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ผลิตไบโอดีเซลด้วยตัวเร่งปฏิกิริยาแบบอัลคาไลน์



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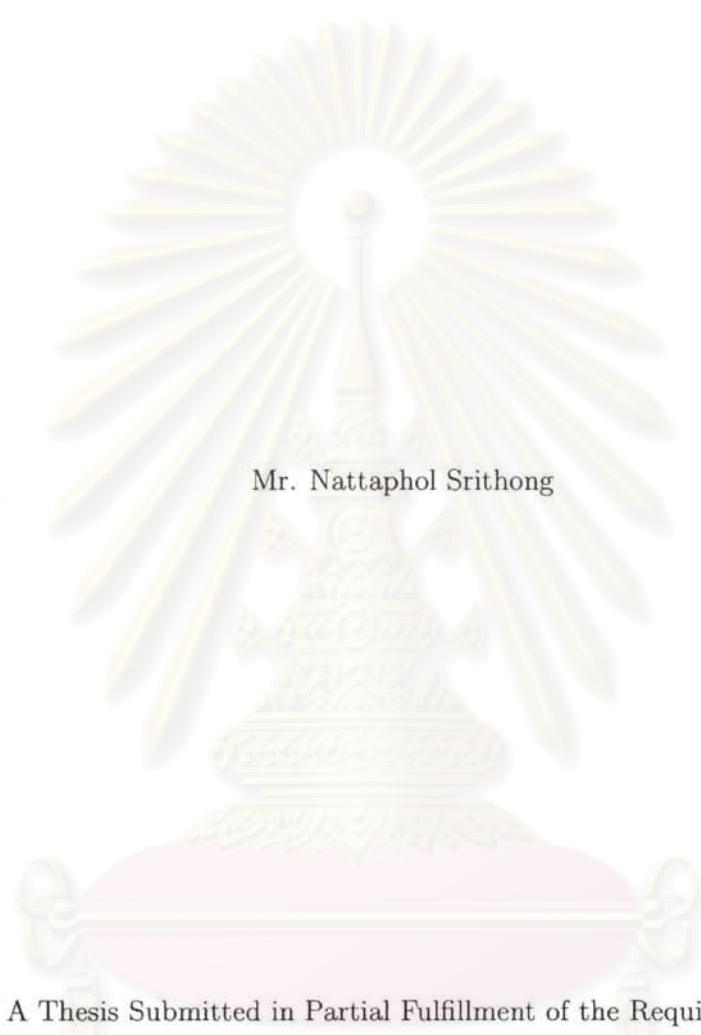
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PLANTWIDE CONTROL DESIGN OF BIODIESEL PRODUCTION  
PROCESS WITH ALKALI-CATALYST



Mr. Nattaphol Srithong

A Thesis Submitted in Partial Fulfillment of the Requirements  
for the Degree of Master of Engineering Program in Chemical Engineering

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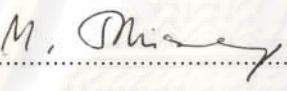
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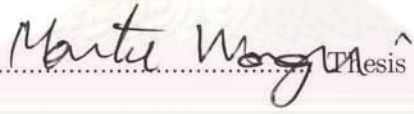
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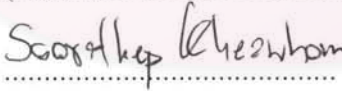
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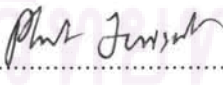
  
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ณัฐพล ศรีทอง: การออกแบบการควบคุมแบบแพลนตัววัดของกระบวนการผลิตไบโอดีเซลด้วย  
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การศึกษานี้คือการใช้ขั้นตอนการควบคุมแบบแพลนตัววัดสำหรับการออกแบบโครงสร้างการ  
ควบคุมกระบวนการผลิตไบโอดีเซลจากน้ำมันปาล์ม ด้วยระบบเบสเป็นตัวเร่งปฏิกิริยา ในการออกแบบ  
โครงสร้างนี้ เพื่อวัตถุประสงค์ที่ตั้งไว้คือ การผลิตไบโอดีเซลให้ได้ความบริสุทธิ์มากกว่า 99.65% และ  
กลีเซอรินให้ได้ความบริสุทธิ์มากกว่า 92% ตามมาตรฐาน ASTM เนื่องด้วยกระบวนการผลิตไบโอดีเซล  
ประกอบไปด้วยหน่วยผลิตหลายหน่วย การศึกษานี้ก็เพื่อคุณูปการของกระบวนการผลิตไบโอดีเซล  
โดยการทดสอบสิ่งรบกวนต่อกระบวนการ ทดสอบปริมาณน้ำมันปาล์มที่เข้าสู่ระบบการผลิตและการ  
เปลี่ยนสถานะของอุณหภูมิในถังปฏิกรณ์ ผลของการทดสอบแสดงให้เห็นว่าโครงสร้างการควบคุมที่ 2  
และ 3 ของวงศ์ศรีให้สมรรถนะของการควบคุมที่ดีกว่าโครงสร้างการควบคุมที่ 1 ของลูย์เบน ตามลำดับ

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จุฬาลงกรณ์มหาวิทยาลัย

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This study is using plantwide control strategies for designing the control structures of an alkali-catalyzed process to produce biodiesel from palm oil that are designed to achieve the control objective. Dynamic behavior of an alkali-catalyzed process produces biodiesel from palm oil is a complex plant consisting of many unit operations. Plantwide control strategies using Luyben's heuristics method and Fixture point of Wongsri were adapted for developed control loops in the biodiesel production plant. In this work presents alkali-catalyzed process to produce biodiesel simulation via using software Aspen HYSYS version 3.1 and design control structure to achieve purity of biodiesel product greater than 99.65%wt. and glycerol by-product greater than 92%wt. (ASTM specification). The control structure for biodiesel production plant showed purity of biodiesel and glycerol by-product greater than ASTM.

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ศูนย์วิทยทรัพยากร  
จุฬาลงกรณ์มหาวิทยาลัย

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# CHAPTER 1

## INTRODUCTION

This chapter consists of importance and reasons for research, objectives of research, scopes of research, contributions of research and research procedures.

### 1.1 Importance and Reasons for Research

Nowadays, industries are very competitive both in quality and cost of production. Therefore, production process should have high quality and high efficiency. The process should always operate under the design condition, use little energy, low waste production and meet the required specification of the products. The quality should not be higher or lower than the specification. If the quality is higher, the cost of production will be high. On the contrary, if the quality is lower, the products can not be sold out. In the real situation, the process will not operate smoothly. All factors do not meet the designed conditions. The process always changes due to disturbance from the external actors and the internal factors. However, not matter what factors cause the change, in case of having deviation or disturbance come in to the process, the effect should be eliminated from the process as soon as possible so that the process will have the least deviation from the designed condition. Moreover, due to the restriction of the environment, safety and operating condition, it is very necessary to have the control system to control the condition and compensate for any deviation occurred.

In general, the production process in all plants do not have single process or have only single unit operation separated from other process but they have several operation units. For example, machines and other equipment that are related and have raw materials, production and energy transferred among the units will work together to change raw materials into production. They try to use production

factors efficiently. Therefore, plant means machines or units or equipments operating together to achieve to production purpose. Previously, the control system of the process to designed to control only each individual unit. Then, each unit in the plant will be combined. As a result, there is the interaction between them. A chemical plant may have thousands of measurements and control loops. By the term plantwide control it is not meant the tuning and behavior of each of these loops, but rather the control philosophy oh the overall plant with the emphasis on the structural decisions. The structural decision include the section/placement of manipulators and measurements as well as the decomposition of the overall problem into smaller subproblems (the control configuration)In this research, it will focus on heuristic-based plantwide control procedure applied to Biodiesel production process with Alkali-catalyzed system. The American Society for Testing and Materials (ASTM) defines biodiesel fuel as monoalkyl esters of long chain fatty acids derived from a renewable lipid feedstock, such as vegetable oil or animal fat. "Bio" represents its renewable and biological source in contrast to traditional petroleum-based diesel fuel; "diesel" refers to its use in diesel engines. As an alternative fuel, biodiesel can be used in neat form or mixed with petroleum-based diesel. Biodiesel, as an alternative fuel, has many merits. It is derived from a renewable, domestic resource, thereby relieving reliance on petroleum fuel imports. It is biodegradable and non-toxic. Compared to petroleum-based diesel, biodiesel has a more favourable combustion emission profile, such as low emissions of carbon monoxide, particulate matter and unburned hydrocarbons. Carbon dioxide produced by combustion of biodiesel can be recycled by photosynthesis, thereby minimizing the impact of biodiesel combustion on the greenhouse effect. Biodiesel has a relatively high flash point which makes it less volatile and safer to transport or handle than petroleum diesel. It provides lubricating properties that can reduce engine wear and extend engine life. In brief, these merits of biodiesel make it a good alternative to petroleumbased fuel and have led to its use in many countries, especially in environmentally sensitive areas.



Biodiesel production process is a complex plant consisting of many unit operations, one recycle stream that create disturbance propagation and the complicated system's dynamic behavior. Therefore, this research will design plantwide control structures of Biodiesel production process and simulate them by using HYSYS simulator in order to study about dynamic behavior and evaluate the performance of the designed structures. An effective designed structure can achieve the control objective to reduce the cost of production and operate the process within safety and environmental constraint.

## 1.2 Research of the Objective

1. To simulate Biodiesel Production Process both steady state and dynamics condition by using HYSYS simulator.
2. To design plantwide control structures of Biodiesel Production Process.
3. To evaluate dynamic performance of the design control structures.

## 1.3 Scopes of the Research

1. The simulator in this research is HYSYS version 3.1 simulator.
2. The design control structures for Biodiesel Production Process are design using luyben's heuristics method.
3. The description of Biodiesel Production Process with Alkali-Catalyzed System is given by Y. Zhang, M.A. Dube', D.D. McLean , M. Kates (2003)
4. The control structures must be able to achieve purification Biodiesel product greater than 99.65% by-product greater than 85% respectively.

## 1.4 Contributions of the Research

1. Process flowsheet diagram of Biodiesel Production Process with Akali-Catalyzed system has been simulated.
2. The control structures are designed by using plantwide control procedure.
3. The dynamic performance of the designed control structure are compared with an earlier control structure in the same process.

## 1.5 Research Procedures

1. Study of plantwide process control theory. Biodiesel Production Process and concerned information
2. Simulation of the Biodiesel Production process at steady state.
3. Simulation of the Biodiesel Production process at dynamic.
4. Design the control structures of Biodiesel Production process.
5. Evaluation of the dynamics performance of the control structures.
6. Collection and summarization of simulation result.

## 1.6 Research Contents

This thesis is divided into six chapters.

**Chapter I** is an introduction to this research. The chapter consists of research objectives, scope of research. contribution of research, and procedure plan.

**Chapter II** reviews the work carried out on biodiesel production and biodiesel process plant, plantwide control design.

**Chapter III** cover some background information of Luyben and theory of Luyben's method for control structure design.

**Chapter IV** transesterification reaction of biodiesel, describes the process description, process simulation via HYSYS, calculation of palm oil properties in HYSYS version 3.1 and selection of Fluid Package in biodiesel production process simulation.

**Chapter V** describes the design of plantwide control structures and dynamic simulation results and compare with control structures of Luyben's method and Wongsri's method.

**Chapter VI** preents the conclusion of this research and makes the recommendations for future work.

This is follow by:

#### **References**

**Appendix A:** Process and Equipment Data

**Appendix B:** Parameter Tuning of Control Structures.

**Appendix C:** Fixture Point Theorem Data

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## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Biodiesel Production

Darnoko and Cheryan presents (2000) methyl esters were produced by transesterification of palm oil with methanol in the presence of a catalyst (KOH). The rate of transesterification in a batch reactor increased with temperature up to 60 °C. Higher temperatures did not reduce the time to reach maximal conversion. The conversion of triglycerides (TG), diglycerides (DG), and monoglycerides (MG) appeared to be second order up to 30 min of reaction time. reaction rate constants for TG, DG, and MG hydrolysis reactions were 0.018-0.191 (wt%.min)<sup>-1</sup>, and were higher at higher temperature and higher for the MG reaction than for TG hydrolysis. Activation energies were 14.7, 14.2 and 6.4 kcal/mol for the TG, DG and MG hydrolysis reactions, respectively. The optimal catalyst concentration was 1% KOH.

Y. Zhang , M.A. Dube' , D.D. McLean and M. Kates (2003) Kates presents four different continuous process flowsheets for biodiesel production from virgin vegetable oil or waste cooking oil under alkaline or acidic conditions on a commercial scale were developed. Detailed operating conditions and equipment designs for each process were obtained. A technological assessment of these four processes was carried out to evaluate their technical benefits and limitations. Analysis showed that the alkali-catalyzed process using virgin vegetable oil as the raw material required the fewest and smallest process equipment units but at a higher raw material cost than the other processes. The use of waste cooking oil to produce biodiesel reduced the raw material cost. The acid-catalyzed process using waste cooking oil proved to be technically feasible with less complexity than the

alkali-catalyzed process using waste cooking oil, thereby making it a competitive alternative to commercial biodiesel production by the alkali-catalyzed process.

P.C. Narváez, S.M. Rincón and F.J. Sánchez (2007) presents kinetics study of palm oil methanolysis was conducted at three different temperatures and three different concentrations of catalyst, sodium hydroxide, keeping constant the molar ratio of methanol to oil and the rotational speed of the impeller (6:1 and 400 rpm). The maximum conversion of palm oil and productivity to methyl esters were obtained at 60°C and 1 wt% of NaOH based on palm oil, and they were 100 and 97.6%, respectively. The statistical analysis of conversions of palm oil and productivities to methyl esters as functions of temperature and concentration of catalyst, after 80 min of reaction, allowed them to fit second order polynomial equations, which adequately describe the experimental behavior. The experimental data appear to be a good fit into a second order kinetic model for the three stepwise reaction, and the reaction rate constants and the activation energies were determined. In this article we present the kinetic constant and activation energies for the experiments with 0.2% wt of NaOH. The effect of molar ratio on the concentration of products was investigated, while the temperature (55°C), the concentration catalyst (0.6% wt of NaOH), and the rotational speed (400 rpm), were held constant. The results showed that the conversion and productivity increased due to methanol excess, and were higher for the reactions with a molar ratio of 6:1.

Alex H. West, Dusko Posarac and Naoko Ellis (2007) presents four continuous biodiesel processes were designed and simulated in HYSYS. The first two employed traditional homogeneous alkali and acid catalysts. The third and fourth processes used a heterogeneous acid catalyst and a supercritical method to convert a waste vegetable oil feedstock into biodiesel. While all four processes were capable of producing biodiesel at high purity, the heterogeneous and supercritical processes were the least complex and had the smallest number of unit operations. Material and energy flows, as well as sized unit operation blocks, were used to

conduct an economic assessment of each process. Total capital investment, total manufacturing cost and after tax rate-of-return were calculated for each process. The heterogeneous acid catalyst process had the lowest total capital investment and manufacturing costs, and had the only positive after tax rate-of-return.

## 2.2 Control Structure Design

The concept plantwide control structure synthesis is not new to the chemical process industry. The first documented plantwide control approach was suggested by Buckley in 1984. In the 1980s the research in this area grew, in part because of the number of heat management systems that were being installed. However, compared to the number of the models available for the synthesis of control structures for individual unit operations. The number of the plantwide control synthesis methods has remained modest. This sparsity reflects the complexity inherent in the plantwide control problem, which includes a multitude of objectives, conflicting objective, interactions, large dimensionality, and non-linearity. The research studies that have appeared in the open literature for plantwide control can be broadly classified into three categories.

Price, R., and Georgakis, C. (1993) presented a five-tiered approach based on inventory (material balance closure), production, and quality control. The control design development is done in stages that correspond to the goals and tasks of the proposed control system. The five tiers: production rate; inventory maintenance; product specification; equipment and operating constraints; and economic performance. There are differences between this method and proposed by Luyben et al.

Luyben, W. L. (1994) presents a mathematical analysis of the problem for several typical kinetic systems. In the simple binary first-order case of  $A \rightarrow B$ ,

an analytical solution can be found for the recycle flow rate as a function of the fresh feed flow rate and fresh feed composition. Two different control structures are explored. It is shown analytically why the control structure proposed by Luyben prevents snowballing and why the conventional structure results in severe snowballing. Two other kinetic systems are studied numerically: consecutive first-order reaction  $A \rightarrow B \rightarrow C$  and a second-order reaction  $A + B \rightarrow C$ . Results confirm that snowball problems can be prevented by using a control structure that fixes the flow rate of one stream somewhere in a liquid recycle loop. In processes with one recycle, the flow rate of the reactor effluent can be set. In processes with two or more recycle streams, the flow rate of each recycle can be fixed.

Luyben and Luyben (1995) examines the plantwide design and control of a complex process. The plant contains two reaction steps, three distillation columns, two recycle streams, and six chemical components. Two methods, a heuristic design procedure and a nonlinear optimization, have been used to determine an approximate economically optimal steady-state design. The designs differ substantially in terms of the purities and flow rates of the recycle streams. The total annual cost of the nonlinear optimization design is about 20 percent less than the cost of the heuristic design. An analysis has also been done to examine the sensitivity to design parameters and specifications. Two effect control strategies have been developed using guidelines from previous plantwide control studies; both require reactor composition control as well as flow control of a stream somewhere in each recycle loop. Several alternative control strategies that might initially have seemed obvious do not work.

Luyben, Tyreus and Luyben (1997) presented a general heuristic design procedure. Their procedure generated an effective plantwide control structure for an entire complex process flowsheet and not simply individual units. The nine steps of the proposed procedure center around the fundamental principles of plantwide control: energy management, production rate, product quality, operational, environmental and safety constraints, liquid-level and gas-pressure invento-

ries, makeup of reactants, component balances and economic or process optimization. Application of the procedure was illustrated with three industrial examples: the vinyl acetate monomer process, Eastman process and HDA process. The procedure produced a workable plantwide control strategy for a given process design. The control system was tested on a dynamic model built with TMODES, Dupont's in-house simulator.

There are two basic control structures for chemical plants: fixed feed and fixed products. Luyben point out that the fixed product, which called on-demand structure, has several inherent dynamics disadvantages compared to the more conventional approach of setting the feed streams to a process. They are illustrated on two processes of increasing complexity. The first is a binary system with the reaction  $A \rightarrow B$  and a plant topology of one reactor, one stripping column, and one recycle stream. The second is a ternary system with the reaction  $A + B \rightarrow C$ , and a floesheet containing one reactor, two distillation columns, and two recycle streams. Dynamic simulations demonstrate that the on-demand structure introduces larger disturbances into the system, which results in more variability in product quality. (Luyben, W. L. 1999)

After that, Luyben (2000) studied the process had the exothermic, irreversible, gas-phase reaction  $A + B \rightarrow C$  occurring in an adiabatic tubular reactor. A gas recycle returns unconverted reactants from the separation section. Four alternative plantwide control structures for achieving reactor exit temperature control had been compared. Manipulation of reactor inlet temperature appeared to be the least attractive scheme. Manipulation of recycle flow rate gave the best control but may be undesirable in some system because of compressor limitations. The on-demand structure provided effective control in the face of feed composition disturbances.

Kapilakarn, K. and Luyben, W. L. present the plant wide control of continuous process multiple products. There are two reversible reactions producing two



products. The control structure must be able to achieve different production rates of the two products. Several conventional control structure are studies in which the flow rates of the fresh feed streams are fixed or manipulated by level or composition controllers and the production rates of the two products are not directly set. They present several "on demand" control structure which both product streams are flow controlled. The control system must adjust the conditions in the plant and the fresh feed streams to achieve the desired product flow rates. The most effective on-demand control structure require on reactor composition analyzer and on recycle of product streams.

Lesson et al. (2003) present control structure selection for a simple plant with a liquid-phase reactor, a distillation column, and recycle of unreacted reactants. The starting point is a clear definition of the operational objectives, constraints, and degrees of freedom. Active constraints should be controller to optimize the economic performance. This implies for this case study that the reactor level should be kept at its maximum, that being economically attractive. Maximizing the reactor holdup also minimizes the "snowball effect". The main focus is not the selection of a suitable controlled variable for the remaining unconstrained degree of freedom, that use the concept of self-optimizing control to search for a constant setpoint strategy with an acceptable economic loss. Both for the case with a given feed rate where the energy costs should be minimized and for the case where the production rate should be maximized, they find that a good controlled variable is the reflux ratio  $L/F$ . this applies to single-loop control as well as multivariable model predictive control.

Murthy Konda et al. (2005) present the novel plantwide control (PDC) methodologies are becoming increasingly important as chemical processes are becoming more and more integrated with recycle for reasons of safety, environmental considerations, and economics. Hence, in the present work, an integrated framework of simulation and heuristics is proposed. The main emphasis here is on vertical integration of simulation and heuristics which exploits the inherent inter-

link between them. By adopting this framework, simulators can be more efficiently utilized and they also offer invaluable support to the decisions taken by heuristics. The proposed framework is then successfully applied to an industrially relevant case study: the hydrodealkylation of toluene (HDA) process. An analysis of results shows that the proposed framework builds synergies between the powers of both the simulation and the heuristics, thereby resulting in a practical PWC methodology that leads to a viable control system.



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## CHAPTER 3

### THEORY

#### 3.1 The main function of control system

In general, the control system installed in process has main function.

##### 3.1.1 To reject disturbance

It is the main objective in installing control system. The external disturbance is uncertain so the operator cannot monitor the changing in process. As a result, the control system must be installed to follow the changing of the process and manipulate the process variable to compensate for the disturbance from external factors.

##### 3.1.2 To maintain stability

The stability is necessary for every process. As a result the control system is set to improve the process stability for the guarantee of quality of product, safety to equipment of process and plant.

##### 3.1.3 To keep the process performing highest efficiency

Besides rejecting disturbance and maintaining stability, the control system can achieve the great profit because it losses less energy and raw materials during the operating. Moreover the product will meet the required specification and have high production rate.

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## 3.2 Integrated Processes

There basic features of integrated chemical processes lie at the root of the need to consider the entire plant's control system.

- (1) The effect of material recycle
- (2) The effect of energy integration
- (3) The need to account for chemical component inventories.

If these issues were not had to worry about, then a complex plantwide control problem was not had to deal with. However, there are fundamental reasons why each there exists in virtually all real processes.

### 3.2.1 Material Recycle

Material is recycle for six basic and important reasons.

- Increase conversion: For chemical involving reversible reaction, conversion of reactant to products is limited by thermodynamic equilibrium constraints. Therefore the reactor effluent by necessity contains both reactants and products. Separation and recycle of reactants are essential if process is to be economically viable.
- Improve economics: In most systems it is simply cheaper to build a reactor with incomplete conversion and recycle reactants than it is to reach the necessary conversion level in one reactor or several in series. A reactor followed by a stripping column with recycle is cheaper than one large reactor or three reactors in series.
- Improve yields: In reaction systems such as  $A \rightarrow B \rightarrow C$ , where B is the desired product, the per-pass conversion of A must be kept low to avoid producing too much of the undesirable product C. Therefore the concentration of B is kept fairly low in the reactor and a large recycle of A is required.
- Provide thermal sink: In adiabatic reactors and in reactors where cooling is difficult and exothermic heat effects are large, it is often necessary to feed

excess material to the reactor (an excess of one reactant or a product or a product) so that the reactor temperature increase will not be too large. High temperature can potentially create several unpleasant events: it can lead to thermal runaways, it can deactivate catalysts, it can cause undesirable side reactions, it can cause mechanical failure of equipment, etc. So the heat of reaction is absorbed by the sensible heat required to rise the temperature of the excess material in the stream flowing through the reactor.

- Provide side reactions: A large excess of one of the reactants is often used so that the concentration of the other reactant is kept low. If this limiting reactant is not kept in low concentration, it could react to produce undesirable products. Therefore the reactant that is in excess must

### 3.2.2 Energy Integration

The fundamental reason for the use of energy integration is to improve the thermodynamic efficiency of the process. This translates into a reduction in utility cost. For energy-intensive processes, the savings can be quite significant.

### 3.2.3 Chemical Component Inventories

A plant's chemical species can be characterized into three types: reactants, product, and inerts. A material balance for each of these components must be satisfied. This is typically not a problem for products and inerts. However, the real problem usually arises when reactants are considered (because of recycle) and accounted for their inventories within the entire process. Every molecule of reactants fed into the plant must either be consumed via reaction or leave as an impurity or purge. Because of their value, the loss of reactants exiting the process must be minimized since this represents a yield penalty. So reactants are prevented from leaving. Energy mole of reactant fed to the process is consumed by the reaction.

This is an important concept and is generic to many chemical component balancing is not a problem because exit streams from the unit automatically adjust their flows and composition. However, when units are connected together with recycle streams, the entire system behaves almost like a pure integrator in the terms of the reactants. If additional reactant is fed into the system without changing reactor condition to consume the reactant, this component will build up gradually within the plant because it has no place to leave the system.

### 3.3 Plantwide process control

Control analysis and control system design for chemical and petroleum processes have traditionally followed the "unit operations approach". First, all of the control loops were established individually for each unit or piece of equipment in the plant. Then the pieces were combined together into an entire plant. This meant that any conflicts among the control loops somehow had to be reconciled. The implicit assumption of this approach was that the sum of the individual parts could effectively comprise the whole of the plant's control system. Over the last few decades, process control researchers and practitioners have developed effective control schemes for many of the traditional chemical unit operations. And for processes where these unit operations are arranged in series, each downstream unit simply sees disturbances from its upstream neighbor.

Most industrial processes contain a complex flowsheet with several recycle streams, energy integration, and many different unit operation. Essentially, the plantwide control problem is how to development the control loops needed to operate an entire process and achieve its design objective. Recycle streams and energy integration introduce a feedback of material and energy among units upstream and downstream. They also interconnect separate unit operations and create a pate for disturbance propagation. The presence of recycle streams profoundly alters that is not localized to an isolated part of the process.

Despite this process complexity, the unit operations approach to control system design has worked reasonably well. In the past, plants with recycle streams contained many surge tanks to buffer disturbance, to minimize interaction, and to isolate units in the sequence of material flow. This allowed each unit to be controlled individually. Prior to the 1970s, low energy costs meant little economic incentive for energy integration. However, there is growing pressure to reduce capital environmental concerns. This has prompted design engineers to start eliminating many surge tanks, increasing recycle streams, and introducing heat integration for both existing and new plants. Often this is done without a complete understanding of their effects on plant operability.

So economic force within the chemical industry are compelling improved capital productivity. Requirements for on-aim product quality control grow increasingly tighter. More energy integration occurs. Improved product yields, which reduce raw material costs, are achieved via lower reactant per-pass conversion and higher material recycle rates through the process. Better product quality, energy integration, and higher yields are all economically attractive in the steady-state flowsheet by they present significant challenges to smooth dynamic plant operation. Hence an effective control system regulating the entire plants operation and a process designed with good dynamic performance play critical parts in achieving the business objectives of reducing operating and capital costs.

Buckley (1964) proposed a control design procedure for the plantwide control problem that consisted of two stages. The first stage determined the material balance control structure to handle vessel inventories for low-frequency disturbances. The second established the product quality control structure to regulate high-frequency disturbances. This procedure has been widely and effectively utilized. It has served as the conceptual framework in many subsequent ideas for developing control systems for complete plants. However, the two-stage Buckley procedure provides little guidance concerning three important aspects of a plantwide control strategy. First, it does not explicitly discuss energy manage-

ment. Second, it does not address the specific issues of recycle systems. Third, it does not deal with component balance in the context of inventory control. By placing the priority on material balance over product quality control, the procedure can significantly limit the flexibility in choosing the latter.

The goals for an effective plantwide process control system include

- (1) Safe and smooth process operation.
- (2) Tight control of product quality in the face of disturbances.
- (3) Avoidance of unsafe process conditions.
- (4) A control system runs in automatic, not manual, requiring minimal operator attention.
- (5) Rapid rate and product quality transitions.
- (6) Zero unexpected environmental releases.

### 3.4 Step of Plantwide Process Control Design Procedure

The nine steps of the design procedure center around the fundamental principles of plantwide control: energy management; production rate; product quality; operational, environmental, and safety component balance; and economic or process optimization.

#### **Step1: Establish control objective**

Assess the steady-state design and dynamic control objective for the process.

