	1	1	1	1					abcd

Table 2.6 Experiment Layout for a 2⁴ Design

Note: Response total is computed by adding the replica row in a given run

Next, the sum of squares is the basis for the analysis of variance computation. The formula for the sum of square is

$$SS = \frac{Contrast^2}{2^{\kappa}n} = \frac{Contrast^2}{Nn}$$

Then, the ANOVA table is computed to determine whether they are the same or at least one mean is different as described above.

c. Response Surface Method

Response surface methods are used to examine the relationship between one or more response variables and a set of quantitative experimental variables or factors. Response surface methods may be used to find factor setting such as operating conditions that produce the best response and satisfy operating or process specifications, identify new operating conditions that produce demonstrated improvement in product quality, and model a relationship between the quantitative factors and the responses.

It can be determined what design is most appropriate for experiment by considering the number of factors that are of interest, the number of runs that can be performed, adequacy coverage of the experimental region of interest, and the other impacts such as cost, time, or availability of facilities. There are other considerations that make a design desirable depending on problems; therefore, design experiment should be chosen to show consistent performance in the criteria. For example, increasing the order of the design sequentially, performing the experiment in orthogonal block which allow for model terms and block effects to be estimated independently and minimize the variation in the estimated coefficients, rotating the design by providing the desirable property of constant prediction variance at all points that are equidistant from the design centre, and detecting model lack of fit.

Response surface methods can be designed into 2 types of central composite and Box-Behnken design (Minitab Inc, 2003). Central composite design can be created in blocked or unblocked central composite design. Central composite designs consist of 2^k or 2^{k-1} factorial points called cube points, axial points called star points, and centre points. A central composite design with two factors is shown below in figure 2.4. Points on the diagrams represent the experimental runs that are performed.



The points in theThe points in the axial (star)Here, the factorial and axialfactorial potion ofportion of the design are at:portions along with the centredesign are coded to be $(+\alpha,0), (-\alpha,0), (0,+\alpha), (0,-\alpha)$ point are shown. The design-1 and +1centre is at (0,0)

Figure 2.4 Central Composite Design with Two Factors

Central composite designs are recommended when design plan calls for sequential experimentation due to these designs can incorporate information from properly planned factorial experiment. The factorial and centre points may serve as a preliminary stage in a first-order or linear model, but still provide the importance of second-order contribution or curvature. It can build the factorial portion of the design into a central composite design to fit a second-degree model by adding axial and centre points. Central composite designs allow for efficient estimation of the quadratic terms in the second-order model and easy to obtain the desirable design properties of orthogonal blocking and rotatability.

Orthogonally blocked designs allow for model terms and block effects to be estimated independently and minimize the variation in the regression coefficients, while rotatable designs provide the desirable property of constant prediction variance at all points that are equidistant from the design center, thus improving the quality of the prediction. Box-Behnken design is used when performing non-sequential experiments. These designs allow efficient estimation of the first and second order coefficients because Box-Behnken design have fewer design points and less expensive to run than central composite designs with the same number of factors. Box-Behnken design with three factors is shown below in figure 2.5.



Figure 2.5 Box-Behnken Design with Three Factors

Box-Behnken design can also prove useful if the safe operating zone for the process is known, while central composite designs usually have axial points outside the cube. These points may not be in the region of interest, or may be impossible to run because they are beyond safe operating limits. Box-Behnken designs do not have axial point, therefore, all design points fall within safe operating zone. Box-Behnken designs also ensure that all factors are never set at high levels simultaneously.

2.1.5 Control Phase

The control phase focuses on maintaining the improvements, and includes putting tool in place to ensure that the key variables remain within the maximum acceptable ranges under the modified process. The improvement might include establishing the new standards and procedures, training the workforce, and instituting controls to make sure that improvement do not die over time. Controls might be simple as using checklists or periodic status reviews to ensure that proper procedures are followed, or implementing statistical process control charts to monitor the performance of key measures. Control is important for 2 reasons. First, it is the basis for effective daily management of work at all levels of an organisation. Second, long-term improvements cannot be made to a process unless the process is the first brought under control. Any control system has 3 components:

- 1. A standard or goal.
- 2. A means of measuring accomplishment.
- 3. Comparison of actual results with the standard, along with feedback to form the basis for corrective action.

Goals and standards establish the accomplished. These goals and standards are reflected by measurable quality characteristics. Measurements supply the information concerning what has actually been accomplished. Workers, supervisors, or managers then assess whether the actual results meet the goals and standards. If not, then corrective action must be taken.

In many industries, data are collected through some type of manual inspection process. Such processes rely on visual interpretation of product characteristics or manual reading of gauges and instruments and may encounter error rates of from 10 to 50 percent. This high rate occurs for several reasons:

- Complexity: The number of defects caught by an inspector decreases with more parts and less orderly arrangement.
- 2. Defect rate: When the product defect rate is low, inspectors tend to miss more defects than when the defect rate is higher.
- 3. Inspection rate: The inspector's performance degrades rapidly as the inspection rate increases.

Short-term corrective action should be taken by those who own the process and are responsible for doing the work. Long-term corrective action is the responsibility of management. The responsibility for control can be determined by checking the 3 components of control systems.

2.1.5.1 Statistical Process Control

Statistical process control (SPC) is a methodology for monitoring a process to identify special cause of variation and signals the need to take corrective action when it is appropriate. When special causes are present, the process is considered as out of control. If the variation in the process is due to common causes alone, the process is considered as in statistical control. A practical definition of statistical control is that both the process averages and variances are constant over time.

2.1.5.1.1 SPC Metrics

Measures and indicators used in SPC fall into one of two categories. An attribute is a performance characteristic that is either present or absent in the product or service under consideration. Attribute measurements are typically expressed a proportions or rates, for example, the fraction of non-conformances in a group of items, number of defects per unit, or rate of errors per opportunity.

The second type of performance characteristic is called a variable which are continuous. Variable measurements are concerned with the degree of conformance to specifications. Variable measurements are generally expressed with such statistics as average and standard deviation.

Collecting attribute data is usually easier than collecting variable data because the assessment can usually be done more quickly by a simple inspection or count, whereas variable data require the use of some type of measuring instrument. In a statistical sense, attribute inspection is less efficient than variable inspection due to it does not provide as much information. This difference means that attribute inspection requires a larger sample than variable inspection to obtain the same amount of statistical information.

2.1.5.1.2 Run Charts and Control Charts

Control chart provides a visual representative of the states of control of a process over time. Control charts are an extension of simple run charts, which are line graphs in which data are plotted over time. The vertical axis represents a measurement while the horizontal axis is the time scale.

The first step in constructing a run chart is to identify the measurement or indicator to be monitored. Constructing the chart consists of the following steps:

Step 1: Collect the data. If samples are chosen, compute the relevant statistic for each sample, such as the average or proportion.

Step 2: Examine the range of the data. Scale the chart so that all data can be plotted on the vertical axis. Provide additional room for new data as they are collected.

Step 3: Plot the points on the chart and connect them. Use graph paper if the chart is constructed by hand; a spreadsheet program is preferable.

Step 4: Compute the average of all plotted points and draw it as a horizontal line through the data. This line denoting the average is called the centre line (CL) of the chart.

Run charts can be used as a basic control mechanism. If the plotted points fluctuate in a stable pattern around the centre line with no large shifts, they indicate that the process is apparently under control. If unusual patterns exist, then the cause for lack of stability should be investigated and corrective action should be taken. However, run charts lack a statistical basis for drawing such as conclusions.

A control chart is simply a run chart to which two horizontal line, called control limits are added: the upper control limit (UCL) and lower control limit (LCL), as illustrated in figure 2.6. Control limits are chosen statistically to provide a high probability (generally greater than 0.99) that points will fall between these limits if the

process is in control. Control limits make it easier to understand patterns in a run chart and draw conclusions about the state of control.



Figure 2.6 Structure of a Control Chart (Evans and Lindsay, 2005: 201)

If sample values fall outside the control limits or if non-random patterns occur in the chart, the special causes may be affecting the process, the process is not stable. The process should be examined and corrective action. If evaluation and correction are done in real time, then the chance of producing nonconforming product is minimised. Thus, as a control tool, control chart allow process owners to identify problems as they occur. Control charts alone cannot determine the source of the problem. Operators, supervisors, and engineers may have to resort to other problem-solving tools to seek the root cause.

Control charts can provide a feedback loop in the DMAIC process. After a process has been improved, a control chart can help to identify further opportunities for improving performance and reducing variation.

2.1.5.2 Constructing and Using Control Charts

Control Charts can be used for 3 purposes:

1. To help identify special causes of variation and establish a state of statistical control.

- 2. To monitor a process and signal when the process goes out of control.
- 3. To determine process capability.

The following is a summary of the steps required to develop and use control charts.

- 1. Preparation
 - a. Choose the variable or attribute to be measured.
 - b. Determine the basis, size, and frequency of sampling.
 - c. Set up the control chart.
- 2. Data collection
 - a. Record the data.
 - b. Calculate relevant statistics: averages, ranges, proportions, and so on.
 - c. Plot the statistics on the chart.
- 3. Determination of trial control limits
 - a. Draw the centre line (process average) on the chart.
 - b. Compute the upper and lower control limits.
- 4. Analysis and interpretation
 - a. Investigate the chart for lack of control.
 - b. Eliminate out of control points.
 - c. Re-compute control limits if necessary.
- 5. Use as a problem-solving tool
 - a. Continue data collection and plotting.
 - b. Identify out of control situations and take corrective action.
- 6. Determination of process capability using the control chart data

2.1.5.2.1 Control Charts for Variable Data

The charts most commonly used for variable data are the \overline{x} - chart (x-bar chart) and the *R*-chart (range chart). The \overline{x} -chart is used to monitor the

centring of the process, and the R-chart is used to monitor the variation in the process. The range is used as a measure of variation simply for convenience performing control chart calculations by hand. For large samples and when data are analyzed by computer programs, the standard deviation is a better measure of variability.

The first step in developing \bar{x} - and *R*-charts is to gather data. Usually, about 25 to 30 samples are collected. Samples between size 3 and 10 are generally used, with 5 being the most common. The number of samples is indicated by *k*, and *n* denotes the sample size. For each sample *i*, the mean, denoted (\bar{x}_i), and the range (R_i) are computed. These values are then plotted on their respective control charts. Next, the overall mean and average range calculations are made. These values specify the centre lines for \bar{x} - and *R*-charts, respectively. The overall mean is the average of the sample means \bar{x}_i :

$$= \frac{\sum_{i=1}^{k} \overline{x}_{i}}{k}$$

The average range is similarly computed, using the formula:

$$\overline{R} = \frac{\sum_{i=1}^{k} R_i}{k}$$

The average range and average mean are used to compute control limits for the *R*- and \bar{x} -charts. Control limits are easily calculated using following formulas:

$$UCL_{R} = D_{4}\overline{R} \qquad UCL_{\overline{x}} = \overline{x} + A_{2}\overline{R}$$
$$LCL_{R} = D_{3}\overline{R} \qquad LCL_{\overline{x}} = \overline{x} - A_{2}\overline{R}$$

Where the constants D_3 , D_4 , and A_2 depend on the sample size and can be found in table 2.7.

x-charts				s-charts				R-charts						
n	Α	A_2	A_{3}	C4	B3	B_4	B_5	B_6	<i>d</i> ₂	d₃	D_1	D_2	D3	D_4
2	2.121	1.880	2.659	0.7979	0	3.267	0	2.606	1.128	0.853	0	3.686	0	3.267
3	1.731	1.023	1.954	0.8862	0	2.568	0	2.276	1.693	0.888	0	4.358	0	2.574
4	1.500	0.729	1.628	0.9213	0	2.266	0	2.088	2.059	0.880	0	4.698	0	2.282
5	1.342	0.577	1.427	0.9400	0	2.089	0	1.964	2.326	0.864	0	4.918	0	2.114
6	1.225	0.483	1.287	0.9515	0.030	1.970	0.029	1.874	2.534	0.848	0	5.078	0	2.004
7	1.134	0.419	1.182	0.9594	0.118	1.882	0.113	1.806	2.704	0.833	0.204	5.204	0.076	1.924
8	1.061	0.373	1.099	0.9650	0.185	1.815	0.179	1.751	2.847	0.820	0.388	5.306	0.136	1.864
9	1.000	0.337	1.032	0.9690	0.239	1.761	0.232	1.707	2.970	0.808	0.547	5.393	0.184	1.816
10	0.949	0.308	0.975	0.9727	0.284	1.716	0.276	1.669	3.078	0.797	0.687	5.469	0.223	1.777
11	0.905	0.285	0.927	0.9754	0.321	1.679	0.313	1.637	3.173	0.787	0.811	5.535	0.256	1.744
12	0.866	0.266	0.886	0.9776	0.354	1.646	0.346	1.610	3.258	0.778	0.922	5.594	0.283	1.717
13	0.832	0.249	0.850	0.9794	0.382	1.618	0.374	1.585	3.336	0.770	1.025	5.647	0.307	1.693
14	0.802	0.235	0.817	0.9810	0.406	1.594	0.399	1.563	3.407	0.763	1.118	5.696	0.328	1.672
15	0.775	0.223	0.789	0.9823	0.428	1.572	0.421	1.544	3.472	0.756	1.203	5.741	0.347	1.653
16	0.750	0.212	0.763	0.9835	0.448	1.552	0.440	1.526	3.532	0.750	1.282	5.782	0.363	1.637
17	0.728	0.203	0.739	0.9845	0.466	1.534	0.458	1.511	3.588	0.744	1.356	5.820	0.378	1.622
18	0.707	0.194	0.718	0.9854	0.482	1.518	0.475	1.496	3.640	0.739	1.424	5.856	0.391	1.608
19	0.688	0.187	0.698	0.9862	0.497	1.503	0.490	1.483	3.689	0.734	1.487	5.891	0.403	1.597
20	0.671	0.180	0.680	0.9869	0.510	1.490	0.504	1.470	3.735	0.729	1.549	5.921	0.415	1.585
21	0.655	0.173	0.663	0.9876	0.523	1.477	0.516	1.459	3.778	0.724	1.605	5.951	0.425	1.575
22	0.640	0.167	0.647	0.9882	0.534	1.466	0.528	1.448	3.819	0.720	1.659	5.979	0.434	1.566
23	0.626	0.162	0.633	0.9887	0.545	1.455	0.539	1.438	3.858	0.716	1.710	6.006	0.443	1.557
24	0.612	0.157	0.619	0.9892	0.555	1.445	0.549	1.429	3.895	0.712	1.759	6.031	0.451	1.548
25	0.600	0.153	0.606	0.9896	0.565	1.435	0.559	1.420	3.931	0.708	1.806	6.056	0.459	1.541

Table 2.7 Factors for Control Charts (Evans and Lindsay, 2005: 318)

Source: Adapted from Table 27 of ASTM STP 15D ASTM Manual on Presentation of Data and Control Chart Analysis. © 1979 American Society for Testing and Materials, Philadelphia, PA.

