ORIGINAL ARTICLE

The application of statistical techniques to reduce ethylene glycol in waste water produced by esterification reaction in polyester manufacturing process

Komson Akesrisakul[1] and Adsada Jiraprayuklert[2]

Abstract

Akesrisakul, K. and Jiraprayuklert, A.

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This study aimed to find the factors that influence the amount of ethylene glycol (EG) in waste water, produced by the esterification reaction in polyester manufacturing process, and to set these influential factors at the proper level, which leads to small amount of EG and low waste water treatment cost but same polyester quality. Several experiments were designed and performed to achieve the research objectives. The study began with reviewing various relevant documents and interviewing specialists to define the potential factors. Consequently, there were 4 potential factors: the reaction temperature, the level of slurry in reactor, the EG feeding rate, and the water temperature in distillation column. To find the influential factors, One-Factor-At-a-Time (OFAT) experiments were applied to define the significant factors. Then, a factorial design experiment was implemented to analyze their interaction effects. The experiments revealed that the reaction temperature, the level of slurry in reactor, and the EG feeding rate best setting condition, the results showed that EG volume was decreased on average 10.15% and that waste water treatment cost was reduced 27.82%.

Key words: Design of Experiment, waste water treatment cost reduction, reduction of ethylene glycol amount, polyester industry

[1]M.Eng. (System and Manufacturing Engineering), [2]Ph.D. (Industrial Engineering),Department of Production Engineering, Faculty of Engineering, King Mongkut's University of Technology Thonburi Thungkru, Bangkok, 10140 Thailand.

Corresponding e-mail: adsada.sae@kmutt.ac.th

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บทคัดย่อ

คมสันต์ เอกศรีสกุล และ อัษฎา จิรประยุกต์เลิศ

การลดปริมาณเอทิลีนไกลคอลในน้ำเสียที่เกิดจากปฏิกิริยาเอสเตอริฟิเคชันของกระบวนการผลิตพอลิเอสเตอร์ด้วยการประยุกต์วิธีทางสถิติ

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การวิจัยครั้งนี้มีวัตถุประสงค์เพื่อศึกษาปัจจัยที่มีอิทธิพลต่อค่าปริมาณเอทิลีนไกลคอลในน้ำเสีย ที่ได้มาจากสายการผลิตพอลิเอสเตอร์ในขั้นตอนการเกิดปฏิกิริยาเอสเตอริฟิเคชัน และศึกษาหาเงื่อนไขในการควบคุมกระบวนการผลิตเพื่อลดต้นทุนการผลิต โดยทำให้ผลิตภัณฑ์พอลิเอสเตอร์ที่ได้มีคุณภาพดีเหมาะสมต่อการใช้งาน การออกแบบการทดลองเป็นเครื่องมือที่ใช้เพื่อให้บรรลุวัตถุประสงค์ของการศึกษานี้ งานวิจัยนี้ได้ศึกษางานวิจัยที่เกี่ยวข้อง และ จากการสัมภาษณ์ผู้เชี่ยวชาญ เพื่อระบุปัจจัยทั้งหมดที่คาดว่าจะมีผลกระทบต่อค่าปริมาณอีจีในน้ำ ซึ่งมี 4 ปัจจัย คือ อุณหภูมิในหม้อปฏิกิริยา ระดับของสารในหม้อปฏิกิริยา อัตราการป้อนอีจี และอุณหภูมิส่วนบนหอกลั่นแยกน้ำ ในการศึกษาหาปัจจัยที่มีอิทธิพล เริ่มจากการทดลองแบบปัจจัยเดียว และทำการวิเคราะห์อิทธิพลร่วมของปัจจัย (interaction effect) ด้วยการทดลองแบบแฟคทอเรียล จากผลการทดลองและวิเคราะห์ด้วยหลักการทางสถิติพบว่ามี 3 ปัจจัยที่มีผลต่อค่าปริมาณอีจีในน้ำเสีย คือ อุณหภูมิในหม้อปฏิกิริยา ระดับของสารในหม้อปฏิกิริยา และอัตราการเติมอีจีในหม้อปฏิกิริยา โดยสามารถหาสภาวะในการปรับตั้งกระบวนการผลิตพอลิเอสเตอร์ที่ทำให้ค่าปริมาณอีจีในน้ำมีค่าน้อยที่สุดคือ อุณหภูมิในหม้อปฏิกิริยา 288 °C ระดับของสารในหม้อปฏิกิริยา 70 % และอัตราการเติมอีจีในหม้อปฏิกิริยา 200 ก.ก/ชั่วโมง โดยเมื่อนำสภาวะการปรับตั้งกระบวนการผลิตใหม่ไปทดสอบเพื่อยืนยันผลแล้วนำค่าเฉลี่ยค่าปริมาณอีจีในน้ำนี้ไปเปรียบเทียบเชิงสถิติพบว่า ค่าเฉลี่ยปริมาณอีจีในน้ำมีค่าลดลง 10.15 %และต้นทุนการบำบัดน้ำเสียลดลง 27.82%