This is probably the most important aspect of the problem because different control objectives lead to different control structures. The “best” control structure for a plant depends upon the design and control criteria established.



These objectives include reactor and separator and separation yields, product quality specifications, product grades and demand determination, environmental restriction, and the range of safe operation conditions.

### **Step2: Determine control degrees of freedom**

Count the number of control valves available.

This is the number of degree of freedom for control, i.e., the number of variables that can be controlled to setpoint. The valves must be legitimate (flow through a liquid-filled line can be regulated by only one control valve). The placement of these control valves can sometimes be made to improve dynamic performance, but often there is no choice in their location.

Most of these valves will be used to achieve basic regulatory control of the process;

- (1) Set production rate
- (2) Maintain gas and liquid inventories
- (3) Control product qualities
- (4) Avoid safety and environmental constraints

Any valves that remain after these vital tasks have been accomplished can be utilized to enhance steady-state economic objectives or dynamic controllability (e.g., minimize energy consumption, maximize yield, or reject disturbances).

### **Step3: Establish energy management system**

Make sure that energy disturbances do not propagate throughout the process by transferring the variability to the plant utility system.

The term energy management is used to describe two functions:

- (1) To provide a control system that removes exothermic heats of reaction from the process. If heat is not removed to utilities directly at the reactor, then it

can be used elsewhere in the process by other unit operations. This heat, however, must ultimately be dissipated to utilities.

(2) To provide a control system that prevents the propagation of thermal disturbances and ensures the exothermic reactor heat is dissipated and not recycled. Process-to-process heat exchangers and heat-integrated unit operation must be analyzed to determine that there are sufficient degrees of freedom for control.

Heat removal in exothermic reactors is crucial because of the potential for thermal runaways. In endothermic reaction, failure to add enough heat simply results in the reaction slowing up. If the exothermic reactor is running adiabatically, the control system must prevent excessive temperature rise through the reactor (e.g., by setting the ratio of the flowrate of the limiting fresh reactant to the flowrate of a recycle stream acting as a thermal sink).

Heat transfer between process stream can create significant interaction. In the case of reactor feed/effluent heat exchangers it can lead to positive feedback and even instability. Where there is partial condensation or partial vaporization in a process-to-process heat exchanger, disturbances can be amplified because of heat of vaporization and temperature effects.

#### **Step4: set production rate**

Establish the variables that dominate the productivity of the reactor and determine the most appropriate manipulator to control production rate.

Throughput changes can be achieved only by altering, either directly or indirectly, conditions in the reactor. To obtain higher production rates, The overall reaction rates must be increased. This can be accomplished by raising temperature (higher specific reaction rate), increasing reactant concentrations, increasing reactor holdup (in liquid-phase reactors), or increasing reactor pressure (in gas-phase reactors).

Our first choice for setting production rate should be to alter one of these variables in the reactor. The variable that is selected must be dominant for the reactor. Dominant reactor variables always have significant effects on reactor performance. For example, temperature is often a dominant reactor variable. In irreversible reactions, specific rates increase exponentially with temperature. As long as reaction rates are not limited by low reactant concentrations, temperature can be increased to increase production rate in the plant. In reversible exothermic reactions, where the equilibrium constant decreases with increasing temperature, reactor temperature may still be a dominant variable. If the reactor is large enough to reach chemical equilibrium at the exit, the reactor temperature can be decreased to increase production.

There are situations where reactor temperature is not a dominant variable or cannot be changed for safety or yield reasons. In these cases, another dominant variable must be found, such as the concentration of the limiting reactant, flowrate of initiator or catalyst to the reactor, reactor residence time, reactor pressure, or agitation rate.

Once the dominant variable must be identified, the manipulators (control valves) must also be identified that are most suitable to control them. The manipulators are used in feedback control loops to hold the dominant variables at setpoint. The setpoints are then adjusted to achieve the desired production rate, in addition to satisfying other economic control objectives.

Whatever variable is chosen, it can provide smooth and stable production rate transitions and to reject disturbances. A variable that has the least effect on the separation section but also has a rapid and direct effect on reaction rate in the reactor without hitting an operational constraint is often wanted to be selected.

When the setpoint of a dominant variable is used to establish plant production rate, the control strategy must ensure that the tight amounts of fresh

reactants are brought into the process. This is often accomplished through fresh reactant makeup control based upon liquid levels or gas pressures that reflect component inventories.

However, design constraints may limit our ability to exercise this strategy concerning fresh reactant makeup. An upstream process may establish the reactant feed flow sent to the plant. A downstream process may require on-demand production, which fixes the product flowrate from the plant. In these cases, the development of the control strategy becomes more complex because the setpoint of the dominant variable on the basis of the production rate that has been specified externally must be somehow adjusted. The production rate with what has been specified externally must be balanced. This cannot be done in an open-loop sense. Feedback of information about actual internal plant conditions is required to determine the accumulation or depletion of the reactant components.

**Step5: Control product quality and handle safety, operational, and environmental constraints**

Select the "best" valves to control each of the product-quality, safety, and environmental variables.

The tight control of these important quantities for economic and operational reasons is wanted. Hence the manipulated variables such that dynamic relationships between the controlled and manipulated variables feature small time constants and deadtimes and large steady-state gains should be selected. The former gives small closed-loop time constants and the latter prevents problem with the range ability of the manipulated variable (control valve saturation)

It should be noted that establishing the product-quality loops first. Before the material balance control structure, is a fundamental different between our plantwide control design procedure and Buckley's procedure. Since product quality considerations have become more important in recent year, this shift in

emphasis follows naturally.

The magnitudes of various flowrates also come into consideration. For example, temperature (or bottoms product purity) in a distillation column is typically controlled by manipulating stem flow to the reboiler (column boilup) and base level is controlled with bottoms product flowrate. However, in column with a large boilup ratio and small bottoms flowrate, these loops should be reversed because boilup has a large effect on base level than bottoms flow (Richardson rule). However, inverse response problem in some columns may occur when base level is controlled by heat input. High reflux ration at the top of a column require similar analysis in selecting reflux or distillate to control overhead product purity.

#### **Step6: Control Inventories (pressures and level) and Fix a Flow in Every Recycle Loop**

Determine the valve to control each inventory variable. These variables include all liquid levels and gas pressures. An inventory variable typically be controlled with the manipulate variable that has the largest effect on it within that unit.

Proportional-only control should be used in nonreactive level loops for cascaded units in series. Even in reactor-level control, proportional control should be considered to help filter flow-rate disturbances to the downstream separation system. There is nothing necessarily sacred about holding reactor level constant.

In most processes a flow controller should be present in all liquid recycle loops. This is a simple and effective way to prevent potentially large changes in recycle flow that can occur if all flow in the recycle loop are controlled by levels. Two benefits result from this flow-control strategy. First, the plant's separation section is not subjected to large load disturbance. Second, consideration must

be given to alternative fresh reactant makeup control strategies rather than flow control. In a dynamic sense, level controlling all flows in a recycle loop is a case of recycling disturbances and should be avoided. Gas recycle loops are normally set at maximum circulation rate, as limited by compressor capacity, to achieve maximum yields.

#### **Step7: Check Component Balances.**

Identify how chemical components enter, leave, and are generated or consumed in the process.

Ensure that the overall component balances for each chemical species can be satisfied either through reaction or exit streams by accounting for the component's composition or inventory at some point in the process. Light, intermediate, and heavy inert components must have an exit path from the system. Reactant must be consumed in the reaction section or leave as impurities in the product streams. Fresh reactant makeup feed streams can be manipulated to control reactor feed composition or a recycle stream composition (or to hold pressure or level as noted in the previous step). Purge streams can also be used to control the amount of high- or low-boiling impurities in a recycle stream.

Component balance can often be quite subtle. They depend upon the specific kinetics and reaction paths in the system. They often affect what variable can be used to set production rate or rate in the reactor.

#### **Step8: Control Individual Unit Operations**

Establish the control loops necessary to operate each of the individual unit operations. Many effective control schemes have been established over the years for chemical units. For example, a tubular reactor usually requires control of inlet temperature. High-temperature endothermic reactions typically have a control system to adjust fuel flow rate to a furnace supplying energy to the reactor.

Crystallizers require manipulation in the stack gas from a furnace is controlled to prevent excess fuel usage. Liquid solvent feed flow to an absorber is controlled as some ratio.

### **Step9: Optimize Economics or Improve Dynamic Controllability**

Establish the best way to use the remaining control degrees of freedom.

After satisfying all of the basic regulatory requirements, an additional degrees of freedom involving control valves that have not been used and setpoints in some controllers that can be adjusted. These can be used either to optimize steady-state economic process performance or to improve dynamic response.

#### **Additional considerations**

Certain quantitative measures from linear control theory may help at various steps to assess relationship between the controlled and manipulated variables. These include steady-state process gains, open-loop time constants, singular value decomposition, condition numbers, eigenvalue analysis for stability, etc. these techniques are described in detail in most process control textbooks. The plantwide control strategy should ultimately be tested on a nonlinear dynamic model that captures the essential process behavior.

## **3.5 Plantwide Control Problem**

### **3.5.1 Units in series**

If process units are arranged in a purely series configuration, where the products of each unit feed downstream units and there is no recycle of material or energy, the plantwide control problem is greatly simplified. If is not had to worry about the issues discussed in the previous section and it can be simply configure

the control scheme on each individual unit operation to handle load disturbances.

If production rate is set at the front end of process, each unit will only see load disturbances coming from its upstream neighbor. If the plant is set up for “on-demand” production, changes in throughput will propagate back through the process. So any individual unit will see load disturbances coming from both its downstream neighbor (flowrate changes to achieve different throughputs) and its upstream neighbor (composition changes as the upstream units adjust to the load changes they see).

Figure 3.1 compares these two possible configurations for a simple plant. A fresh feed stream containing a mixture of chemical components A, B and C is fed into a two-column distillation train. The relation volatility are  $\alpha_A > \alpha_B > \alpha_C$  and the “direct” (or “light-out-first”) separation sequence is selected: A is taken out the top of the first column and B out the top of the second column.

Figure 3.1 (a) shows the situation where the fresh feed stream is flow-controlled into the process. The inventory loops (liquid levels) in each unit are controlled by manipulating flows leaving that unit. All disturbances propagate from unit to unit down the series configuration. The only disturbances that each unit sees are changes in its feed conditions.

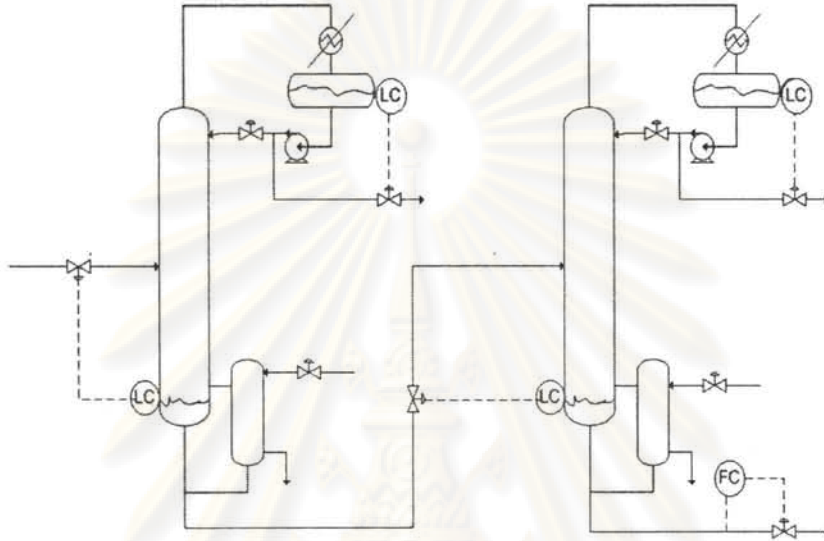
Figure 3.1 (b) shows the on-demand situation where the flowrate of product C leaving the bottom of the second column is set by the requirements of a downstream unit. Now some of the inventory loops (the base of both columns) are controlled by manipulating the feed into each column.

When the units are arranged in series with no recycles, the plantwide control problem can be effectively broken up into the control of each individual unit operation. There is no recycle effect, no coupling, and no feedback of material from downstream to upstream units. The plant’s dynamic behavior is governed

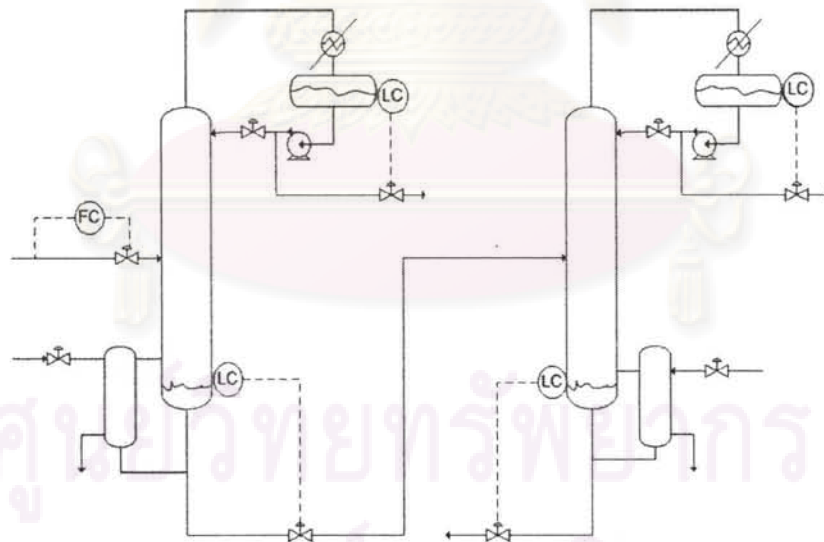


by the individual unit operations and the only path for disturbance propagation is linear along the process.

(a)



(b)



**Figure 3.1** Unit in series. (a) Level control in direction of flow; (b) level control in direction opposite flow.

### 3.5.2 Effects of recycle

Most real processes contain recycle streams. In this case the plantwide control problem becomes much more complex and its solution is not intuitively obvious. The presence of recycle streams profoundly alters the plant's dynamic and steady-state behavior. To gain an understanding of these effects, some very simple recycle systems are looked. The insight they are obtained from these idealized, simplistic systems can be extended to the complex flowsheets of typical chemical processes. First the groundwork must be laid and had some feel for the complexities and phenomena that recycle stream produce in a plant.

Two basic effects of recycle:

(1) Recycle has an impact on the dynamics of the process. The overall time constant can be much different than the sum of the time constants of the individual units.

(2) Recycle leads to the "snowball" effect. This has two manifestations, one steady state and one dynamic. A small change in throughput or feed composition can lead to a large change in steady-state recycle stream flowrates. These disturbances can lead to even larger dynamic changes in flows, which propagate around the recycle loop. Both effects have implications for the inventory control of components.

### 3.5.3 Snowball effects

Another interesting observation that has been made about recycle systems is their tendency to exhibit large variations in the magnitude of the recycle flows. Plant operators report extended periods of operation when very small recycle flows occur. It is often difficult to turn the equipment down to such low flowrates. Then, during other periods when feed conditions are not very different, recycle flowrates increase drastically, usually over a considerable period of time. Often

the equipment cannot handle such a large load.

This high sensitivity of the recycle flowrate to small disturbances is called the snowball effect. It is important to note that this is not a dynamic effect; it is a steady-state phenomenon. But it does have dynamic implications for disturbance propagation and for inventory control. It has nothing to do with closed-loop stability. However, this does not imply that it is independent of the plant's control structure. On the contrary, the extent of the snowball effect is very strongly dependent upon the control structure used.

The large swings in recycle flowrates are undesirable in a plant because they can overload the capacity of the separation section or move the separation section into a flow region below its minimum turndown. Therefore it is important to select a plantwide control structure that avoids this effect. As the example below illustrates and as more complex processes discussed in later chapters also show, a very plant wide control heuristic "A stream somewhere in each liquid recycle loop should be flow controlled"

Let us consider one of the simplest recycle processes imaginable: a continuous stirred tank reactor (CSTR) and a distillation column. As shown in Figure 3.2, a fresh reactant stream is fed into the reactor. Inside the reactor, a first-order isothermal irreversible reaction of component A to produce component B occurs  $A \rightarrow B$ . The specific reaction rate is  $k(h^{-1})$  and the reactor holdup is  $V_R(\text{moles})$ . The fresh feed flowrate is  $F_0(\text{moles/h})$  and its composition is  $z_0(\text{mole fraction component A})$ . The system is binary with only two components: reactant A and product B. The composition in the reactor is  $Z$  (mole fraction A). The reactor effluent, with flowrate  $F$  (moles/h) is fed into a distillation column that separates unreacted A from product B.

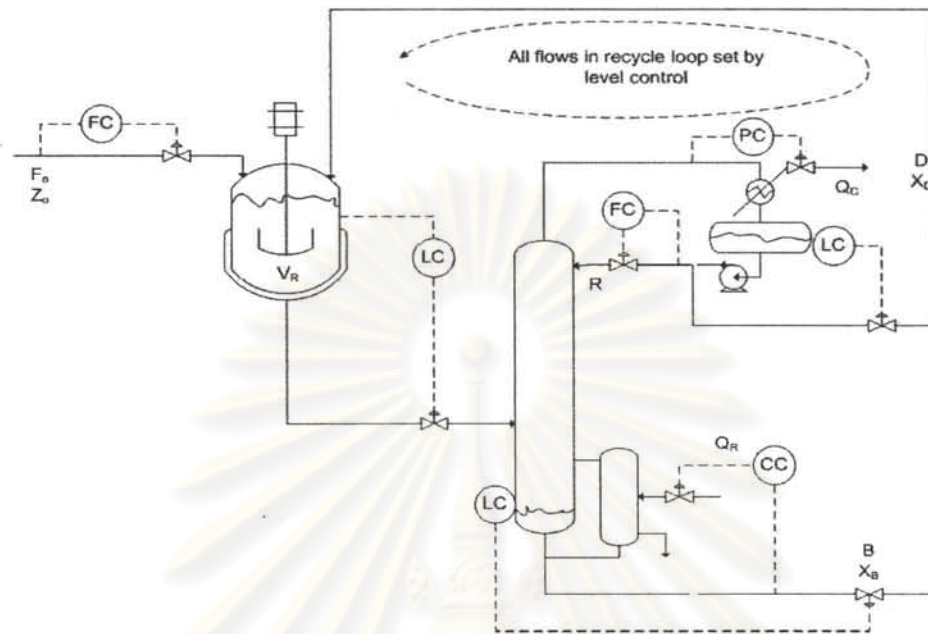
The relative volatilities are such that A is more volatile than B, so the bottom from the column is the product stream. Its flowrate is  $B$  (moles/h) and

its composition is (mole fraction A). the amount of A impurity in this product stream is an important control objective and must be maintained at some specified level to satisfy the product quality requirements of the customer.

The overhead distillate stream from the column contains almost all of component A that leaves the reactor because of the purity specification on the bottoms stream. It is recycled back to the reactor at a flowrate  $D$  and with a composition  $x_D$  (mole fraction A). the column has trays and the feed tray is  $N_F$  (counting from the bottom). The reflux flowrate is  $R$  and the vapor boilup is  $V$  (moles/h).

The two alternative control structures for this process: Conventional control structure As shown in Fig. 3.2, the following control loops are chosen:

1. Fresh feed flow is controlled.
2. Reactor level is controlled by manipulating reactor effluent flow.
3. Bottoms product purity is controlled by manipulating heat input to the reboiler.
4. Distillate purity is controlled by manipulating reflux flow. Note that dual composition control (controlling both distillate and bottoms purities) have been chosen to use in the distillation column, but there is no a priori reason for holding the composition of the recycle stream constant since it does not leave the process. It may be useful to control the composition of this recycle stream for reactor yield purposes or for improved dynamic response. The "best" recycle purity levels in both the design and operation of the plant are been often free to find.
5. Reflux drum level is held by distillate flow (recycle).
6. Base level is held by bottoms flow.
7. Column pressure is controlled by manipulating coolant flowrate to the condenser.



**Figure 3.2** Conventional control structure with fixed reactor holdup

This control scheme is probably what most engineers would devise if given the problem of designing a control structure for this simple plant. Our tendency is to start with setting the flow of the fresh reactant feed stream as the means to regulate plant production rate, and then work downstream from there as if looking at a steady-state flowsheet and simply connect the recycle stream back to the reactor based upon a standard control strategy for the column.

However, this strategy is no flow controller anywhere in the recycle loop. The flows around the loop are set based upon level control in the reactor and reflux drum. This control structure is expected to find that exhibiting the snowball effect. By writing the various overall steady-state mass and component balances around the whole process and around the reactor and column, the flow of the recycle stream can be calculated at steady state for any given fresh reactant feed flow and composition.

With the control structure in Fig. 3.2 and the base-case fresh feed flow and composition, the recycle flowrate is normally 260.5 moles/h. However, the

recycle flow must decrease to 205 moles/h when the fresh feed composition is 0.80 mole fraction A. It must increase to 330 moles/h when the fresh feed composition changes to pure A. Thus a 25 percent change in the disturbance (fresh feed composition) results in a 60 percent change in recycle flow. With this same control structure and the base-case fresh reactant feed composition, the recycle flow drops to 187 moles/h if the fresh feed flow changes to 215 moles/h. It must increase to 362 moles/h when the fresh feed flowrate is changed to 265 moles/h. Thus a 23 percent change in fresh feed flowrate results in a 94 percent change in recycle flowrate. These snowball effects are typical for many recycle systems when control structure such as that shown in Figure 3.2 are used and there is no flow controller somewhere in the recycle loop.

**Variable reactant holdup structure** An alternative control structure is shown in Figure 3.3. This strategy differs from the previous one in two simple but important ways.

1. Reactor effluent flow is controlled.
2. Reactor holdup is controlled by manipulating the fresh reactant feed flowrate.

All other control loops are the same. The production rate cannot change directly by manipulating the fresh feed flow, because it is used to control reactor level. However, the plant throughput can be achieved indirectly in this scheme by changing the setpoint of the reactor level controller. Using the same numerical case considered previously, the recycle flowrate does not change at all when the fresh feed composition changes. To alter production rate from 215 moles/h to 265 moles/h (a 23 percent change), the reactor holdup must be changed from 1030 moles/h to 1520 moles/h (a 48 percent change). Recycle flow also changes, but only from 285 to 235 moles/h. This is an 18 percent change in recycle flow compared with 94 percent in the alternative strategy.

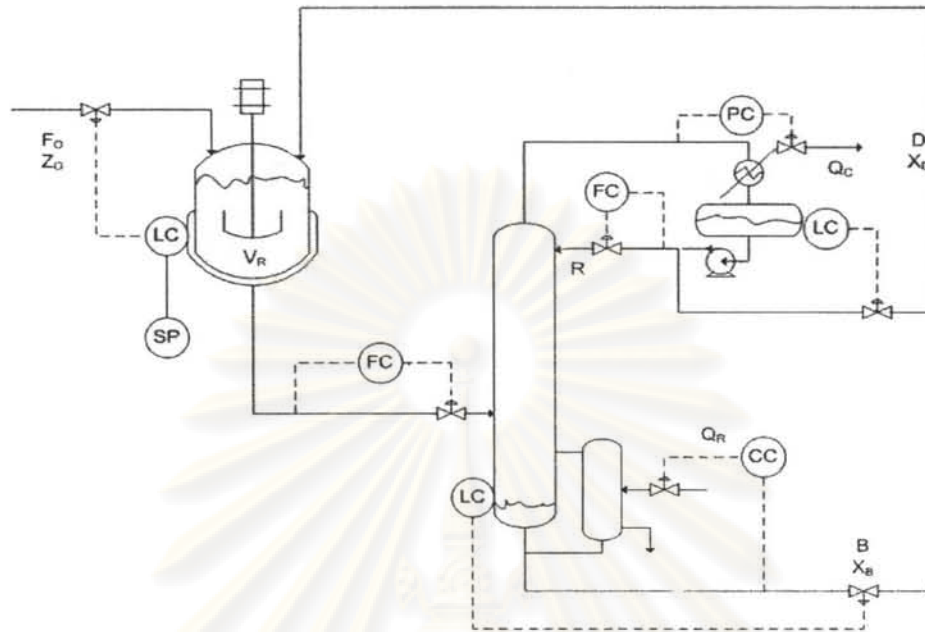


Figure 3.3 Control structure with variable reactor hold

### 3.5.4 Reaction/Separation Section Interaction

For the process considered in the previous section where the reaction is  $R = V_R k z$ , the overall reaction rate depends upon reactor holdup, temperature (rate constant), and reactant composition (mole fraction A)  $R = V_R k z$ . The two control structures considered above produce fundamentally different behavior in handling disturbances. In the first, the separation section must absorb almost all of the changes. For example, to increase production rate of component B by 20 percent, the overall reaction rate must increase by 20 percent since both reactor temperature and reactor holdup  $V_R$  are held constant, reactor composition  $z$  must increase 20 percent. This translates into a very significant change in the composition of the feed stream to the separation section. This means the load on the separation section changes significantly, producing large variations in recycle flowrates.

In the second structure, both reactor holdup and reactor composition  $z$  can change, so the separation section sees a smaller load disturbance. This reduces

the magnitude of the resulting change in recycle flow because the effects of the disturbance can be distributed between the reaction and separation sections.

If the tuning of the reactor level controller in the conventional structure (Fig. 3.2) is modified from normal PI to P only, then changes in production rate also produce changes in reactor holdup. This tends to compensate somewhat for the required changes in overall reaction rate and lessens the impact on the separation section. So both control system structure and the algorithm used in the inventory controller of the reactor affect the amount of this snowball phenomenon.

This example has a liquid-phase reactor, where volume can potentially be varied. If the reactor were vapor phase, reactor volume would be fixed. However, an additional degree of freedom are had and could vary reactor pressure to affect reaction rate.

A very useful general conclusion from this simple binary system can be depicted that is applicable to more complex processes: changes in production rate can be achieved only by changing conditions in the reactor. This means something that affects reaction rate in the reactor must vary: holdup in liquid-phase reactor, pressure in gas-phase reactors, temperature, concentrations of reactants (and products in reversible reactions), and catalyst activity or initiator addition rate. Some of these variables affect the conditions in the reactor more than others. Variable with a large effect are called dominant. By controlling the dominant variables in a process, partial control is achieved. The term partial control arises because it typically have fewer available manipulators than variables that would like to control. The setpoints of the partial control loops are then manipulated to hold the important economic objectives in the desired ranges.

The plantwide control implication of this idea is that production rate changes should preferentially be achieved by modifying the setpoint of a partial



control loop in the reaction section. This means that the separation section will not be significantly disturbed. Using the control structure in Fig. 3.2, changes in production rate require large changes in reactor composition, which disturb the column. Using the control structure shown in Fig. 3.3, changes in production rate are achieved by altering the setpoint of a controlled dominant variable, reactor holdup, with only small changes in reactor composition. This means that the column is not disturbed as much as with the alternative control scheme.

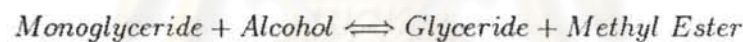
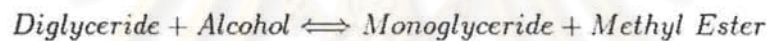
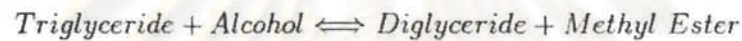


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Methyl esters derived from vegetable oil (biodiesel) have good potential as an alternative diesel fuel. The cetane number, energy content, viscosity, and phase changes of biodiesel are similar to those of petroleum-based diesel fuel. Biodiesel is produced by transesterification of large, branched triglycerides (TG) into smaller, straight-chain molecules of methyl esters, using an alkali or acid as catalyst. There are three stepwise reactions with intermediate formation of diglycerides (DG) and monoglycerides (MG) resulting in the production of 3 mol of methyl esters (EM) and 1 mol of glycerol (GL) as follows (Figure 2).