The control limits represent the range between which all points are expected to fall if the process is in statistical control. If any points fall outside the control limits or if any unusual patterns are observed, then some special cause has probably affected the process. The process should be studied to determine the cause. If special causes are present, then they are not representative of the true state of statistical control and the calculations of the centre line and control limits will be biased. The corresponding data points should be eliminated and new values for \overline{x} , \overline{R} , and the control limits should be computed.

In determining whether a process is in statistical control, the *R*-chart is always analysed first. Because the control limits in the \bar{x} -chart depend on the average range, special causes in the *R*-chart may produce unusual patterns in the \bar{x} -charts, even when the centring of the process is in control.

2.1.5.2.2 Interpreting Patterns in Control Charts

When the process is in statistical control, the points on a control chart fluctuate randomly between the control limits with no recognisable pattern. The following checklist provides a set of general rules for examining a process to determine whether it is in control.

- 1. No points are outside control limits.
- 2. The number of points above and below the centre line is about the same.
- 3. The points seem fall randomly above and below the centre line.
- 4. Most points, but not all, are near the centre line, and only a few are close to the control limits.

The underlying assumption behind these rules is that the distribution of sample means is normal. The upper and lower control limits are computed to be three standard deviations from the overall mean. Thus, the probability that any sample means fall outside the control limits is small. This probability is the origin of rule

one. Because the normal distribution is symmetric, about the same number of points fall above as below the centre line. Also, because the mean of the normal distribution is the median, about half the points fall on either side of the centre line.

Finally, about 68 percent of a normal distribution falls within one standard deviation of the mean. Thus, most points should be close to the centre line. These characteristics will hold provided that mean and variance of the original data have not changed during the time the data were collected, the process is stable. The most common indicators of an out of control condition are summarised below.

a. One Point Outside Control Limits

A single point outside the control limits is usually produced by a special cause. The *R*-chart provides a similar indication. However, such points are a normal part of the process and occur simply by chance. A common reason for a point falling outside a control limit is an error in the calculation of \overline{x} or *R* for the sample. Other possible causes are sudden power surge, a broken tool, measurement error, or an incomplete or omitted operation in the process.

b. Sudden Shift in the Process Average

An unusual number of consecutive points falling on one side of the centre line are usually an indication that the process average has suddenly shifted. Typically, this occurrence is the result of an external influence that has affected the process, which would be considered as a special cause. In both the \bar{x} - and *R*-charts, possible causes might be a new operator, a new inspector, a new machine setting, or a change in the setup or method.

If the shift is up in the *R*-chart, the process has become less uniform. Typical causes are carelessness of operators, poor or inadequate maintenance, or possibly a fixture in need of repair. If the shift is down in the *R*-chart, the uniformity of the process has improved. This shift might be the result of improved workmanship or better machines or materials. Three rules of thumb are used for early detection of process shifts. A simple rule is that if eight consecutive points fall on one side of the centre line, one could conclude that mean has shifted. Second, divide the region between the centre line and each control limit into three equal parts. Then if two of three consecutive points fall in the outer one-third region between the centre line and one of the control limits or four of five consecutive points fall within the outer two-thirds region, one would also conclude that the process has gone out of control.

c. Cycles

Cycles are short which repeated patterns in the chart. These patterns are the result of causes that come and go on a regular basis. In the \bar{x} -chart, cycles may be the result of operator rotation or fatigue at the end of a shift, different gauges used by different inspectors, seasonal effects such as temperature or humidity, or differences between day and night shifts. In the *R*-chart, cycles can occur from maintenance schedules, rotation of fixtures or gauges, differences between shifts, or operator fatigue.

d. Trends

A trend is the result of some cause that gradually affects the quality characteristics of the product and causes the points on a control chart to gradually move up or down from the centre line. For example, a new group of operators gains experience on the job or maintenance of equipment improves over time, a trend may occur. In the \bar{x} -chart, trends may be the result of improving operator skills, dirt or chip build up in fixtures, tool wear, changes in temperature or humidity, or aging of equipment. In the *R*-chart, an increasing trend may be due to a gradual decline in material quality, operator fatigue, loosening of a fixture or a tool, or dulling of a tool. A decreasing trend often is the result of improved operator skill or work methods, better materials, or improved or more frequent maintenance.

After a process is determined to be in control, the charts should be used on a routine basis to monitor performance, identify any special causes that might arise, and make corrections only a necessary. Control charts indicate when to take action, and move importantly, when to leave a process alone.

The data in a control chart may be used to estimate short-term process capability. It is a quick and useful method providing the distribution of the original data. Under the normality assumption, the standard deviation of the original data can be estimated as follows:

$$\hat{\sigma} = \overline{R} / d_2$$

Where d_2 is a constant that depends on the sample size and is also given in table 2.7. Process capability is therefore given by $6\hat{\sigma}$. The natural variation of individual measurements is given by $x \pm 3\hat{\sigma}$.

2.1.5.2.3 Control Charts for Attributes

Attribute data assume only two values – good or bad, pass or fail, and so on. Attributes usually cannot be measured, but they can be observed and counted. Several different types of control charts are used for attribute data. One of the most common is the p-chart. One distinction between the terms defects and defectives must be clearly understood. A defect is a single nonconforming quality characteristic of an item. An item may have several defects. The term defective refers to items having one or more defects. Because certain attribute charts are used for defectives while others are used for defects, one must understand the difference. The term nonconforming is often used instead of defective.

A p-chart monitors the proportion of nonconforming items produced in a lot. Often it is also called a fraction nonconforming or fraction defective chart. As with variable data, a p-chart is constructed by first gathering 25 to 30 samples of the attribute being measured. The size of each sample should be large enough to have several nonconforming items. If the probability of finding a nonconforming item is small, a large sample size is usually necessary. Suppose that *k* samples, each of size *n*, are selected. If *y* represents the number nonconforming in a particular sample, the proportion nonconforming is y/n. Let p_i be the fraction nonconforming in the *i*th sample; the average fraction nonconforming for the group of *k* samples then is

$$\overline{p} = \frac{p_1 + p_2 + \dots + p_k}{k}$$

This statistic reflects the average performance of the process. One would expect a high percentage of samples to have a fraction nonconforming within three standard deviations of \overline{p} . An estimate of the standard deviation is given by

$$s_{\overline{p}} = \sqrt{\frac{\overline{p}(1-\overline{p})}{n}}$$

Therefore, upper and lower control limits are given by

$$UCL_{p} = \overline{p} + 3s_{\overline{p}}$$
$$LCL_{p} = \overline{p} - 3s_{\overline{p}}$$

If LCL_p is less than zero, a value of zero is used.

Analysis of a p-chart is similar to that of an \bar{x} - or *R*-chart. Points outside the control limits signify an out of control situation. Patterns and trends should be sought to identify special causes. However, a point on a p-chart below the lower control limit or the development of trend below the centre line indicates that the process might have improved based on an ideal of zero defectives. Caution is advised before such conclusions are drawn, because errors may have been made in computation.

2.1.5.2.4 Attributes Charts with Variable Sample Size

Often 100 percent inspection is performed on process output during fixed sampling periods; however, the number of units produced in each sampling period may vary. In this case, the p-chart would have a variable sample size. This variation is to compute a standard deviation for each individual sample. Thus, if the number of observations in the *i*th sample is n_i , control limits are given by

$$\overline{p} \pm 3 \sqrt{\frac{\overline{p}(1-\overline{p})}{n_i}}$$
where $\overline{p} = \sum \text{number nonconforming}}$

$$\sum n_i$$

An alternative approach is to use the average sample size, \overline{n} , to compute approximate control limits. Using the average sample size, the control limits are computed as

$$UCL_{p} = \overline{p} + 3\sqrt{\frac{\overline{p}(1-\overline{p})}{\overline{n}}}$$
$$LCL_{p} = \overline{p} - 3\sqrt{\frac{\overline{p}(1-\overline{p})}{\overline{n}}}$$

2.1.5.2.5 Other Types of Control Charts

Several alternatives to the popular \overline{x} -, *R*-, and p-charts are available and others are used for different types of data as follows:

a. s-Charts

An alternative to using the *R*-chart along with the \bar{x} -chart is to compute and plot the standard deviation *s* of each sample. The range involves less computational effort and is easier for shop-floor personnel to understand. However, the sample standard deviation is a more sensitive and better indicator of process variability than the range, especially for larger sample sizes. Thus, when tight control of variability is required, s-charts should be used. With the availability of modern calculators and personal computers, the computational burden of computing *s* is reduced or eliminated, and *s* has thus become a viable alternative to *R*-charts.

b. Individual (x) Charts

With the development of automated inspection for many processes, manufactures can now easily inspect and measure quality characteristics on every item produced. Hence, the sample size for process control is n = 1 and a control chart for individual measurements which also called an x-chart can be used. Other

examples in which x-charts are useful includes accounting data such as shipments, orders, absences, and accidents; production records of temperature, humidity, voltage, or pressure; and the results of physical or chemical analyses. However, samples of size 1 do not furnish enough information for process variability measurement. Process variability can be determined by using a moving average of ranges or a moving range of n successive observations.

c. np-Charts

Instead of using a chart for the fraction nonconforming (p-chart), an equivalent alternative or a chart for the number of nonconforming items is useful. Such a control chart is called an np-chart. The np-chart is a control chart for the number of nonconforming items in a sample. To use the np-chart, the size of each sample must be constant. Suppose that two samples of sizes 10 to 15 each have four nonconforming items. The fraction nonconforming in each sample is different, which would be reflected in a p-chart. However, an np-chart would indicate no difference between samples. Thus, equal sample sizes are necessary to have a common base for measurement. Equal sample sizes are not required for p-charts because the fraction nonconforming is invariant to the sample size.

The np-chart is a useful alternative to the p-chart because it is often easier to understand for production personnel and the number of nonconforming items is more meaningful than a fraction.

d. Charts for defects

In some situations, one may be interested not only in whether an item is defective but also in how many defects it has. Two charts can be applied in such situations. The c-chart is used to control the total number of defects per unit when subgroup size is constant. If subgroup sizes are variable, a u-chart is used to control the average number of defects per unit. Figure 2.7 provides guidelines for selecting the proper type of chart in a control application.



Figure 2.7 Guidelines for Control Chart Selection (Evans and Lindsay, 2005: 216)

2.2 Literature Review

There have been many of the research studying cost reduction and defect reduction which are listed as below.

1. Narongsak Nanthagasigorn (2000) proposed the cost reduction plan for printed circuit cable assembling industry by using 4 major steps. Measurement step used process analysis information in order to understand current cost for each process by using Methods-Time Measurement technique (MTM-2). Analysis step used MTM-2 to find out the root cause of costing and applied grant chart technique for cost reduction plan. Improve step is to implement plan to production line using Six Sigma philosophy combined with MTM-2. Cost step is to control actual cost. The result indicates that the cost reduction using process analysis yields more efficient and make planning efficiency that it is helpful to understand the source of cost and be able to exactly determine product cost reduction plan.
2. Prasert Ngamviseschaikul (2000) studied improvements in reflective glass production cost system and loss control to identify production cost by product category and controlled by estimated cost. The concepts of loss reduction are also applied to resolve the problem. After implementation it was found that the new production cost system can reflect actual cost by product category. As the result of loss control, the defect occurrences in production process become 7.26% compared the prior loss of 13.81%. The actual process time process increased to 316.42 hours per month instead of 263.33 hours per month or an increasing of 20.16% of the production time.

3. Usanee Thinkohkaew (2002) applied six sigma approaches to reduce defect and causes of failure mode in can production process. It consists 4 phases, which are measurement phase, analyse phase, improvement phase and control phase. In each phase of Six Sigma approach mainly applies the statistical techniques to make decisions. Measurement phase is to determine the repeatability and reproducibility of attribute Gauge R&R study. Key factors are listed by cause and effect diagram and FMEA (Failure Mode and Effects Analysis). Analyse phase is to use statistic to analyse the actual root causes. Improvement phase is to improve the entire key factor to reduce defect and control in the acceptance level by control phase. The sigma level improves from 2.85 to be 2.986. In each day, the average defect which occurred from inspection process was around 1,200 DPM. If unnecessary inspection is reduced, the defect will decrease 50% and remain only 2000 DPM or sigma level at 3.092. Finally, the variance of processes will be decreased by 1.5sigma, which the sigma level become 4.592.

There have been many of the research using Six Sigma to improve manufacturing processes which are listed as below.

1. Chanchai Bovornchokchai (2002) reduced a number of suspension defect which have Pitch Static Attitude (PSA) out of product specification limit by applying Six Sigma methodology to study factors that influence PSA variation and identify appropriate operative conditions in order to reduce its variation. The measure of improvement in this project is a number of defects in DPPM unit measured, and the current process has 4,456 Defect Part Per Million (DPPM). The step of study will follow 5 phases improvement model of Six Sigma methodology which begins with define phase, measure phase, analyze phase. After finishing analyze phase, laser welding shows the highest standard deviation value of suspension angle and key process input variables (KPIVs) are Y-distance, Diameter of welding point and laser welding voltage, then perform an experiment of these KPIVs in improvement phase and from the experiment, the appropriate operating condition are laser welding voltage 280 volt, Diameter of welding point 0.234 mm. and Y-distance 2.017 mm. Then setting the control system for these KPIVs in control phase which is the last step of Six Sigma improvement model. The data of PSA defect after process improvement show 997 DPPM which is equal to 77.63% of amount of defect before process improvement.

2. Chanat Rojanaburanon (2003) reduced loss reduction in the 4 colour offset printing process. Six Sigma method is used as a process tools in this research, which consists 5 phases. 1. Define phase: to define problem, objective and scope. 2. Measuring phase: to define key process input variable (KPIV) are listed by cause and effect diagram, cause and effect matrix and FMEA and analyze the precision of measurement system. 3. Analyzing phase: to do hypothesis test for screening significant KPIV (4 factors). 4. Improving phase: to use design of experiment (DOE) 2k 3 centre point with 3 replicate to analyze interested KPIV. The experiment results are curvature and improve all the key process input to increase process capability of print contrast. 5. Controlling phase: control the acceptance level with work instruction. Finally, printing machine set up time after improve average are 0.21 Hours/Colour which is equal to 20.92% better than company target is 0.25 hours/colour.