ภาควิชาวิศวกรรมอุตสาหการ คณะวิศวกรรมศาสตร์ มหาวิทยาลัยเทคโนโลยีพระจอมเกล้าธนบุรี บางมด ทุ่งครุ กรุงเทพฯ 10140

Polyester Industry is an essential business in Thailand.It grows rapidly,and there are extremely high demands in textile, garment, and packaging industries. Also, it is highly competitive industry. In order to stay in business,a company in the polyester industry decides to use quality and price as the competition strategies. One of the best means is finding the ways to reduce cost,but gain high customers satisfaction.Therefore, the major objective of this study focuses on not only the cost reduction but also the same quality level or higher.

The research started from finding an opportunity for cost reduction. The result showed that waste water treatment contributed the highest cost variant. To discover the causes of high cost, several statistical techniques were applied, and then an appropriate working condition was established to eliminate the causes. Consequently, the waste water treatment cost was significantly reduced to the target

Problem Identification

This is a case study of an anonymous polyester manufacturing company.In the problem exploration, the product quality was firstly analyzed using benchmarking with competitors.The results of benchmarking performed by a Thai organization supporting textile industry in 2002 showed that the studied company provided the best quality compared to others in the same industry.Therefore, quality issue was not the major problem, but after exploring manufacturing cost researchers found high cost variant in the supporting part. After stratifying the supporting cost, it showed that the actual cost of waste water treatment was 3.824 million baht higher than the cost should be as shown in Table 1. As the result,this study focused on the reduction of the waste water treatment cost.

In polyester production, the studied company controlled the amount of polyester produced, so the volume of waste water was constant.However, the cost of waste water treatment varied over time. It depends on the amount of organic compounds in water, which is expressed in term of Biochemical Oxygen Demand (BOD).After performing laboratory analysis, it was found that Ethylene Glycol (EG) was the major component in the waste water. From statistical analysis, EG amount and BOD showed a positive relationship, which is the higher EG,the higher BOD. In addition,the waste water was produced from esterification reaction,which produced less EG in waste water.

Statistical techniques are the efficient tools only when the problem is stable and predictable. Therefore, it was necessary to investigate the stability of EG amount produced by the reaction.To analyze the stability, the amount of EG was collected during November 2002 to October 2003, and was displayed by a control chart in Figure 1. It presented a stability pattern, and the amount of EG was around 70.47 mg/L

Table 1. The supporting cost in three quarters in 2003 (scale: million baht)

Type of Supporting Cost	1st quarter	2nd quarter	3rd quarter	Total cost in three quarters	Budget for three quarters	Difference between actual and the budget
Electricity	2.602	2.565	2.723	7.890	8.550	0.660
Nitrogen Gas	0.254	0.252	0.259	0.765	0.900	0.135
Waste water treatment	**5.313**	**6.191**	**5.820**	**17.324**	**13.500**	**-3.824**
Others	0.006	0.006	0.006	0.018	0.027	0.009