The stepwise reactions are:



**Figure 4.2** The Stepwise Transesterification Reaction

The governing set of second-order rate equations characterizing the stepwise reactions for transesterification of TG, without the shunt reaction, are as following (Equation 4.1 to 4.6).

$$\frac{d[TG]}{dt} = -k_1[TG].[M] + k_{-1}[DG].[ME] - k_4[TG].[M] + k_{-4}[G].[ME] \quad (4.1)$$

$$\frac{d[DG]}{dt} = k_1[TG].[M] - k_{-1}[DG].[ME] - k_2[DG].[M] + k_{-2}[MG].[ME] \quad (4.2)$$

$$\frac{d[MG]}{dt} = k_2[DG].[M] - k_{-2}[MG].[ME] - k_3[MG].[M] + k_{-3}[G].[ME] \quad (4.3)$$

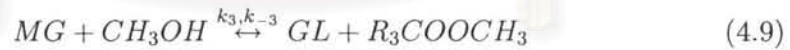
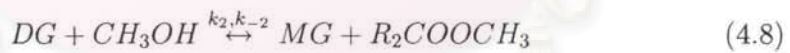
$$\begin{aligned} \frac{d[ME]}{dt} = & k_1[TG].[M] - k_{-1}[DG].[ME] + k_2[DG].[M] - k_{-2}[MG].[ME] \quad (4.4) \\ & + k_3[MG].[M] - k_{-3}[G].[ME] + k_4[TG].[M] - k_{-4}[G].[ME] \end{aligned}$$

$$\frac{d[G]}{dt} = k_3[MG].[M] - k_{-3}[G].[ME] + k_4[TG].[M] - k_{-4}[G].[ME] \quad (4.5)$$

$$\frac{d[M]}{dt} = -\frac{d[ME]}{dt} \quad (4.6)$$

Where  $k_i$  are reaction rate constants (L/mol.s); [TG], [DG], [MG], [GL], [M] and [ME] are mole concentrations of triglyceride, diglyceride, monoglyceride, glyceride, methanol and methyl ester in a reaction mixture (mol/L).

Model formulation this research. Kinetic parameters for palm oil transesterification have been obtained from D. Darnoko and Munir Cheryan (2000) who used sodium hydroxide (NaOH) as catalyst. In particular, the following reaction system, reaction rate equations and kinetic parameters were employed:



with

$$-r_{[4.7]} = k_1 C_{TG} C_{CH_3OH} - k_{-1} C_{DG} C_{R_1COOCH_3} \quad (4.10)$$

$$-r_{[4.8]} = k_2 C_{DG} C_{CH_3OH} - k_{-2} C_{MG} C_{R_2COOCH_3} \quad (4.11)$$

$$-r_{[4.9]} = k_3 C_{MG} C_{CH_3OH} - k_{-3} C_{GL} C_{R_3COOCH_3} \quad (4.12)$$

Where  $C_{TG}$  represents concentration of triglyceride,  $C_{DG}$  dyglyceride, and  $C_{MG}$  monoglyceride, and  $C_{GL}$  represents glycerol.  $C_{R_3COOCH_3}$  are the respective methyl esters (biodiesel). Also  $k_i$  represents the respective kinetic constant that follows the Arrhenius equation:

$$k_i = k_i^\infty e^{-\frac{E}{RT}} \quad (4.13)$$

**Table 4.1** Kinetic Parameters.

Reaction	$E$ activation energy(kcal/mol)	$k$ values at 60°C(1/mol min)
TG → DG	13.145	0.050
DG → MG	19.860	0.215
MG → GL	6.421	0.242

Where  $E$  stands for the activation energy,  $R$  is the ideal gas constant,  $T$  is the absolute temperature, and  $k_i^\infty$  is the pre-exponential factor. The kinetic parameters values used in this study are as shown in Table 4.1 above.

Glycerides were represented by pure components within the process simulator. Those are palm oil  $C_{51}H_{98}O_6$  for triglycerides,  $C_{35}H_{68}O_5$  for diglycerides,  $C_{19}H_{38}O_4$  for monoglycerides, and biodiesel  $C_{17}H_{34}O_2$  for all methyl esters.

## 4.2 Biodiesel Production Process Simulation.

Process simulations support the gathering of information on the process behavior under specific operating conditions. This work builds on the use of specialized tools called simulators, which include thermodynamic packages and unit operation models, as well as calculation strategies for their resolution. In this study, the HYSYS Version 3.1 tool was used to perform the simulation of a process proposed for the generation of fatty acid methyl esters from palm oil in steady state conditions and dynamic mode.

#### **4.2.1 Calculation of Palm Oil Properties in HYSYS Version 3.1**

The procedure needed to perform the simulation implies the definition of substances involved, the selection of a thermodynamic package and process units, including operating conditions such as temperature, pressure, material flow, and alike. In this case, the simulator databases have comprehensive information available on methanol, glycerol, and water. As for palm oil and its methyl ester, such databases do not include the information required for the simulation. Considering that triglycerides with radicals from oleic and palmitic acids feature the highest ratios within the composition of palm oil (Baileys, 1996; Goncalves, 2004), palm oil was represented as a mixture of triolein and tripalmitin, and its methyl ester as a mixture of methyl oleate and methyl palmitate. Parameters were set for the calculation of properties of these compounds through the introduction of properties such as boiling point, combustion heat, and density, specific heat and viscosity against temperature. Once the parameters were calculated, the calculation models and routes were defined. Finally, the values estimated by the simulator were matched against data gathered via the experiments. This is research values estimated

#### **4.2.2 Selection of Fluid Package in Biodiesel Production Process Simulation**

Considering the process operation characteristics, the presence of two non-polar compounds (palm oil and methyl ester) and two highly-polar compounds (methanol and glycerin), and the fact that the esterification reaction takes place in the liquid phase, the decision was made to use a method based on the calculation of activity coefficients (Aspen Technology Inc., 2001; Carlson, 1996). Within the group of possibilities available in the simulator, the UNIFAC method was

chosen, since, in contrast with the UNIQUAC and NRTL models which require molecule-interaction parameters - this is a group-contribution model. Literature has reported that predictions on liquid-liquid balance approximate to experimental data when using models such as UNIQUAC, NRTL and UNIFAC for systems covering compounds similar to those that make up the process (Batista et al., 1999a; Batista et al., 1999b; Goncalves, 2004). The percentage differences between the thermodynamic and transportation properties calculated by the simulator and the properties found in literature for glycerin and methanol, as experimentally defined for the oil and ester, are less than 6%. For calculation of liquid-liquid balance by the UNIFAC model, a set of binary-interaction parameters was found for the groups that make up the molecules.

### 4.3 Biodiesel Production Process Description

In biodiesel production process, it's have six unit:

#### 4.3.1 Transesterification

A continuous alkali-catalyzed process flowsheet using palm oil was developed (Fig. 4.2). The reaction was carried out with a 6:1 molar ratio of methanol to oil, 1% sodium hydroxide (based on oil), 60 °C and 400 kPa. Fresh feed methanol at 117 kg/h, recycled methanol recovery at 111 kg/h and anhydrous sodium hydroxide at 10 kg/h were mixed prior to being pumped into transesterification reactor by pump. palm oil was heated in exchanger before entering transesterification reactor. In reactor, from reserach 95% of oil was converted to FAME, producing glycerol as a by-product. Botton stream from the reactor was introduced to methanol recovery distillation.

### 4.3.2 Methanol recovery

In methanol recovery column, five theoretical stages and a reflux ratio of 2 were used to obtain a good separation between methanol and other components. Top stream was a pure methanol distillate, containing 94% of the total methanol in stream from transesterification reactor. Vacuum distillation was used to keep the bottom temperature under 150 °C. Pure methanol from methanol recovery column was mixed with fresh make-up methanol and then charged back into transesterification reactor. Bottom stream of methanol column was sent to liquid-liquid extraction column after being cooled in exchanger to 60 °C.

### 4.3.3 Water washing (liquid-liquid extraction)

The purpose of this step was to separate the biodiesel from the glycerol, methanol and catalyst. Although separation using a gravity settler was proposed, a complete separation could not be achieved based on our simulation results. Consequently, a water washing column (liquid-liquid extraction with four theoretical stages) was used in this study. The biodiesel in bottom stream from methanol recovery column was separated from the glycerol, methanol and catalyst by adding 11 kg/h water (25 °C). The amounts of unconverted oil, methanol and water in top stream from liquid-liquid extraction were all less than 6%. All of the glycerol remained in the bottom stream from liquid-liquid extraction.

### 4.3.4 Biodiesel purification

In order to obtain a final biodiesel product adhering to ASTM specifications (greater than 99.6% pure), biodiesel distillation with four theoretical stages and a reflux ratio of 2 were used. Stream from liquid-liquid column was forwarded to biodiesel column. Biodiesel column was operated under vacuum to keep temperatures low enough to prevent degradation of the biodiesel. A partial condenser was used to provide easy separation of the biodiesel from water and methanol in the column overhead. Water and methanol were removed as vent gases. Biodiesel



product (99.65% purity) was obtained in top stream as a liquid distillate (194 °C and 10 kPa). Unconverted oil remained at the bottom of biodiesel column. Since only a small amount of unconverted oil, it was treated as a waste. When oil conversion in transesterification reactor was low and oil recycling was necessary for waste reduction, a cooler and a pump were required to pump the unconverted oil back to the transesterification reactor. Superheated high pressure steam was the heating medium for the reboiler.

#### 4.3.5 Alkali removal (Neutralization reactor)

Bottom stream from liquid-liquid extraction column was fed to neutralization reactor to remove sodium hydroxide by adding phosphoric acid (100% purity). The resulting  $\text{Na}_3\text{PO}_4$  was removed in gravity separator, centrifuges. When potassium hydroxide is used as an alkali catalyst, the resulting potassium phosphate may be used as a valuable by-product. That is fertilizer.

#### 4.3.6 Glycerol purification

After removing the sodium hydroxide, stream 305 contained 85% glycerol. If a glycerine by-product with a higher grade was preferred, this stream would pass to glycerol column for further removal of water and methanol by distillation. Glycerine purification column was designed with four theoretical stages and a reflux ratio of 2. Water and methanol were removed in distillate top stream column. At the bottom, 92% glycerine was obtained as a high quality by-product to follow standard ASTM.

#### 4.3.7 Waste treatment

The compositions of streams from top of biodiesel column, unconverted palm oil from biodiesel column and top of glycerol column are listed in APPENDIX A. Because of their small flows, these streams were treated as hazardous gas or liquid wastes. However, reusing these streams may be advantageous in the

future, especially for larger scale processes. For example, top stream of glycerol column can be returned to liquid-liquid extraction column as a washing solvent instead of fresh water. Recovery of the solid waste from neutralization process as a possible fertilizer credit is also feasible. Overall, these changes offer potential approaches for reducing waste treatment loads.

#### 4.4 Condition of Biodiesel Production and Separater

The objective of separation and distillation units is to separate and purify the ester attained in the reaction of the other components in the mixture, methanol in excess, oil that did not react, glycerin produced, and water employed in the rinsing stages. Distillation columns are also targeted to recover methanol, which is further recirculated, and to purify glycerin, which is a sub-product of significant industrial importance. The distillation is favoured by the difference in the points of boil of the methanol ( $64.7^{\circ}\text{C}$ ), glycerine ( $287.85^{\circ}\text{C}$ ) and methyl esters ( $194^{\circ}\text{C}$ ). However, it should be noted that these operations use vacuum pressures, since both glycerol and the ester may decompose at high temperatures. Figure 4.3 shows a flowsheet for the process, as well as the simulation results. The simulated process proved to be suitable for the production of high quality, palm-oil methyl esters featuring all the characteristics included in APPENDIX A.

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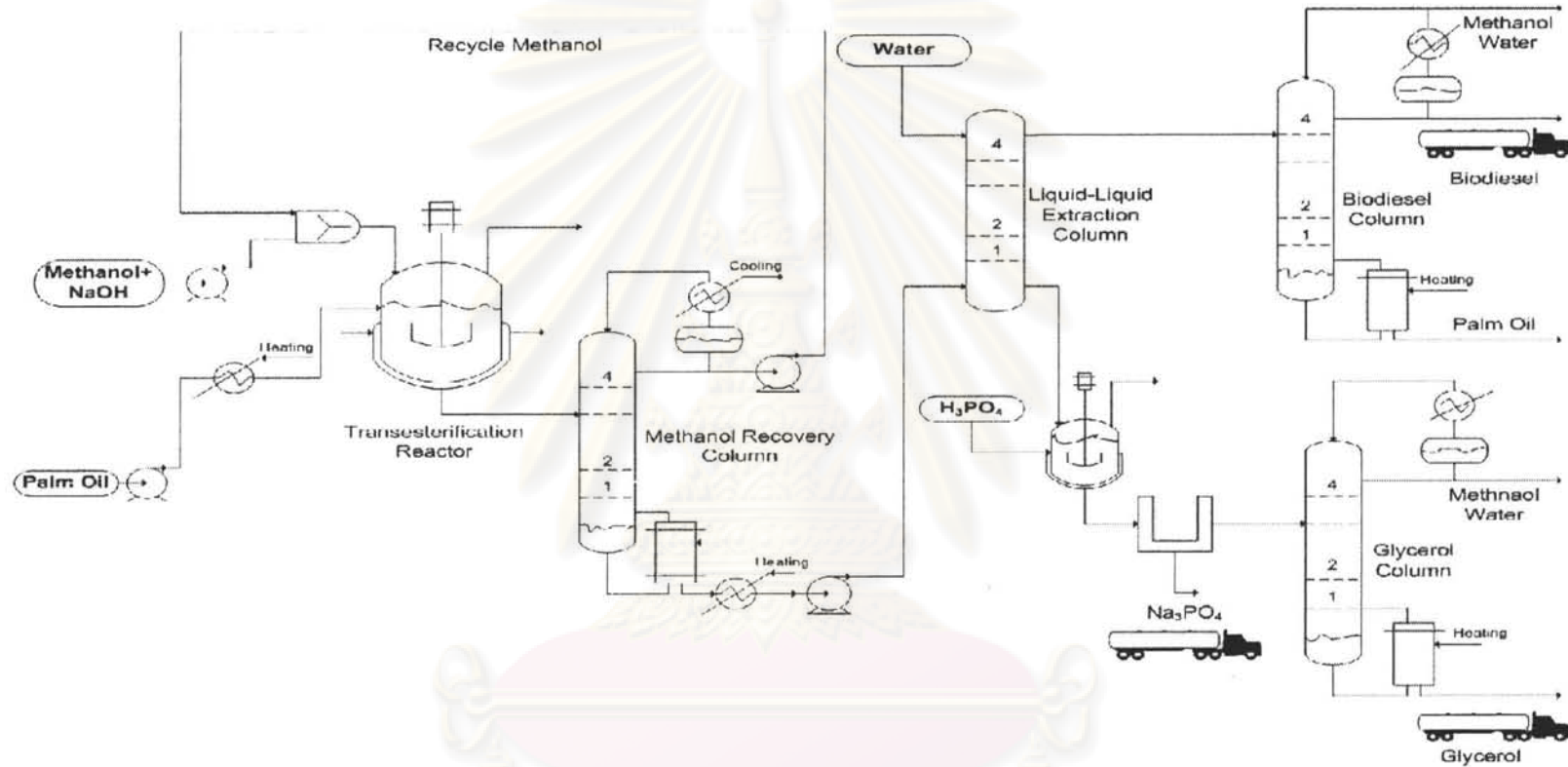


Figure 4.3 Biodiesel Production Process Plant

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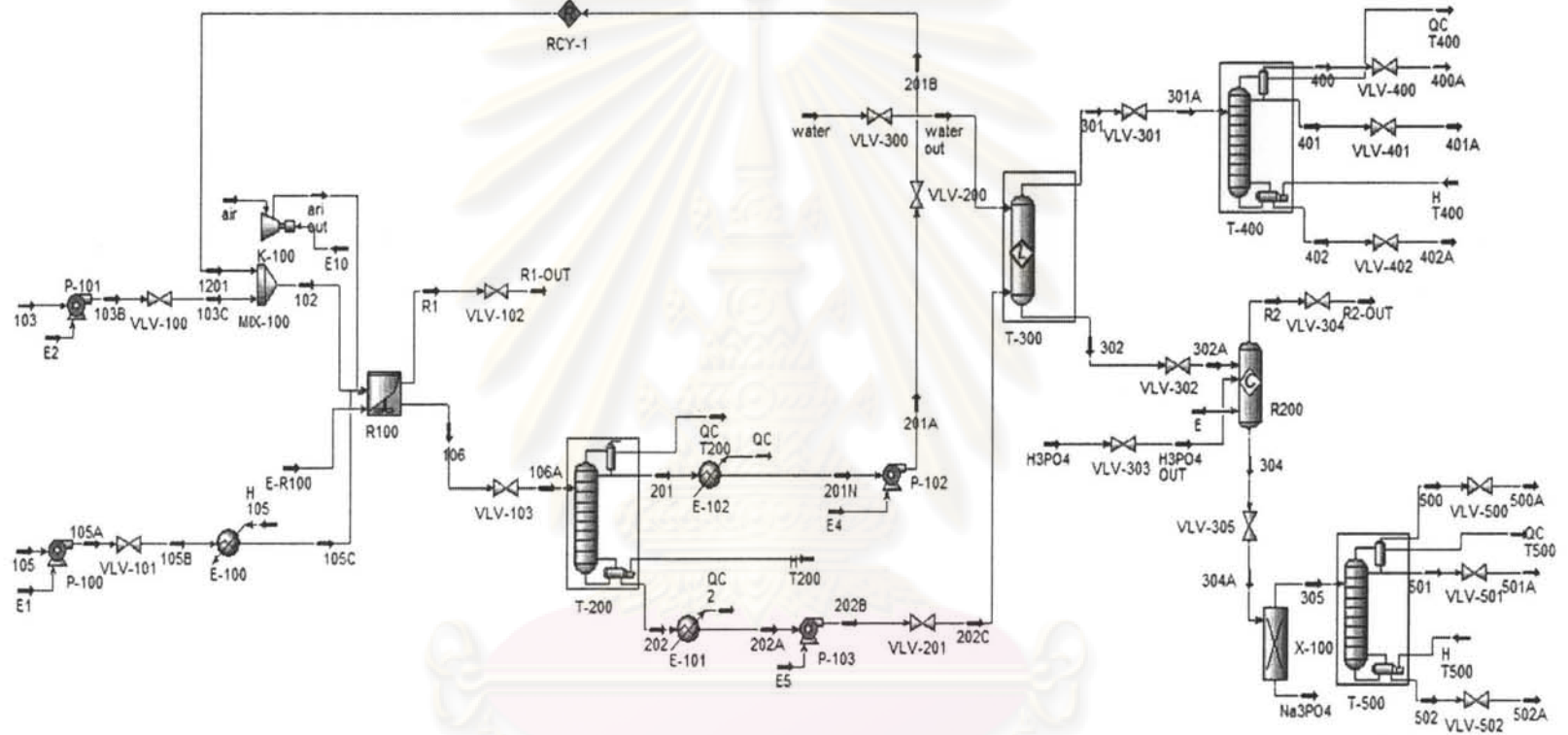


Figure 4.4 Biodiesel Production Process Plant flowsheet via HYSYS version 3.1

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# CHAPTER V

## CONTROL STRUCTURE DESIGN AND DYNAMIC SIMULATION

### 5.1 Introduction

Maintaining the plant energy and mass balances are the essential task of plantwide for a complex plant consists of recycle streams and energy integration when the disturbance load come through the process. The control system is needed to reject loads and regulate an entrie process into a design condition to achieve its objectives therefore our purpose of this chapter is to present the new control structures of energy integrated process. Moreover, the three designed control structures are also compared between three structures on rigorous dynamic simulation by using the commercial software HYSYS version 3.1.

### 5.2 Plantwide control design procedure

The plantwide control structures can be applied to the modules. Here, the nine-step approach of Luyben (1999) CS1 and Fixtrue point theorem CS2 and CS3 (Wongsri, 2008).

#### 5.2.1 Nine-step approach of Luyben

This approach is used to design the first control structure of the biosiesel production process and will use this control structure to design the continue control structure. The approach discussed below.

### Step 1. Establish Control Objectives

1. For this process, we want to achieve the biodiesel and glyceride production at 99.6%, 85% respectively.
2. Temperature of glyceride column and biodiesel column at reboiler is not more than boiling point, it should be noted that these operations use vacuum pressures, since both glyceride and biodiesel may be decompose at high temperatures

### Step 2. Determine Control Degree of Freedom

**Table 5.1** Degree of freedom of biodiesel production process.

Unit Operation	Position of Valves	Degree of Freedom
Transesterification Reactor	fresh feed valves for palm oil (VLV-101)and methanol (VLV-100),vent valve at reactor (VLV-102),heater at reactor (E-3 valve)	4
Methanol Recovery Column	fresh feed valve from CSTR (VLV-103), distillate valve (VLV-200),QC T-200 valve H T-200 valve	4
Water Washing	fresh feed valves for column T-200 (VLV-201), and water (VLV-300)	2
Neutralization reactor	stream from water washing T-300 (VLV-302), H <sub>3</sub> PO <sub>4</sub> fresh feed (VLV-303), vessel presure of reactor (VLV-304)	3
Glyceride Column	fresh feed valves for column T-300 (VLV-305), distillate valve (VLV-501), bottom production rate (VLV-502), QC T-500 and H T-300 valves	5
Biodiesel Column	fresh feed valves for column T-300 (VLV-301), vapor outlet (VLV-400), distillate valve VLV-401),bottom production rate (VLV-402), QC T-400 and H T-400 valves	6
Cooler and heater	H 1,QC 2	2
All	Degree of freedom = 26	

Degree of freedom = 26

### Step 3. Establish Energy Management system

Establish energy management able to divide two part, In this work, we no designed the new heat exchanger network, since this biodiesel process is low temperature operation.

Transesterification reactor energy management, since biodiesel reaction is endothermic heat reaction. The endothermic heat of reaction must be add in CSTR reactor, and the transesterification reactor must be heated to a high enough temperature to initiate the reaction at  $60^{\circ}\text{C}$ , since temperature  $60^{\circ}\text{C}$  is the best temperature for transesterification reaction of biodiesel. if temperature get below  $60^{\circ}\text{C}$ , it will be down conversion of biodiesel and if temperature upper  $60^{\circ}\text{C}$ , it will be increase conversion of biodiesel but it don't above boiling point of methanol at pressure condition 400 kPa.

### Step 4. Set Production Rate

There are not constrained to set production either by supply or demand, then the production rate can be set by palm oil and methanol that it's raito 1:6. Considering of the kinetics equation is found that the two variable, concentration of palm oil, methanol and temperature. The most important considerations within a reactor are the extent of reaction of the reactants, which is known as conversion, and the selectivity of the reaction to the desired products, which is known as the selectivity. Key reactor variables that dictate conversion and selectivity are temperature, pressure, reaction time (residence time), and degree of mixing. In general, increasing the reaction temperature increases the reaction rate and, hence, the conversion for a given reaction time. However, if more than one reaction is taking place, the selectivity to the desired products can be impacted by changing the reaction temperature. In the transesterification reaction, the selectivity of the reaction is not negatively impacted by increasing temperature. Increasing temperature in the transesterification reaction does impact the operating pressure.

The reaction is a liquid phase reaction, so the pressure in the reactor must be maintained at a level that keeps the methanol in the liquid phase. Therefore, as the reaction temperature is increased the pressure must also be increased. Another means to increase the conversion in a reactor is to increase the reaction time. For a given temperature, conversion will increase as the reaction time increases. Increasing the reaction time has the negative effect of decreasing the chemical throughput in a given plant or increasing the reactor size for a given chemical throughput. A final very important parameter in the reactor is the degree of mixing or mass transfer.

$$-r = k_1 C_{TG} C_{CH_3OH} - k_{-1} C_{DG} C_{R_1COOCH_3}$$

$$-r = k_2 C_{DG} C_{CH_3OH} - k_{-2} C_{MG} C_{R_2COOCH_3}$$

$$-r = k_3 C_{MG} C_{CH_3OH} - k_{-3} C_{GL} C_{R_3COOCH_3}$$

From upper equation show signs of something biodiesel rate that biodiesel rate is function with methanol and palm oil concentration. Then feed rate of palm oil and methanol increased in transesterification reaction, product as a result of productivity of biodiesel. On the other hand, then increasing temperature of transesterification reaction result in expedite reaction and rising product (conversion of biodiesel), since reaction rate constant of transesterification reaction rise to follow temperature and endothermic reaction.

$$k_i = k_i^\infty e^{-\frac{E}{RT}}$$

Reaction rate constant that follows the Arrhenius equation.

So rate production is able to control, methanol feed control valve (V2), palm oil feed control valve (V1) and temperature control valve (E-Reactor).



## Step 5. Control Production Quality and Address Safety, Operational, and Environmental

### Control production quality

- Biodiesel and glycerol purity must be maintained at 99.6% and 85.0% respectively for this research. We can control by manipulating biodiesel and glycerol column reboiler duty to maintain temperature in the column or use reflux to manipulate purity.
- In water washing unit, the purpose of this step was to separate the biodiesel from the glycerol, methanol and catalyst. Although separation using a gravity settler. So the separation of biodiesel from the glycerol, methanol and catalyst (NaOH) depends on the quantity of water fed into the washing unit. Control of separation is able to use control valve VLV-300.

### Safety

- Biodiesel production process in transesterification reactor unit operates at a pressure of 400 kPa and this pressure is high pressure. So control is able to control by vent gas valve VLV-102.

### Operational

- Temperature of glyceride column and biodiesel column at reboiler is not more than boiling point, it should be noted that these operations use vacuum pressures, since both glyceride and biodiesel may be decomposed at high temperatures forenamed in objective of the first step. So operational control is able to control by QC T-200, QC T-400 and QC T-500.

### Environmental constraint

- In this research, it has a neutralization reactor to clean up sodium hydroxide that is catalyst (NaOH).

## Step 6. Control Inventories and Fix a Flow in Every Recycle Loop

### Control pressure

- four pressures must be controlled: in the column of methanol recovery, biodiesel, glycerol distillation and transesterification reactor. In column pressure control able to controlled by manipulating the vent stream of condenser or manipulating the cooling stream in condenser. And pressure control in transesterification reactor able to controlled by the vent stream of reactor or able to controlled by the compressor.

### Control level

- eight liquid levels are in the process: transesterification reactor, neutralization reactor, methanol recovery column, biodiesel column and glycerol column in each column it compose condenser and reboiler that it must be holdup liquid in vessel. Level control design in biodiesel production process can be controlled by the down stream of each vessel. The most direct way to control the remaining levels would be with the exit valves.

### Recycle loop control

- All of the flows around the liquid recycle would be set on the basis of level, which would lead to undesirable propagation of disturbances. So a flow somewhere in this loop should be controlled that in this biodiesel production process plant will be controlled level of condenser of methanol recovery column.

## Step 7. Check Component Balance

- Palm oil is raw material of transesterification reaction and it will be converted biodiesel about 98%, 2% unconverted palm oil ,it can be removed from the process biodiesel column at down stream, so purge flow used to control at down stream.

- Methanol is raw material of transesterification reaction. The reaction was carried out with 6:1 molar ratio of methanol to palm oil, unconverted methanol it can be removed from the process by biodiesel column at vent stream of condenser and glycerol column at vent stream of condenser, so purge flow is used to control methanol composition.
- Biodiesel is product, it can be removed from catalyst (NaOH), glycerol, methanol by liquid-liquid extraction unit T-300 and biodiesel get into biodiesel column for biodiesel purity, so control purity of biodiesel is used to control temperature at reboiler H T-400.
- Glycerol is by-product, it can be removed from biodiesel by liquid-liquid extraction unit T-300 and glycerol get into glycerol column for purity of glycerol is used to control temperature at reboiler by H T-500
- Sodium hydroxide is catalyst, it not reaction in system, but it help to accelerate transesterification reaction of biodiesel, it can be removed from biodiesel with liquid-liquid extraction unit and remove glycerol with neutralization reactor to remove sodium hydroxide by adding phosphoric acid (100% purity) The resulting Sodium phosphate  $\text{Na}_3\text{PO}_4$  was removed in gravity separation X-100 or centrifuges, When potassium hydroxide is used as an alkali catalyst, the resulting potassium phosphate may be used as a valuable by-product (fertilizer).
- Water used to washing for separate biodiesel from glycerol and catalyst, so water will be mixed with biodiesel and glycerol, it can be control to removed in vent stream valves in column.

## Step 8. Control Individual Unit Operations

### Heat exchanger E-100

- In transesterification reactor unit, palm oil stream that it's raw material, and this stream will be have heat exchanger for pre-heat before get into reactor, because palm oil stream then happen energy disturbances, heat exchanger able to control temperature at palm oil stream for protect disturbances propagation into temperature in reactor that it have effect conversion of transesterification reaction.