3. Pattara Aryuwat (2003) reduced defect of gramload out of specification and identify the appropriate operative conditions for reducing defects in head stack assembly process. The study has been proceeded according to the 5 phases improvement models of Six Sigma methodology. The results of the process are to determine KPIVs that significantly effect to increase gramload value in head stack assembly process. Four KPIVs have been used to perform and experiment with response surface in improvement phase. It is found that the appropriate average

gramlaod is 2.5 gram, the base plate height is 12.170 millimetre, the 1st key thickness is 2.274 millimetre, the comb tower pin slot gap is 7.655 millimetre for shuttle setting and swaging machine speed is 2,600 rpm. The preliminary experiments are conducted to confirm the results before applying to production line. Finally, the results of statistical analysis are set at the process of control phase. The data of gramload defect after process improvement show 720 DPPM which is equal to 91.88% of amount of defect before process improvement.

There have been many of the research using failure modes and effects analysis to solving production problem which are listed as below.

1. Kittisak Anuraksakul (2002) analysed and reduced defect for automotive body press part by using FMEA technique to improve and reduce the defect. By using such technique for improve and reducing of defects result can be shown as the follows: 1. The draw process the percentage of defects before improve is 2.02% after improve is 0.79%, 0.24% and 0.22% on December 2002, January and February 2003; 2. The trim/pierce the percentage of defects before improve is 2.20% after improve is 0.70%, 0.25% and 0.22% on December 2002, January and February 2003; 3. The separate process the percentage of defects before improve is 2.25% after improve is 1.06%, 0.20% and 0.18% on December 2002, January and February 2003.

2. Piyawat Rattanasupar (2002) developed process standard including standard work instruction, check sheet, and preventive maintenance plan for colour control in tinted products in paint manufacturing. Based on the study, it was found that there are 5 major problems that extremely impact to colour deviation including quality of raw material, precision of tinting formulation, inaccuracy of tinter dispensing machine, insufficiency work instruction, and human error. The results of these problems lead to 2-3 times for colour adjustment. Consequently, it impacts to productivity in production line. The result of analysis by means of using the Cause and Effect Diagram and FMEA technique have leaded to the establishment of the quality assurance system for tinted alkyds products which include standard work instruction, check sheet, and preventive maintenance plan. Based on the result, process time in tinting section reduce from 233

minutes to 147 minute per batch. Moreover, in terms of RPN (Risk Priority Number) improvement, the percentage of RPN for each criteria process comparing between before and after implementation decrease 73% to 95%.

3. Sunchai Paisarn (2004) used FMEA to reduce defect in extruding process of tire manufacturing and to improve processes which have RPN higher value than 100. The improvements improved the Tuber machine and concerned equipments which make the standard of properly method, training, and etc. The results of the improvement operation are the percent defects in extruding process from 26.07% to 14.82%, scrap component are reduced from 2.09% to 0.74%, processing return component are reduced from 25.08% to 11.24%, and RPN are reduced about 29% to 80% from previous RPN.

4. Sunya Sirichanyakul (2004) applied FMEA technique for solving breakage problem in the PP-Band production in the oven in the stretching process and reducing the material loss from breakage. The application of FMEA technique began with forming an FMEA team, which consisted of people from various departments. The team conducted brainstorming to identify all possible causes that could potentially lead to PPband breakage problems with the aid of a detailed process flowchart and fishbone diagram. To analyze the real cause of breakage, a three-factor experiment was designed. After the cause of breakage is found, the corrective action is implemented to eliminate the problem. The finding from the experiment revealed that the real cause of the breakage of PP bands at the stretching oven was the bubbles which occurred in chilled water from chillers to quenching bath, which cooled down PP bands drawn from the extruder. Tiny bubble particles would combine to form big ones and touch the surface of PP bands in the bath. This causes abnormal surface of PP bands that can be easily broken during stretching process. After the problem of bubbles was solved, it was found that the breakage problem was eliminated. The results of this research help the case company reduce the material loss from breakage for approximately 265,000 baht a year even though this loss can be recycled. The company also gains benefit from increased productivity and reduced maintenance time.

There have been many of the research using design of experiment which are listed as below.

1. Aik Silavisesrith (2000) determined the suitable conditions of the molar ratio of formalin to melamine crystal, the acid-base indicator of melamine crystal, formalin, and water, and volume of sodium hydroxide for the reactor process in melamine compound process to reduce the variation of melamine compound's curing time. This research starts from selecting the factors which involve the change in volume of sodium hydroxide that has an effect on the curing time. Those factors are the molar ratio of formalin to melamine crystal, the acid-base indicator of melamine crystal, formalin, and water. The factorial designed experiments for the 4 factors are performed and can be concluded that only 2 factors, which are the molar ratio of formalin to melamine crystal and the acid-base indicator of melamine crystal, influence the curing time and there is no interaction effect between the 2 factors. Consequently, the 2 factor factorial designed experiment is employed to find the suitable conditions by using levels of the molar ratio and more replicates. Finally, the confirmation experiment with the hypotheses testing brings about the conclusions that the 2 curing time means and variances in each condition, resulting from the previous experiments, could be reliable to be applied to the melamine compound process. The results of this research can be concluded as the 6 suitable conditions at the reactor. And these suitable conditions will be applied to the company's process, leading to the reduction in the curing time variation from about 30 seconds to about 20 seconds.

2. Tossapol Kiatcharoenpol (1995) studied factors that had effects on lacquering process on tin plate and determine the suitable condition in order to get good quality of lacquering and that the outcome data could be referenced in practice. The principle of design and analysis of experiments was used to study 4 factors which were lacquer types, lacquer film weight, curing temperature and curing time. Lacquer coating was tested in 6 characteristics: flexibility test, scratch resistance test, rub test, blushing resistance test, adhesion test and cooking resistance test. Finally effecting factors and suitable condition were analyzed. The experiment showed important factors affecting the result of flexibility test and rub test which were lacquer types, lacquer film

weight, curing temperature as well as curing time. Factors signify result on scratch resistance test and blushing resistance test were lacquer types, lacquer film weight and curing temperature. Comparing with other factors, the curing time had only little effect. In the result of adhesion test and cooking resistance test, there was no lacquer removal. Referring to the result of experiment in 6 characteristics of lacquer coating, the suitable conditions was lacquer type "Z" with lacquer film weight of 8-9 grams per square meter, the curing temperature of 205 Celsius and holding time of 13 minutes

There have been many of the research using statistical process control to control process or manufacturing industries which are listed as below.

1. Boonsom Prasertakarakul (1996) studied appropriate statistical process control (SPC) method and evaluated of SPC effective in refrigerator compressor factory. According to survey and study in cylinder, piston scotch and slider production line, some production line used SPC but not correct and appropriate. So that, I evaluated machine capability by using CP and evaluated process capability by using CPK, for designing the appropriate SPC. The results of this project are as the follows: 1. Using 2 kinds of SPC for controlling the production process by using X - R Chart 2 stations and using CPS check sheet 11 stations 2. Using 4 kinds of SPC evaluated by using CP or CPK in production lines, using accuracy or checking in checking point, using percentage of defective and using total of production. Summations from SPC are using X - R chart, % defect isn't different, but % total productive is decreased, while using CSP check sheet, checking accuracy is increased.

2. Woraphot Rattanasaengsakulthai (1998) developed SPC for automotive part industry in leaf spring process and to evaluate the effectiveness of SPC in a sample factory. The results for this thesis are as the follows: 1. Using 2 kinds of SPC controlling and monitoring the production process by using X-R chart at 10 stations and using CSP (Continuous Sampling Plan) checksheet at 5 stations 2. Implementing improvement method for 3 processes (punching of centre hole, eye forming and primer coating) using Cp, Cpk in production line and, percent defective product in process for evaluation. From the implementation, it is found that Cp and Cpk are increased and percent defective product in the process is decreased.



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CHAPTER III

SIX SIGMA APPROACH FOR DOP MANUFACTURING PROCESS

3.1 Dioctyl Phthalate Manufacturing Process

Dioctyl Phthalate (DOP) is the most commonly used plasticizer in the PVC industry. It is a colourless high boiling point liquid, highly stable to light and soluble in most common solvents. DOP is also used in lacquers to improve resistance to abrasion and is compatible with ethyl cellulose to improve its low temperature flexibility. DOP manufacturing processes can be divided into 4 parts as follows:

3.1.1 Reaction Part

The procedure for this part can be described as follows:

1. Set up temperature of reactor (R-411/R-412) to 160 °C by opening control valve of hot oil. Hot oil temperature is controlled at 550 °C.

2. Transfer raw materials, Phthalic Anhydride (PA) and Octyl Alcohol (OA), to reactor by using ratio of PA and OA as follows:

- a. 4500 litre of PA,
- b. 16000 litre of OA

Colour of raw materials specification is 25 APHA maximum.

3. After reactor temperature reach 160 °C, finish mono-esterification and start di-esterification. During di-esterification, water will be generated from the reaction and evaporated. Evaporated water and OA will be condensed and returned to the reactor via condenser (E-411/E-412).

- 4. Increase reactor temperature to 190 °C.
- 5. Add catalyst to the reactor. Quantity of catalyst is 5 kg.
- 6. Continuously increase reactor temperature to 215 °C.
- 7. Take sample and check AV and colour.

8. Calculate Molar Ratio (MR). If value of MR is between 2.35 and 2.65, start vacuum pump. If not, take sample and calculate MR every 30 minutes.

9. After start vacuum pump, slightly set vacuum pressure at the reactor for 2 hours.

10. Take sample and check AV. If AV is lower than 0.8 mg KOH/g sample, start stripping process by setting vacuum pressure to 200 torr. If not, take sample and check AV every 30 minutes.

11. Start stripping process about 1 hour.

12. Take sample and check AV before transfer to neutralization tank (T-411A/T-411B).

3.1.2 Neutralization Part

The procedure for this part can be described as follows:

1. Transfer crude DOP from each reactor (R-411/R-412) to neutralization tank (T-411A/T-411B).

2. Open agitator with speed of 60 rpm (round per minute) in neutralization tank.

3. Calculate quantity of sodium hydroxide (NaOH) to neutralize crude DOP as shown in table 3.1

Table 3.1 Quantity of NaOH for Crude DOP Neutralization

Quantity of NaOH (kg)	Acid Value
$1.5 \times AV \times Crude DOP Volume$	0.03 – 0.19
$1.8 \times AV \times Crude DOP Volume$	0.20 – 0.29
$2.0 \times AV \times Crude DOP Volume$	0.30 – 0.50
$2.5 \times AV \times Crude DOP Volume$	> 0.50

Note: AV is the acid value of crude DOP after finish reaction part.

4. Control temperature of neutralization tank at 90 - 99 °C by using cooling water flow to coil inside the tank.

5. Add NaOH as per calculated from table 3.2 and demineralization water to neutralization tank. Quantity of water is 2.0 m^3

6. After finish adding demineralization water, open agitator for 15 minutes.

7. Leave crude DOP and water to completely separate about 3 hours.

8. After crude DOP is settled about 3 hours, drain all water and check quality which is AV, Colour, and Specific Gravity (SG) before crude DOP is transferred to storage tank (T-412).

3.1.3 Distillation Part

The procedure for this step can be described as follows:

1. Transfer crude DOP from storage tank (T-412) to stripping column by feed pump (P-301) to remove the residual volatile component such as water and OA in crude DOP.

2. Feed steam to stripping column and control temperature at 175 – 190 °C.

3. Open vacuum pump (VP-301) and control pressure at 30 torr.

4. Transfer DOP to another storage tank (T-424) and check crude DOP quality which is water, OA and DOP content.

5. During start-up period, crude DOP will be circulated from T-412 to T-424 until crude DOP is meet specification at this part.

3.1.4 Filtration Part

The procedure for this step can be described as follows:

1. Transfer crude DOP from storage tank (T-424) to preparing tank (T-431).

2. Add 15 kg of filter aid and open agitator in T-431

3. Open filtering pump (P-431) to pump crude DOP through filter plate and store in finished product tank (T-433).

4. Check quality of DOP finished product which is AV, Colour, water content,

%OA, %DOP and Resistivity Value (VR) before transfer DOP to storage tank at tank farm.

5. In case of high DOP colour, 20 kg of activated carbon will be added to decolour of DOP finished product.

In the study company, 2 reactors are operated and 6 batches are produced a day. Approximately 2 batches of DOP finished product can be produced a day.

3.2 Define Phase of DOP Manufacturing Process

The company encounters high DOP finished product colour and low resistivity value problems, which lead to high production cost on reprocessing of nonconforming product.

Reprocessing of high DOP colour can be done by adding activated carbon, while reprocessing of low resistivity value can be done by adding filter aid and replacing filter paper. It also has loss opportunity of production due to reprocessing both high DOP colour and low resistivity value product, which can be determined based on numbers of failure batch.