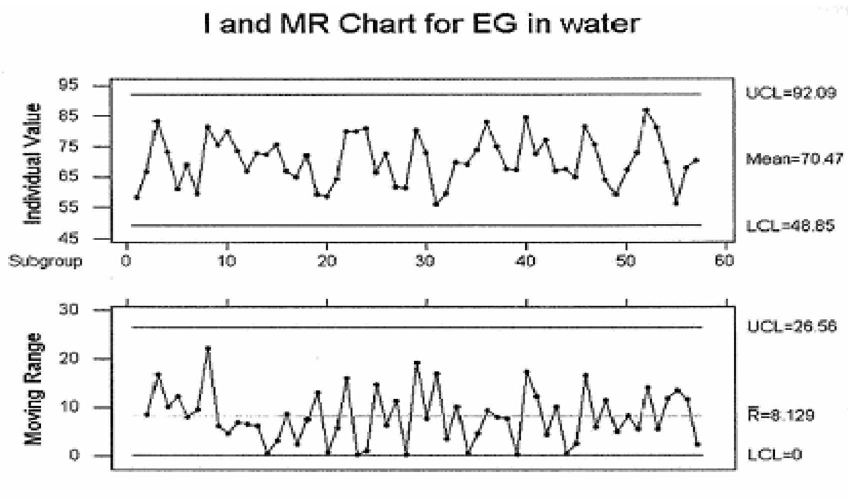

Figure 1. Control Chart of EG amount during November 2002 to October 2003

Methodology

After understanding the problem, the amount of EG was treated as the response variable. Next,the measurement system,which consisted of equipment,operators,and methods of measurement, were analyzed to assure that measured data did not carry errors. The results of the measurement system analysis showed that the system provided accurate and precise data, unbiased and with low variation. Now,researchers were confident that the data,EG amount,did not contain errors,but it represented only the result of the reaction.

To locate input factors,which produce a lower amount of EG, all potential input factors in the esterification reaction were selected by interviewing process experts and reviewing related literatures. Experimental design was the statistical technique applied to analyze and obtain the solutions. As mentioned in Montgomery (2000), OFAT is a good strategy and economical way to do screening experiments at the starting point.These experiments showed how input factors affect the response variable,and then critical factors were selected.However, OFAT is poor to detect interaction among factors.To correct this problem,factorial experiment, full 2^kfactorial design with single replicate,was used to study interaction effect among critical factors Then, a model of the relationship was created, and the best condition was chosen.

Selecting the potential input factors

To define possibly influential factors,researchers reviewed several related materials (Hovenkamp and Munting (1970); Ravindranath and Marshelkar(1982); Immanuel and Gupta (1986); Bradrup *et.al.*(1999)),and interviewed the process experts to focus only on the potential factors. Interviewing the process experts was necessary because they have knowledge from the real process condition and the intrinsic technology.After interviewing, there were four potential factor schosen as shown in Figure 2.Those factors were 1) reaction temperature, which was set at the heat exchanger,displayed by number 1;2) level of material (slurry) in heat exchanger shown by number 2;3) EG feeding rate displayed by number 3; and 4) temperature at vapor separator depicted by number 4.These factors were studied within their process specification,so the highest,the lowest,and the middle levels were picked and set in the OFAT experiments (Table 2).The level outside specification was prohibited in this study because all experiments were performed on-line, so varying outside specification may cause high losses in production.