### Heat exchanger E-101

- Heat exchanger E-101, it have to function control temperature at stream 202 (drown stream methanol recovery column), this temperature and pressure 122.34°C and 30 kPa respectively, so methanol will be vapourization, it's impact with pump because then happen gass and liquidmphase in stream 202, it happen cavitation in pump, pump maybe disservice.

## Step 9. Optimize Economics or Improve Dynamic Controlability

This step is not considered in this work.

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### 5.2.2 Fixture Point Theorem

For CS2 and CS3 control structure, we use the Fixture point theorem (Wongsri, 2008).

The fixture point theorem is provided by Wongsri, 2008 to define the control variable that the most sensitivity. Dedined control variable should consider to control and pairing with manipulate variable (MV) in the first.

#### Fixture point theorem analysis

1. Consideration in dynamic mode of simulation until process set up to steady state.
2. Control variable (CV) can be arranged to follow the most sensibility of the process variable by step change the MV (change only one MV, the other should be fixed then alternate to other until complete). Study the magnitude of integral absolute error (IAE) of all process variables that deviates from steady state.
3. Consider CV that give the most deviation from steady state (high IAE score) to match with MV. CV and MV should be directing interactive together, after that will consider the next CV to match with other MV.

### 5.3 Design of plant control structure

In this current work, we apply the first control structure of Luyben (1998) namely control structure 1 (CS1), the second control structure (CS2) and the third control structure (CS3), we apply control structure of Wongsri, 2008. In all of these control structures, the same loops are used as follows:

#### Trasesterification reactor unit

- The reactor pressure is controlled manipulating the vent stream from reactor.
- The palm oil stream (raw material) exit temperature is controlled manipulating by the heating utility vale.
- The palm oil and methanol mixed with sodium hydroxide is controlled via ratio control between palm oil stream and methanol stream.

#### Methanol recovery column unit

- The column pressure is controlled manipulating by the cooler utility vale at condenser.
- The column temperature is controlled manipulating by the heating utility vale at reboiler.

#### Liquid-Liquid extraction unit

- The water freash feed flow rate is controlled manipulating valve VLV-300.
- The top stream outlet from liquid-liquid extraction is controlled manipulating valve VLV-301.

#### Neutralization reactor unit

- Sodium hydroxide is controlled via ratio control between liquid exit from liquid-liquid extraction cloumn stream and phospheric acid stream.

#### Biodieel column unit

- The column pressure is controlled manipulating by the cooler utility vale at condenser.

- The column temperature is controlled manipulating by the heating utility vale at reboiler.

#### Glycerol column unit

- The column pressure is controlled manipulating by the cooler utility vale at condenser.
- The column temperature is controlled manipulating by the heating utility vale at reboiler.
- The biodiesel level in condenser can be controlled with the liquid exit valves from its units.

#### 5.3.1 Refereance control structure I (CS1)

The control structure of biodiesel production process is designed by Luyben method and showed in figure 5.1. this structure is liquid exit stream in transesterification reactor valve to control base level and freash feed of palm oil and methanol valve to control production rate of biodiesel, recycle flow rate is controlled by level of condenser of methanol recovery column. In neutralization reactor is not control temperature and not control presure because both temperature and pressure is operate at room temperature and atmosphas pressure.

#### 5.3.2 Refereance control structure II (CS2)

The control structure of biodiesel process is designed by Wongsri method and showed in fixgure 5.2. this structure is palm oil freash feed get into transesterification reactor valve to control level, control ratio of palm oil and methanol carried out with a 6:1 molar controlled by ratio control, palm stream is load stream and methanol stream is manipulated stream. Control flow rate of liquid exit from transesterification reactor for protect material disturbance into methanol recovery column. Control level of condenser and reboiler liquid exit of each vessel. Control of removing of catalyst by neutralization reactor with control ratio of drown

stream from liquid-liquid extraction unit T-300 and fresh feed phosphoric acid by down stream from T-300 is load stream and fresh feed phosphoric acid is manipulated stream. Control level of neutralization reactor by down stream from T-300. Control level of reboiler of glycerol column can be manipulated liquid exit from neutralization reactor.

### 5.3.3 Reference control structure III (CS3)

The control structure of biodiesel process is designed by Wongsri method and as showed in figure 5.3. this structure is palm oil fresh feed get into transesterification reactor valve to control level, the same control structure in transesterification reactor of CS2 , but except loop of liquid exit from reactor by control level of reboiler of methanol recovery column. At flow rate of recycle stream and liquid exit stream from reboiler of methanol recovery column manipulated constant by level condenser and level reboiler respectively. Control of removing of catalyst by neutralization reactor with control ratio of down stream from liquid-liquid extraction unit T-300 and fresh feed phosphoric acid by down stream from T-300 is load stream and fresh feed phosphoric acid is manipulated stream. Control level of neutralization reactor by liquid exit from reactor.

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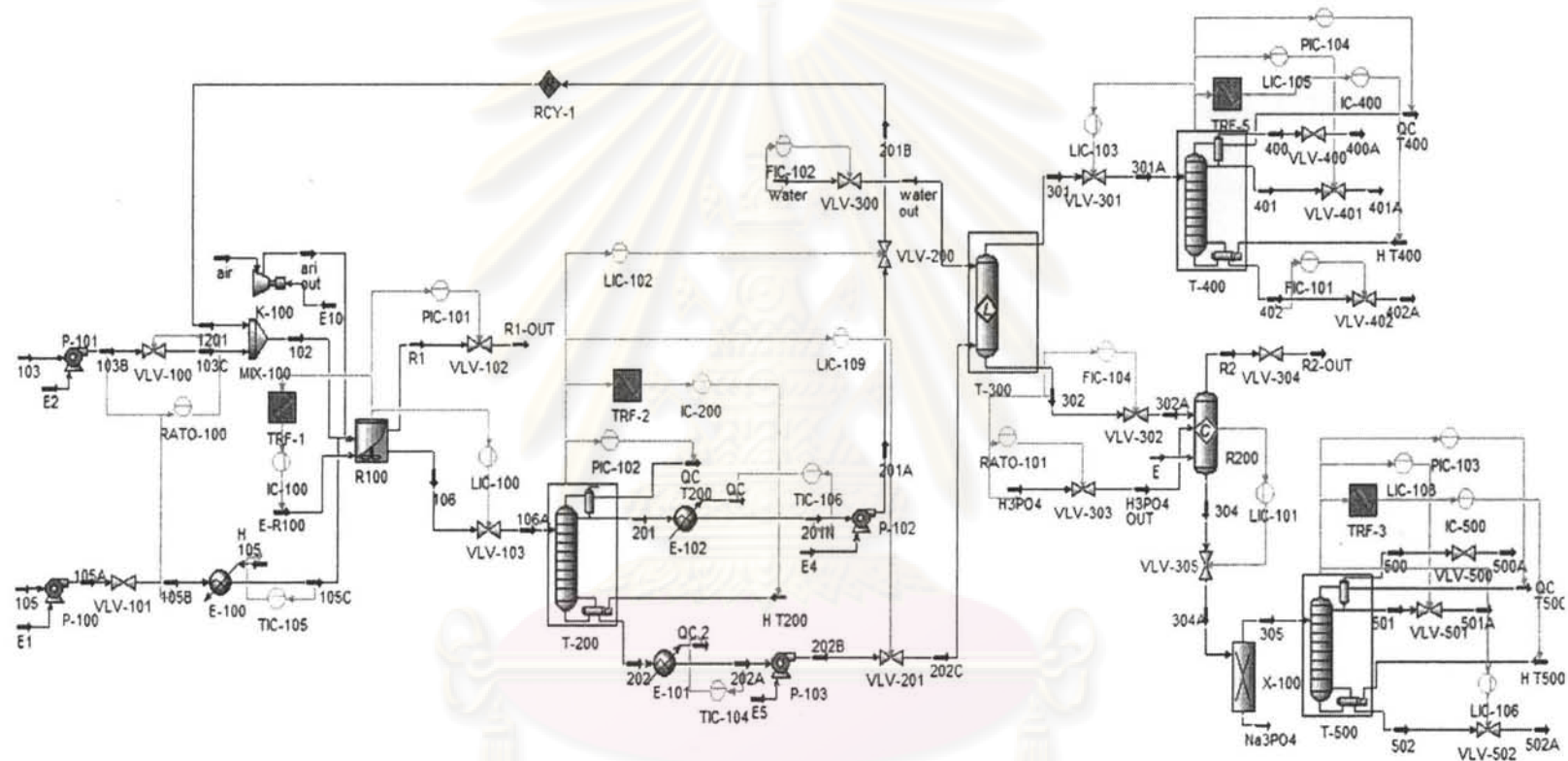


Figure 5.1 Designed control structure I (CS1) of biodiesel production process

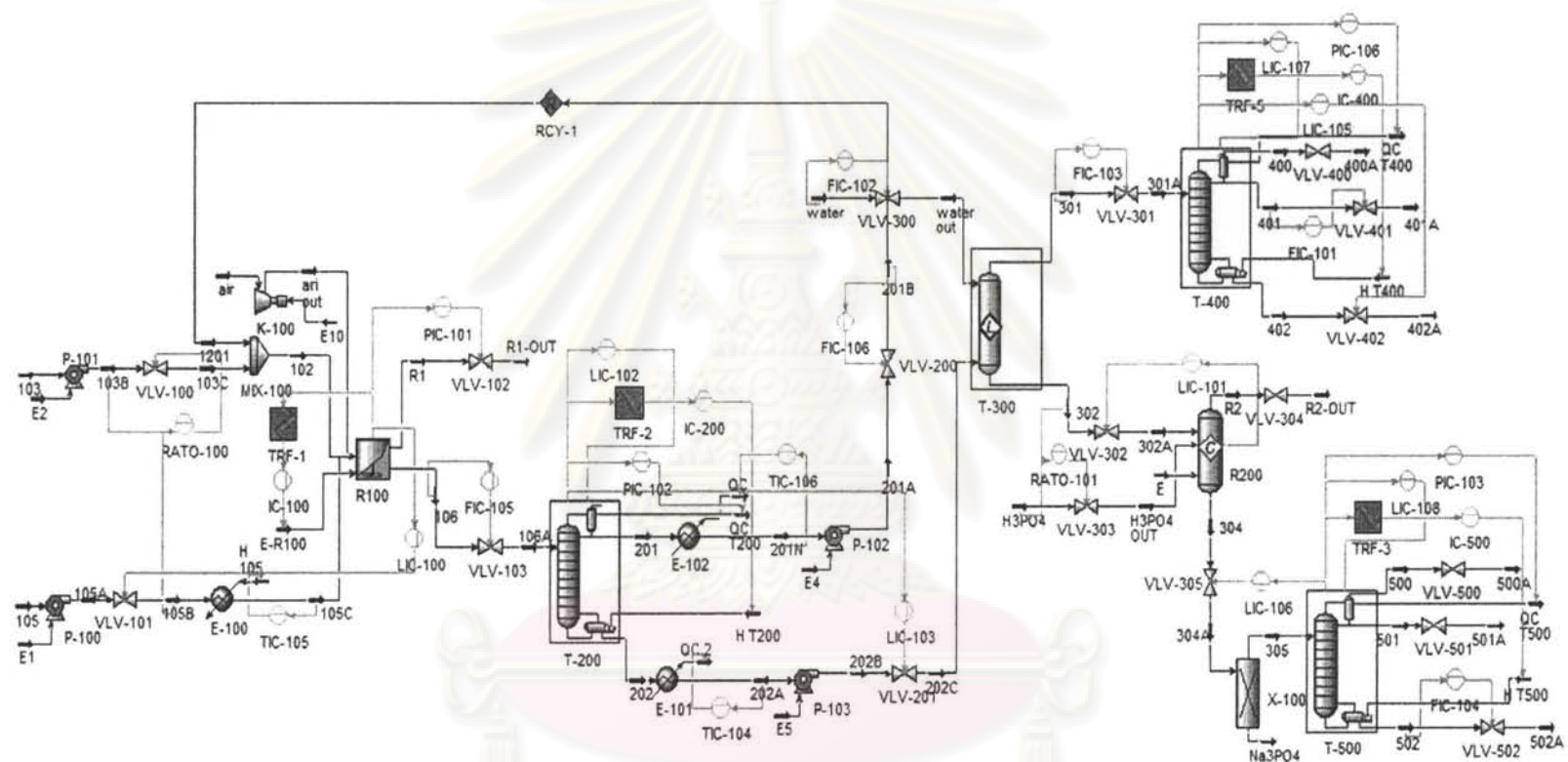


Figure 5.2 Designed control structure II (CS2) of biodiesel production process

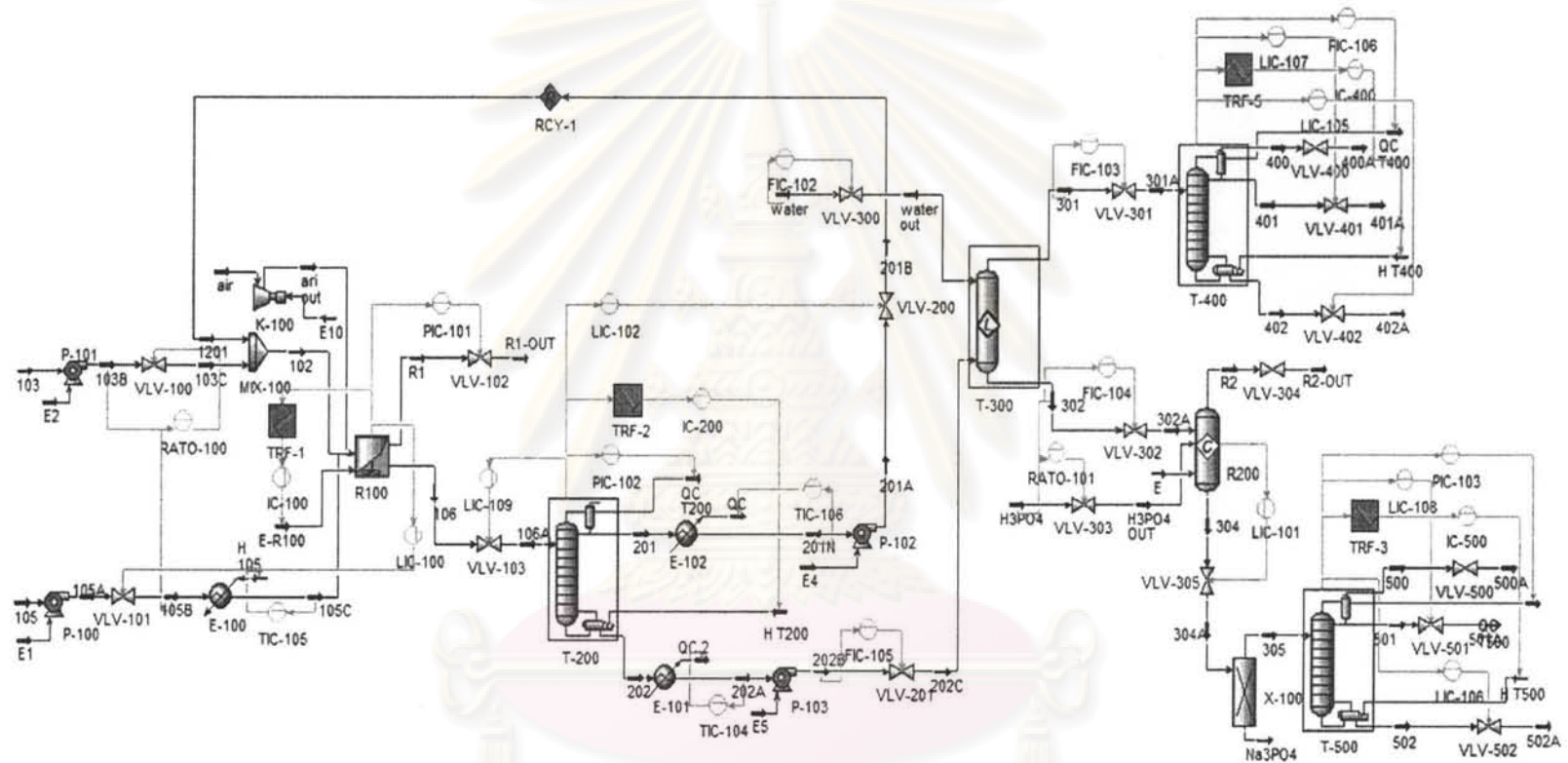


Figure 5.3 Designed control structure III (CS3) of biodiesel production process

## 5.4 Dynamic simulation results

In order to illustrate the dynamic behaviors of our control structures by control structure design Luyben method (1998) and Wongsri method (2008), two types of disturbance: thermal and material disturbances are used to test response of system. Temperature controllers are PIDs which are tuned using relay feedback. Temperature measurement lags of 0.5 minute are included in the temperature loops. Flow and pressure controller are PIs and their parameters are heuristics values. Proportional-only level controllers are used and their parameters are heuristics values. All control valves are half-open at nominal operating condition.

**Testing of disturbances, in two types disturbance:**

- Change in the mass flow rate CS1 to CS3 in biodiesel production process plant (CS1; Luyben et al.,1998, CS2 to CS3; Wongsri.,2008),the fresh feed inlet into transesterification reactor is increased 1050 kg/h to 1150 kg/h at time equals 50 to 200 minutes, decreased 1050 kg/h to 945 kg/h at time equals 200 to 350 minutes and increased 945 kg/h to 1050 kg/h at time equals 350 to 500 minutes.
- Change in the thermal disturbance of hot stream for CS1 to CS3 in biodiesel production process plant (CS1; Luyben et al.,1998, CS2 to CS3; Wongsri.,2008). The transesterification reactor outlet temperature is increased from  $60^{\circ}C$  to  $70^{\circ}C$  at time equals 50 to 200 minutes, decreased from  $70^{\circ}C$  to  $50^{\circ}C$  at time equals 200 to 350 minutes and  $50^{\circ}C$  to  $60^{\circ}C$  at time equals 350 to 500 minutes

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#### 5.4.1 Change in the mass flow rate CS1 to CS3 in biodiesel production process plant (CS1; Luyben et al.,1998, CS2 to CS3; Wongsri.,2008)

Figure 5.4 to 5.17 show dynamic responses of biodiesel production plant in the material balance. This disturbance made as follows: The palm oil and methanol fresh feed is increased from 1050 kg/h to 1150 kg/h at equals 50 to 200 minutes, decreased from 1150 kg/h to 945 kg/h at equals 200 to 350 minutes and increased from 945 kg/h to 1050 kg/h at equals 350 to 500 minutes.

From change in the mass flow rate in transesterification reactor, production rate of biodiesel direct variation concentration of palm oil and methanol when increased palm oil in stream 105, ratio control that it used to control ratio between palm oil and methanol, it is going to affect with methanol stream adjustment to follow ratio palm oil:methanol (1:6), by palm oil stream is disturbance load and methanol stream is manipulated. Control structure design CS1 to CS3 in the transesterification reactor part, it same control structure in ratio control part, but it contrast in liquid exit from transesterification reactor. That is CS1 liquid exit stream used to level control of transesterification reactor, CS2 liquid exit stream used to fresh feed control before into methanol recovery column and CS3 liquid exit stream used to level of reboiler of methanol recovery column when biodiesel into column, methanol recovery column change of temperature, since biodiesel feed from reactor change of material, CS1 will be change of condition, but CS2 to CS3 not change because it's controlled via feed flow and level reboiler of methanol recovery column respectively. In neutralization reactor is clean up sodium hydroxide catalyst to controlled via ratio control between liquid exit from liquid-liquid extraction and phosphoric acid stream. Biodiesel will be change of biodiesel flow rate for CS1, but CS2 to CS3 not change. And glycerol will be change flow rate for CS1, but CS2 to CS3 not change.

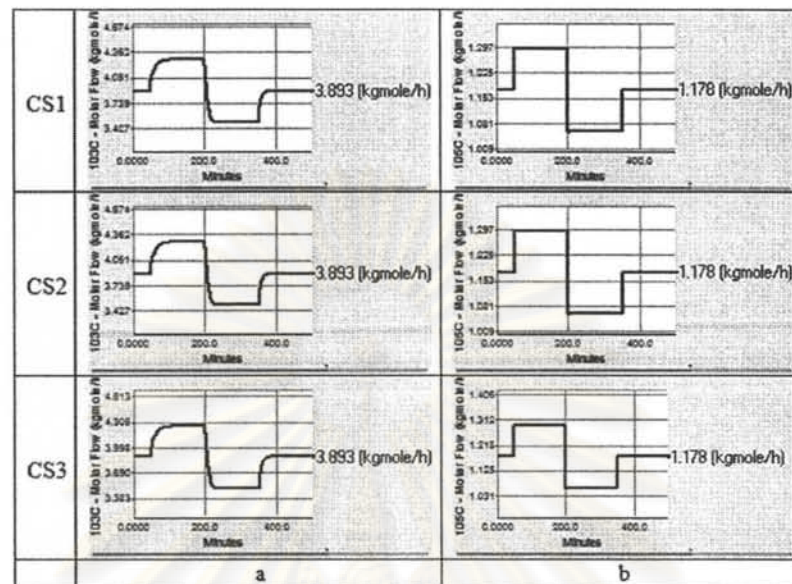


Figure 5.4 Dynamic responses of fish feed when change  $\pm 10\%$  kmole/h fresh feed in transesterification (a) fresh feed methanol (b) fresh feed palm oil

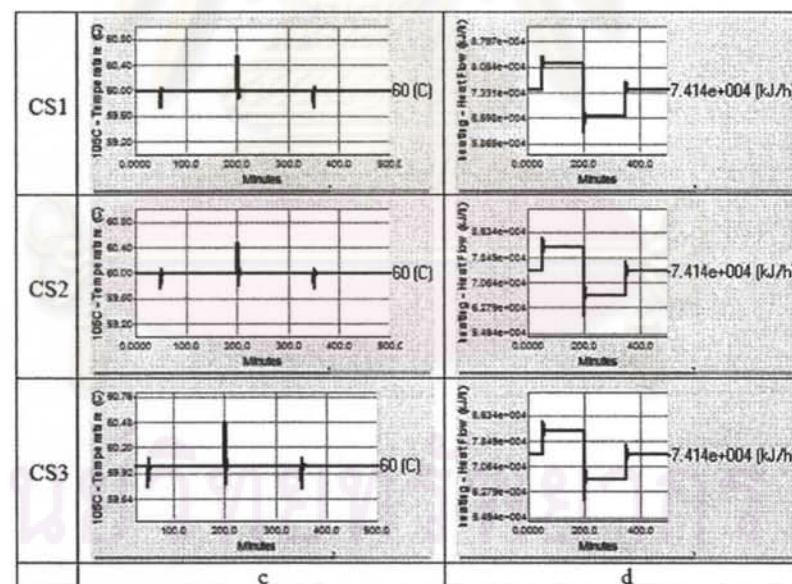


Figure 5.5 Dynamic responses of pre-heat palm oil stream when change  $\pm 10\%$  kmole/h fresh feed in reactor (c) temperature of pre-heat palm oil stream (d) heater duty of E-100

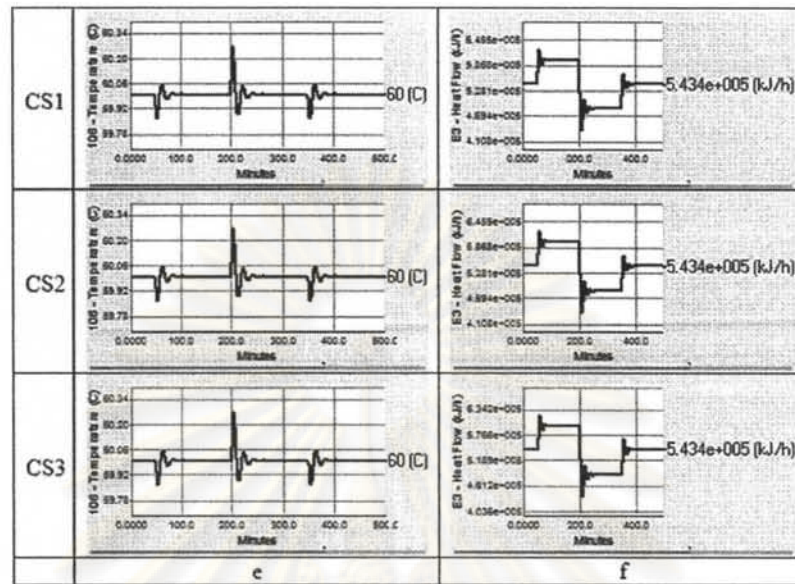


Figure 5.6 Dynamic responses of temperature outlet when change  $\pm 10\%$  kmole/h fresh feed in reactor(e) temperature outlet of transesterification reactor (f) heater duty of E-R100

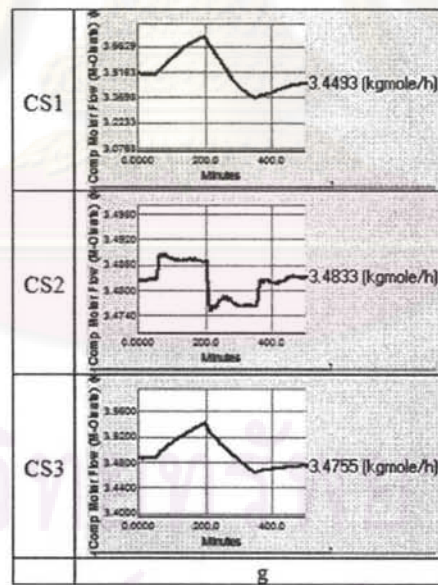


Figure 5.7 Dynamic responses of production flowrate after outlet reactor when change  $\pm 10\%$  kmole/h fresh feed in reactor (g) biodiesel production flowrate

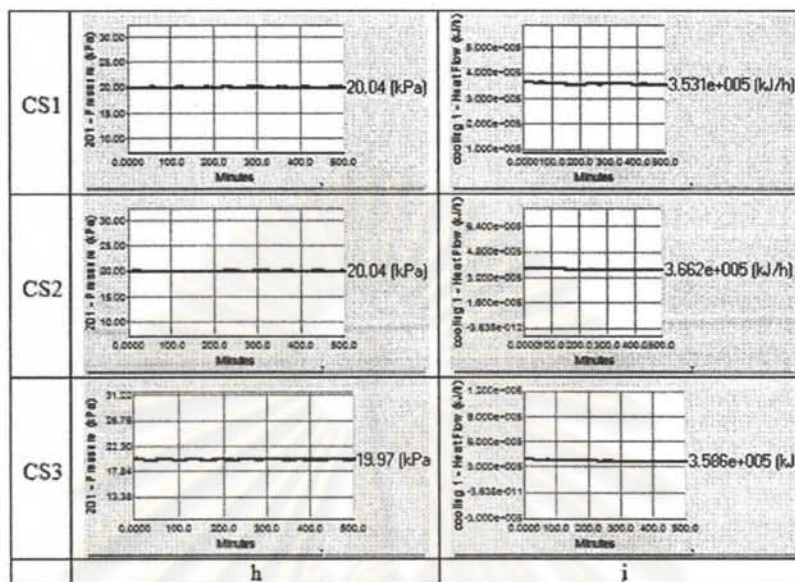


Figure 5.8 Dynamic responses of pressure of methanol recovery column when change  $\pm 10\%$  kmole/h fresh feed in reactor (h) pressure of methanol recovery column (i) duty of condenser of methanol recovery column

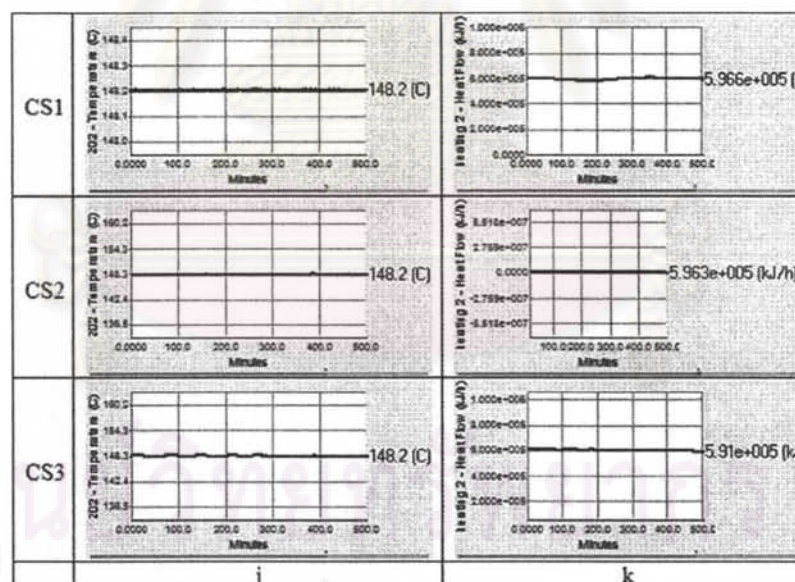


Figure 5.9 Dynamic responses of temperature in methanol column when change  $\pm 10\%$  kmole/h fresh feed in reactor (j) temperature of reboiler (k) duty of reboiler of methanol recovery column