Failure cost due to reprocessing of high DOP colour and low resistivity value material and loss opportunity of production can be summarized in table 3.2 as follows:

(anno	1555 M				Total per
	Jan	Feb	Mar	Average	Quarter
Number of production, batch	70	67	57	65	194
Production consumption, m ³	2,100	2,010	1,710	1,940	5,820
High colour product consumption, m ³	30	90	150	90	270
Low VR product consumption, m ³	180	240	30	150	450
Lost production opportunity on reprocessing, m ³	120	180	90	130	390
Net sales, baht	5,806,080	5,557,248	4,727,808	5,363,712	16,091,136
High colour product reprocessing cost, baht	8,560	25,680	42,800	25,680	77,040
Low VR product reprocessing cost, baht	68,822	91,763	11,470	57,352	172,056
Lost production opportunity on reprocessing cost,	331,776	497,664	248,832	359,424	1,078,272
baht		0	16	5	
Total of failure cost, baht	409,158	615,107	303,102	442,456	1,327,368
Percentage of failure cost, baht	7.0%	11.1%	6.4%	8.2%	8.2%

Table 3.2 Failure Cost Production on Reprocessing of Colour and VR Material

Notes:

- 8. Volume of production, high colour product and low VR product per batch is 30 m³
- 9. Lost production opportunity is based on half number of high DOP colour and low VR reprocessing by considering about using half production time to reprocess
- 10. Cost of high DOP colour product reprocessing is based on material cost of activated carbon and high DOP colour product consumption as shown in table 3.3.
- 11. Cost of low VR product reprocessing is based on material cost of filter aid and filter paper and low VR product consumption as shown in table 3.3.
- 12. Labour cost of normal operation and reprocessing are same due to using same operator in shift, therefore, labour cost is not considered.
- 13. Rejected product cost is not considered due to finished product has been checked before selling to customers.
- 14. Percentage of failure cost is determined by net sales and total of failure cost.
- 15. DOP price at 79 USD per metric tonnes (35 Baht/USD).
- 16. Production consumption = Number of production \times Volume of production (30 m³)
- 17. High colour product consumption = Number of batch of high colour \times 30 m³
- 18. Low VR product consumption = Number of batch of low VR \times 30 m³
- 19. Lost production opportunity on reprocessing = (Number of batch of high colour + Number of batch of low VR)/2 × 30 m³ (if half number of batch of high colour and low VR is not integer, round the number up to the nearest integer)
- 20. Net sales = DOP price × Production consumption
- 21. High colour product reprocessing cost = Number of batch of high colour × Activated carbon cost
- 22. Low VR product reprocessing cost = Number of batch of low VR × (Filer aid cost + Filter paper cost)
- 23. Lost production opportunity on reprocessing cost = Lost production opportunity on reprocessing × DOP price
- 24. Total of failure cost = High colour product reprocessing cost + Low VR product reprocessing cost + Lost production opportunity on reprocessing cost
- 25. Percentage of failure cost = Total of failure cost / Net sales × 100

Items	Cost					
Activated Carbon	8,560	baht per batch				
Filter Aid	10,700	baht per batch				
Filter Paper	770	baht per batch				

Table 3.3 Material Cost of DOP Reprocessing

In table 3.2, estimated failure cost due to reprocessing of DOP product per quarter is 1,327,368 baht or 8.2% of net sales. Therefore, the company sets the objectives to reduce the reprocessing cost of DOP product by implementing Six Sigma approach. By making a successful project, project charter has been used to identify business cases, problem statements, goals, team members, and timeline to obtain the commitment from all team members within a specific project and agreement upon scopes and objectives. Project charter is created in table 3.4 as follows:

Business Case:	Failure Cost in Dioctyl Phthalate Manufa	acturing Process is significant and the Six						
	Sigma is an effective approach to reduce	failure cost.						
Problem Statement:	The company encounters high DOP color	ur and low resistivity value problems, which						
	lead to failure cost of 491,760 and 835,6	08 baht, respectively or total failure cost of						
	1,327,368 baht per quarter on reproce	ssing of nonconforming product and lost						
	production opportunity. And also proces	ss capability of DOP colour and resistivity						
	value is lower than 1.33.							
Objective:	Reduce failure cost of reprocessing on nonconforming product due to colour ar							
	resistivity value parameters problem. The	target is to reduce failure cost of 1,327,368						
สถา	to 398,210 baht per quarter or 70% reduction and increase process capabilities to							
	least 1.33 by the first quarter of 2008.							
Team Members:	Production manager	Production department						
N N I 6 N	Process engineer	Production department						
9	DOP operators	Production department						
	QC supervisor	Quality Control department						
	QC operators	Quality Control department						
	Instrument engineer	Maintenance department						
	mechanical engineer	Maintenance department						
	Logistics supervisor	Logistics department						
	Sales representative	Sales department						

Table 3.4 Project Charter

	Milestone	Responsible Person	Date
Define I	Phase		
-	Collect process data	Process engineer	1 – 31 July 2007
		DOP operators	
Measur	e Phase		
-	Identify process capability	Process Engineer	1 August 2007 –
-	Verify measurement system	QC supervisor	30 September 2007
		QC operators	
		Instrument engineer	
		mechanical engineer	
Analyze	Phase		
-	Identify possible causes of the two defect types	All team members	1 October 2007 –
	using Fishbone diagrams.		30 November 2007
-	Prioritize the causes of defects by applying failure		
	modes and effects analysis		
Improve	e Phase		
-	Set up design of experiment	Production manager	1 November 2007 –
-	Determine the effect of factors on process capability	Process engineer	31 December 2007
-	Define the suitable method to improve process	DOP operators	
	capability		
-	Implement into the process		
Control	Phase		
-	Confirm results from design of experiment	Production manager	1 January –
-	Compare process capability and failure costs before	Process engineer	31 March 2008
	and after improvement	DOP operators	
-	Select key parameter and prepare suitable statistical		
	process control chart		

Table 3.4 Project Charter (Cont)

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3.3 Measure Phase of DOP Manufacturing Process

3.3.1 Process Capability of DOP Finished Product

Quality of DOP finished product has been checked before filled into drum and delivered to customers. The results of six specifications of DOP finished product which are Acid Value (AV), Colour, water content, %OA, %DOP and Resistivity Value (VR) are collected and determined process capability as shown in table 3.5.

Items	Specification	Process Capability
Acid Value (AV)	< 0.04 mgKOH/gDOP	3.25
Colour	< 25 APHA	0.92
Water Content	< 0.05%	1.38
%OA	< 0.05%	3.51
%DOP	> 99.5%	5.56
Resistivity Value (VR)	$> 1.0 \times 10^{11} \Omega.cm$	1.03

Table 3.5 Summary of Process Capability of DOP Finished Product

In table 3.5, it shows that process capability (Cpk) for colour of DOP finished product is 0.92 and process capability (Cpk) for resistivity value of DOP finished product is 1.03 which are lower than 1.33, while process capability of other DOP specifications is higher than 1.33. Normally, process capability which is lower than 1.33 has high variation in process, therefore, these process capabilities should be improved and prevented product quality problem which lead to reprocessing of nonconforming product and failure cost problem.

3.3.2 Measurement System Evaluation and Verification

Due to accurate Six Sigma performance depends on reliable measurement systems, measuring quality characteristics generally requires the use of the human senses which are seeing, hearing, feeling, tasting, and smelling and the use of some type of instrument or gauge to measure the magnitude of the characteristic. In the company, there are many types of instrumentation such as pressure gauge, pressure transmitter, temperature gauge, temperature transmitter and flow meter to measure parameters in the process. Calibration of measurement transducers is a vital part of instrument maintenance and should be performed on a regular basis (Gas Processors Association, 2004). To obtain accurate and precise data and to prevent data error from the instrumentation, all instruments have been calibrated annually by instrument and maintenance departments. For instruments in laboratory which cannot be calibrated by our instrument and maintenance due to the regulation of laboratory equipment calibration, all instruments and equipments have been sent to calibrate by certified lab having the international standard of calibration procedure.

For DOP quality measurement which using human senses such as seeing the colour and the values of resistivities, there are procedure to set up the standard to make sure that all operators can check the quality without error. Gage R&R method has been selected to analyse the precision of DOP quality measurement. Ten batches with one sample of DOP have been sampled and checked three times each for the colour and resistivity value by six operators. The results of analysis show in table 3.6 and 3.7.

r				r						r								
Batch	Op	perato	ator 1 Operator 2		Op	Operator 3 Operator 4		Operator 5		Operator 6		or 6						
	С	heck	#	С	heck	#	С	heck	#	С	heck	#	Check #			Check #		
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
1	20	20	20	22	20	20	20	18	20	20	20	20	20	22	20	20	20	20
2	18	18	17	20	18	18	18	18	18	18	18	17	18	18	18	17	18	18
3	15	15	15	15	15	15	15	15	17	15	15	15	17	15	15	15	15	15
4	20	20	20	20	20	20	20	22	20	20	20	22	20	20	20	22	20	20
5	17	17	18	17	17	17	17	17	17	18	17	17	17	17	17	17	17	17
6	18	18	18	17	18	18	18	18	18	18	18	18	18	17	18	18	18	20
7	18	18	18	18	18	18	18	18	17	18	18	18	18	18	18	18	18	18
8	20	22	22	22	22	22	22	22	22	22	22	23	20	22	22	22	22	22
9	20	20	20	22	20	20	20	20	22	20	20	20	20	20	20	20	20	20
10	15	15	15	15	15	15	15	15	15	15	15	15	15	17	15	15	15	15

Table 3.6 The Results of Analysis on DOP Colour

Batch	Operator 1		or 1	Op	perato	or 2	Operator 3		Operator 4		Operator 5		Operator 6					
	С	heck	#	С	heck	#	С	heck	#	Check #		Check #		Check #		#		
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
1	3.24	3.30	3.33	3.30	3.29	3.24	3.29	3.20	3.16	3.20	3.24	3.30	3.16	3.24	3.20	3.29	3.20	3.24
2	2.83	2.71	2.82	2.83	2.92	2.83	2.73	2.83	3.01	2.92	2.82	2.68	2.73	2.82	2.71	2.83	2.82	2.71
3	3.77	3.86	3.81	3.76	3.67	3.77	3.82	3.76	3.76	3.77	3.53	3.63	3.76	3.81	3.82	3.77	3.91	3.86
4	3.16	3.06	2.92	3.06	3.01	3.29	3.18	3.01	3.06	3.06	3.07	3.16	2.92	3.06	3.20	3.06	3.09	3.07
5	2.40	2.36	2.35	2.36	2.40	2.31	2.40	2.35	2.36	2.35	2.36	2.40	2.36	2.35	2.73	2.31	2.54	2.36
6	3.06	2.92	3.18	3.16	3.06	3.07	3.09	3.06	3.01	3.06	3.07	3.01	3.20	3.16	3.06	3.01	2.92	3.29
7	2.82	2.83	2.73	2.83	3.06	2.92	2.82	2.71	2.73	2.59	2.82	2.73	2.71	2.83	2.92	2.82	2.83	2.68
8	3.20	3.30	3.24	3.29	3.20	3.24	3.16	3.20	3.06	3.30	3.29	3.20	3.16	3.30	3.20	3.20	3.24	3.29
9	2.31	2.35	2.40	2.31	2.59	2.36	2.40	2.49	2.35	2.12	2.36	2.35	2.36	2.40	2.20	2.26	2.36	2.40
10	1.64	1.65	1.60	1.53	1.64	1.69	1.41	1.65	1.55	1.51	1.64	1.53	1.60	1.64	1.79	1.65	1.55	1.51

Table 3.7 The Results of Analysis on DOP Resistivity Value

From data in table 3.6 and 3.7, Gage R&R module in Minitab Software was used to analyze the precision of DOP quality measurement by considering Total Gage R&R %Contribution value. The results from Minitab Software are shown as follows:



Gage R&R Study - ANOVA Method

Two-Way ANOVA Table With Interaction

Source	DF	SS	MS	F	P
Batch	9	808.606	89.8451	237.437	0.000
Operator	5	0.917	0.1833	0.485	0.786
Batch * Operator	45	17.028	0.3784	0.987	0.506
Repeatability	120	46.000	0.3833		
Total	179	872.550			

Two-Way ANOVA Table Without Interaction

Source	DF	SS	MS	F	P
Batch	9	808.606	89.8451	235.205	0.000
Operator	5	0.917	0.1833	0.480	0.791
Repeatability	165	63.028	0.3820		
Total	179	872.550			

Gage R&R

	50	Contribution		
Source	VarComp (of VarComp)		
Total Gage R&R	0.38199	7.14		
Repeatability	0.38199	7.14		
Reproducibility	0.00000	0.00		
Operator	0.00000	0.00		
Part-To-Part	4.97017	92.86		
Total Variation	5.35216	100.00		
		Study Var	%Study Var	%Tolerance
Source	StdDev (SD)	(6 * SD)	(%SV)	(SV/Toler)
Total Gage R&R	0.61805	3.7083	26.72	24.72
Repeatability	0.61805	3.7083	26.72	24.72
Reproducibility	0.00000	0.0000	0.00	0.00
Operator	0.0000	0.0000	0.00	0.00
Part-To-Part	2.22939	13.3763	96.37	89.18
Total Variation	2.31347	13.8808	100.00	92.54

Number of Distinct Categories = 5

Figure 3.1 Gage R&R Results on DOP Colour from Minitab

The results from Minitab[™] in figure 3.1 shows that the p-value for Batch * Operator interaction in the ANOVA Table is larger than 0.25 based on criteria suggested by Minitab, therefore, Minitab omits this from the full model and performs the ANOVA Table without the interaction because the p-value is 0.506. The measurement system of DOP colour is acceptable when Total Gage R&R %Contribution is between 1% and 9% referred in Chapter 2 (AIAG, 2002). Due to the percent contribution from Part-to-Part (92.86) is much larger than that of Total Gage R&R (7.14). It means that much of the variation is due to differences between parts. For this data, the number of distinct categories is five (5). It means that the measurement system is acceptable due to it needs at least five (5) distinct categories to have an adequate measuring system (AIAG, 2002).

Gage R&R Study - ANOVA Method

Two-Way ANOVA Table With Interaction

Source	DF	SS	MS	F	P
Batch	9	58.8666	6.54073	932.777	0.000
Operator	5	0.0723	0.01447	2.064	0.088
Batch * Operator	45	0.3155	0.00701	0.903	0.645
Repeatability	120	0.9322	0.00777		
Total	179	60.1867			

Two-Way ANOVA Table Without Interaction

Source	DF	SS	MS	F	P
Batch	9	58.8666	6.54073	864.937	0.000
Operator	5	0.0723	0.01447	1.913	0.095
Repeatability	165	1.2477	0.00756		
Total	179	60.1867			

Gage R&R

	50	Contribution		
Source	VarComp	(of VarComp)		
Total Gage R&R	0.007792	2.10		
Repeatability	0.007562	2.04		
Reproducibility	0.000230	0.06		
Operator	0.000230	0.06		
Part-To-Part	0.362954	97.90		
Total Variation	0.370746	100.00		
		Study Var	%Study Var	%Tolerance
Source	StdDev (SD)	(6 * SD)	(%SV)	(SV/Toler)
Total Gage R&R	0.088274	0.52965	14.50	10.59
Repeatability	0.086960	0.52176	14.28	10.44
Reproducibility	0.015174	0.09105	2.49	1.82
Operator	0.015174	0.09105	2.49	1.82
Part-To-Part	0.602457	3.61474	98.94	72.29
Total Variation	0.608889	3.65334	100.00	73.07

Number of Distinct Categories = 9

Figure 3.2 Gage R&R Results on DOP Resistivity Value from Minitab

The results from MinitabTM in figure 3.2 shows that the p-value for Batch * Operator interaction in the ANOVA Table is larger than 0.25, therefore, Minitab omits this from the full model and performs the ANOVA Table without the interaction because the p-value is 0.645. The measurement system of DOP resistivity value is acceptable when

Total Gage R&R %Contribution is between 1% and 9% referred in Chapter 2 (AIAG, 2002). Due to the percent contribution from Part-to-Part (97.90) is much larger than that of Total Gage R&R (2.10). It means that much of the variation is due to differences between parts.

