การออกแบบโครงสร้างการควบคุมของกระบวนการผลิตไบโอดีเซลที่มีการแยกเฟส และมีรีไซเคิลในระบบปฏิกรณ์

นายธนผลิน ยิ้มละมัย

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต
สาขาวิชาวิศวกรรมเคมี ภาควิชาวิศวกรรมเคมี
คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2555

CONTROL STRUCTURE DESIGN OF BIODIESEL PRODUCTION PROCESS WITH PHASE SPLIT AND RECYCLE IN THE REACTOR SYSTEM

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A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Engineering Program in Chemical Engineering
Department of Chemical Engineering
Faculty of Engineering
Chulalongkorn University
Academic Year 2012

Thesis Title	CONTROL STRUCTURE DESIGN OF BIODIESEL PRODUCTION PROCESS WITH PHASE SPLIT IN THE REACTOR SYSTEM
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ธนผลิน ยิ้มละมัย : การออกแบบโครงสร้างการควบคุมของกระบวนการผลิตไบโอดีเซลที่มีการแยกเฟสและมีรีไซเคิลในระบบปฏิกรณ์. (CONTROL STRUCTURE DESIGN OF BIODIESEL PRODUCTION PROCESS WITH PHASE SPLIT AND RECYCLE IN THE REACTOR SYSTEM) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: ผศ.ดร.มนตรี วงศ์ศรี, 143 หน้า.

งานวิจัยนี้จึงได้ทำการออกแบบกระบวนการผลิตไบโอดีเซลที่มีการแยกเฟสและมีรีไซเคิลในระบบปฏิกรณ์ ไตรกลีเซอไรด์จะทำปฏิกิริยากับเมทานอลเกิดเป็นเมทิลเอสเทอร์และกรีเซอรอล กระบวนการดังกล่าวจะถูกนำมาออปติไมซ์เพื่อลดการใช้พลังงาน โดยจะต้องไม่ละเมิดข้อจำกัดในการออปติไมซ์ทั้ง 30 ข้อ โดยผลจากการออปติไมซ์แสดงให้เห็นว่า กระบวนการดังกล่าวต้องการพลังงาน 371.43 กิโลวัตต์ เพื่อหลีกเลี่ยงการละเมิดข้อจำกัดในการดำเนินการในสภาวะพลวัต สภาวะที่ได้ทำการออปติไมซ์จะถูกแบ็คออฟ ซึ่งผลจากการแบ็คออฟทำให้กระบวนการผลิตไบโอดีเซลต้องการพลังงานเพิ่มเป็น 445.67 กิโลวัตต์ การมีรีไซเคิลสารและการบูรณาการการใช้พลังงานภายในกระบวนการส่งผลทำให้ระบบมีความซับซ้อนเชิงพลวัตที่เพิ่มมากขึ้น ดังนั้นวิธีการออกแบบระบบควบคุมแบบแพลนท์ไวด์ของวงศ์ศรีจึงถูกนำมาใช้เพื่อออกแบบโครงสร้างการควบคุมของกระบวนการผลิตไบโอดีเซล ผลจากการจำลองกระบวนการในสภาวะพลวัตแสดงให้เห็นว่า ทั้ง 6 โครงสร้างการควบคุมที่ได้ทำการออกแบบสามารถทำงานได้อย่างมีประสิทธิภาพ ประสิทธิภาพการควบคุมของโครงสร้างการควบคุมที่ออกแบบได้จะถูกคำนวณในรูปของค่าความคลาดเคลื่อนสัมบูรณ์ถ่วงน้ำหนัก นอกจากนี้ความต้องการสาธารณูปโภคของแต่ละโครงสร้างการควบคุมนั้นไม่แตกต่างกัน

ภาควิชา วิศวกรรมเคมี ลายมือชื่อนิสิต ....
สาขาวิชา วิศวกรรมเคมี ลายมือชื่อ อ.ที่ปรึกษาวิทยานิพนธ์หลัก ....
ปีการศึกษา 2555

##5370436121 : MAJOR CHEMICAL ENGINEERING
KEYWORDS : PLANTWIDE CONTROL STRUCTURE / FIXTURE PLANT / BIODIESEL / DISTURBANCE REJECTION / CONTROL STRUCTURE DESIGN

TANAPALIN YIMLAMAI : CONTROL STRUCTURE DESIGN OF BIODIESEL PRODUCTION PROCESS WITH PHASE SPLIT AND RECYCLE IN THE REACTOR SYSTEM. ADVISOR : ASST. PROF. MONTREE WONGSRI, D.Sc., 143 pp.