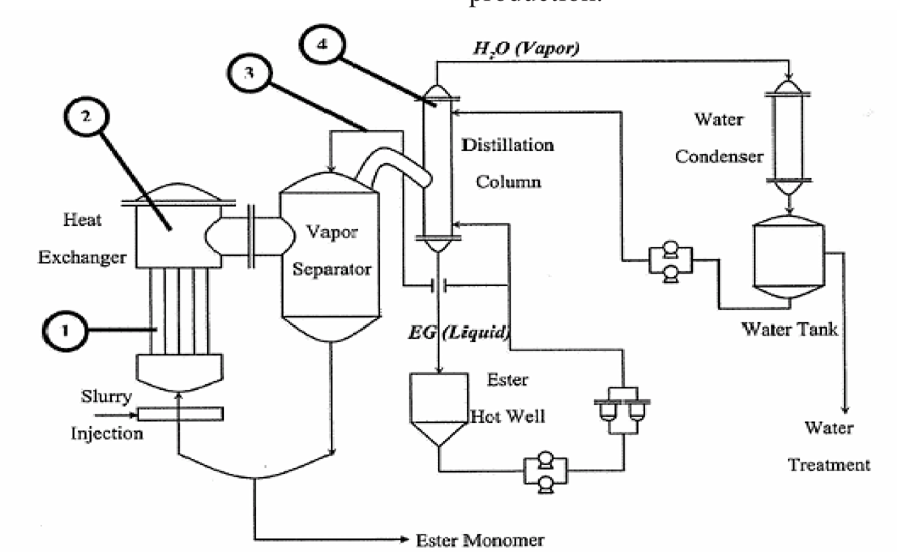

Figure 2. Potential factors influencing EG amount in waste water produced by esterification eaction in a polyester manufacturing process from the studied

OFAT is the experiment that varies each factor and holds others constant at regularly used levels. The objective of doing OFAT was to screen the significant factors by finding the influential factors. Before performing the experiments, it was important to choose appropriate sample size of each variable to increase power of the test. Then, Analyzing the experiments were done for each factor with the specified sample sizes. Results from ANOVA showed that the reaction temperature, the level of slurry in heat exchanger, and EG feeding rate were statistically significant factors, low p-value,at significant level 0.05 as shown in Table-3.It meant that these factors showed significant influence on the EG amount in waste water. The results also showed that high reaction temperature, low level of slurry, and high EG feeding rate tend to produce high amount of EG in waste water. Since OFAT is unable to detect interaction effect among these influential factors, a factorial experiment was designed to study the interaction effect.

Studying interaction effect

At this step, researchers used a 2^3 factorial design with single replicate because of time and cost limitation. Levels of all factors are shown in Table 4, and the experiment conditions and results are shown in Table 5.

Analyzing the experiments

From the 2^3 factorial experiment, since the experiments were performed with a single replicate, it was impossible to analyze the residuals, which meant that it was unable to verify the data qualification. Therefore, to obtain qualified data, these experiments had to be done with strictly controlled conditions. Pareto analysis was used to define significant factors (Figure 3). The Pareto analysis showed that there was no significant interaction effect, but the main effects from all three factors showed significant influences over the EG amount in waste water.Then,the non-significant effects were reduced to be residual error for the model. Results of the Analysis of Variance (ANOVA) after reducing the model are displayed in Figure 4. Figure 4 showed that the three factors were statistically significant, p-value = 0.00,at significant level 0.05. The analysis in Figure 4 also gave a model showing the relationship between EG amount and three input factors. The equation of the relationship is displayed in Equation 1. All main effect plots are depicted in Figure 5. From the main effect plots, researcher could summarize that the lowest EG amount within specified process specifications could occur when setting the reaction temperature at 288 °C, the level of slurry at 70%, and EG feeding rate at 200 kg/hr.

Table 2. Levels of each potential factor in the screening experiments (OFAT)

Potential factors	Process Specification	Experiment levels	Unit
Reaction temperature	288-292	288, 290, 292	^{0}C
Level of slurry	67-70	67, 68, 69, 70	%
EG feeding rate	200-300	200, 250, 300	kg/hr
Temperature at vapor separator	103-109	103, 106, 109	^{0}C

Table 3. Summary of the results from ANOVA in screening experiments (OFAT)

Potential factors	Sample size	p-value	Interpretation
Reaction temperature	5	0.00	Statistically significant
Level of slurry	6	0.00	Statistically significant
EG feeding rate	5	0.00	Statistically significant
Temperature at vapor separator	5	0.14	Not statistically significant

Table 4. Levels of each critical factor for 2^k factorial design

Critical factors	Experiment levels	Unit
Reaction temperature	288, 292	^{0}C
Level of slurry	67, 70	%
EG feeding rate	200, 300	kg/hr