For this data, the number of distinct categories is nine (9). It means that the measurement system is acceptable due to it needs at least five (5) distinct categories to have an adequate measuring system (AIAG, 2002).

3.4 Analysis Phase of DOP Manufacturing Process

3.4.1 Identification of Causes of Defects

According to the estimated failure cost of DOP reprocessing product per quarter of 1,327,368 baht or 8.2% of profit, the company has set up Six Sigma team from each related discipline to identify and evaluate the DOP product problem. Team members include the following personnel:

- Production manager, process engineer, and DOP operators from Production department
- 2. QC supervisor and QC operators from Quality Control department
- 3. Instrument engineer and mechanical engineer from Maintenance department
- 4. Logistics supervisor from Logistics department
- 5. Sales representative from Sales department

All representatives from all departments conduct the meeting to brainstorm and prepare cause and effect diagrams, known as fishbone diagrams in order to list out the potential causes of two types of defect, which are DOP high color and DOP low resistivity value. Normally, fishbone diagrams can be divided into 6 causes which are people, methods, materials, machines, measurements, and environment. Fishbone diagrams are separated into 2 diagrams for each defect type. The diagrams can be shown in figure 3.3 and 3.4.



Figure 3.3 Fishbone Diagrams for DOP High Colour



Figure 3.4 Fishbone Diagrams for DOP Low Resistivity Value

After preparing Fishbone diagrams, the team then identifies key failure causes and rank each failure cause listed in table 3.8 and 3.9.

Potential Failure Causes	Key(Yes/No)	Description
Reactor temperature too high	Yes	High potential to change reactor
/ low	Sollin.	temperature range from 210 – 220 $^{\circ}$ C
Hot oil temperature too high /	Yes	High potential to change hot oil
low		temperature range from 530 – 550 °C
Quantity of catalyst too low	Yes	High potential to change quantity of
		catalyst range from 5 – 10 kg
Ratio of Material (PA & OA)	No	Excess quantity of OA
too high		
High raw material (PA & OA)	Yes	Currently set at maximum standard
colour	a ha man	colour of 25 APHA. High potential to
	228/23/2 Ju	reduce from 25 to 20 APHA
Low quality of catalyst	Yes	Low quality of catalyst causes high colour
Reactor seal leakage	No	Follow maintenance schedule every 3
8		months to check reactor leakage
Data not filled correctly	No	Follow production procedure and do
		review / check by supervisor
Lack of monitoring and	No	Follow production procedure and do
controlling	29115	review / check by supervisor
Lack of training	No	Set up internal training to improve
		operator performance
Inaccuracy and Imprecision	No	Perform measurement evaluation and
of measurement		verification to prevent errors
Storage condition on	No	Check storage area periodically ex. roof
temperature and moisture		and ventilation

Table 3.8 List of Key Potential Failure Causes of DOP High Colour

Potential Failure Causes	Key(Yes/No)	Description
Neutralization Temperature	Yes	High potential to change neutralization
too high / low		temperature range from 90 – 99 $^{\circ}$ C
Quantity of NaOH too low	No	Excess quantity of NaoH based on Acid
		Value in table 3.1
Quantity of water too low	Yes	High potential to change quantity of
		water range from 2.0 – 3.0 m^3
Agitator speed too high / low	No	Limitation on agitator speed modification
Agitator time too high / low	Yes	High potential to change agitator time
		range from 10 – 20 minutes
Settling time too low	Yes	High potential to change settling time
		range from 2 – 4 hours
Quantity of filter aid too low	Yes	High potential to change quantity of filter
	a ha Omi	aid range from 10 – 20 kg
Differential pressure of filter	Yes	High potential to change differential
too high	6464889777	pressure of filter to prevent leakage of
		filtration
Low density of filter paper	Yes	Low density of filter paper causes low
		performance of filtration leading to
		increase resistivity value
Low quality of filter aid	Yes	Low density of filter aid causes low
6161 I U	2112	performance of filtration leading to
ລາທາລາຄ	ະກິງທ	increase resistivity value
Agitator blade design	No	Limitation on agitator blade design
		modification
Filter type	No	Limitation on filter type modification
Data not filled correctly	No	Follow production procedure and do
		review / check by supervisor
Lack of monitoring and	No	Follow production procedure and do
controlling		review / check by supervisor

Table 3.9 List of Key Potential Failure Causes of DOP Low Resistivity Value

Potential Failure Causes	Key(Yes/No)	Description
Lack of training	No	Set up internal training to improve
		operator performance
No accuracy and precision	No	Perform measurement evaluation and
of measurement		verification to prevent errors
Storage condition on	No	Check storage area periodically ex. roof
temperature and moisture		and ventilation

Table 3.9 List of Key Potential Failure Causes of DOP Low Resistivity Value (Cont)

3.4.2 Failure Mode and Effects Analysis

This research used failure mode and effects analysis (FMEA) worksheet to evaluate the potential and actual effects of failure of DOP colour and resistivity value referred in Chapeter 2, section 2.1.3.2.6 (Basem and David, 2005 and AIAG, 2002).

In severity rating table, it is used from Chapter 2 (Basem and David, 2005) as follows:

Effect	Severity of Effect Defined	Rating
None	No effect.	1
Very Minor	Very minor effect on product quality and/or reduced level of process	2
	performance. Product may have to be reprocessed.	
Minor	Minor effect on product quality and/or reduced level of process	3
	performance. Product may have to be reprocessed.	
Very Low	Very low effect on product quality and/or reduced level of process	4
0019	performance. Product may have to be reprocessed.	
Low	Low effect on product quality and/or reduced level of process	5
9	performance. Product may have to be reprocessed.	
Moderate	Moderate effect on product quality and/or reduced level of process	6
	performance. Product may have to be reprocessed.	
High	High effect on product quality and/or reduced level of process	7
	performance. Product may have to be reprocessed.	

Effect	Severity of Effect Defined	Rating
Very High	Very high effect on product quality and/or equipment damaged.	8
	Product can not achieve their specification. They are treated as	
	waste.	
Serious	Potential Hazardous effect. Able to stop production without mishap;	9
	safety related. Disruption to subsequent process operation. Failure	
	mode involves non-compliance with government regulation.	
Hazardous	Hazardous effect. Safety related – sudden failure in process	10
	production. Failure mode involves non-compliance with government	
	regulation.	

In occurrence rating table, it is scaled based on the failure rate extracted from historical data and percentage of occurrence is used from Chapter 2 (Basem and David, 2005) as follows:

Table 3.11	Occurrence	Rating
------------	------------	--------

Probability of Failure	Occurrence	Rating
Almost Certain	Failure almost certain. It is inevitable. History of failures exists	10
C	from previous or similar design. (1 in 2 or 50%)	
Very High	Very high number of failure likely. (1 in 10 or 10%)	9
High	High number of failure likely. (1 in 20 or 5%)	8
Moderately High	Moderately high number of failure likely. (1 in 50 or 2%)	7
Moderate	Moderate number of failure likely. (1 in 100 or 1%)	6
Low	Low number of failure likely. (1 in 500 or 0.2%)	5
Very Low	Very low number of failure likely. (1 in 2,000 or 0.05%)	4
Remote	Remote number of failure likely. (1 in 5,000 or 0.02%)	3
Very Remote	Very remote number of failure likely. (1 in 20,000 or 0.005%)	2
Absolute	Failure unlikely. History shows no failures. (1 in 10 ⁶ or 0.001%)	1
Uncertainty		

Detection rating table is described more details about likelihood of detection and shown in table 3.12 as follows:

Detection	Likelihood of Detection	Rating
Almost Certain	Design control will almost certainly detect a potential	1
	cause/mechanism and subsequent failure mode. There are	
	automatic controller and low/high alarm device to control and	
	monitor process parameters including shut-down the system.	
	Historical data is automatically recorded in the system.	
Very High	Very high chance the design control will detect a potential	2
	cause/mechanism and subsequent failure mode. There are	
	automatic controller and low/high alarm device to control and	
	monitor process parameters. Historical data is automatically	
	recorded in the system.	
High	High chance the design control will detect a potential	3
	cause/mechanism and subsequent failure mode. There are	
	automatic controller to control and monitor process parameters.	
	Historical data is automatically recorded in the system	
Moderately High	Moderately high chance the design control will detect a potential	4
6	cause/mechanism and subsequent failure mode. There are	
	automatic controller to control and monitor process parameters.	
	Historical data is recorded by operator.	
Moderate	Moderate chance the design control will detect a potential	5
	cause/mechanism and subsequent failure mode. Semi-automatic	
ыв	controller to control process parameters. Semi-automatic	
00000	recorder to record process parameters.	
Low	Low chance the design control will detect a potential	6
9	cause/mechanism and subsequent failure mode. Semi-automatic	
	controller to control process parameters. Process parameters are	
	recorded by operator.	
Very Low	Very low chance the design control will detect a potential	7
	cause/mechanism and subsequent failure mode. No automatic	
	controller to control process parameters. Process parameters are	
	manually controlled and recorded by operator.	

Detection	Likelihood of Detection	Rating
Remote	Remote chance the design control will detect a potential	8
	cause/mechanism and subsequent failure mode. No	
	automatic controller to control process parameters.	
	Process parameters are manually controlled by	
	operator. No historical data record	
Very Remote	Very remote chance the design control will detect a	9
	potential cause/mechanism and subsequent failure	
	mode. No controller to control process parameters.	
Absolute	Design control will not and/or can not detect a potential	10
Uncertainty	cause/mechanism and subsequent failure mode; or	
	there is no design control. No controller to control	
	process parameters. No historical data record	

Table 3.12 Detection Rating

Once setting up the severity, occurrence, and detection rating table above, team rank each table by investigating in enough detail, providing a description and getting consensus understanding of what is actually taking place. The severity rating of DOP finished product on failure cause of high colour and low resistivity values is considered as rating number 7 due to both failure causes are high effect on product quality and have to be reprocessed.

For the occurrence and detection rating table, the team ranks each criterion in table 3.13 and 3.14. The occurrence rating of DOP finished product on each failure cause can be shown in table 3.13.

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Potential Failure Causes		Occurrence				
	Jan	Feb	Mar	%		
High Colour						
Reactor temperature too high/low	0	0	1	0.52	5	
Hot oil temperature too high/low	1	1	1	1.55	6	
Quantity of catalyst too low	0	0	1	0.52	5	
High PA raw material colour	1	2	3	3.09	7	
High OA raw material colour	0	0	0	0.00	1	
Low quality of catalyst	0	0	0	0.00	1	
Low Resistivity Value						
Neutralisation temperature too	1	2	0	1.55	6	
high/low						
Quantity of water too low	1	0	0	0.52	5	
Agitator time too high/low	2	3	1	3.09	7	
Settling time too low	1	2	1	2.06	7	
Quantity of filter aid too low	0	0	0	0.00	1	
Differential pressure of filter too	0	0	0	0.00	1	
high						
Low density of filter paper	0	0	0	0.00	1	
Low quality of filter aid	0	0	0	0.00	1	

Table 3.13 Occurrence Rating of DOP Finished Product

Notes: 1. Causes of high DOP colour and low resistivity values are possible to be more than one cause.

2. The number of batches is 194 batches.

The percentage of occurrence is determined from the number of occurrence for each potential failure cause divided by the number of DOP production. In this research, collecting data on DOP production started from January to March 2007 which is equal to 194 batches. For example, percentage of occurrence on reactor temperature is $1 / 194 \times 100 = 0.52\%$.

The detection rating of DOP finished product on each failure cause can be shown in table 3.14.

Potential Failure Causes	ses Detection			
High Colour				
Reactor temperature too	Use control valve to control temperature at 215 °C.	3		
high/low	Temperature profile is automatically recorded by			
	the system.			
Hot oil temperature too high	Use control valve to control temperature at 550 °C.	3		
low	Temperature profile is automatically recorded by			
	the system.			
Quantity of catalyst too low	Use operator to fill up quantity of catalyst by adding	7		
	5 kg per batch. Quantity of catalyst is recorded by			
	operator.			
High PA raw material colour	Follow quality control system to check quality of raw	5		
	material based on raw material specification at 25			
	APHA maximum. It is considered as semi-automatic			
	control system.			
High OA raw material colour	Follow quality control system to check quality of raw	5		
	material based on raw material specification at 25			
	APHA maximum. It is considered as semi-automatic			
	control system.			
Low quality of catalyst	Follow quality control system to check quality of	5		
สถาเ	catalyst. It is considered as semi-automatic control			
	system.			

Table 3.14 Detection Rating of DOP Finished Product



Potential Failure Causes	Detection	Rating
Low Resistivity Value		
Neutralisation temperature too	Use control valve to control temperature at 90-99	4
high/low	^o C. Temperature profile is recorded by operator.	
Quantity of water too low	Use operator to fill up quantity of water by adding	7
	2.0 m ³ per batch. Quantity of water is recorded by	
	operator.	
Agitator time too high/low	Use operator to control agitator time of 15 minutes.	7
	Agitator time is recorded by operator.	
Settling time too low	Use operator to control settling time of 3 hours.	7
	Settling time is recorded by operator.	
Quantity of filter aid too low	Use operator to fill up quantity of filter aid by adding	7
	15 kg per batch. Quantity of filter aid is recorded by	
	operator.	
Differential pressure of filter too	Use operator to monitor differential pressure of filter	7
high	at 0.6 kg/cm ² maximum. Differential pressure of filter	
	is recorded by operator.	
Low density of filter paper	Follow quality control system to check quality of	5
	filter paper. It is considered as semi-automatic	
	control system.	
Low quality of filter aid	Follow quality control system to check quality of	5
	filter aid. It is considered as semi-automatic control	
	system.	