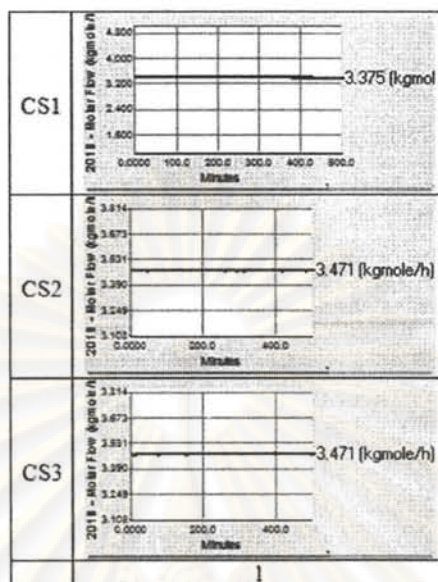


Figure 5.10 Dynamic responses of methanol recycle flowrate when change  $\pm 10\%$  kmole/h fresh feed in reactor (l) recycle flowrate of methanol

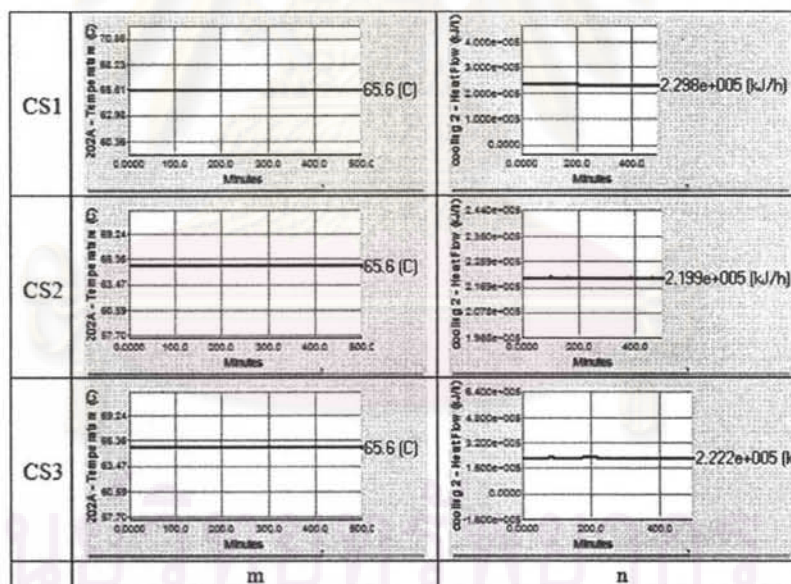


Figure 5.11 Dynamic responses of temperature cooler 202 stream when change  $\pm 10\%$  kmole/h fresh feed in reactor (m) temperature of stream 202 (n) Cooler duty of E-101

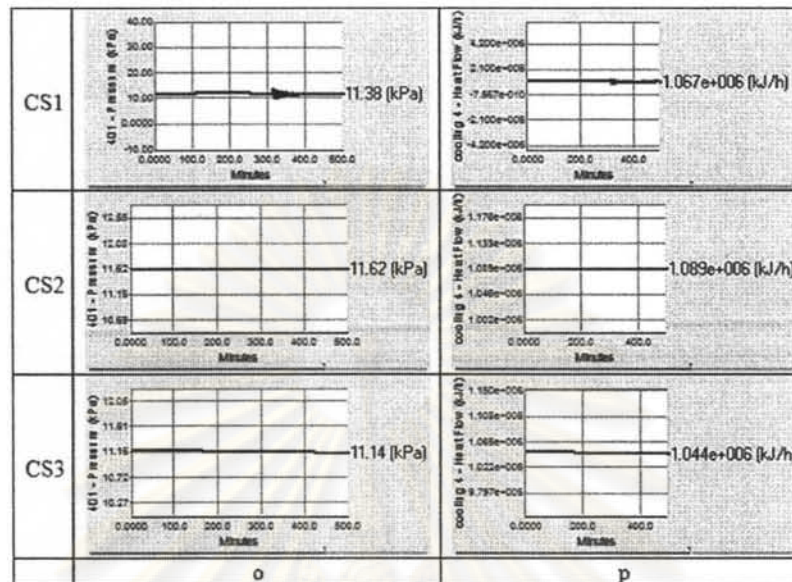


Figure 5.12 Dynamic responses of pressure of biodiesel column change  $\pm 10\%$  kmole/h fresh feed in reactor (o) pressure of biodiesel column (p) duty of condenser of biodiesel column

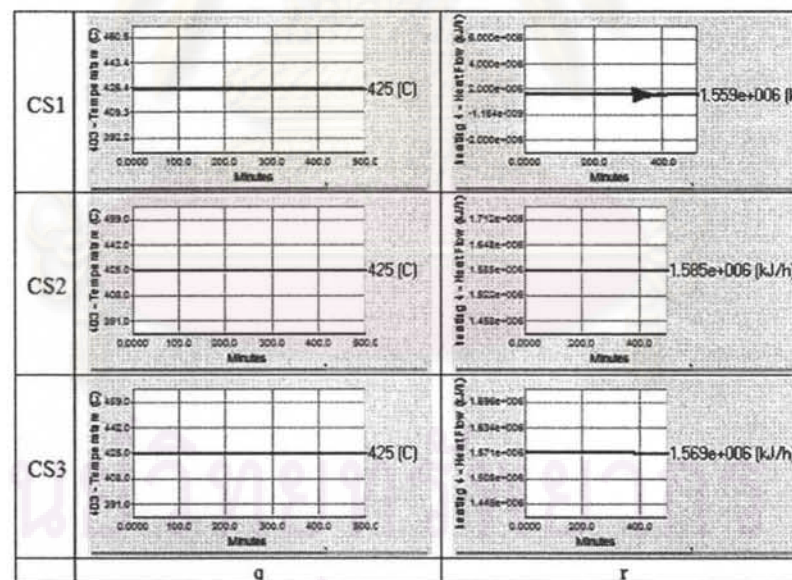


Figure 5.13 Dynamic responses of temperature in biodiesel column when change  $\pm 10\%$  kmole/h fresh feed in reactor (q) temperature of reboiler (r) duty of reboiler of biodiesel column

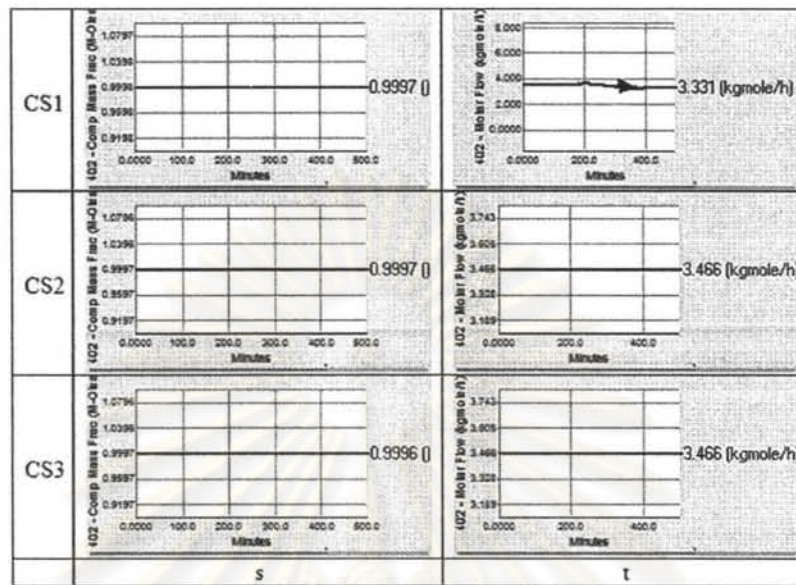


Figure 5.14 Dynamic responses of product purity and product flowrate when change  $\pm 10\%$  kmole/h fresh feed in reactor (s) purity of biodiesel (t) biodiesel product flowrate

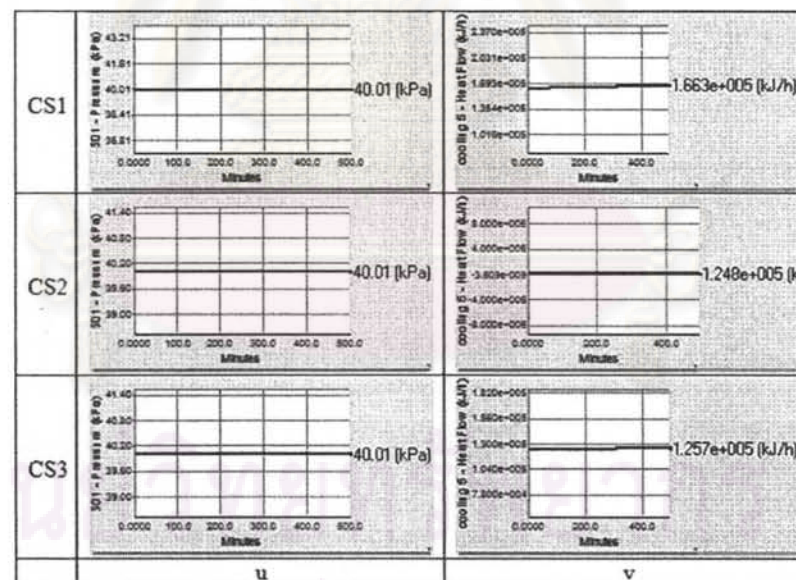


Figure 5.15 Dynamic responses of pressure of glycerol column change  $\pm 10\%$  kmole/h fresh feed in reactor (u) pressure of glycerol column (v) duty of condenser of glycerol column

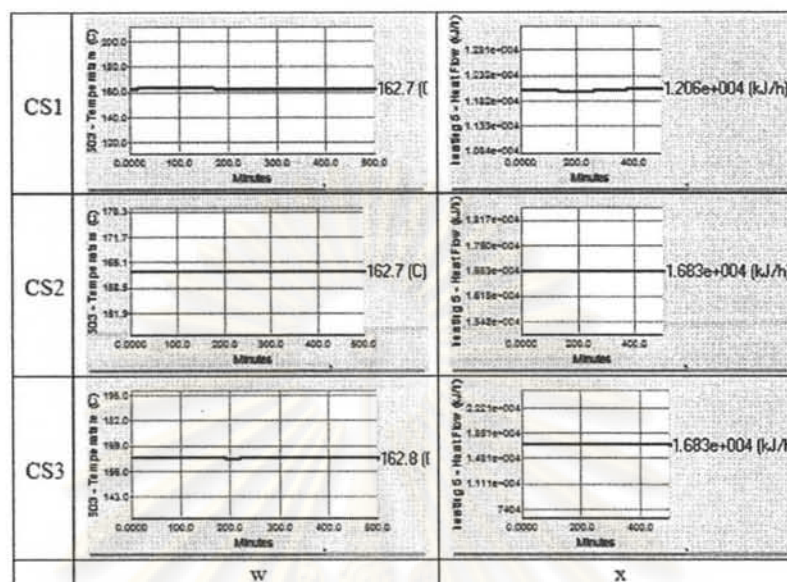


Figure 5.16 Dynamic responses of temperature in glycerol column when change  $\pm 10\%$  kmole/h fresh feed in reactor (w) temperature of reboiler (x) duty of reboiler of glycerol column

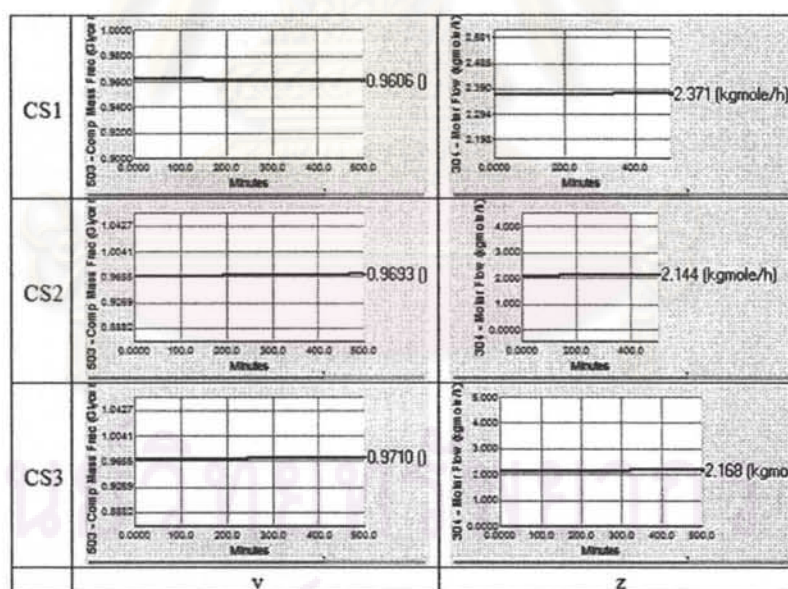


Figure 5.17 Dynamic responses of product purity and product flowrate when change  $\pm 10\%$  kmole/h fresh feed in reactor (y) purity of glycerol (z) glycerol product flowrate

#### 5.4.2 Change in the thermal disturbance of hot stream for CS1 to CS3 in biodiesel production process plant (CS1; Luyben et al.,1998, CS2 to CS3; Wongsri.,2008)

Figure 5.18 to 5.31 show dynamic responses of biodiesel production plant in the energy balance. The transesterification reactor outlet temperature is increased from  $60^{\circ}\text{C}$  to  $70^{\circ}\text{C}$  at time equals 50 to 200 minutes, decreased from  $70^{\circ}\text{C}$  to  $50^{\circ}\text{C}$  at time equals 200 to 350 minutes and  $50^{\circ}\text{C}$  to  $60^{\circ}\text{C}$  at time equals 350 to 500 minutes

From change in the temperature outlet of transesterification reactor when increased temperature  $70^{\circ}\text{C}$  at reactor, it will be increase conversion of biodiesel more than 98%, usually at temperature  $60^{\circ}\text{C}$  conversion of biodiesel equal 98%, so increasing temperature more than  $60^{\circ}\text{C}$ , it will be less increase conversion of biodiesel, on the other hand outlet temperature of reactor decreased temperature at  $50^{\circ}\text{C}$ , conversion of biodiesel is decrease in reaction, from Figure 5.9 (e), biodiesel flow rate is decrease at equal time 200 to 250 minute and increase at 230 to 350 minute at temperature  $50^{\circ}\text{C}$  when change of temperature  $60^{\circ}\text{C}$  conversion of biodiesel remain as ever, when temperature alternate at reactor, it's effect in methanol recovery column, but not effect in biodiesel column and glycerol column, since temperature is controlled via temperature control at reactor, temperature control at reboiler and temperature control at stream 202C.

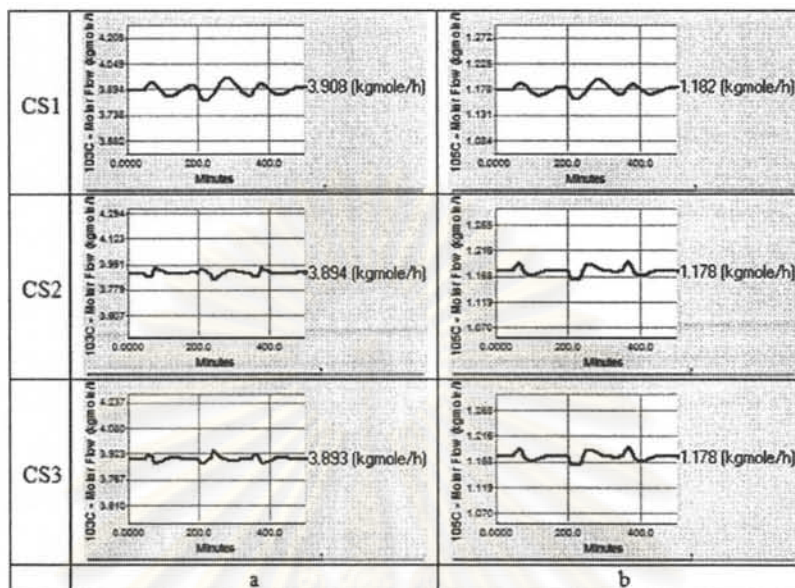


Figure 5.18 Dynamic responses of fresh feed when change temperature  $\pm 10^{\circ}\text{C}$  in transesterification (a) fresh feed methanol (b) fresh feed palm oil

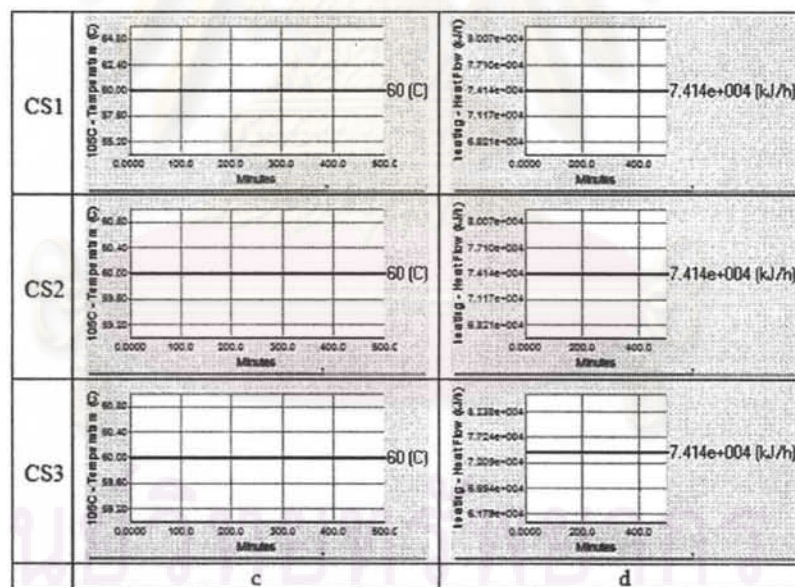


Figure 5.19 Dynamic responses of pre-heat palm oil stream when change temperature  $\pm 10^{\circ}\text{C}$  in transesterification (c) temperature of pre-heat palm oil stream (d) heater duty of E-100

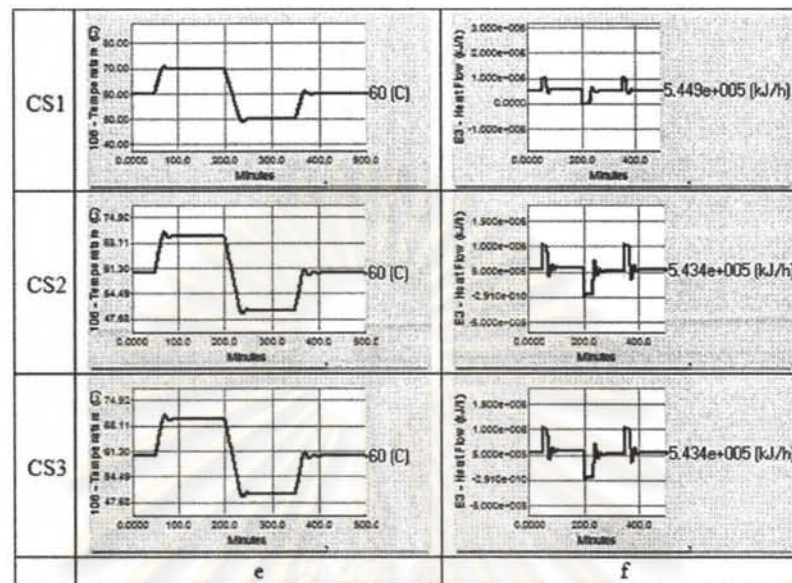


Figure 5.20 Dynamic responses of temperature outlet when change temperature  $\pm 10^{\circ}\text{C}$  in transesterification (e) temperature outlet of transesterification reactor (f) heater duty of E-R100

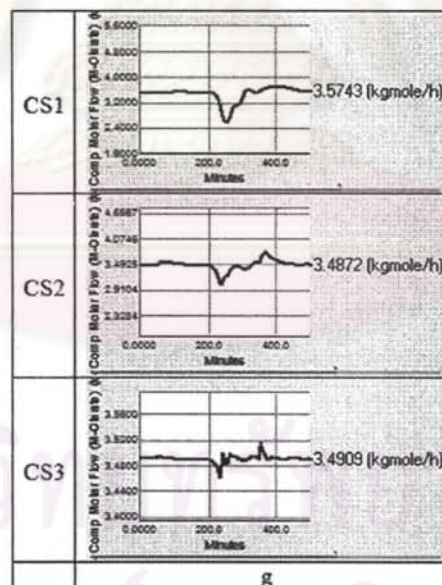


Figure 5.21 Dynamic responses of production flowrate after outlet reactor when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (g) biodiesel production flowrate

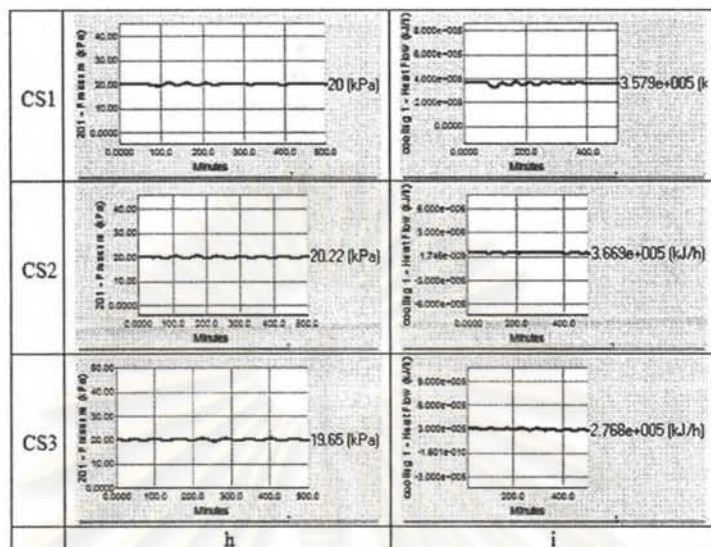


Figure 5.22 Dynamic responses of pressure of methanol recovery column when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (h) pressure of methanol recovery column (i) duty of condenser of methanol recovery column

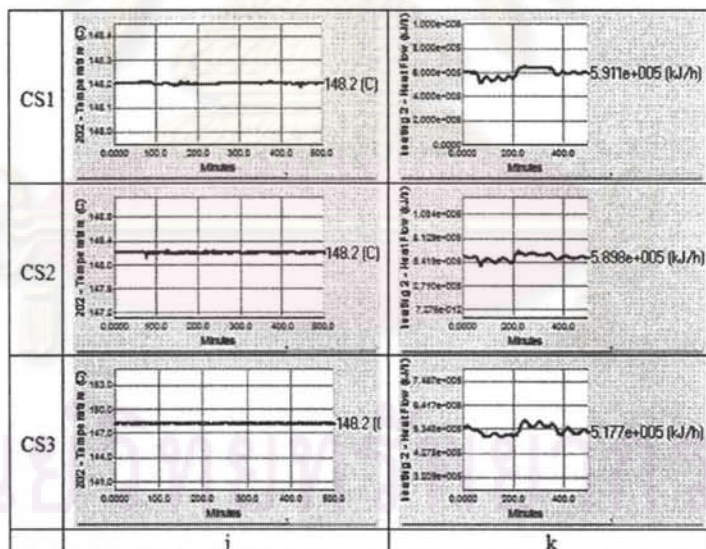


Figure 5.23 Dynamic responses of temperature in methanol column when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (j) temperature of reboiler (k) duty of reboiler of methanol recovery column



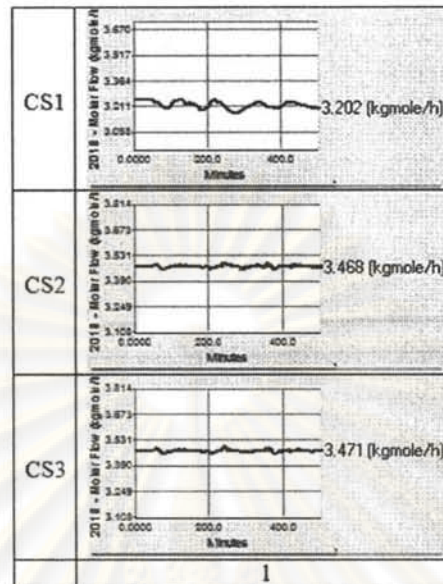


Figure 5.24 Dynamic responses of methanol recycle flowrate when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (l) recycle flowrate of methanol

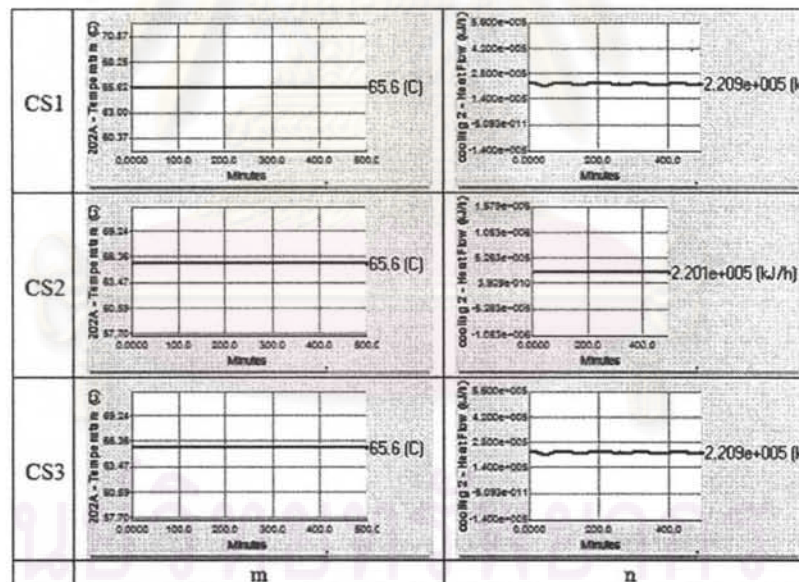


Figure 5.25 Dynamic responses of temperature cooler 202 stream when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (m) temperature of stream 202 (n) Cooler duty of E-101

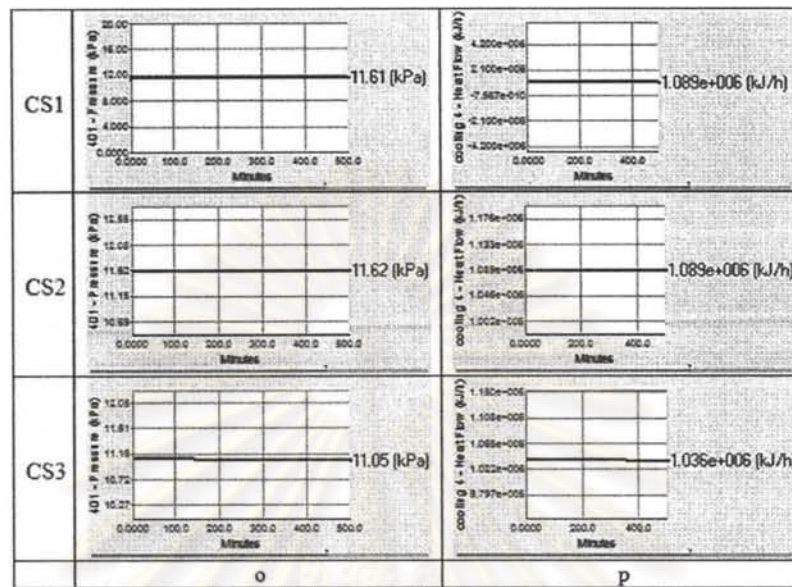


Figure 5.26 Dynamic responses of pressure of biodiesel column when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (o) pressure of biodiesel column (p) duty of condenser of biodiesel column