Table 5. Experiment conditions and results

Order	Reaction Temperature (^{0}C)	Level of Slurry (%)	EG Feeding Rate (kg/hr)	EG weight in water (mg/L)
1	292	70	200	68.1
2	288	67	300	67.0
3	292	67	300	75.2
4	292	67	200	73.6
5	288	67	200	65.0
6	288	70	300	62.9
7	288	70	200	61.5
8	292	70	300	70.1

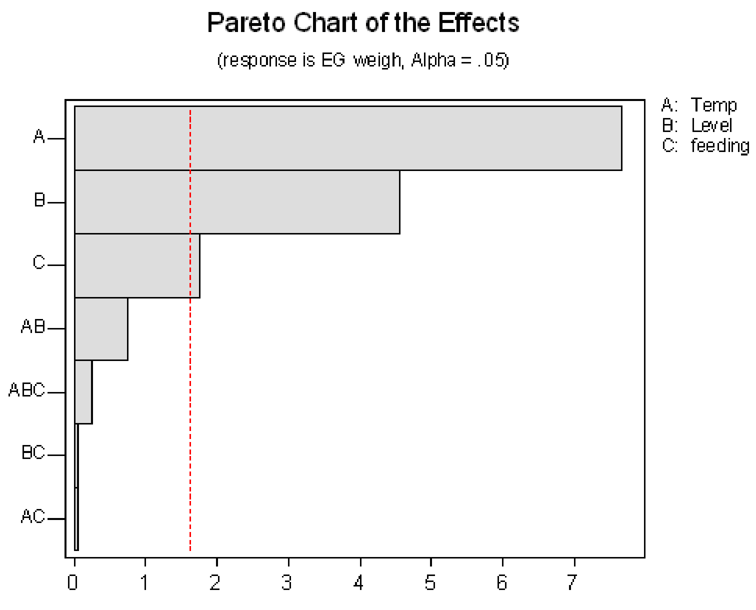

Figure 3. Pareto Chart of the effects from the experiment

Fractional Factorial Fit EG weight versus Temp,Level, feeding rate

Estimated Effects and Coefficients for EG (coded units)

Term	Effect	Coef	SE Coef	T	P
Constant		67.925	0.1984	342.31	0.000
Temp	7.650	3.825	0.1984	19.28	0.000
Level	- 4.550	- 2.275	0.1984	-11.45	0.000
Feeding	1.750	0.875	0.1984	4.41	0.012

Analysis of Variance for EG (code units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	164.575	164.575	54.8583	174.15	0.000
Residual Error	4	1.260	1.260	0.3150		
Total	7	165.835				

Estimated Coefficients for EG using data in uncoded units

Term	Coef
Constant	-387.183
Temp	1.91250
Level	-1.51667
Feeding	0.0175000

Figure 4. Analysis of Variance of the reduced model

Main Effects Plot (data means) for EG Weight

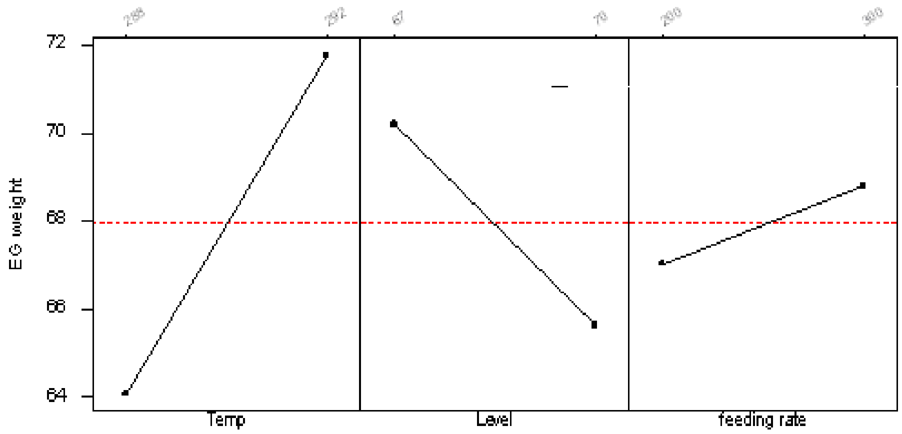

Figure 5. Main effect plots of three input factors

Based on these results, several trial runs were performed during January 16th- Jan. 30th, 2004 to confirm the required results. The trial runs showed stability pattern of the EG amount in production, and the average amount of EG was at 63.33 mg/L, which previously was 70.47 mg/L, as shown in Figure 6. This led to low BOD in waste water, but polyester quality was still the same.