Table 3.14 Detection Rating of DOP Finished Product (Cont)

After finishing the ratings of severity, occurrence and detection scores, the team performs FMEA table in table 3.15 as follows:

Sheet 1 of 2	Actions Recommended	Set up new reactor temperature in range of 210 - 220 °C based on recommendation of original design engineering.	Set up new hot oil temperature in range of 530 - 550 °C based on capability of control range.	Set up new quantity of catalyst in range of 5 - 10 kg based on recommendation of catalyst vendor and use log book to check back of catalyst usage with warehouse.	Set up new raw material specification if possible	Keep control at current specification	Keep control at current specification			
	ччz	105	126	245	245	35	35			
	Ош⊢	3	3	7	5	5	5			
	Current Controls	Use control valve to control temperature at 215 °C. Temperature profile is automatically recorded by the system.	Use control valve to control temperature at 550 °C. Temperature profile is automatically recorded by the system.	Use operator to fill up quantity of catalyst by adding 5 kg per batch. Quantity of catalyst is recorded by operator.	Follow quality control system to check quality of raw material based on raw material specification at 25 APHA maximum. It is considered as semi- automatic control system.	Follow quality control system to check quality of raw material based on raw material specification at 25 APHA maximum. It is considered as semi- automatic control system.	Follow quality control system to check quality of catalyst. It is considered as semi-automatic control system.			
	000	5	9	5	7	-				
	Potential Causes	Reactor temperature too high/low	Hot oil temperature too high/low	Quantity of catalyst too low	High PA raw material colour	High OA raw material colour	Low quality of catalyst			
	с ш >			٦						
	Potential Failure Effect	ิลเ ไาลง	Defect and reprocess							
	Potential Failure Mode		High Colour							
	Process Step			DOP finished	product					

FAILURE MODE AND EFFECTS ANALYSIS WORKSHEET

Table 3.15 Failure Mode and Effects Analysis Table for DOP Product

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Actions Recommended	Set up new neutralization temperature in range of 90 – 99 °C based on recommendation of original design engineering.	Set up new quantity of water in range of 2.0 - 3.0 m3 to clean crude DOP in neutralisation tank and use stop watch to record the time.	Set up new agitator time in range of 10 - 20 minutes to reduce emulsion formation and to ensure that NaOH and crude DOP are mixed together nd use stop watch to record the time.	Set up new settling time in range of 2 -4 hours to allow crude DOP more settling and reduce emulsion carry over to downstream nd use stop watch to record the time.	Keep control at current specification	Keep control at current specification	Keep control at current specification	Keep control at current specification		
α σ z	168	245	343	343	49	49	35	35		
∩ш⊢	• 4	~	~	2	2	2	5	ى ك		
Current Controls	Use control valve to control temperature at 90-99 °C. Temperature profile is recorded by operator.	Use operator to fill up quantity of water by adding 2.0 m3 per batch. Quantity of water is recorded by operator.	Use operator to control agitator time of 15 minutes. Agitator time is recorded by operator.	Use operator to control settling time of 3 hours. Settling time is recorded by operator.	Use operator to fill up quantity of filter aid by adding 15 kg per batch. Quantity of filter aid is recorded by operator.	Use operator to monitor differential pressure of filter at 0.6 kg/cm2 maximum. Differential pressure of filter is recorded by operator.	Follow quality control system to check quality of filter paper. It is considered as semi-automatic control system.	Follow quality control system to check quality of filter aid. It is considered as semi-automatic control system.		
000	ο ο	ى ك	~	7	-	-	-	-		
Potential Causes	Neutralisation temperature too high/low	Quantity of water too low	Agitator time too high/low	Settling time too low	Quantity of filter aid too low	Differential pressure of filter too high	Low density of filter paper	Low quality of filter aid		
с ш >				٦	-					
Potential Failure Effect				Defect and	reprocess	3				
Potential Failure Mode	রা	Low Resistivity Value								
Process Step	DOP finished product									

Table 3.15 Failure Mode and Effects Analysis Table for DOP Product (Cont)

The team identifies risk priority number (RPN) on each rating table and concludes that the acceptable rating of severity, occurrence, and detection is 4, 5, and 5 respectively; therefore, the RPN value is 100. From above table, the team focuses on RPN that higher than 100 which is considered as high potential failure causes of DOP finished product problem.

3.5 Improvement Phase of DOP Manufacturing Process

From analysis phase, the potential failure modes of high colour of DOP finished product cause from temperature of reactor, temperature of hot oil, high colour of PA raw material, and quantity of catalyst, while the potential failure modes the low resistivity of DOP finished product cause from temperature of neutralization, quantity of water, agitator time, and settling time.

Design of experiment (DOE) is used to determine significant causes that affect the quality of DOP finished product and set up new variable to improve quality and prevent non-conforming product.

3.5.1 Design of Experiment for High Colour Failure

This design of experiment is used to find new suitable operating condition to improve colour of DOP finished product. There are four (4) factors to be tested to determine suitable operating conditions as follows:

- 1. Temperature of reactor
- 2. Temperature of hot oil
- 3. Quantity of catalyst
- 4. High colour of PA raw material

Box-Behnken response surface design was used with four factors listed above to determine the factors affecting to the colour of DOP finished product. Each factor and three levels for each factor are set as follows:

Factor	Description	Low Level (-1)	Centre Level (0)	High Level (+1)
А	Temperature of reactor	210 °C	215 °C	220 °C
В	Temperature of hot oil	530 °C	540 °C	550 °C
С	Quantity of catalyst	5 kg	7.5 kg	10 kg
D	Colour of PA raw material	20 APHA	22.5 APHA	25 APHA

Table 3.16 Colour Failure Factors and Three levels for Each Factor

According to determination of the appropriate number of replicates by using Box-Behnken response surface design was not mentioned in Minitab[™] software and any documents, this research will determine the number of replicates of Box-Behnken design by using power and sample size module of 2-Level factorial design in Minitab[™] software.

Due to DOP production capacities is 3 batches per day, the number of replicates is vary from 3, 6 and 9 batches. This enables the experiment to be set up and controlled easily and correctly. The power value from Minitab will show the probability of detecting specified effect when various numbers of replicates are used. Figure 3.5 shows the result from power and sample size module for colour experiment with the following input. There are four factors in the experiment. It is interested in detecting a difference in colour that is greater than 2 between the low and high levels of colour. From previous data, 3.07 is a reasonable estimates of standard deviation for colour experiment.

Power and Sample Size

2-Level Factorial Design Alpha = 0.05 Assumed standard deviation = 3.07 Factors: 4 Base Design: 4, 16 Blocks: none Including a term for center points in model. Center Total Effect Points Runs Reps Power 3 2 3 0.592036 51 3 2 6 99 0.883753 9 3 2 0.972558 147

Figure 3.5 Results of Power and Sample Size Design of Colour

According to power and sample size above, the number of replicates of 6 provides 88% chance of detecting the specified effect. Design matrix and data obtained from DOP colour experiment are shown in table 3.17.

Table 3.17 Design Matrix and Results of Box-Behnken from DOP Colour Experiment

Box-Behnken Design

Factors:	4	Replicates:	6
Base runs:	27	Total runs:	162
Base blocks:	1	Total blocks:	6
Center points	: 18		

	Factors				Replicas					
Run No.	А	В	С	D	1	2	3	4	5	6
1	-1	-1	0	0	25	22	23	23	27	22
2	1	-1	0	0	22	27	25	23	23	22
3	-1	1	0	0	22	23	22	20	23	22
4	1	1	0	0	23	22	25	22	25	23
5	0	0	-1	-1	15	12	13	13	13	15
6	0	0	1	-1	13	15	12	13	15	13
7	0	0	-1	1	22	23	22	22	22	20
8	0	0	1	1	22	20	22	23	22	22
9	-1	0	-1	0	18	20	22	20	20	22
10	1	0	-1	0	22	23	20	22	22	23
11	-1	0	1	0	20	22	22	18	22	20
12	1	0	1	0	23	23	22	20	22	20
13	0	-1	0	-1	20	20	20	18	22	20
14	0	1	0	-1	18	20	18	22	20	20
15	0	-1	0	1	27	23	22	25	22	23
16	0	1	0	1	23	22	25	23	22	23
17	-1	0	0	-1	17	15	18	17	17	17
18	1	0	0	-1	20	18	20	20	22	20
19	-1	0	0	o* 1	23	23	23	22	25	22
20	1	0	0	1	20	23	22	22	25	22
21	0	-1	-1	0	20	20	20	22	20	18
22	0	1	-1	0	20	20	22	20	20	20
23	0	-1	1	0	22	20	18	20	22	20
24	0	1	1	0	18	22	20	23	20	20
25	0	0	0	0	17	18	15	17	18	18
26	0	0	0	0	18	15	17	17	18	17
27	0	0	0	0	17	18	18	15	17	18
Box-Behnken Design

Factors:	4	Replicates:	6
Base runs:	27	Total runs:	162
Base blocks:	1	Total blocks:	6

Center points: 18

Response Surface Regression: DOP Colour versus Block, Reactor Temp, ...

The analysis was done using coded units.

Estimated Regression Coefficients for DOP Colour

Term	Coef	SE Coef	Т	P
Constant	17.1111	0.3733	45.843	0.000
Block 1	-0.0741	0.2782	-0.266	0.790
Block 2	-0.0000	0.2782	-0.000	1.000
Block 3	-0.0370	0.2782	-0.133	0.894
Block 4	-0.2593	0.2782	-0.932	0.353
Block 5	0.6296	0.2782	2.263	0.025
Reactor Temp	0.5417	0.1866	2.902	0.004
Hot Oil Temp	-0.2083	0.1866	-1.116	0.266
Catalyst	0.0417	0.1866	0.223	0.824
PA Colour	2.6806	0.1866	14.363	0.000
Reactor Temp*Reactor Temp	3.1458	0.2799	11.238	0.000
Hot Oil Temp*Hot Oil Temp	3.2292	0.2799	11.535	0.000
Catalyst*Catalyst	0.2708	0.2799	0.967	0.335
PA Colour*PA Colour	0.6042	0.2799	2.158	0.033
Reactor Temp*Hot Oil Temp	0.3333	0.3232	1.031	0.304
Reactor Temp*Catalyst	-0.1667	0.3232	-0.516	0.607
Reactor Temp*PA Colour	-0.9583	0.3232	-2.965	0.004
Hot Oil Temp*Catalyst	-0.0417	0.3232	-0.129	0.898
Hot Oil Temp*PA Colour	-0.0833	0.3232	-0.258	0.797
Catalyst*PA Colour	0.0000	0.3232	0.000	1.000

S = 1.584 R-Sq = 76.5% R-Sq(adj) = 73.3%

Analysis of Variance for DOP Colour

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Blocks	5	14.52	14.52	2.904	1.16	0.333
Regression	14	1143.39	1143.39	81.671	32.57	0.000
Linear	4	541.72	541.72	135.431	54.01	0.000
Square	4	576.08	576.08	144.021	57.43	0.000
Interaction	б	25.58	25.58	4.264	1.70	0.125
Residual Error	142	356.09	356.09	2.508		
Lack-of-Fit	130	340.76	340.76	2.621	2.05	0.081
Pure Error	12	15.33	15.33	1.278		
Total	161	1514.00				

Unusual Observations for DOP Colour

Obs	Std0rder	DOP Colour	Fit	SE Fit	Residual	St Resid
15	15	27.000	23.843	0.567	3.157	2.14 R
20	20	20.000	23.051	0.567	-3.051	-2.06 R
29	29	27.000	23.903	0.567	3.097	2.09 R
32	32	12.000	15.264	0.567	-3.264	-2.21 R
60	60	12.000	15.310	0.567	-3.310	-2.24 R
95	95	22.000	17.880	0.567	4.120	2.79 R
121	121	22.000	19.019	0.567	2.981	2.02 R

R denotes an observation with a large standardized residual.

Estimated Regression Coefficients for DOP Colour using data in uncoded units

Term Constant Block 1 Block 2 Block 3 Block 4 Block 5	Coef 15599.5 -0.0740741 -4.39682E-16 -0.0370370 -0.259259 0.629630
Reactor Temp	-55.7750
Hot Oil Temp	-36.2417
Catalyst	3.13333
PA Colour	15.0056
Reactor Temp*Reactor Tem	np 0.125833
Hot Oil Temp*Hot Oil Tem	np 0.0322917
Catalyst*Catalyst	0.0433333
PA Colour*PA Colour	0.0966667
Reactor Temp*Hot Oil Tem	np 0.00666667
Reactor Temp*Catalyst	-0.0133333
Reactor Temp*PA Colour	-0.0766667
Hot Oil Temp*Catalyst	-0.00166667
Hot Oil Temp*PA Colour	-0.00333333
Catalyst*PA Colour	6.02284E-17

MinitabTM software's response optimizer was used to provide optimal solution for the input variable combinations and optimization plot. The results from response optimizer of DOP colour with the desirability of 1.0 are shown in figure 3.6.



Figure 3.6 Response Optimization Results of DOP Colour

The best factor levels should be set at the values as shown in figure 3.6 to obtain minimum DOP colour at 14.8 APHA. That is, reactor temperature would be set at 214 °C, hot oil temperature at 540 °C, catalyst quantity at 7.0 kg, and PA raw material colour at 20 APHA.

Due to PA raw material colour can be blended to vary from 20 – 25 APHA depended on market and supplier, if adjusting the factor setting of PA raw material colour by increasing to 25 APHA at maximum colour specification, it shows in figure 3.7 that desirability reduces a bit from 1.0 to 0.925 at DOP colour of 20.4 APHA with reactor temperature at 215.3 °C, hot oil temperature at 540.2 °C, catalyst quantity at 7.35 kg which still meets the DOP finished product colour at 25 APHA maximum.



Figure 3.7 Response Optimization Results of DOP Colour at Max PA Raw Material Colour

From the result above in figure 3.6 and 3.7, it can be concluded that the new suitable operating condition would be set. That is, reactor temperature would be set at 214°C, hot oil temperature at 540 °C, catalyst quantity at 7.0 kg, and PA raw material colour at 20 APHA to minimize DOP colour at 14.8 APHA. However, if PA raw material

colour increase to 25 APHA, reactor temperature would be set at 215.5 °C, hot oil temperature at 540 °C, catalyst quantity at 7.35 kg.

Therefore, if PA raw material colour varies from 20 to 25, reactor temperature would be set at 214 - 216 °C, hot oil temperature at 540 °C, catalyst quantity at 7.0 - 7.5 kg which still meet DOP finished product colour at 25 APHA maximum and it needs to ensure that new operating condition is under controlled to avoid high DOP colour problem.