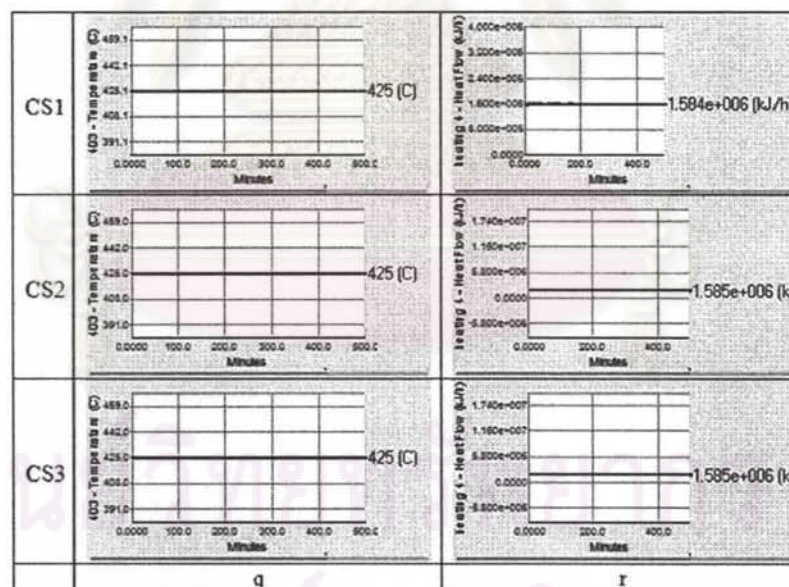


Figure 5.27 Dynamic responses of temperature in biodiesel column when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (q) temperature of reboiler (r) duty of reboiler of biodiesel column

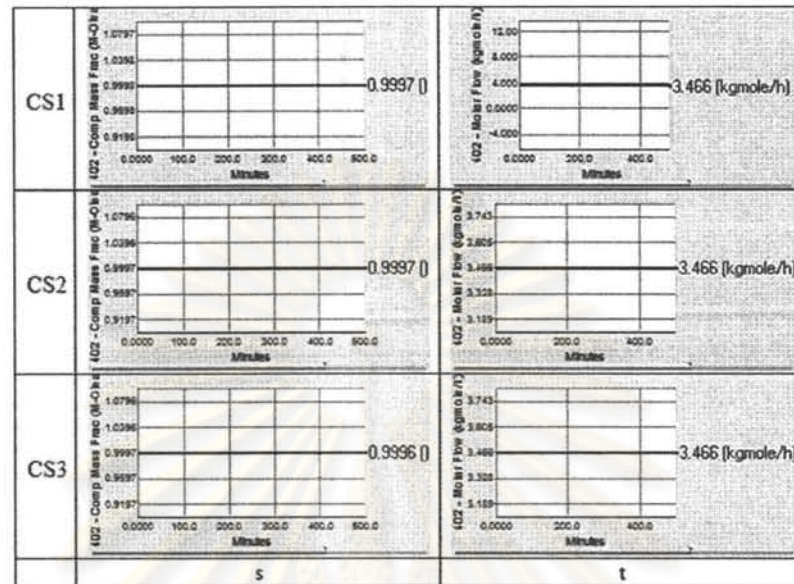


Figure 5.28 Dynamic responses of product purity and product flowrate when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (s) purity of biodiesel (t) biodiesel product flowrate

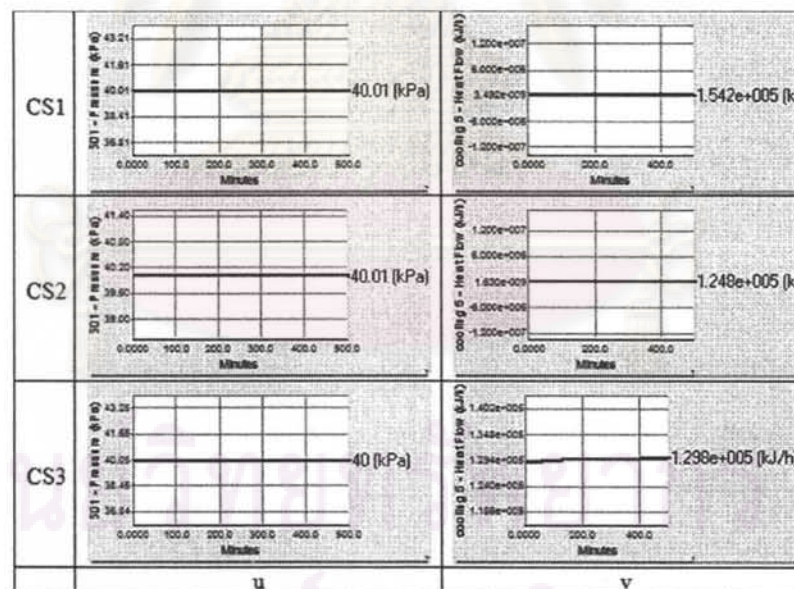


Figure 5.29 Dynamic responses of pressure of glycerol column when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (u) pressure of glycerol column (v) duty of condenser of glycerol column

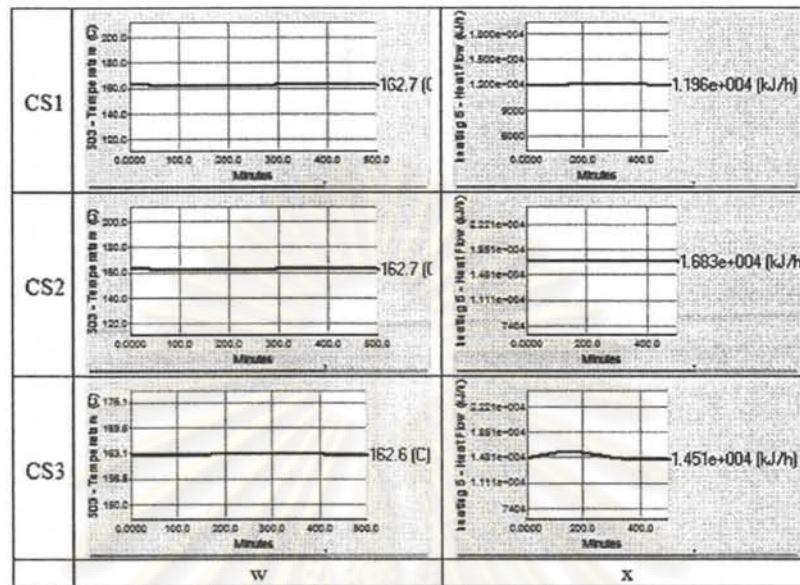


Figure 5.30 Dynamic responses of temperature in glycerol column when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (w) temperature of reboiler (x) duty of reboiler of glycerol column

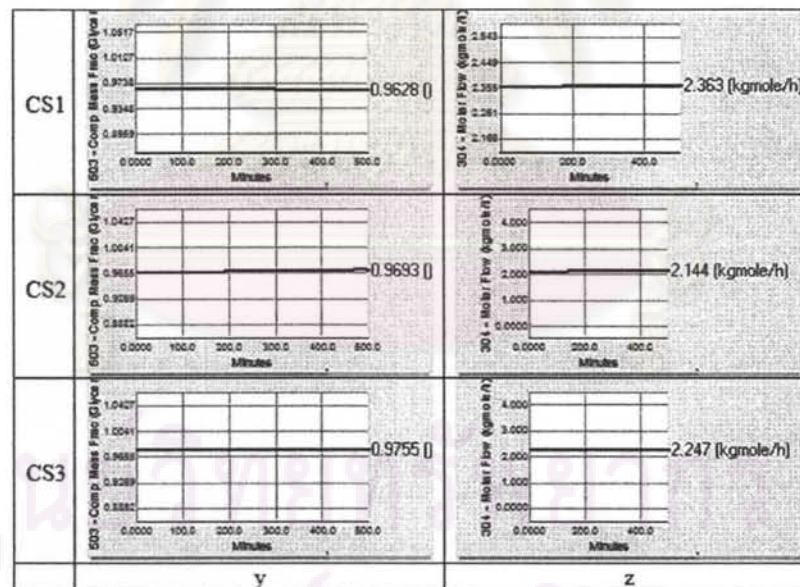


Figure 5.31 Dynamic responses of product purity and product flowrate when temperature change  $\pm 10^{\circ}\text{C}$  in transesterification (y) purity of glycerol (z) glycerol product flowrate

## 5.5 Evaluation of the Dynamic Performance

The dynamic performance index is focused on time relate characteristics of the controller's response to setpoint changes or deterministic disturbances. There exit several candidate performance measures such as setting time and integral absolute error (IAE). Integral absolute error is well known and widely used. For the formulation of a dynamic performance as written below:

$$IAE = \int |e(t)|dt$$

Note that  $e(t)=y_{sp}(t)-y(t)$  is the deviation (error) of the response from the desired setpoint.

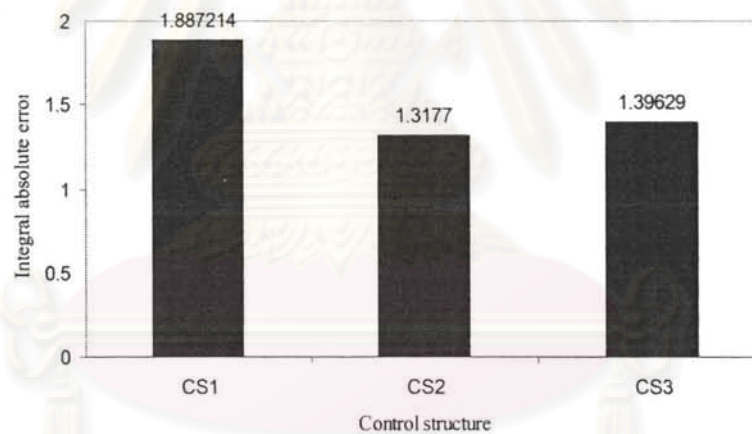
In this work, IAE method is used to evaluate the dynamic performance of the designed control systems. Table 5.2 show the IAE value result for the change in material disturbance of palm oil fresh feed stream in transesterification reactor and Table 5.3 show the IAE value result for the change in thermal disturbance of transesterification reactor outlet temperature.

In material disturbance, CS2 can manage flow disturbance better than other control structure, because flow rate of stream 106 is controlled remain as over before into methanol recovery coulmn, then not effect with liquid-liquid extraction, biodiesel column, neutralization reactor and glycerol column. In this case, we can find IAE value of CS1 close to CS3. Thus, CS2 is the best control structure for handle disturbances due to it gives better control performances.

In thermal disturbance, CS2 is the best controller. When we compare CS1 to CS3, CS2 gives smaller IAE value than CS1 and CS3.

**Table 5.2** The IAE Result of the biodiesel production process, control structure CS1 to CS3 in the mass disturbance of raw material before entering transesterification reactor.

Controller	CS1	CS2	CS3
Temperature of 105C stream	0.25198	0.25825	0.26813
Temperature of reactor (R100)	0.14753	0.15131	0.14808
Temperature of 202 stream	0.50825	0.50192	0.56092
Temperature of 202C stream	0.02936	0.02690	0.02998
Temperature of 402 stream	0.51317	0.00000	0.00075
Temperature of 502 stream	0.43679	0.37875	0.38842
SUM	1.88721	1.31770	1.39629

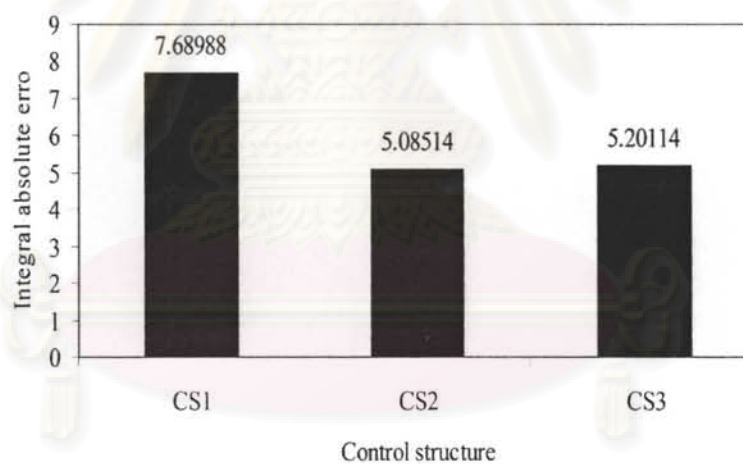


**Figure 5.32** The IAE results of the control systems to  $\pm 10$  of fresh feed flow for CS1 to CS3 in biodiesel production process

From Table 5.2 show IAE value is used to evaluate the dynamic performance of the designed control system, CS2 least value then compare control structure CS1 and CS3. So control structure CS2 is the best control structure. and the dynamic performance of the designed control system.

**Table 5.3** The IAE Result of the biodiesel production process, control structure CS1 to CS2 in the thermal disturbance of temperature in transesterification reactor.

Controller	CS1	CS2	CS3
Temperature of 105C stream	0.00349	0.00000	0.00000
Temperature of reactor (R100)	6.47447	4.23636	4.22348
Temperature of 202 stream	1.10342	0.80123	0.86308
Temperature of 202C stream	0.00453	0.04755	0.04650
Temperature of 402 stream	0.08375	0.00000	0.01458
Temperature of 502 stream	0.02475	0.38858	0.05350
SUM	7.68988	5.08514	5.20114



**Figure 5.33** The IAE results of the control systems to  $\pm 10$  of temperature for CS1 to CS3 in biodiesel production process

# CHAPTER VI

## CONCLUSION AND RECOMMENDATIONS

### 6.1 Conclusion

In this research has discussed control structure design for biodiesel production process, using the design procedure of Luyben (1998) and Wongsri (2008), Luyben's method has nine step for control structure design. Wongsri's method is the process variable should be control are selected by the "Fixture point theorem". The procedure is base on the selection of controlled variable that the most disturbances propagate to plantwide, previously using heuristic approach. The maximum (scale) gain is used to pairing controlled variables with manipulated variable,

In material disturbance, CS2 can manage flow disturbance better than other control structure, because flow rate of stream 106 is controlled remain as over before into methanol recovery column, then not effect with liquid-liquid extraction, biodiesel column, neutralization reactor and glycerol column. In this case, we can find IAE value of CS1 close to CS3. Thus, CS2 is the best control structure for handle disturbances due to it gives better control performances.

In thermal disturbance, CS2 is the best controller. When we compare CS1 to CS3, CS2 gives smaller IAE value than CS1 and CS3.

when against Luyben's method ,control structure CS2 to CS3 designed via Wnogsri performance more than control structure CS1 designed via Luyben



## 6.2 Recommendations

1. Study and design of control structure via Luyben's method and Wongsri's method
2. Performance evaluation of biodiesel production process via integral absolute error (IAE)



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**APPENDICES**

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## APPENDIX A

### PROCESS STREAM DATA FOR SIMULATION AND EQUIPMENT DATA SPECIFICATION

Table A.1 Data of Biodiesel production process

Stream name	101	102	105B	106	201	202	301A
Temperature (°C)	25.0	26.7	60.0	60.0	28.2	122.34	60.0
Pressure(kPa)	100	400	400	400	20	30	110
Molar flow(kg-mol/h)	3.85	7.13	1.19	8.51	3.47	5.04	3.60
Mass flow (kg/h)	118.30	238.39	1050.00	1288.40	111.19	1177.20	1060.21
Liquid volume flow (m <sup>3</sup> /h)	0.153	0.287	1.167	1.440	0.140	1.300	1.108
Component mass fraction							
Methanol	0.921	0.956	0.000	0.092	1.000	0.006	0.003
Palm oil	0.000	0.000	1.000	0.041	0.000	0.045	0.050
FAME (biodiesel)	0.000	0.000	0.000	0.779	0.000	0.853	0.946
Glycerol	0.000	0.044	0.000	0.081	0.000	0.088	0.000
NaOH	0.079	0.000	0.000	0.008	0.000	0.008	0.000
H <sub>2</sub> O	0.000	0.000	0.000	0.000	0.000	0.000	0.001
H <sub>3</sub> PO <sub>4</sub>	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Na <sub>3</sub> PO <sub>4</sub>	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Stream name	305	306	401A	401	402	501	502
Temperature (°C)	60.0	60.0	193.7	193.7	414.7	56.2	112
Pressure(kPa)	110	110	10	10	20	40	50
Molar flow(kg-mol/h)	2.04	0.084	0.155	3.384	0.06	0.42	1.52
Mass flow (kg/h)	122.31	13.73	7.82	999.88	52.50	9.02	113.29
Liquid volume flow (m <sup>3</sup> /h)	0.153	0.287	1.167	1.440	0.140	1.300	1.108
Component mass fraction							
Methanol	0.032	0.000	0.388	0.000	0.000	0.363	0.000
Palm oil	0.000	0.000	0.000	0.000	1.000	0.000	0.000
FAME (biodiesel)	0.002	0.000	0.504	0.997	0.000	0.000	0.000
Glycerol	0.850	0.000	0.000	0.000	0.000	0.000	0.085
NaOH	0.000	0.000	0.000	0.008	0.000	0.000	0.000
H <sub>2</sub> O	0.117	0.000	0.107	0.003	0.002	0.637	0.150
H <sub>3</sub> PO <sub>4</sub>	0.000	0.005	0.000	0.000	0.000	0.000	0.000
Na <sub>3</sub> PO <sub>4</sub>	0.000	0.995	0.000	0.000	0.000	0.000	0.000

**Table A.2** Column Specifications of Biodiesel Production Process

Main Process Equipment			
Separation Unit	T-200	Temperature, °C	28/122
		Pressure, kPa	20/30
		Condenser duty, MJ	0.4
		Reboiler duty, MJ	0.5
		Normal vapor flowrate, kg/h	330
		Size ( <i>DxH</i> )	0.6x10
	T-300	Total flowrate, kg/h	1190
		Size ( <i>DxH</i> )	0.8x10
	T-400	Temperature, °C	194/415
		Pressure, kPa	10/20
		Condenser duty, MJ	1.3
		Reboiler duty, MJ	1.7
		Normal vapor flowrate, kg/h	3100
		Size ( <i>DxH</i> )	1.2x12
	T-500	Temperature, °C	46/114
		Pressure, kPa	40/50
		Condenser duty, MJ	1.0
		Reboiler duty, MJ	1.0
		Normal vapor flowrate, kg/h	740
		Size ( <i>DxH</i> )	0.8x10

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**Table A.3** Reactor Specifications of Biodiesel Production Process

Main Process Equipment			
Transesterification Reactor	R-100	Temperature, °C	60
		Pressure, kPa	400
		Size ( <i>DxH</i> )	1.8x5.4
Neutralization Reactor	R-300	Temperature, °C	Room Temperature
		Pressure, kPa	Atmosphas
		Size ( <i>DxH</i> )	1x3

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## APPENDIX B

### PARAMETER TUNING OF CONTROL STRUCTURES

#### B.1. Tuning Flow, Level, Pressure and Temperature Loops

##### Flow Controllers

The dynamics of flow measurement are fast. The time constants for moving control valves are small. Therefore, the controller can be tuned with a small integral or reset time constants  $\tau_I$ . A value of  $\tau_I = 0.3$  minutes works in most flow controllers. The value of controller gain should be kept modest because flow measurement signals are sometime noisy due to the turbulent flow through the orifice plant. A value of controller gain of  $K_c$  is often used. Derivative action should not be used.

In a real plant application, filtering of the flow signal is also recommended because of the noise. So filtering is put at controller output signal and a good number to use for a flow loop is  $\tau_F=0.1$  minute.

##### Level Controllers

Most level controllers should use proportional-only action with a gain of 1 to 2. This provides the maximum amount of flow smooting. Proportional control means there will be steady-state offset (the level will not be returned to its setpoint value). However, maintaining a liquid level at a certain value is often not necessary when the liquid capacity is simply being used as surge volume. So the recommended tuning of a level controller is  $K_c=2$ .

##### Pressure Controllers

Setting the integral time equal to about 2 to 4 times the process time constant and using a reasonable controller gain usually gives satisfactory pressure



control. Of course the gain used depends on the span of the pressure transmitter. Some simple step tests can be used to find the value of controller gain that yields satisfactory pressure control. Typical pressure controller tuning constants for columns and tanks are  $K_c=2$  and  $\tau_I=20$  minute.

### Temperature Controllers

Temperature dynamic responses are generally slow, so PID control is used. Typically, the controller gain,  $K_c$ , should be set between 2 to 10, the integral time,  $\tau_I$ , should be set between 2 to 10 minutes, and the derivative time  $\tau_d$ , should be set between 0 and 5 minutes.

### B.2 Relay-Feedback Testing

If we have a controller that needs tuning and after we have inserted reasonable lags and deadtimes, we need a quick and simple method for identifying the dynamic parameters that are important for designing a feedback controller. The relay-feedback test is a tool that serves this purpose well. The results of the test are the ultimate gain and the ultimate frequency. This information is usually sufficient to permit us to calculate some reasonable controller tuning constants.

The method consists of merely inserting an on-off relay in the feedback loop. The only parameter that must be specified is the high of the relay  $h$ . This height is typically 5 to 10% of the controller-output scale. The loop starts to oscillate around the setpoint, with the controller output switching every time the process variable (PV) signal crosses the setpoint.

The maximum amplitude of the PV signal is used to calculate the ultimate gain  $K_u$  from the equation:

$$K_u = \frac{4h}{a\pi}$$

The period of the output PV curve is the ultimate period  $P_u$ . From these two parameters, controller tuning constants can be calculated for PI or PID controllers,

using a variety of tuning methods proposed in the literature that require only the ultimate gain and ultimate frequency, e.g., Ziegler-Nichols, Tyreus-Luyben, etc.

The test has many positive features that have led to its widespread use in real plants as well in simulation studies:

1. Only one parameter has to be specified (relay height)
2. The time it takes to run the test is short, particularly compared to the extended periods required for methods like PRBS.
3. The test is closed loop, so the process is not driven away from the setpoint.
4. The information obtained is very accurate in the frequency range that is important for the design of a feedback controller (the ultimate frequency)
5. The impact of load changes that occur during the test can be detected by a change to asymmetric in the manipulated variable.

All these features make relay-feedback testing a useful identification tool.

Knowing the ultimate gain  $K_u$  and ultimate period  $P_u$  permits us to calculate controller setting. There are several methods that require only these two parameters. The Ziegler-Nichols tuning equations for a PI controller are:

$$K_c = \frac{K_u}{2.2}$$

$$\tau_I = \frac{P_u}{1.2}$$

These tuning constants are frequently too aggressive for many chemical engineering applications. The Tyreus-Luyben tuning method provides more conservative setting with increased robustness. The TL equations for a PI controller are:

$$K_c = \frac{K_u}{3.2}$$

$$\tau_I = 2.2P_u$$

Relay feedback testing can be done in HYSYS. It simply click the /tuning bottom on the controller faceplate, select Autotuning and click the strat autotuning botton. The loop will strat to oscillate. After several cycles, the tuning is stopped and some recommended settings for a PID controller are suggested.



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Table B.1 Parameter tuning of biodiesel production process CS1

Controller	Controlled variable	Manipulate variable	Control action	$K_C$	$\tau_I$	$\tau_d$
RATIO-100	Ratio control between palm oil and methanol	VLV-100	direct	0.5	0.3	-
TIC-105	Preheat temperature	H 105	reverse	13.2	0.483	0.107
PIC-101	Pressure of transesterification reactor	E-R100	direct	2	10	-
TIC-100	Temperature of transesterification reactor	VLV-103	direct	2	-	-
LIC-100	Level of condenser at T-200	VLV-200	direct	2	-	-
LIC-109	Level of reboiler at T-200	VLV-201	direct	2	-	-
TIC-200	Temperature reboiler at T-200	H T200	reverse	13.2	0.302	0.0671
PIC-102	Pressure of condenser at T-200	QC T200	direct	2	10	-
TIC-104	temperature at stream 202C	QC2	direct	7.23	0.583	0.129
FIC-102	water flow rate	VLV-300	reverse	0.5	0.3	-
FIC-104	liquid flow rate from T-300	VLV-302	direct	0.5	0.3	-
RATIO-101	ratio control between stream 302 and H <sub>3</sub> PO <sub>4</sub>	VLV-303	direct	0.5	1	-
LIC-103	Level of reboiler at T-400	VLV-301	reverse	2	-	-
PIC-104	Pressure of condenser at T-400	QC T400	direct	2	10	-
LIC-105	Level of condenser at T-400	VLV-401	direct	2	-	-
TIC-400	Temperature reboiler at T-400	H T400	reverse	13.2	0.5	0.1
FIC-101	Unconvert oil flow rate	VLV-402	reverse	0.5	0.3	-
PIC-103	Pressure of condenser at T-500	QC T500	direct	2	10	-
LIC-103	Level of condenser at T-500	VLV-501	direct	2	-	-
TIC-500	Temperature reboiler at T-500	H T500	reverse	1.75	7.5	0.015

Table B.2 Parameter tuning of biodiesel production process CS2

Controller	Controlled variable	Manipulate variable	Control action	$K_C$	$\tau_I$	$\tau_d$
RATIO-100	Ratio control between palm oil and methanol	VLV-100	direct	0.5	0.3	-
TIC-105	Preheat temperature	H 105	reverse	13.2	0.483	0.107
PIC-101	Pressure of transesterification reactor	VLV-102	direct	2	10	-
TIC-100	Temperature of transesterification reactor	E-R100	direct	2	10	-
FIC-105	liquid exit flow rate from R100	VLV-103	reverse	0.5	0.3	-
LIC-102	Level of condenser at T-200	Reflux	direct	2	-	-
TIC-200	Temperature reboiler at T-200	H T200	reverse	13.2	0.302	0.0671
PIC-102	Pressure of condenser at T-200	QC T200	direct	2	10	-
FIC-106	Recycle flow rate	VLV-200	reverse	0.5	0.3	-
LIC-103	Level of reboiler at T-200	VLV-201	direct	2	-	-
TIC-104	Temperature of stream 202C	QC2	reverse	0.1	0.1	-
FIC-102	water flow rate	VLV-300	reverse	0.5	0.3	-
FIC-103	liquid flow rate exit from T-300	VLV-301	reverse	0.5	0.3	-
PIC-106	Pressure of condenser at T-400	QC T400	direct	2	10	-
LIC-107	Level of condenser at T-400	Reflux	direct	2	-	-
TIC-400	Temperature reboiler at T-400	H T400	reverse	13.2	0.302	0.0671

**Table B.2** Parameter tuning of biodiesel production process CS2 (Cont)

Controller	Controlled variable	Manipulate variable	Control action	$K_C$	$\tau_I$	$\tau_d$
FIC-101	Biodiesel flow rate	VLV-401	reverse	0.5	0.3	-
LIC-105	Level of reboiler at T-400	VLV-402	direct	2	-	-
LIC-106	Level of reboiler at T-500	VLV-305	reverse	2	-	-
PIC-103	Pressure of condenser at T-500	QC T500	direct	2	10	-
LIC-500	Level of condenser at T-500	Reflux	direct	2	-	-
TIC-500	Temperature reboiler at T-500	H T500	reverse	13.2	0.302	0.0671
FIC-104	Glycerol flow rate	VLV-502	reverse	0.5	0.3	-

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Table B.3 Parameter tuning of biodiesel production process CS3

Controller	Controlled variable	Manipulate variable	Control action	$K_C$	$\tau_I$	$\tau_d$
RATIO-100	Ratio control between palm oil and methanol	VLV-100	direct	0.5	0.3	-
TIC-105	Preheat temperature	H 105	reverse	13.2	0.483	0.107
PIC-101	Pressure of transesterification reactor	VLV-102	direct	2	10	-
TIC-100	Temperature of transesterification reactor	E-R100	direct	2	10	-
FIC-105	Level of reboiler at T-200	VLV-103	reverse	2	-	-
TIC-200	Temperature reboiler at T-200	H T200	reverse	13.2	0.302	0.0671
PIC-102	Pressure of condenser at T-200	QC T200	direct	2	10	-
TIC-104	Temperature of stream 202C	QC2	reverse	0.1	0.1	-
FIC-105	liquid exit from stream 202	VLV-201	reverse	0.5	0.3	-
FIC-102	water flow rate	VLV-300	reverse	0.5	0.3	-
FIC-103	liquid flow rate exit from T-300	VLV-301	reverse	0.5	0.3	-
PIC-106	Pressure of condenser at T-400	QC T400	direct	2	10	-
LIC-107	Level of condenser at T-400	VLV-401	direct	2	-	-
TIC-400	Temperature reboiler at T-400	H T400	reverse	13.2	0.302	0.0671
FIC-101	Biodiesel flow rate	VLV-401	reverse	0.5	0.3	-
LIC-105	Level of reboiler at T-400	VLV-402	direct	2	-	-
LIC-106	Level of reboiler at T-500	VLV-502	reverse	2	-	-
PIC-103	Pressure of condenser at T-500	QC T500	direct	2	10	-
LIC-500	Level of condenser at T-500	VLV-501	direct	2	-	-
TIC-500	Temperature reboiler at T-500	H T500	reverse	13.2	0.302	0.0671