Figure 5 and Equation show the directtion of new setting condition. They indicatee that the low level of reation temperature and EG feeding rate but the high level of slurry cause small amount of EG in wast water. At this setting condition, it is possible that the reaction is well complete, so there is only small amount od EG left water.

EG = -387.183 + 1.9125A ñ 1.5167B + 0.0175C ... (1)

where EG = Amount of EG in waste water (mg/L)

A = Reaction temperature (^{0}C)

B = Level of slurry (%)

C = EG feeding rate (kg/hr)

Results of the implementation

After setting reaction temperature at 288 ^{o}C, level of slurry at 70 %, and EG feeding rate at 200 kg/hr, the average amount of EG significantly decreased from 70.47 mg/L to 63.32 mg/L as shown in Figure 7. Also, BOD and waste water treatment cost were decreased as seen in Figure 8 and 9. Figure 9 showed that cost of waste water treatment was decreased from 1.927 million baht per month on average to 1.391 million baht per month in January after improvement, which is lower than the budget cost.

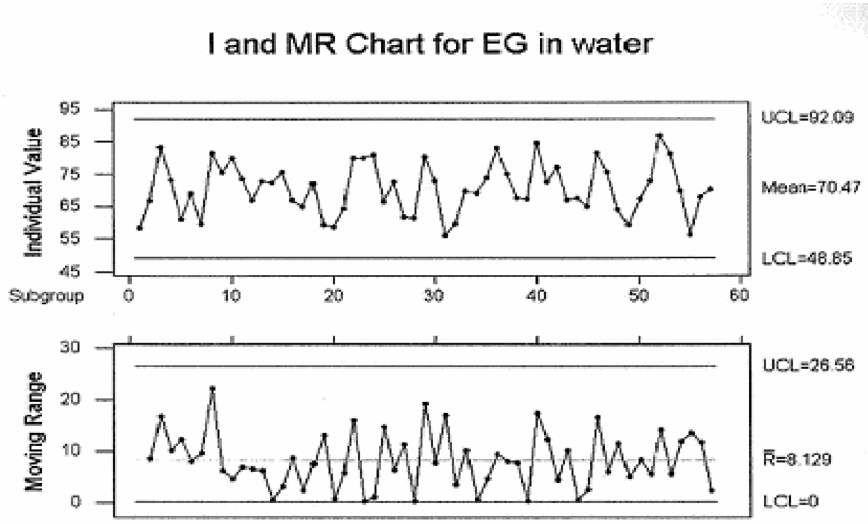

Figure 6. Conrol Chart of the EG amount after setting at the recommend condition

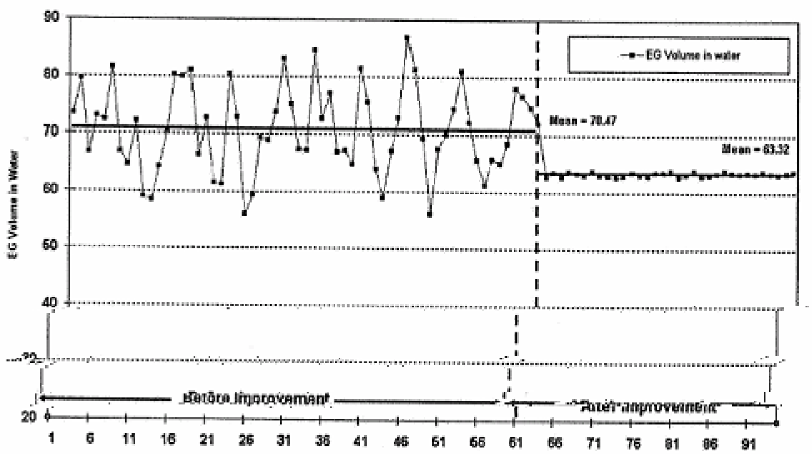

Figure 7. Amount of EG before and after improvement

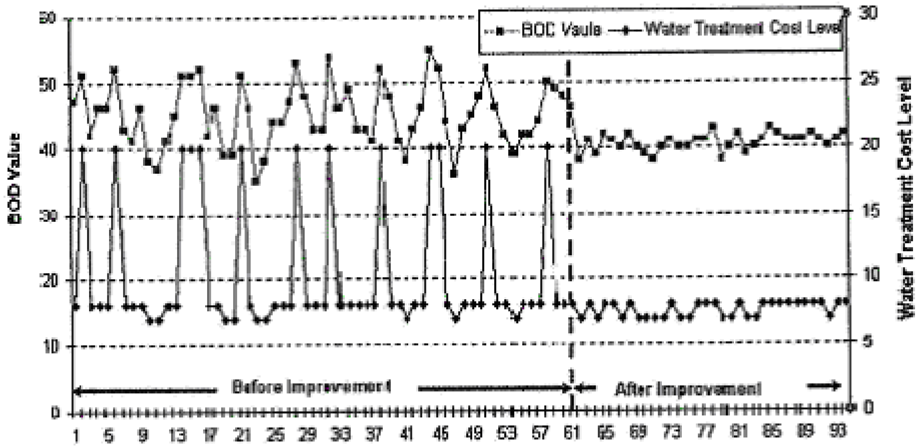