3.5.2 Design of Experiment for Resistivity Value Failure

This design of experiment is used to find new suitable operating condition to improve resistivity value of DOP finished product. There are four (4) factors to be tested to determine suitable operating conditions as follows:

- 1. Temperature of neutralization tank
- 2. Quantity of water
- 3. Agitator time of neutralization tank
- 4. Settling time of neutralization tank

Box-Behnken response surface design was used with four factors listed above to determine the factors affecting to the resistivity value of DOP finished product. Each factor and three levels for each factor are set as follows:

Factor	Description	Low Level (-1)	Centre Level (0)	High Level (+1)
A	Temperature of	90 °C	94.5 °C	99 °C
9	neutralization tank			
В	Quantity of water	2 m ³	2.5 m^3	3 m ³
С	Agitator time of	10 minutes	15 minutes	20 minutes
	neutralization tank			
D	Settling time of	2 hours	3 hours	4 hours
	neutralization tank			

Table 3.18 Resistivity Value Failure Factors and Three levels for Each Factor

Figure 3.8 shows the result from power and sample size module for resistivity value experiment with the following input. There are four factors in the experiment. It is interested in detecting a difference in resistivity value that is greater than 0.5 between the low and high levels of resistivity value. From previous data, 0.65 is a reasonable estimates of standard deviation for resistivity value experiment.

Power and Sample Size

2-Level Factorial Design
Alpha = 0.05 Assumed standard deviation = 0.65
Factors: 4 Base Design: 4, 16
Blocks: none
Including a term for center points in model.

		Total	
Effect	Reps	Runs	Power
0.5	3	51	0.735273
0.5	6	99	0.961133
0.5	9	147	0.995617
	Effect 0.5 0.5 0.5	Effect Reps 0.5 3 0.5 6 0.5 9	Total Effect Reps Runs 0.5 3 51 0.5 6 99 0.5 9 147

Figure 3.8 Results of Power and Sample Size Design of Resistivity Value

According to power and sample size above, the number of replicates of 3 provides 74% chance of detecting the specified effect. Design matrix and data obtained from DOP resistivity value experiment are shown in table 3.19.

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Table 3.19 Design Matrix and Results of Box-Behnken from DOP VR Experiment

Box-Behnken Design

Factors:	4	Replicates:	3
Base runs:	27	Total runs:	81
Base blocks:	1	Total blocks:	3
Center points	: 9		

	Factors				Replicas		
Run No.	А	В	С	D	1	2	3
1	-1	-1	0	0	2.54	2.31	2.36
2	1	-1	0	0	2.12	1.79	1.98
3	-1	1	0	0	1.88	2.24	2.12
4	1	1	0	0	1.65	2.03	1.41
5	0	0	-1	-1	1.64	1.74	1.65
6	0	0	1	-1	1.13	0.90	0.89
7	0	0	-1	1	2.83	2.71	2.82
8	0	0	1	1	2.54	2.45	2.73
9	-1	0	-1	0	2.17	2.26	2.12
10	1	0	-1	0	2.10	1.84	1.98
11	-1	0	1	0	1.79	1.88	1.65
12	1	0	1	0	1.64	1.36	1.41
13	0	-1	0	-1	1.60	1.74	1.79
14	0	1	0	-1	1.88	1.84	2.03
15	0	-1	0	1	2.36	2.40	2.35
16	0	1	0	1	3.18	3.09	2.83
17	-1	0	0	0 -1 0	1.41	1.42	1.32
18	1	0	0	-1	0.85	0.94	1.01
19	-1	0	0	1	2.26	2.12	2.07
20	1	0	0	001	1.98	2.07	1.88
21	0	-1	-1	0	2.45	2.59	2.36
22	0	1	-1	0	3.20	3.29	2.92
23	0	-1	1	0	1.88	2.12	2.07
24	0	1	1	0	2.49	2.59	2.40
25	0	0	0	0	2.73	2.92	2.83
26	0	0	0	0	2.83	3.06	2.71
27	0	0	0	0	2.78	2.59	2.68

Box-Behnken Design

Factors:	4	Replicates:	3
Base runs:	27	Total runs:	81
Base blocks:	1	Total blocks:	3

Center points: 9

Response Surface Regression: DOP Vr versus Block, Neutralizati, Water, ...

The analysis was done using coded units.

Estimated Regression Coefficients for DOP Vr

Term	Coef	SE Coef	Т	P
Constant	2.79222	0.06913	40.390	0.000
Block 1	0.01432	0.03259	0.439	0.662
Block 2	0.02840	0.03259	0.871	0.387
Neutralization Temp	-0.16333	0.03457	-4.725	0.000
Water	0.11833	0.03457	3.423	0.001
Agitator Time	-0.24306	0.03457	-7.032	0.000
Settling Time	0.52472	0.03457	15.181	0.000
Neutralization Temp*	-0.69556	0.05185	-13.415	0.000
Neutralization Temp				
Water*Water	-0.03222	0.05185	-0.621	0.536
Agitator Time*Agitator Time	-0.25264	0.05185	-4.873	0.000
Settling Time*Settling Time	-0.50847	0.05185	-9.807	0.000
Neutralization Temp*Water	0.01417	0.05987	0.237	0.814
Neutralization Temp*Agitator Time	-0.02333	0.05987	-0.390	0.698
Neutralization Temp*Settling Time	0.06917	0.05987	1.155	0.252
Water*Agitator Time	-0.05000	0.05987	-0.835	0.407
Water*Settling Time	0.11417	0.05987	1.907	0.061
Agitator Time*Settling Time	0.12250	0.05987	2.046	0.045

S = 0.2074 R-Sq = 89.9% R-Sq(adj) = 87.4%

Analysis of Variance for DOP Vr

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Blocks	2	0.0766	0.0766	0.03829	0.89	0.416
Regression	14	24.4419	24.4419	1.74585	40.59	0.000
Linear	4	13.5032	13.5032	3.37581	78.49	0.000
Square	4	10.5058	10.5058	2.62645	61.06	0.000
Interaction	6	0.4328	0.4328	0.07214	1.68	0.141
Residual Error	64	2.7527	2.7527	0.04301		
Lack-of-Fit	58	2.6187	2.6187	0.04515	2.02	0.190
Pure Error	6	0.1341	0.1341	0.02234		
Total	80	27.2712				

Unusual Observations for DOP Vr

Obs	Std0rder	DOP Vr	Fit	SE Fit	Residual	St Resid
1	1	2.540	2.138	0.097	0.402	2.19 R
3	3	1.880	2.346	0.097	-0.466	-2.54 R
4	4	1.650	2.048	0.097	-0.398	-2.17 R
58	58	1.410	1.991	0.097	-0.581	-3.17 R

R denotes an observation with a large standardized residual.

Estimated Regression Coefficients for DOP Vr using data in uncoded units

Term	Coef
Constant	-303.170
Block 1	0.0143210
Block 2	0.0283951
Neutralization Temp	6.40926
Water	-0.0988889
Agitator Time	0.329056
Settling Time	1.18472
Neutralization Temp*	-0.0343484
Neutralization Temp	
Water*Water	-0.128889
Agitator Time*Agitator Time	-0.0101056
Settling Time*Settling Time	-0.508472
Neutralization Temp*Water	0.00629630
Neutralization Temp*Agitator Time	-0.00103704
Neutralization Temp*Settling Time	0.0153704
Water*Agitator Time	-0.0200000
Water*Settling Time	0.228333
Agitator Time*Settling Time	0.0245000

MinitabTM software's response optimizer was used to provide optimal solution for the input variable combinations and optimization plot. The results from response optimizer of DOP resistivity value are shown in figure 3.9.



Figure 3.9 Response Optimization Results of DOP Resistivity Value

The best factor levels should be set at the values as shown in figure 3.13 to obtain maximum DOP resistivity value at $3.1 \times 10^{11} \Omega$.cm. That is, neutralization temperature would be set at 94.2 °C, water quantity at 3.0 m³, agitator time at 13 minutes, and settling time at 3.6 hours.

Therefore, it can be concluded that the new suitable operating condition would be set. That is, neutralization temperature would be set at 94.2 °C, water quantity at 3.0 m³, agitator time at 13 minutes, and settling time at 3.6 hours to maximize DOP resistivity value and it needs to ensure that new operating condition is under controlled to avoid low DOP resistivity value problem.

3.6 Control Phase of DOP Manufacturing Process

From design of experiment in improvement phase, new operating conditions are implemented to DOP manufacturing process as follows:

DOP Colour Improvement	New Operating Conditions
Temperature of reactor	214 – 216 °C
Temperature of hot oil	540 °C
Quantity of catalyst	7.0 – 7.5 kg
Colour of PA raw material	20 – 25 APHA
DOP Resistivity Value Improvement	New Operating Conditions
Temperature of neutralization tank	94.2 °C
Quantity of water	3.0 m ³
Agitator time of neutralization tank	13 minutes
Settling time of neutralization tank	3.6 hours

Table 3.20 New Operating Conditions for DOP Manufacturing Process

Then, statistical process control is used for monitoring a process to identify special cause of variation and take corrective action when it is appropriate and determine process capability. The charts most commonly used for variable data are the I-MR chart which is used for individual plot statistics from measurement data to monitor the centring of the process.

Starting with using control chart on data set of DOP finished product colour and resistivity value on January - March 2007, the data is shown in table 3.21 and 3.22 respectively.

From the data in table 3.21 and 3.22, plot I-MR chart of DOP finished product colour and resistivity value including mean, upper control limit, and lower control limit as shown in figure 3.10 and 3.11.



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Table 3.21 DOP Finished Product Colour on January - March 2007 before Six Sigma

Batch No	Colour								
1/1	17	1/41	18	2/11	23	2/51	18	3/24	23
1/2	17	1/42	22	2/12	23	2/52	22	3/25	32
1/3	17	1/43	22	2/13	22	2/53	22	3/26	27
1/4	17	1/44	22	2/14	18	2/54	23	3/27	27
1/5	22	1/45	22	2/15	22	2/55	23	3/28	23
1/6	18	1/46	22	2/16	22	2/56	22	3/29	23
1/7	22	1/47	22	2/17	22	2/57	22	3/30	23
1/8	22	1/48	22	2/18	22	2/58	22	3/31	23
1/9	22	1/49	22	2/19	23	2/59	22	3/32	23
1/10	22	1/50	22	2/20	22	2/60	22	3/33	25
1/11	23	1/51	22	2/21	22	2/61	22	3/34	25
1/12	17	1/52	18	2/22	22	2/62	22	3/35	23
1/13	27	1/53	18	2/23	22	2/63	22	3/36	23
1/14	22	1/54	18	2/24	22	2/64	22	3/37	23
1/15	18	1/55	18	2/25	22	2/65	18	3/38	22
1/16	18	1/56	17	2/26	22	2/66	23	3/39	23
1/17	18	1/57	22	2/27	22	2/67	23	3/40	23
1/18	22	1/58	22	2/28	22	3/1	23	3/41	23
1/19	18	1/59	18	2/29	22	3/2	22	3/42	23
1/20	18	1/60	17	2/30	18	3/3	23	3/43	23
1/21	17	1/61	22	2/31	18	3/4	23	3/44	23
1/22	17	1/62	22	2/32	18	3/5	28	3/45	23
1/23	18	1/63	18	2/33	18	3/6	22	3/46	23
1/24	17	1/64	18	2/34	18	3/7	23	3/47	25
1/25	18	1/65	17	2/35	18	3/8	23	3/48	23
1/26	23	1/66	17	2/36	18	3/9	22	3/49	23
1/27	22	1/67	17	2/37	18	3/10	17	3/50	23
1/28	23	1/68	18	2/38	18	3/11	18	3/51	23
1/29	22	1/69	18	2/39	22	3/12	22	3/52	22
1/30	23	1/70	22	2/40	18	3/13	22	3/53	23
1/31	23	2/1	18	2/41	18	3/14	23	3/54	25
1/32	23	2/2	18	2/42	17	3/15	23	3/55	27
1/33	25	2/3	17	2/43	17	3/16	22	3/56	25
1/34	23	2/4	17	2/44	23	3/17	23	3/57	23
1/35	13	2/5	17	2/45	22	3/18	23		
1/36	22	2/6	18	2/46	18	3/19	23		
1/37	23	2/7	23	2/47	18	3/20	22		
1/38	18	2/8	37	2/48	23	3/21	23		
1/39	18	2/9	32	2/49	22	3/22	23		
1/40	18	2/10	27	2/50	22	3/23	23		

Table 3.22 DOP Finished Product VR on January – March 2007 before Six Sigma

Batch No	VR								
1/1	2.83	1/41	2.36	2/11	0.94	2/51	1.88	3/24	2.92
1/2	3.20	1/42	1.84	2/12	0.99	2/52	2.12	3/25	2.83
1/3	2.49	1/43	1.41	2/13	1.65	2/53	1.84	3/26	2.36
1/4	2.49	1/44	1.41	2/14	1.64	2/54	1.88	3/27	2.12
1/5	1.41	1/45	1.13	2/15	1.41	2/55	1.88	3/28	2.36
1/6	1.08	1/46	1.41	2/16	1.41	2/56	1.65	3/29	2.59
1/7	1.32	1/47	1.13	2/17	1.60	2/57	1.69	3/30	2.36
1/8	1.42	1/48	1.41	2/18	1.41	2/58	2.26	3/31	3.29
1/9	1.65	1/49	0.47	2/19	1.51	2/59	2.36	3/32	3.20
1/10	1.41	1/50	1.18	2/20	1.41	2/60	2.36	3/33	2.54
1/11	2.03	1/51	1.41	2/21	1.18	2/61	2.68	3/34	2.59
1/12	1.88	1/52	1.18	2/22	1.18	2/62	2.49	3/35	2.26
1/13	2.12	1/53	1.51	2/23	1.41	2/63	2.26	3/36	2.31
1/14	2.83	1/54	1.27	2/24	1.18	2/64	2.78	3/37	2.24
1/15	2.12	1/55	1.41	2/25	1.22	2/65	0.64	3/38	2.71
1/16	2.83	1/56	2.07	2/26	1.41	2/66	0.71	3/39	2.36
1/17	2.83	1/57	1.74	2/27	1.51	2/67	1.08	3/40	3.09
1/18	2.71	1/58	1.98	2/28	1.88	3/1	1.88	3/41	2.83
1/19	2.36	1/59	1.79	2/29	1.41	3/2	2.24	3/42	2.71
1/20	3.06	1/60	2.12	2/30	1.69	3/3	2.12	3/43	1.88
1/21	2.36	1/61	2.17	2/31	2.36	3/4	2.36	3/44	3.09
1/22	2.83	1/62	1.88	2/32	1.88	3/5	2.24	3/45	2.83
1/23	2.59	1/63	2.26	2/33	1.53	3/6	3.67	3/46	2.71
1/24	1.88	1/64	1.79	2/34	2.12	3/7	1.18	3/47	1.88
1/25	1.41	1/65	2.21	2/35	1.88	3/8	1.41	3/48	2.26
1/26	1.18	1/66	1.41	2/36	1.88	3/9	0.99	3/49	2.45
1/27	1.13	1/67	1.98	2/37	2.40	3/10	1.32	3/50	2.71
1/28	0.89	1/68	1.65	2/38	2.59	3/11	1.42	3/51	2.45
1/29	1.00	1/69	1.88	2/39	2.35	3/12	1.65	3/52	2.73
1/30	0.71	1/70	1.88	2/40	2.36	3/13	1.41	3/53	2.36
1/31	0.94	2/1	2.12	2/41	2.83	3/14	2.03	3/54	2.36
1/32	1.01	2/2	1.18	2/42	2.59	3/15	1.88	3/55	2.73
1/33	0.94	2/3	1.41	2/43	2.45	3/16	2.12	3/56	2.83
1/34	1.36	2/4	1.46	2/44	2.36	3/17	2.64	3/57	2.83
1/35	1.01	2/5	1.55	2/45	1.36	3/18	2.92		
1/36	1.18	2/6	0.85	2/46	1.41	3/19	2.92		
1/37	0.90	2/7	0.57	2/47	1.60	3/20	2.54		
1/38	1.98	2/8	0.85	2/48	1.88	3/21	2.45		
1/39	1.64	2/9	1.13	2/49	2.36	3/22	2.59		
1/40	1.65	2/10	0.85	2/50	1.88	3/23	2.73		



Figure 3.10 I-MR chart of DOP Finished Product Colour on January – March 2007



Figure 3.11 I-MR chart of DOP Finished Product Resistivity Value on January – March

From figure 3.10 and 3.11, it shows that DOP finished product colour and resistivity value fall outside the upper and lower control limits. Moreover, DOP finished product colour falls outside the upper specification limit at 25 APHA, while DOP finished product resistivity value falls outside the lower specification limit at $1 \times 10^{11} \Omega$.cm, which means that the process is not stable

Comparing to the data of DOP finished product colour and resistivity value on January – March 2008 in table 3.23 and 3.24, I-MR chart of DOP finished product colour and resistivity value are plotted and shown in figure 3.12 and 3.13, respectively.