## APPENDIX C

### FIXTURE POINT THEOREM DATA

**Table C.1** : List of Manipulated Variables for the biodiesel Production Process.

Manipulated Variables	Description
VLV-100	Methanol freash feed valve
VLV-101	Palm oil freash feed valve
VLV-102	Pressure transesterification reactor valve
VLV-103	Pruge transesterification reactor bottom valve
VLV-200	Methanol recovery column feed recycle vale
VLV-201	Pruge methanol recovery bottom vale
VLV-300	Water freash feed valve
VLV-301	Pruge liquid-liquid extraction column upper valve
VLV-302	Pruge liquid-liquid extraction column bottom valve
VLV-303	Freash phosphoric acid valve
VLV-304	Pressure neutralization reactor valve
VLV-305	Pruge neutralization reactor bottom valve
VLV-400	Prage biodiesel column upper valve
VLV-401	Prage biodiesel column feed valve
VLV-402	Pruge biodiesel column bottom vale
VLV-500	Prage glycerol column upper valve
VLV-501	Prage glycerol column upper valve
VLV-502	Prage glycerol column bottom valve
E-R100	Heat duty of transesterification reactor valve
H 105	Heat duty of stream 105 (palm oil freash feed)
H T200	Heat duty of methanol recovery column



Table C.1 (Cont)

Manipulated Variables	Description
H T400	Heat duty of biodiesel column
H T500	Heat duty of glycerol column
QC 201	Condenser duty of stream 201(methanol recovery stream)
QC 202	Condenser duty of stream 202(bottom stream)
QC T200	Condenser duty of methanol recovery column
QC T400	Condenser duty of biodiesel column
QC T500	Condenser duty of glycerol column



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Table C.2 IAE Results of Flow Rate Deviation for the Process Stream

Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
103B-M	0.1480	0.0450	0.1084	0.0746	0.0521	0.0527	0.0528	0.0149	0.0528	0.0528	0.0419	0.0523	0.0525	0.0525
103C-M	0.1480	0.0450	0.1084	0.0746	0.0521	0.0526	0.0528	0.0149	0.0527	0.0528	0.0419	0.0523	0.0525	0.0525
105A-M	0.3283	0.8322	0.6881	0.4733	0.3304	0.3344	0.3348	0.0943	0.3349	0.3342	0.2662	0.3316	0.3327	0.3327
105B-M	0.3286	0.8322	0.6881	0.4733	0.3304	0.3341	0.3348	0.0943	0.3484	0.3345	0.2660	0.3316	0.3327	0.3327
105C-M	0.3424	0.8771	0.7015	0.4858	0.3442	0.3476	0.3486	0.0981	0.3484	0.3487	0.2769	0.3453	0.3464	0.3464
106-M	1.8109	1.7799	1.5846	5.7924	1.8094	1.8019	1.7663	0.5120	1.7990	1.7872	1.4521	1.7723	1.7801	1.7781
106A-M	1.8109	1.7806	1.5846	5.7924	1.8127	1.8019	1.7670	0.5107	1.8020	1.7872	1.4508	1.7723	1.7801	1.7781
201-M	0.2597	0.2618	0.2785	0.2648	0.3466	0.2571	0.2503	0.0722	0.2590	0.2573	0.2099	0.2558	0.2609	0.2664
201A-M	0.2375	0.2301	0.2657	0.2475	0.3179	0.2339	0.2309	0.0646	0.2340	0.2363	0.1865	0.2349	0.2403	0.2412
201B-M	0.2375	0.2293	0.2646	0.2400	0.3195	0.2331	0.2359	0.0663	0.2331	0.2358	0.1899	0.2349	0.2403	0.2412
201N-M	0.2375	0.2301	0.2646	0.2474	0.3195	0.2328	0.2311	0.0661	0.2340	0.2358	0.1899	0.2349	0.2403	0.2412
202B-M	2.8520	2.8540	2.7730	3.4434	2.8658	3.4310	2.8901	0.8083	2.8638	2.9066	0.5927	2.9081	2.8425	2.8435
202C-M	0.4232	0.4277	0.4207	0.5827	0.4250	0.3980	0.4273	0.1194	0.4249	0.4298	0.3428	0.4281	0.4282	0.4270
301-M	1.4421	1.4262	1.4317	1.2974	1.4441	1.4389	1.4417	2.3256	1.4451	1.4428	1.2069	1.4339	1.4386	1.4386
301A-M	1.4421	1.4262	1.4317	1.2986	1.4431	1.4402	1.4417	2.4654	1.4451	1.4451	1.2069	1.4339	1.4386	1.4386
302-M	0.0018	0.0018	0.0018	0.0017	0.0018	0.0018	0.0018	0.0005	0.0018	0.0018	0.0015	0.0018	0.0018	0.0018
302A-M	0.0018	0.0018	0.0018	0.0017	0.0018	0.0018	0.0018	0.0005	0.0018	0.0018	0.0015	0.0018	0.0018	0.0018
304-M	0.0022	0.0022	0.0022	0.0020	0.0022	0.0022	0.0022	0.0006	0.0022	0.0022	0.0018	0.0022	0.0022	0.0022
304A-M	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
401-M	0.5192	0.5132	0.5145	0.4672	0.5201	0.5175	0.5185	0.2324	0.5195	0.5195	0.4316	0.5156	0.5173	0.5173
401A-M	0.5189	0.5128	0.5148	0.4676	0.5181	0.5175	0.5206	0.2324	0.5199	0.5199	0.4316	0.5156	0.5173	0.5165

Table C.2 IAE Results of Flow Rate Deviation for the Process Stream (Cont)

Stream	VLV-305	QC	QC 2	QC T200	QC T400	H T200	H T400	E-R100	QC T500	H T500	VLV-501	VLV-502	SUM
103B-M	0.0525	0.0749	0.0528	0.0354	0.0538	0.0532	0.0590	0.0831	0.0538	0.0532	0.0525	0.0525	1.5295
103C-M	0.0525	0.0749	0.0529	0.0355	0.0538	0.0533	0.0591	0.0831	0.0538	0.0533	0.0525	0.0525	1.5301
105A-M	0.3327	0.1867	0.3351	0.2185	0.3409	0.3378	0.3741	0.5277	0.3409	0.3378	0.3327	0.3327	9.3457
105B-M	0.3327	0.1867	0.3344	0.2185	0.3409	0.3378	0.3741	0.5277	0.3409	0.3378	0.3327	0.3327	9.3586
105C-M	0.3464	0.1867	0.3483	0.2276	0.3550	0.3511	0.3895	0.5410	0.3550	0.3511	0.3464	0.3464	9.7017
106-M	1.7827	2.1255	1.8088	2.9632	1.8544	1.8598	2.0307	2.0074	1.8544	1.8598	1.7781	1.7827	50.9337
106A-M	1.7827	2.1255	1.8084	2.9936	1.8544	1.8602	2.0351	2.0074	1.8544	1.8602	1.7781	1.7827	50.9739
201-M	0.2585	0.4153	0.2656	7.0342	0.2709	0.2728	0.2939	0.2735	0.2709	0.2728	0.2664	0.2585	13.6536
201A-M	0.2350	0.3468	0.2388	7.9607	0.2439	0.2413	0.2616	0.2562	0.2439	0.2413	0.2412	0.2350	13.9470
201B-M	0.2350	0.3389	0.2388	7.9586	0.2439	0.2413	0.2561	0.2562	0.2439	0.2413	0.2412	0.2350	13.9315
201N-M	0.2350	0.3468	0.2388	7.9607	0.2439	0.2413	0.2628	0.2562	0.2439	0.2413	0.2412	0.2350	13.9520
202B-M	0.9059	4.1783	3.2469	1.4282	3.0330	2.6522	3.2154	2.8600	3.0330	2.6522	2.8435	2.9059	71.8291
202C-M	0.4303	0.6044	0.6933	0.2905	0.4381	0.4182	0.4777	0.4120	0.4381	0.4182	0.4270	0.4303	11.1831
301-M	1.4386	1.1988	1.4408	0.9622	1.4599	1.4537	1.5154	1.4200	1.4599	1.4537	1.4386	1.4386	37.3335
301A-M	1.4386	1.1820	1.4408	0.9622	1.5051	1.4537	1.5157	1.4200	1.5051	1.4537	1.4386	1.4386	37.5511
302-M	0.0018	0.0026	0.0018	0.0012	0.0019	0.0019	0.0021	0.0018	0.0019	0.0019	0.0018	0.0018	0.0463
302A-M	0.0018	0.0026	0.0018	0.0012	0.0019	0.0019	0.0021	0.0018	0.0019	0.0019	0.0018	0.0018	0.0463
304-M	0.0022	0.0031	0.0022	0.0015	0.0022	0.0022	0.0025	0.0022	0.0022	0.0022	0.0022	0.0022	0.0554
304A-M	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0006
401-M	0.5173	0.4441	0.5181	0.3475	0.5420	0.5231	0.5565	0.5107	0.5420	0.5231	0.5173	0.5173	12.8826
401A-M	0.5173	0.4448	0.5181	0.3475	0.5420	0.5213	0.5565	0.5107	0.5420	0.5213	0.5165	0.5173	12.8789

Table C.2 IAE Results of Flow Rate Deviation for the Process Stream (Cont)

Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
402-M	42.6746	42.0021	42.2809	38.3486	42.5402	42.4436	42.9415	45.2652	42.8231	42.7339	44.8450	42.6312	42.7531	42.7541
402A-M	42.5867	42.0883	42.3682	38.3486	42.5980	42.5309	42.5857	42.2730	42.6452	42.7339	44.8450	42.6312	42.7531	42.7541
403-M	0.2892	0.2858	0.2871	0.2604	0.2899	0.2885	0.2903	1.8148	0.2904	0.2895	0.2408	0.4254	0.2889	0.2889
403A-M	0.2895	0.2861	0.2871	0.2607	0.2896	0.2875	0.2902	1.8481	0.2900	0.2900	0.2408	0.4254	0.2889	0.2889
H3PO4-1M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0000	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
H3PO4-2M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0000	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
R1-1M	0.0206	0.0183	0.0859	0.0302	0.0208	0.0210	0.0211	0.0059	0.0211	0.0077	0.0168	0.0209	0.0209	0.0209
R1-2M	0.0208	0.0185	0.0861	0.0304	0.0209	0.0212	0.0213	0.0060	0.0212	0.0213	0.0169	0.0211	0.0211	0.0211
R2-1M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0000	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
R2-2M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0000	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
WATER-1M	0.0019	0.0019	0.0019	0.0017	0.0019	0.0019	0.0019	0.0005	0.0019	0.0019	0.0015	0.0019	0.0019	0.0019
WATER-2M	0.0019	0.0019	0.0019	0.0017	0.0019	0.0019	0.0019	0.0005	0.0019	0.0019	0.0015	0.0019	0.0019	0.0019
501-M	0.5192	0.5132	0.5145	0.4672	0.5201	0.5175	0.5185	0.2324	0.5195	0.5195	0.4316	0.5156	0.5173	0.5173
501A-M	0.5189	0.5128	0.5148	0.4676	0.5181	0.5175	0.5206	0.2324	0.5199	0.5199	0.4316	0.5156	0.5173	0.5165
502-M	0.2892	0.2858	0.2871	0.2604	0.2899	0.2885	0.2903	1.8148	0.2904	0.2895	0.2408	0.4254	0.2889	0.2889
502A-M	0.2895	0.2861	0.2871	0.2607	0.2896	0.2875	0.2902	1.8481	0.2900	0.2900	0.2408	0.4254	0.2889	0.2889
503-M	42.6746	42.0021	42.2809	38.3486	42.5402	42.4436	42.9415	42.7537	42.8231	42.7339	44.8450	42.6312	42.7531	42.7541
503A-M	42.5867	42.0883	42.3682	38.3486	42.5980	42.5309	42.5857	42.2730	42.6452	42.7339	42.7537	42.6312	42.7531	42.7541

Table C.2 IAE Results of Flow Rate Deviation for the Process Stream (Cont)

Stream	VLV-305	QC	QC 2	QC T200	QC T400	H T200	H T400	E-R100	QC T500	H T500	VLV-501	VLV-502	SUM
402-M	42.7537	42.4699	42.3553	28.3462	42.4809	42.5440	42.0182	42.2021	42.4809	42.5440	42.7541	42.7537	1092.7400
402A-M	72.7537	41.5611	42.4410	28.2895	42.4809	42.9487	42.0988	42.2021	42.4809	42.9487	42.7541	42.7537	1089.4551
403-M	0.2889	0.2338	0.2885	0.1931	0.3045	0.2916	0.2958	0.2851	0.3045	0.2916	0.2889	0.2889	8.9850
403A-M	0.2889	0.2342	0.2885	0.1935	0.3045	0.2919	0.2958	0.2851	0.3045	0.2919	0.2889	0.2889	9.0194
H3PO4-1M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0021
H3PO4-2M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0021
R1-1M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0021
R1-2M	0.0209	0.0119	0.0211	0.0137	0.0215	0.0213	0.0235	0.0310	0.0215	0.0213	0.0209	0.0209	0.5818
R2-1M	0.0211	0.0119	0.0212	0.0138	0.0217	0.0214	0.0238	0.0312	0.0217	0.0214	0.0211	0.0211	0.5995
R2-2M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0019
WATER-1M	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0019
WATER-2M	0.0019	0.0027	0.0019	0.0013	0.0019	0.0019	0.0021	0.0019	0.0019	0.0019	0.0019	0.0019	0.0482
501-M	0.0019	0.0027	0.0019	0.0013	0.0019	0.0019	0.0021	0.0019	0.0019	0.0019	0.0019	0.0019	0.0482
501A-M	0.5173	0.4441	0.5181	0.3475	0.5420	0.5231	0.5565	0.5107	0.5420	0.5231	0.5173	0.5173	12.8826
502-M	0.5173	0.4448	0.5181	0.3475	0.5420	0.5213	0.5565	0.5107	0.5420	0.5213	0.5165	0.5173	12.8789
502A-M	0.2889	0.2338	0.2885	0.1931	0.3045	0.2916	0.2958	0.2851	0.3045	0.2916	0.2889	0.2889	8.9850
503-M	42.7537	42.4699	42.3553	28.3462	42.4809	42.5440	42.0182	42.2021	42.4809	42.5440	42.7541	44.8450	1092.3198
503A-M	42.7537	41.5611	42.4410	28.2895	42.4809	42.9487	42.0988	42.2021	42.4809	42.9487	42.7541	44.8450	1089.4551

Table C.3 IAE Results of Pressure Deviation for the Process Stream

Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
103B-P	0.00823	0.00260	0.00548	0.00380	0.00292	0.00285	0.00306	0.00087	0.00356	0.00302	0.00247	0.00302	0.00304	0.00304
103C-P	0.00437	0.00387	0.00821	0.00568	0.00435	0.00425	0.00456	0.00129	0.00529	0.00450	0.00367	0.00450	0.00453	0.00452
105A-P	0.00236	0.00604	0.00443	0.00307	0.00235	0.00230	0.00247	0.00070	0.00000	0.00243	0.00199	0.00244	0.00245	0.00245
105B-P	0.00325	0.00401	0.00611	0.00423	0.00324	0.00316	0.00340	0.00096	0.00471	0.00335	0.00273	0.00335	0.00338	0.00337
105C-P	0.00437	0.00387	0.00821	0.00568	0.00435	0.00424	0.00456	0.00129	0.00530	0.00450	0.00367	0.00450	0.00453	0.00452
106-P	0.00437	0.00387	0.00821	0.00568	0.00435	0.00425	0.00456	0.00129	0.00530	0.00450	0.00367	0.00450	0.00453	0.00452
106A-P	0.30378	0.30840	0.26705	0.31077	0.30187	0.28777	0.30472	0.08841	0.35963	0.30646	0.25357	0.30706	0.30711	0.30675
201-P	0.45272	0.46860	0.40334	0.46184	0.45401	0.43059	0.45175	0.13095	0.54439	0.45594	0.38543	0.45633	0.45828	0.46085
201A-P	0.00596	0.00595	0.00729	0.00551	0.01131	0.00564	0.00596	0.00169	0.00703	0.00600	0.00489	0.00602	0.00615	0.00620
201B-P	0.00437	0.00387	0.00821	0.00747	0.00435	0.00425	0.00456	0.00129	0.00530	0.00450	0.00368	0.00450	0.00453	0.00452
201N-P	0.56849	0.57080	0.50734	0.51623	0.58386	0.53305	0.56751	0.16258	0.66431	0.57165	0.47189	0.57550	0.58848	0.59118
202B-P	0.03170	0.03197	0.02763	0.03456	0.03147	0.06193	0.03292	0.00922	0.03787	0.03267	0.73104	0.03299	0.03250	0.03226
202C-P	0.13076	0.13362	0.11423	0.14293	0.13032	0.15067	0.13591	0.03745	0.15529	0.13607	0.10947	0.13626	0.13371	0.13350
301-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
301A-P	0.41881	0.42291	0.37518	0.34141	0.41558	0.40040	0.42940	0.38657	0.50019	0.42466	0.36469	0.42555	0.42855	0.42773
302-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
302A-P	0.00003	0.00003	0.00003	0.00002	0.00003	0.00003	0.00003	0.00001	0.00004	0.00003	0.00002	0.00003	0.00003	0.00003
304-P	0.00006	0.00006	0.00006	0.00005	0.00006	0.00006	0.00007	0.00002	0.00008	0.00006	0.00005	0.00006	0.00007	0.00007
304A-P	0.00039	0.00040	0.00035	0.00032	0.00039	0.00038	0.00040	0.00011	0.00047	0.00040	0.00033	0.00040	0.00040	0.00040
401-P	0.58098	0.58619	0.51924	0.47319	0.57691	0.55397	0.59458	0.35894	0.69276	0.58815	0.50955	0.59001	0.59416	0.59303
401A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000

Table C.3 IAE Results of Pressure Deviation for the Process Stream (Cont)

Stream	VLV-305	QC	QC 2	QC T200	QC T400	H T200	H T400	E-R100	QC T500	H T500	VLV-501	VLV-502	SUM
103B-P	0.00302	0.00356	0.00292	0.00148	0.00298	0.00308	0.00308	0.00444	0.00298	0.00308	0.00304	0.00302	0.08463
103C-P	0.00450	0.00531	0.00435	0.00221	0.00444	0.00459	0.00460	0.00663	0.00444	0.00460	0.00452	0.00450	0.11831
105A-P	0.00243	0.00113	0.00235	0.00116	0.00240	0.00248	0.00248	0.00358	0.00240	0.00248	0.00245	0.00243	0.06324
105B-P	0.00335	0.00155	0.00323	0.00160	0.00331	0.00341	0.00341	0.00494	0.00331	0.00341	0.00337	0.00335	0.08751
105C-P	0.00450	0.00208	0.00434	0.00215	0.00444	0.00458	0.00458	0.00663	0.00444	0.00458	0.00452	0.00450	0.11497
106-P	0.00450	0.00208	0.00434	0.00214	0.00444	0.00458	0.00458	0.00663	0.00444	0.00458	0.00452	0.00450	0.11497
106A-P	0.30522	0.36818	0.29779	0.10157	0.30613	0.33840	0.31543	0.28310	0.30613	0.31543	0.30675	0.30522	7.56272
201-P	0.46188	0.55372	0.44823	0.14790	0.45960	0.51190	0.47981	0.42575	0.45960	0.47981	0.46085	0.46188	11.36596
201A-P	0.00600	0.00570	0.00585	0.09056	0.00602	0.00607	0.00611	0.00661	0.00602	0.00611	0.00620	0.00600	0.24283
201B-P	0.00450	0.00208	0.00434	0.00214	0.00444	0.00458	0.00458	0.00663	0.00444	0.00458	0.00452	0.00450	0.11676
201N-P	0.57293	0.73288	0.55796	1.32699	0.57376	0.59568	0.57822	0.54550	0.57376	0.57822	0.59118	0.57293	15.27286
202B-P	0.03279	0.03911	0.03417	0.01117	0.03306	0.03272	0.03326	0.02985	0.03306	0.03326	0.03226	0.03279	1.53820
202C-P	0.13571	0.15894	0.14546	0.04863	0.13627	0.12441	0.13639	0.12440	0.13627	0.13639	0.13350	0.13571	3.33227
301-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
301A-P	0.42549	0.28930	0.40874	0.20664	0.42839	0.43155	0.40579	0.39116	0.42839	0.40579	0.42773	0.42549	10.43610
302-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
302A-P	0.00003	0.00004	0.00003	0.00001	0.00003	0.00003	0.00003	0.00003	0.00003	0.00003	0.00003	0.00003	0.00075
304-P	0.00006	0.00008	0.00006	0.00003	0.00006	0.00007	0.00007	0.00006	0.00006	0.00007	0.00007	0.00006	0.00159
304A-P	0.00040	0.00047	0.00039	0.00020	0.00039	0.00041	0.00041	0.00037	0.00039	0.00041	0.00040	0.00040	0.00977
401-P	0.58991	0.41855	0.56594	0.28726	0.59438	0.59814	0.56004	0.54232	0.59438	0.56004	0.59303	0.58991	14.30555
401A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000

Table C.3 IAE Results of Pressure Deviation for the Process Stream (Cont)

Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
402-P	0.58098	0.58516	0.51999	0.47319	0.57607	0.55446	0.59757	0.32268	0.69337	0.58815	0.50955	0.59001	0.59416	0.59303
402A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
403-P	0.26672	0.26911	0.23893	0.21743	0.26509	0.25481	0.27428	3.28512	0.31870	0.27005	0.23087	0.27088	0.27279	0.27227
403A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
H3PO4-1P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
H3PO4-2P	0.00006	0.00006	0.00006	0.00005	0.00006	0.00006	0.00007	0.00002	0.00008	0.00006	0.00005	0.00006	0.00007	0.00007
R1-1P	0.00499	0.00443	0.00939	0.00649	0.00497	0.00485	0.00521	0.00147	0.00606	0.00514	0.00420	0.00514	0.00518	0.00517
R1-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R2-1P	0.00006	0.00006	0.00006	0.00005	0.00006	0.00006	0.00007	0.00002	0.00008	0.00006	0.00005	0.00006	0.00007	0.00007
R2-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
WATER-1P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
WATER-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
501-P	0.14525	0.14655	0.12981	0.11830	0.14423	0.13849	0.14865	0.08973	0.17319	0.14704	0.12739	0.14750	0.14854	0.14826
501A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
502-P	0.14525	0.14629	0.13000	0.11830	0.14402	0.13861	0.14939	0.08067	0.17334	0.14704	0.12739	0.14750	0.14854	0.14826
502A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
503-P	0.10669	0.10765	0.09557	0.08697	0.10604	0.10192	0.10971	1.31405	0.12748	0.10802	0.09235	0.10835	0.10911	0.10891
503A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000



Table C.3 IAE Results of Pressure Deviation for the Process Stream (Cont)

Stream	VLV-305	QC	QC 2	QC T200	QC T400	H T200	H T400	E-R100	QC T500	H T500	VLV-501	VLV-502	SUM
402-P	0.58991	0.42498	0.56594	0.28701	0.59438	0.59571	0.55947	0.54232	0.59438	0.55947	0.59303	0.58991	14.27489
402A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
403-P	0.27084	0.17644	0.26017	0.00000	0.27224	0.27482	0.26190	0.24899	0.27224	0.26190	0.27227	0.27084	9.54967
403A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
H3PO4-1P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
H3PO4-2P	0.00006	0.00008	0.00006	0.00003	0.00006	0.00007	0.00007	0.00006	0.00006	0.00007	0.00007	0.00006	0.00159
R1-1P	0.00514	0.00238	0.00496	0.00247	0.00507	0.00524	0.00524	0.00758	0.00507	0.00524	0.00517	0.00514	0.13142
R1-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R2-1P	0.00006	0.00008	0.00006	0.00003	0.00006	0.00007	0.00007	0.00006	0.00006	0.00007	0.00007	0.00006	0.00159
R2-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
WATER-1P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
WATER-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
501-P	0.14748	0.10464	0.14148	0.07182	0.14860	0.14953	0.14001	0.13558	0.14860	0.14001	0.14826	0.14748	3.57639
501A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
502-P	0.14748	0.10624	0.14148	0.07175	0.14860	0.14893	0.13987	0.13558	0.14860	0.13987	0.14826	0.14748	3.56872
502A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
503-P	0.10834	0.07058	0.10407	0.00000	0.10890	0.10993	0.10476	0.09960	0.10890	0.10476	0.10891	0.10834	3.81987
503A-P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000

Table C.4 IAE Results of Temperature Deviation for the Process Stream

Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
103B-T	0.00079	0.00023	0.00059	0.00042	0.00027	0.00027	0.00027	0.00007	0.00027	0.00027	0.00020	0.00027	0.00027	0.00027
103C-T	0.00079	0.00023	0.00059	0.00042	0.00027	0.00027	0.00027	0.00007	0.00027	0.00027	0.00020	0.00027	0.00027	0.00027
105A-T	0.00042	0.00107	0.00089	0.00065	0.00042	0.00044	0.00044	0.00012	0.70162	0.00044	0.00032	0.00043	0.00043	0.00043
105B-T	0.00042	0.00107	0.00089	0.00065	0.00042	0.00044	0.00044	0.00012	0.03968	0.00044	0.00032	0.00043	0.00043	0.00043
105C-T	0.03937	0.12129	0.09757	0.06858	0.03964	0.04056	0.04012	0.01114	0.03968	0.04012	0.02935	0.03975	0.03978	0.03987
106-T	0.03852	0.14071	0.08412	0.06502	0.03934	0.04010	0.03958	0.01105	0.03935	0.03997	0.02919	0.03982	0.03977	0.03975
106A-T	0.16370	0.27312	0.21688	0.21079	0.16962	0.17262	0.16731	0.04807	0.17148	0.16855	0.12797	0.16725	0.16896	0.17153
201-T	5.55475	5.71933	5.48223	6.51629	5.61297	5.62437	5.37017	1.53689	5.54641	5.52244	4.23857	5.46808	5.45596	5.50380
201A-T	1.06806	1.03595	1.06264	1.39708	1.10137	1.06923	1.05847	0.28969	1.04663	1.07134	0.77034	1.06371	1.06015	1.05412
201B-T	1.06806	1.03353	1.06016	1.02630	1.10396	1.06668	1.07042	0.29320	1.04419	1.06882	0.77822	1.06371	1.06015	1.05412
201N-T	1.06604	1.03649	1.06109	1.39439	1.10484	1.06041	1.06167	0.29267	1.04722	1.06943	0.77866	1.06431	1.06076	1.05474
202B-T	2.17449	2.20439	2.18483	3.21423	2.18205	2.05497	2.18180	0.60591	2.16466	2.18213	14.69918	2.18300	2.17851	2.17767
202C-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
301-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
301A-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
302-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
302A-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
304-T	0.00012	0.00012	0.00012	0.00011	0.00012	0.00012	0.00012	0.00003	0.00012	0.00012	0.00009	0.00012	0.00012	0.00012
304A-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
401-T	14.06086	13.97562	13.95203	13.57783	14.07266	14.04932	13.88403	39.19361	13.89000	14.02656	13.04883	14.04573	14.05031	14.08517
401A-T	13.99207	13.90725	13.95777	13.59874	13.81773	14.02551	14.11664	33.91307	13.91211	14.04888	13.02940	14.02231	14.02703	13.95267

Table C.4 IAE Results of Temperature Deviation for the Process Stream (Cont)