Figure 8. BOD and waste water treatment cost before and after improvement

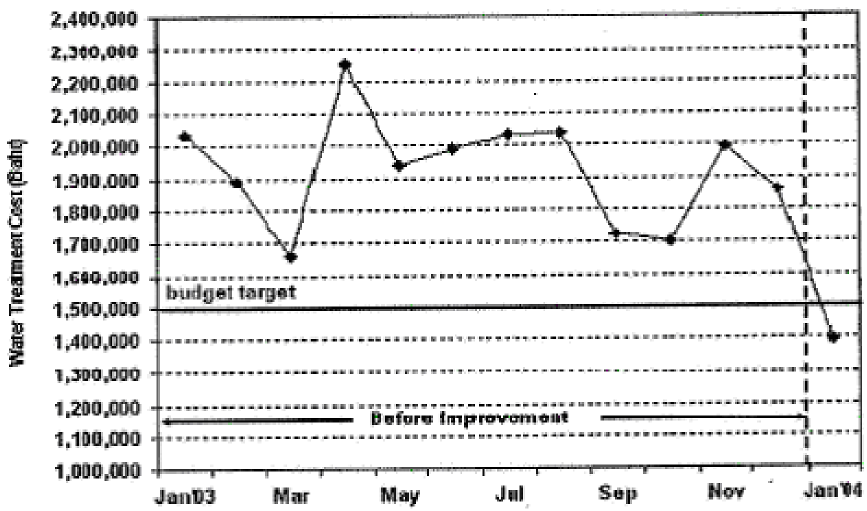

Figure 9. Cost of waste water before and after improvement

Conclusion

This study aimed to reduce the waste water treatment cost. The experimental designs were the methodology used to collect data because they could lead to the required results with minimum unexplained error.The results showed that the reaction temperature,the level of slurry in reactor, and the EG feeding rate are input variables affecting amount of EG, which high amount of EG leads to high cost of treatment. In addition, the results reveal the best setting condition. This study was performed under the current process specifications because the study outside the process specifications may cause high losses.Searching the best result within the current process specification is called looking for the best practice.Therefore, in this study,researchers searched for the best practice in the esterification reaction.In the other words,researchers looked for the best setting condition giving the lowest waste water treatment cost, and put the findings to the standard of the current process. Obviously,this study shows an efficient methodology in cost reduction and standardization using statistics.

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