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Table 3.23 DOP Finished Product Colour on January – March 2008 after Six Sigma

Batch No	Colour								
1/1	18	1/41	17	2/19	18	3/4	20	3/44	18
1/2	18	1/42	18	2/20	15	3/5	20	3/45	20
1/3	22	1/43	18	2/21	20	3/6	18	3/46	20
1/4	17	1/44	17	2/22	18	3/7	22	3/47	20
1/5	15	1/45	22	2/23	20	3/8	20	3/48	20
1/6	20	1/46	17	2/24	20	3/9	20	3/49	20
1/7	17	1/47	18	2/25	18	3/10	20	3/50	22
1/8	22	1/48	20	2/26	20	3/11	18	3/51	20
1/9	17	1/49	17	2/27	18	3/12	20	3/52	20
1/10	17	1/50	17	2/28	18	3/13	22	3/53	20
1/11	15	1/51	17	2/29	20	3/14	20	3/54	20
1/12	15	1/52	17	2/30	20	3/15	20	3/55	20
1/13	17	1/53	18	2/31	18	3/16	20	3/56	22
1/14	18	1/54	18	2/32	20	3/17	20	3/57	20
1/15	20	1/55	22	2/33	17	3/18	20	3/58	20
1/16	17	1/56	17	2/34	18	3/19	18	3/59	20
1/17	17	1/57	18	2/35	20	3/20	20	3/60	20
1/18	17	1/58	17	2/36	18	3/21	20	3/61	20
1/19	17	1/59	18	2/37	17	3/22	20	3/62	20
1/20	18	1/60	17	2/38	18	3/23	25		
1/21	18	1/61	17	2/39	18	3/24	20		
1/22	20	1/62	18	2/40	20	3/25	25		
1/23	17	2/1	20	2/41	20	3/26	20		
1/24	17	2/2	20	2/42	20	3/27	20		
1/25	17	2/3	18	2/43	18	3/28	20		
1/26	20	2/4	15	2/44	20	3/29	20		
1/27	17	2/5	17	2/45	20	3/30	20		
1/28	13	2/6	20	2/46	20	3/31	18		
1/29	17	2/7	18	2/47	20	3/32	20		
1/30	18	2/8	20	2/48	20	3/33	20		
1/31	13	2/9	20	2/49	18	3/34	20	0.1	
1/32	17	2/10	22	2/50	20	3/35	18		
1/33	18	2/11	20	2/51	23	3/36	20		
1/34	17	2/12	20	2/52	20	3/37	20		
1/35	17	2/13	20	2/53	20	3/38	18		
1/36	17	2/14	20	2/54	20	3/39	18		
1/37	20	2/15	17	2/55	20	3/40	20		
1/38	15	2/16	15	3/1	20	3/41	20		
1/39	13	2/17	18	3/2	25	3/42	18		
1/40	18	2/18	17	3/3	20	3/43	20		

Batch No	VR								
1/1	3.24	1/41	2.29	2/19	2.56	3/4	2.92	3/44	2.24
1/2	2.29	1/42	2.82	2/20	2.79	3/5	3.76	3/45	2.71
1/3	2.76	1/43	2.86	2/21	3.27	3/6	2.54	3/46	2.36
1/4	3.71	1/44	3.24	2/22	2.66	3/7	3.30	3/47	3.29
1/5	2.30	1/45	2.30	2/23	2.80	3/8	2.45	3/48	3.09
1/6	1.83	1/46	2.77	2/24	3.17	3/9	2.59	3/49	2.83
1/7	1.83	1/47	2.29	2/25	2.64	3/10	3.06	3/50	2.71
1/8	2.67	1/48	2.06	2/26	2.39	3/11	3.53	3/51	3.77
1/9	2.53	1/49	2.77	2/27	2.80	3/12	2.73	3/52	2.88
1/10	2.77	1/50	2.29	2/28	2.80	3/13	3.53	3/53	2.26
1/11	2.18	1/51	3.00	2/29	3.27	3/14	2.92	3/54	2.45
1/12	2.06	1/52	3.24	2/30	3.03	3/15	3.06	3/55	2.83
1/13	3.47	1/53	2.76	2/31	3.27	3/16	2.83	3/56	2.71
1/14	3.24	1/54	2.77	2/32	3.03	3/17	2.83	3/57	2.83
1/15	2.77	1/55	2.76	2/33	2.56	3/18	3.06	3/58	2.73
1/16	3.71	1/56	2.77	2/34	3.27	3/19	2.92	3/59	2.45
1/17	2.29	1/57	2.06	2/35	2.80	3/20	3.18	3/60	2.73
1/18	3.10	1/58	1.83	2/36	3.27	3/21	3.39	3/61	3.30
1/19	2.29	1/59	2.77	2/37	2.74	3/22	3.29	3/62	2.83
1/20	2.76	1/60	3.77	2/38	3.03	3/23	3.29		
1/21	2.29	1/61	3.81	2/39	2.33	3/24	2.36		
1/22	2.76	1/62	2.77	2/40	2.79	3/25	3.29		
1/23	3.24	2/1	2.33	2/41	3.27	3/26	2.92		
1/24	3.71	2/2	2.33	2/42	2.50	3/27	3.53		
1/25	3.70	2/3	2.56	2/43	2.68	3/28	2.36		
1/26	3.71	2/4	2.32	2/44	3.26	3/29	3.44		
1/27	2.76	2/5	2.70	2/45	3.27	3/30	3.77		
1/28	2.77	2/6	3.17	2/46	2.74	3/31	3.06		
1/29	2.76	2/7	2.83	2/47	2.73	3/32	3.77		
1/30	2.76	2/8	2.73	2/48	2.74	3/33	2.36		
1/31	2.77	2/9	1.62	2/49	2.73	3/34	3.29	01	
1/32	2.30	2/10	1.38	2/50	3.26	3/35	3.30		
1/33	2.86	2/11	1.03	2/51	3.28	3/36	3.20		
1/34	2.77	2/12	1.62	2/52	3.27	3/37	3.29		
1/35	3.00	2/13	2.32	2/53	3.36	3/38	3.34		
1/36	2.77	2/14	2.33	2/54	2.74	3/39	2.54		
1/37	3.00	2/15	2.57	2/55	3.15	3/40	2.59		
1/38	3.24	2/16	2.65	3/1	2.36	3/41	2.26		
1/39	3.71	2/17	2.79	3/2	3.06	3/42	2.31		
1/40	2.29	2/18	2.65	3/3	2.92	3/43	3.29		



Figure 3.12 I-MR chart of DOP Finished Product Colour on January – March 2008



Figure 3.13 I-MR chart of DOP Finished Product Resistivity Value on January – March

From figure 3.12 and 3.13, it shows that there are 179 batches of DOP produced in January – March 2008, and there are 6 batches of DOP finished product colour and 4 batches of DOP finished product resistivity value fall outside the upper and lower control limits but all DOP finished product fall inside specification limits, which means that the process is in statistical control. DOP colour falls outside the upper and lower control limits due to high colour of PA raw material fluctuation, while DOP resistivity value falls outside the lower control limit due to motor of agitator failure leading to low mixing quality between NaOH, water, and crude DOP.

Figure 3.14 and 3.16 show process capability of DOP finished product colour and resistivity value on January – March 2007 before Six Sigma approach, comparing to process capability of DOP finished product colour and resistivity value on January – March 2008 after Six Sigma approach in figure 3.15 and 3.17. It shows that the process capability (Cpk) after Six Sigma approach of DOP finished product colour increases from 0.92 to 1.48 and from 1.03 to 1.57 for DOP finished product resistivity value.

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Figure 3.14 Process Capability of DOP Finished Product Colour before Six Sigma

Approach



Figure 3.15 Process Capability of DOP Finished Product Colour after Six Sigma



Figure 3.16 Process Capability of DOP Finished Resistivity Value before Six Sigma

Approach



Figure 3.17 Process Capability of DOP Finished Resistivity Value after Six Sigma

Once process capability of DOP finished product on colour and resistivity value have been improved, failure cost production of reprocessing will be reduced. It can be seen that there are no nonconforming product in January – March 2008. It means that if the company continue control DOP product quality and process capability in this level, the company will have no nonconforming product and have unnecessary for reprocessing. Therefore, the percentage of failure cost production of reprocessing will be reduced from 8.2% quarterly of 2007 to 0% in first quarter of 2008, which can reduce the failure cost of the company about 1,327,368 baht per quarter.



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CHAPTER IV

CONCLUSION AND RECOMMENDATION

4.1 Conclusion

This research has implemented Six Sigma approach to Dioctyl Phthalate (DOP) manufacturing process which is the most commonly used plasticizer in the PVC industry. Six Sigma approach, consisting of 5 phases which are define, measure, analyze, improve, and control phase, is used to reduce process variation, improve process capability, and reduces nonconforming product which helps reduce the failure cost production in DOP manufacturing process. Techniques in Six Sigma such as Prioritization of causes of defects, Design of Experiment (DOE), Statistical Process Control (SPC) are used in each phase of Six Sigma approach as explained in Chapter 3.

Due to the company encounters high DOP colour and low resistivity value problems, which lead to high production cost of 491,760 and 835,608 baht, respectively or total failure cost of 1,327,368 baht per quarter on reprocessing of nonconforming product and lost production opportunity. The company would like to reduce failure cost of reprocessing on nonconforming product of colour and resistivity value.

The research has determined process capability (Cpk) of DOP finished product and considered to improve DOP quality which has process capability lower than 1.33. The company would like to improve process capability of DOP colour and resistivity value to at least 1.33.

After defining the problems, this research continues the Six Sigma approach to the next phase, which is measurement phase by using calibration method to verify the accuracy of instrumentation and using Gage R & R method to evaluate the precision of the measurement system of DOP manufacturing process. The results in Chapter 3 show that measurement system is acceptable. Next, analysis phase will identify the potential causes of DOP colour and resistivity value defect from team brainstorming by using fishbone diagrams. Then, team will identify high potential causes by using FMEA method. The results from this phase are the high potential causes of DOP colour defect which are temperature of reactor, temperature of hot oil, high colour of PA raw material, and quantity of catalyst, while the high potential causes of DOP resistivity defect are temperature of neutralization, quantity of water, agitator time, and settling time.

In improvement phase, design of experiment (DOE) is used to determine new suitable operating condition to improve quality and prevent non-conforming product. This research use Box-Behnken response surface design to set up the experiment and determine the operating condition to optimum DOP colour and resistivity value.

Finally, I-MR chart has been used and plotted for monitoring parameter of DOP product quality on colour and resistivity value. There are no DOP product colour and resistivity value fall outside the upper and lower control limit after using new operating condition determined from design of experiment. Process capability of DOP colour and resistivity values also increase from 0.92 to 1.48 and 1.03 to 1.57, respectively.

During implementing Six Sigma in DOP manufacturing process, operating cost of the company has slightly increased due to overtime of operators on some experiments such as measurement evaluation and verification by using Gage R&R. The company have no significant of investment cost due to the company used and modified existing equipments and instruments to change operating condition.

From the Six Sigma approach in this research, the company can improve DOP product quality and process capability leading to reduce failure cost production of 1,327,368 baht quarterly of 2007 to 0 baht in first quarter of 2008 and expecting to have no failure cost production in 2008.

4.2 Recommendation

There are some recommendations of Six Sigma approach for failure cost reduction in DOP manufacturing process as follows:

1. The results from Chapter 3 show that there are no DOP failures in January 2008. However, it might have some DOP failures in the future from potential causes with low RPN, which are not considered in the improvement phase. If there are opportunities in the future, the team should look back and perform continuous improvement to reduce the potential causes if it is economical.

2. Response Optimizer can be used to find the suitable operating conditions. However, if there are some problems such as high PA raw material colour, the new operating conditions such as reactor temperature, hot oil temperature, and quantity of catalyst should be set to operate at high PA raw material colour to get the minimum DOP colour. In other cases, if there is hot oil system problem, which cannot increase hot oil temperature at 540 °C, the new operating conditions should be re-set to get minimum DOP colour.

3. After performing the Improvement phase for DOP manufacturing process in this research, the most important thing is continuous monitoring to maintain consistency, stability, performance, efficiency, process capability, and provide feed back to the system.

4. Six Sigma approach should be implemented for other projects such as efficiency of equipment to improve reliability and efficiency of equipment which increasing DOP productivity.

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BIOGRAPHY

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