Stream	VLV-305	QC	QC 2	QC T200	QC T400	H T200	H T400	E-R100	QC T500	H T500	VLV-501	VLV-502	SUM
103B-T	0.00027	0.00027	0.00027	0.00028	0.00024	0.00027	0.00035	0.00044	0.00024	0.00035	0.00027	0.00027	0.00795
103C-T	0.00027	0.00027	0.00027	0.00028	0.00024	0.00027	0.00035	0.00044	0.00024	0.00035	0.00027	0.00027	0.00795
105A-T	0.00043	0.00017	0.00044	0.00057	0.00039	0.00044	0.00057	0.00066	0.00039	0.00057	0.00043	0.00043	0.71363
105B-T	0.00043	0.00017	0.00044	0.00057	0.00039	0.00044	0.00057	0.00066	0.00039	0.00057	0.00043	0.00043	0.05169
105C-T	0.03974	0.02026	0.04003	0.03724	0.03550	0.04004	0.05265	0.06968	0.03550	0.05265	0.03987	0.03974	1.18971
106-T	0.03969	0.01750	0.03936	0.03472	0.03544	0.04141	0.05196	0.75231	0.03544	0.05196	0.03975	0.03969	1.86553
106A-T	0.17084	0.13664	0.17085	0.37664	0.15251	0.18458	0.22730	0.65276	0.15251	0.22730	0.17153	0.17084	5.19219
201-T	5.56924	5.88221	5.65933	3.26208	5.01631	7.48013	7.48013	5.39474	5.01631	6.01989	5.50380	5.56924	141.00569
201A-T	1.05316	0.86960	1.04870	1.23210	0.94236	1.54726	1.38558	1.02244	0.94236	1.38558	1.05412	1.05316	27.68519
201B-T	1.05316	0.85863	1.04615	1.54355	0.94236	1.23210	1.37344	1.02244	0.94236	1.37344	1.05412	1.05316	27.28641
201N-T	1.05376	0.87046	1.04678	0.52333	0.94291	1.23289	1.38965	1.02320	0.94291	1.38965	1.05474	1.05376	26.67677
202B-T	2.18663	2.16427	3.53373	2.27275	1.94057	2.10373	2.85872	2.06620	1.94057	2.85872	2.17767	2.18663	70.67797
202C-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
301-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
301A-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
302-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
302A-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
304-T	0.00012	0.00012	0.00012	0.00012	0.00010	0.00012	0.00015	0.00013	0.00010	0.00015	0.00012	0.00012	0.00299
304A-T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
401-T	14.03399	14.37269	13.81591	14.30446	14.27742	13.93670	14.74962	13.67247	14.27742	14.74962	14.08517	14.03399	390.22199
401A-T	14.01074	14.43519	13.79243	14.28014	14.25724	13.71113	14.72074	13.64966	14.25724	14.72074	13.95267	14.01074	384.11981

Table C.4 IAE Results of Temperature Deviation for the Process Stream (Cont)

Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
402-T	11.84101	11.70388	11.81647	11.43910	11.80164	11.87857	11.89412	4.30795	11.72698	11.80870	10.85370	11.80566	11.81136	11.84067
402A-T	10.64621	10.58167	10.68694	10.31363	10.65666	10.73946	10.60218	3.81549	10.51567	10.65228	10.02932	10.66103	10.66570	10.69217
403-T	18.46008	18.34817	18.47336	17.88340	18.53514	18.55357	18.60454	7.49577	18.44024	18.44246	15.15925	18.53352	18.53667	18.58267
403A-T	18.74770	18.63405	18.69117	18.16219	18.68309	18.53452	18.80605	7.16315	18.59512	18.77793	15.36851	18.76141	18.76428	18.81083
H3PO4-1T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
H3PO4-2T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R1-1T	0.03849	0.14103	0.08409	0.06476	0.03941	0.03995	0.03997	0.01098	0.03952	0.03942	0.02934	0.03978	0.03973	0.03972
R1-2T	0.03780	0.14056	0.08532	0.06517	0.03814	0.03940	0.03933	0.01087	0.03854	0.03920	0.02887	0.03915	0.03910	0.03908
R2-1T	0.00012	0.00012	0.00012	0.00011	0.00012	0.00012	0.00012	0.00003	0.00012	0.00012	0.00009	0.00012	0.00012	0.00012
R2-2T	0.00013	0.00013	0.00013	0.00013	0.00014	0.00014	0.00013	0.00004	0.00013	0.00013	0.00010	0.00013	0.00013	0.00013
WATER-1T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
WATER-2T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
501-T	140.06086	13.97562	13.95203	13.57783	14.07266	14.04932	13.88403	14.74962	13.89000	14.02656	13.04883	14.04573	14.05031	14.08517
501A-T	13.99207	13.90725	13.95777	13.59874	13.81773	14.02551	14.11664	14.72074	13.91211	14.04888	13.02940	14.02231	14.02703	13.95267
502-T	11.84101	11.70388	11.81647	11.43910	11.80164	11.87857	11.89412	4.30795	11.72698	11.80870	10.85370	11.80566	11.81136	11.84067
502AT	10.64621	10.58167	10.68694	10.31363	10.65666	10.73946	10.60218	3.81549	10.51567	10.65228	10.02932	10.66103	10.66570	10.69217
503-T	42.67461	42.00215	42.28089	38.34861	42.54016	42.44363	42.94154	42.75373	42.82309	42.73391	44.84501	42.63117	42.75307	42.75405
503A-T	42.58671	42.08830	42.36816	38.34861	42.59799	42.53091	42.58575	42.27303	42.64523	42.73391	42.75373	42.63117	42.75307	42.75405

Table C.4 IAE Results of Temperature Deviation for the Process Stream (Cont)

Stream	VLV-305	QC	QC 2	QC T200	QC T400	H T200	H T400	E-R100	QC T500	H T500	VLV-501	VLV-502	SUM
402-T	11.79764	12.87095	11.65379	12.03372	11.06843	11.59139	12.91311	11.49373	11.06843	12.91311	11.84067	11.79764	299.57240
402A-T	10.65332	11.91688	10.52945	10.80481	10.27998	10.58578	11.33984	10.37888	10.27998	11.33984	10.69217	10.65332	270.71265
403-T	18.51515	17.12952	18.16734	18.88171	19.33181	18.35870	15.04152	18.03819	19.33181	15.04152	18.58267	18.51515	458.98392
403A-T	18.74248	17.41906	18.37637	19.02640	19.70534	18.64540	15.31049	18.25967	19.70534	15.31049	18.81083	18.74248	464.55436
H3PO4-1T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
H3PO4-2T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R1-1T	0.03966	0.01759	0.03932	0.03495	0.03541	0.04150	0.05179	0.75228	0.03541	0.05179	0.03972	0.03966	1.86522
R1-2T	0.03906	0.01732	0.03869	0.03415	0.03485	0.04088	0.05113	0.74877	0.03485	0.05113	0.03908	0.03906	1.84947
R2-1T	0.00012	0.00012	0.00012	0.00012	0.00010	0.00012	0.00015	0.00013	0.00010	0.00015	0.00012	0.00012	0.00299
R2-2T	0.00013	0.00013	0.00013	0.00014	0.00012	0.00013	0.00018	0.00013	0.00012	0.00018	0.00013	0.00013	0.00340
WATER-1T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
WATER-2T	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
501-T	14.03399	14.37269	13.81591	14.30446	14.27742	13.93670	14.74962	13.67247	14.27742	39.19361	14.08517	14.03399	390.22199
501A-T	14.01074	14.43519	13.79243	14.28014	14.25724	13.71113	14.72074	13.64966	14.25724	33.91307	13.95267	14.01074	384.11981
502-T	11.79764	12.87095	11.65379	12.03372	11.06843	11.59139	12.91311	11.49373	11.06843	12.91311	11.84067	11.79764	299.57240
502AT	10.65332	11.33984	10.52945	10.80481	10.27998	10.58578	11.33984	10.37888	10.27998	11.91688	10.69217	10.65332	270.71265
503-T	42.75373	42.46990	42.35530	28.34620	42.48093	42.54396	42.01821	42.20206	42.48093	42.54396	42.75405	44.84501	1092.31984
503A-T	42.75373	41.56108	42.44105	28.28945	42.48093	42.94875	42.09876	42.20206	42.48093	42.94875	42.75405	44.84501	1089.45515

Table C.5 IAE Results of Biodiesel Process Deviation for the Process Stream

Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
103B-M	0.14799	0.04503	0.10836	0.07459	0.05206	0.05266	0.05276	0.01485	0.05276	0.05276	0.04192	0.05228	0.05245	0.05245
103C-M	0.14799	0.04503	0.10839	0.07456	0.05206	0.05263	0.05283	0.01485	0.05273	0.05276	0.04192	0.05228	0.05245	0.05245
105A-M	0.32829	0.83225	0.68811	0.47334	0.33037	0.33443	0.33476	0.09426	0.33487	0.33421	0.26621	0.33160	0.33268	0.33268
105B-M	0.32862	0.83225	0.68811	0.47334	0.33037	0.33410	0.33476	0.09426	0.34836	0.33454	0.26595	0.33160	0.33268	0.33268
105C-M	0.34243	0.87711	0.70150	0.48578	0.34421	0.34757	0.34858	0.09806	0.34836	0.34868	0.27694	0.34528	0.34641	0.34641
106-M	1.81086	1.77991	1.58459	5.79236	1.80943	1.80191	1.76629	0.51204	1.79901	1.78717	1.45214	1.77231	1.78007	1.77810
106A-M	1.81086	1.78056	1.58459	5.79236	1.81273	1.80191	1.76695	0.51074	1.80197	1.78717	1.45084	1.77231	1.78007	1.77810
201-T	5.55475	5.71933	5.48223	6.51629	5.61297	5.62437	5.37017	1.53689	5.54641	5.52244	4.23857	5.46808	5.45596	5.50380
201A-T	1.06806	1.03595	1.06264	1.39708	1.10137	1.06923	1.05847	0.28969	1.04663	1.07134	0.77034	1.06371	1.06015	1.05412
201B-T	1.06806	1.03353	1.06016	1.02630	1.10396	1.06668	1.07042	0.29320	1.04419	1.06882	0.77822	1.06371	1.06015	1.05412
201N-T	1.06604	1.03649	1.06109	1.39439	1.10484	1.06041	1.06167	0.29267	1.04722	1.06943	0.77866	1.06431	1.06076	1.05474
202B-M	2.85197	2.85403	2.77302	3.44342	2.86581	3.43101	2.89006	0.80833	2.86382	2.90658	0.59274	2.90814	2.84248	2.84346
202C-M	0.42323	0.42767	0.42066	0.58266	0.42500	0.39802	0.42731	0.11943	0.42491	0.42983	0.34277	0.42809	0.42822	0.42699
301-M	1.44211	1.42620	1.43174	1.29739	1.44411	1.43890	1.44174	2.32565	1.44507	1.44276	1.20685	1.43388	1.43856	1.43856
301A-M	1.44211	1.42620	1.43174	1.29858	1.44313	1.44021	1.44174	2.46537	1.44507	1.44507	1.20685	1.43388	1.43856	1.43856
302-M	0.00184	0.00182	0.00183	0.00166	0.00185	0.00184	0.00184	0.00052	0.00184	0.00184	0.00146	0.00182	0.00183	0.00183
302A-M	0.00184	0.00182	0.00183	0.00166	0.00185	0.00184	0.00184	0.00052	0.00184	0.00184	0.00146	0.00182	0.00183	0.00183
304-M	0.00220	0.00218	0.00219	0.00198	0.00221	0.00220	0.00221	0.00062	0.00220	0.00220	0.00175	0.00218	0.00219	0.00219
304A-P	0.00039	0.00040	0.00035	0.00032	0.00039	0.00038	0.00040	0.00011	0.00047	0.00040	0.00033	0.00040	0.00040	0.00040
401-T	14.06086	13.97562	13.95203	13.57783	14.07266	14.04932	13.88403	39.19361	13.89000	14.02656	13.04883	14.04573	14.05031	14.08517
401A-T	13.99207	13.90725	13.95777	13.59874	13.81773	14.02551	14.11664	33.91307	13.91211	14.04888	13.02940	14.02231	14.02703	13.95267

Table C.5 IAE Results of Biodiesel Process Deviation for the Process Stream (Cont)

Stream	VLV-305	QC	QC 2	QC T200	QC T400	H T200	H T400	E-R100	QC T500	H T500	VLV-501	VLV-502	SUM
103B-M	0.05245	0.07490	0.05283	0.03543	0.05375	0.05321	0.05903	0.08310	0.05375	0.05321	0.05245	0.05245	0.14799
103C-M	0.05247	0.07494	0.05288	0.03547	0.05383	0.05330	0.05914	0.08310	0.05383	0.05330	0.05245	0.05247	0.14799
105A-M	0.33268	0.18665	0.33509	0.21853	0.34094	0.33782	0.37406	0.52774	0.34094	0.33782	0.33268	0.33268	0.83225
105B-M	0.33268	0.18665	0.33443	0.21853	0.34094	0.33782	0.37406	0.52774	0.34094	0.33782	0.33268	0.33268	0.83225
105C-M	0.34641	0.18665	0.34826	0.22758	0.35501	0.35108	0.38951	0.54097	0.35501	0.35108	0.34641	0.34641	0.87711
106-M	1.78268	2.12552	1.80876	2.96316	1.85443	1.85983	2.03068	2.00742	1.85443	1.85983	1.77810	1.78268	5.79236
106A-M	1.78268	2.12552	1.80843	2.99362	1.85443	1.86016	2.03509	2.00742	1.85443	1.86016	1.77810	1.78268	5.79236
201-T	5.56924	5.88221	5.65933	3.26208	5.01631	7.48013	7.48013	5.39474	5.01631	6.01989	5.50380	5.56924	7.48013
201A-T	1.05316	0.86960	1.04870	1.23210	0.94236	1.54726	1.38558	1.02244	0.94236	1.38558	1.05412	1.05316	1.54726
201B-T	1.05316	0.85863	1.04615	1.54355	0.94236	1.23210	1.37344	1.02244	0.94236	1.37344	1.05412	1.05316	1.54355
201N-T	1.05376	0.87046	1.04678	0.52333	0.94291	1.23289	1.38965	1.02320	0.94291	1.38965	1.05474	1.05376	1.39439
202B-M	2.90588	4.17825	3.24687	1.42816	3.03300	2.65217	3.21539	2.86000	3.03300	2.65217	2.84346	2.90588	4.17825
202C-M	0.43031	0.60442	0.69335	0.29048	0.43812	0.41824	0.47771	0.41198	0.43812	0.41824	0.42699	0.43031	0.69335
301-M	1.43856	1.19879	1.44075	0.96219	1.45990	1.45372	1.51537	1.42000	1.45990	1.45372	1.43856	1.43856	2.32565
301A-M	1.43856	1.18199	1.44075	0.96219	1.50511	1.45372	1.51574	1.42000	1.50511	1.45372	1.43856	1.43856	2.46537
302-M	0.00183	0.00261	0.00184	0.00124	0.00188	0.00186	0.00206	0.00181	0.00188	0.00186	0.00183	0.00183	0.00261
302A-M	0.00183	0.00261	0.00184	0.00124	0.00188	0.00186	0.00206	0.00181	0.00188	0.00186	0.00183	0.00183	0.00261
304-M	0.00219	0.00313	0.00221	0.00148	0.00224	0.00222	0.00246	0.00216	0.00224	0.00222	0.00219	0.00219	0.00313
304A-P	0.00040	0.00047	0.00039	0.00020	0.00039	0.00041	0.00041	0.00037	0.00039	0.00041	0.00040	0.00040	0.00047
401-T	14.03399	14.37269	13.81591	14.30446	14.27742	13.93670	14.74962	13.67247	14.27742	14.74962	14.08517	14.03399	39.19361
401A-T	14.01074	14.43519	13.79243	14.28014	14.25724	13.71113	14.72074	13.64966	14.25724	14.72074	13.95267	14.01074	33.91307

Table C.5 IAE Results of Biodiesel Process Deviation for the Process Stream (Cont)

Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
402-M	42.67461	42.00215	42.28089	38.34861	42.54016	42.44363	42.94154	45.26517	42.82309	42.73391	44.84501	42.63117	42.75307	42.75405
402A-M	42.58671	42.08830	42.36816	38.34861	42.59799	42.53091	42.58575	42.27303	42.64523	42.73391	44.84501	42.63117	42.75307	42.75405
403-T	18.46008	18.34817	18.47336	17.88340	18.53514	18.55357	18.60454	7.49577	18.44024	18.44246	15.15925	18.53352	18.53667	18.58267
403A-T	18.74770	18.63405	18.69117	18.16219	18.68309	18.53452	18.80605	7.16315	18.59512	18.77793	15.36851	18.76141	18.76428	18.81083
H3PO4-1M	0.00008	0.00008	0.00008	0.00007	0.00008	0.00008	0.00008	0.00002	0.00008	0.00008	0.00007	0.00008	0.00008	0.00008
H3PO4-2M	0.00008	0.00008	0.00008	0.00007	0.00008	0.00008	0.00008	0.00002	0.00008	0.00008	0.00007	0.00008	0.00008	0.00008
R1-1P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R1-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R2-1P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R2-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
WATER-1M	0.00191	0.00189	0.00190	0.00172	0.00192	0.00191	0.00192	0.00054	0.00191	0.00191	0.00152	0.00190	0.00190	0.00190
WATER-2M	0.00191	0.00189	0.00190	0.00172	0.00192	0.00191	0.00192	0.00054	0.00191	0.00191	0.00152	0.00190	0.00190	0.00190
501-T	14.06086	13.97562	13.95203	13.57783	14.07266	14.04932	13.88403	14.74962	13.89000	14.02656	13.04883	14.04573	14.05031	14.08517
501A-T	13.99207	13.90725	13.95777	13.59874	13.81773	14.02551	14.11664	14.72074	13.91211	14.04888	13.02940	14.02231	14.02703	13.95267
502-T	11.84101	11.70388	11.81647	11.43910	11.80164	11.87857	11.89412	4.30795	11.72698	11.80870	10.85370	11.80566	11.81136	11.84067
502AT	10.64621	10.58167	10.68694	10.31363	10.65666	10.73946	10.60218	3.81549	10.51567	10.65228	10.02932	10.66103	10.66570	10.69217
503-M	42.67461	42.00215	42.28089	38.34861	42.54016	42.44363	42.94154	2.75373	42.82309	42.73391	44.84501	42.63117	42.75307	42.75405
503A-M	42.58671	42.08830	42.36816	38.34861	42.59799	42.53091	42.58575	42.27303	42.64523	42.73391	42.75373	42.63117	42.75307	42.75405



Table C.5 IAE Results of Biodiesel Process Deviation for the Process Stream (Cont)

Stream	VLV-305	QC	QC 2	QC T200	QC T400	H T200	H T400	E-R100	QC T500	H T500	VLV-501	VLV-502	SUM
402-M	42.75373	42.46990	42.35530	28.34620	42.48093	42.54396	42.01821	42.20206	42.48093	42.54396	42.75405	42.75373	45.26517
402A-M	42.75373	41.56108	42.44105	28.28945	42.48093	42.94875	42.09876	42.20206	42.48093	42.94875	42.75405	42.75373	44.84501
403-T	18.51515	17.12952	18.16734	18.88171	19.33181	18.35870	15.04152	18.03819	19.33181	15.04152	18.58267	18.51515	19.33181
403A-T	18.74248	17.41906	18.37637	19.02640	19.70534	18.64540	15.31049	18.25967	19.70534	15.31049	18.81083	18.74248	19.70534
H3PO4-1M	0.00008	0.00012	0.00008	0.00006	0.00008	0.00008	0.00009	0.00008	0.00008	0.00008	0.00008	0.00008	0.00012
H3PO4-2M	0.00008	0.00012	0.00008	0.00006	0.00008	0.00008	0.00009	0.00008	0.00008	0.00008	0.00008	0.00008	0.00012
R1-1P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R1-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R2-1P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
R2-2P	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
WATER-1M	0.00190	0.00272	0.00192	0.00128	0.00195	0.00193	0.00214	0.00188	0.00195	0.00193	0.00190	0.00190	0.00272
WATER-2M	0.00190	0.00272	0.00192	0.00128	0.00195	0.00193	0.00214	0.00188	0.00195	0.00193	0.00190	0.00190	0.00272
501-T	14.03399	14.37269	13.81591	14.30446	14.27742	13.93670	14.74962	13.67247	14.27742	39.19361	14.08517	14.03399	39.19361
501A-T	14.01074	14.43519	13.79243	14.28014	14.25724	13.71113	14.72074	13.64966	14.25724	33.91307	13.95267	14.01074	33.91307
502-T	11.79764	12.87095	11.65379	12.03372	11.06843	11.59139	12.91311	11.49373	11.06843	12.91311	11.84067	11.79764	12.91311
502AT	10.65332	11.33984	10.52945	10.80481	10.27998	10.58578	11.33984	10.37888	10.27998	11.91688	10.69217	10.65332	11.91688
503-M	42.75373	42.46990	42.35530	28.34620	42.48093	42.54396	42.01821	42.20206	42.48093	42.54396	42.75405	44.84501	44.84501
503A-M	42.75373	41.56108	42.44105	28.28945	42.48093	42.94875	42.09876	42.20206	42.48093	42.94875	42.75405	44.84501	44.84501

Table C.6 IAE Results of Temperature Deviation at Methanol Recovery, Biodiesel and Glycerol Column

T-200 (Methanol Recovery Column)														
Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
Tray 1	16.9591	17.3372	16.8171	20.0082	17.1026	16.881	16.8032	17.2389	17.0208	16.8032	17.4096	16.8249	16.8551	17.035
Tray 2	18.2997	18.7251	18.2334	21.5795	18.5033	18.2056	18.2047	18.5791	18.4088	18.2047	18.8177	18.2299	18.1087	18.2964
Tray 3	56.4346	56.3607	56.4024	50.9806	56.2622	56.269	56.7096	56.5398	56.6865	56.7096	57.0935	57.0956	57.1005	57.1161
Tray 4	56.1633	56.1115	56.1714	50.4808	56.0022	56.0074	56.4348	56.2751	56.4258	56.4348	56.8126	56.8116	56.8212	56.835
T-400 (Biodiesel Column)														
Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
Tray 1	44.125	44.125	44.125	44.125	44.125	44.125	44.337	147.884	43.957	44.013	48.424	44.426	44.415	44.415
Tray 2	40.131	40.131	40.131	40.131	40.131	40.133	40.311	154.458	39.988	40.035	43.632	40.386	40.377	40.377
Tray 3	37.225	37.225	37.225	37.225	37.225	37.225	37.383	145.8	37.099	37.14	40.211	37.45	37.442	37.442
Tray 4	35.509	35.509	35.508	35.509	35.509	35.509	35.651	180.506	35.395	35.433	38.117	35.711	35.701	35.703
T-500 (Glycerol Column)														
Stream	VLV-100	VLV-101	VLV-102	VLV-103	VLV-200	VLV-201	VLV-300	VLV-301	VLV-302	VLV-400	VLV-401	VLV-402	VLV-303	VLV-304
Tray 1	20.0109	20.6607	20.0066	23.3221	20.3863	19.9879	19.8577	20.5344	20.2564	19.8577	20.6543	19.8816	19.925	20.1323
Tray 2	34.098	34.099	34.098	34.099	34.099	34.099	34.233	34.233	33.991	34.027	36.414	34.289	34.28	34.282
Tray 3	35.509	35.509	35.508	35.509	35.509	35.509	35.651	35.651	35.395	35.433	38.117	35.711	35.701	35.703
Tray 4	37.225	37.225	37.225	37.225	37.225	37.225	37.383	37.383	37.099	37.14	40.211	37.45	37.442	37.442

**Table C.6** IAE Results of Temperature Deviation at Methanol Recovery, Biodiesel and Glycerol Column (Cont)

T-200 (Methanol Recovery Column)													
Stream	VLV-305	VLV-500	VLV-501	VLV-502	QC 2	QC	QC T200	QC T400	QC T500	H T200	H T400	H T500	SUM
Tray 1	17.0495	16.8551	17.035	17.0495	17.035	17.0495	14.6695	17.175	17.0495	18.037	17.2145	17.2145	444.5295
Tray 2	18.3967	18.1087	18.2964	18.3967	18.2964	18.3967	15.8371	18.5941	18.3967	19.5546	18.6908	18.6908	480.0523
Tray 3	57.0877	57.1005	57.1161	57.0877	57.1161	57.0877	59.6256	57.0985	57.0877	71.293	56.4169	56.4169	1488.2951
Tray 4	56.8078	56.8212	56.835	56.8078	56.835	56.8078	60.3009	56.8178	56.8078	71.2513	56.1517	56.1517	1482.1833
T-400 (Biodiesel Column)													
Stream	VLV-305	VLV-500	VLV-501	VLV-502	QC 2	QC	QC T200	QC T400	QC T500	H T200	H T400	H T500	SUM
Tray 1	44.415	44.415	44.415	44.415	44.415	44.415	43.901	46.142	43.901	44.088	39.959	39.959	1251.061
Tray 2	40.377	40.377	40.377	40.377	40.377	40.377	39.941	41.831	39.941	40.099	36.544	36.544	1157.514
Tray 3	37.442	37.442	37.442	37.442	37.442	37.442	37.057	38.723	37.057	37.196	34.035	34.035	1076.072
Tray 4	35.703	35.703	35.703	35.703	35.703	35.703	35.358	36.86	35.358	35.481	32.621	32.621	1067.787
T-500 (Glycerol Column)													
Stream	VLV-305	VLV-500	VLV-501	VLV-502	QC 2	QC	QC T200	QC T400	QC T500	H T200	H T400	H T500	SUM
Tray 1	20.2051	19.925	20.1323	20.2051	20.1323	20.2051	17.1865	20.3967	20.2051	21.3534	20.5496	20.5496	526.5197
Tray 2	34.282	34.282	34.282	34.282	34.282	34.282	33.954	35.406	33.954	34.072	30.16	30.16	883.739
Tray 3	35.703	35.703	35.703	35.703	35.703	35.703	35.358	36.86	35.358	35.481	32.621	32.621	922.932
Tray 4	37.442	37.442	37.442	37.442	37.442	37.442	37.057	37.057	38.723	37.196	34.035	34.035	967.655

Table C.7 IAE Results of Level Deviation for the Process Stream

Stream	VLV-100	VLV-101	VLV-103	VLV-200	VLV-201	VLV-302	VLV-401	VLV-402	VLV-303	VLV-305
L-R100	0.0121	0.1887	1.5664	0.023	0.0373	0.0372	0.0375	0.0372	0.0372	0.0372
L-R101	0.0038	0.0038	0.0038	0.0038	0.0038	0.3128	0.0038	0.0038	0.085	0.2135
L-C-T200	16.0442	15.0803	18.5305	15.9068	15.8469	15.4553	15.3814	15.4553	15.4553	15.4553
L-C-T400	0.0035	0.0051	0.0033	0.0045	0.0003	0.0029	18.5056	0.0016	0.0029	0.0029
L-C-T500	0.0246	0.0246	0.0246	0.0246	0.0246	0.0237	0.0246	0.0246	0.0299	0.072
L-R-T200	16.0442	15.0803	18.5305	15.9068	15.8469	15.4553	15.3814	15.4553	15.4553	15.4553
L-R-T400	0.0291	0.0291	0.0291	0.0291	0.0291	0.0291	0.2816	0.1892	0.0291	0.0291
L-R-T500	0.0173	0.0173	0.0173	0.0173	0.0173	0.018	0.0173	0.0173	0.0117	0.0743
Stream	VLV-501	VLV-502	QC T200	QC T400	QC T500	H T200	H T400	H T500	MAX	
L-R100	0.0372	0.0372	0.7606	0.0378	0.0372	0.0533	0.0375	0.0373	1.5664	
L-R101	0.0038	0.0038	0.0038	0.0038	0.0038	0.0038	0.0038	0.314	0.314	
L-C-T200	15.4553	15.4553	54.3565	15.4694	15.4553	16.2485	14.7874	15.5254	54.3565	
L-C-T400	0.0029	0.0029	0.0059	4.2504	0.0003	4.2507	0.7358	0.0003	18.5056	
L-C-T500	0.0442	0.0245	0.03	0.0246	0.0246	0.0246	0.0246	0.0635	0.072	
L-R-T200	15.4553	15.4553	54.3565	15.4694	15.4553	16.2485	14.7874	15.5254	54.3565	
L-R-T400	0.0291	0.0291	0.0301	0.9803	0.0291	0.9803	1.0272	0.0291	1.0272	
L-R-T500	0.0167	0.5479	0.024	0.0173	0.0173	0.0173	0.0173	0.091	0.5479	

## VITA

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