This work studies control structure design of a biodiesel production process with phase split and recycle in the reactor system. Triglyceride reacts with methanol to form methyl esters and glycerol. The process is optimized to minimize total equivalent energy subject to 30 optimization constraints. The optimization result shows that the energy required is about 371.43 kW. To avoid the control constraint violation while the process is operated dynamically, the process is backed-off making the energy required increases to 445.67 kW. A presence of the material recycles and energy integrations make the process has more complexity in dynamic behavior; therefore, the control system is designed in plantwide perspective using Wongsri's plantwide control design procedure. The simulation result shows that six viable control structures have good control performances. The control performances of all control structures are evaluated in term of the weighted integral absolute errors. In addition, utility requirements for all control structures are about the same.

Department: Chemical Engineering Student's Signature ....
Field of Study: Chemical Engineering Advisor's Signature ....
Academic Year: 2012

ACKNOWLEDGEMETS

First of all, I gratefully acknowledge my thesis advisor, Assistant Professor Dr. Montree Wongsri, for his support, invaluable suggestions, guidance, and useful discussions during this research.

I would also like to thank the committee members, Associate Professor Dr. Bunjerd Jongsomjit, as the chairman, Assistant Professor Dr. Soorathep Kheawhom and Dr. Veerayut Lersbamrungsuk, as the examiners for their constructive comments and valuable guidance.

Two-year scholarship from Chemical Engineering Department, Faculty of Engineering, Chulalongkorn University for my study in master's degree is gratefully acknowledged. I appreciate the technical support on the use of the commercial process simulation program and the encouragement from my friends and those who helped me during my research period.

Finally, I would like to extremely express my deepest gratitude to my parent for their support and encouragement to pursue a degree of Master of Engineering and their unconditional love.

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LIST OF ABBREVIATIONS

SYMBOL

A_i	Pre-exponential factor (L/mol min K)
DG	Diglyceride
GL	Glycerol
$e(t)$	error or difference between controlled variable and its setpoint
E_i	Activation energy (J/mol)
FAME	Fatty acid methyl ester
IAE_i	Integral absolute error of a controller in a controller group
K_C	Controller gain
k_i	Rate constant(L/mol min)
MeOH	Methanol
MG	Monoglyceride
n	Number of output streams from a splitter
n_i	number of controllers in a controller group
NaOH	Sodium hydroxide
$\bar{p}$	Nominal value of a controller output
$p(t)$	Controller output
PB	Proportional band
$PV_{max,i}$	maximum value of a controlled variable in a controller group
$PV_{min,i}$	minimum value of a controlled variable in a controller group
Q_C	Cooling duty (Watt)
Q_H	Heating duty (Watt)
R	Universal gas constant (8.314 J/mol)
T	Reaction temperature (K)
T_C	Heat source temperature for heat pump or heat sink temperature for heat engine (298.15 K)
T_H	Heat source temperature for heat engine or heat sink temperature for heat pump, operating temperature of any unit (K)
TG	Triglyceride

SYMBOL

W	Total equivalent work (Watt)
W_{comp}	Compressor work (Watt)
W_{DG}	Diglyceride mass fraction
W_{eq}	Equivalent work (Watt)
W_{GL}	Glycerin mass fraction
W_i	Weight factor
W_{MG}	Monoglyceride mass fraction
W_{pump}	Pump work (Watt)
W_{TG}	Triglyceride mass fraction
$WIAE_i$	Weighted integral absolute error of considered controller groups
$y_m(t)$	Measured value of a controlled variable
$y_{sp}(t)$	Setpoint of a controlled variable
η	Efficiency of Carnot cycle heat pump or heat engine
τ_d	Derivation time (minute)
τ_i	Integral time (minute)

CHAPTER I

INTRODUCTION

This chapter introduces to the importance of process control, plantwide control, and biodiesel, objectives, scopes, contributions, procedures, and framework of this research.

1.1 Introduction

Most of chemical processes consist of a lot of units connected into a process. To ensure that those units operate correctly, the process control system is required. The primary objective of process control is to maintain a process at the desired operation conditions, safely and efficiently, while satisfying environmental and product quality requirements. In the past, the control system is designed separately for each individual unit. However, the control system for each individual unit is not satisfied to achieve its goals or may have poor performance because of the presences of material recycles and energy integrations. The material recycles and energy integrations are used for economic improvement, these make the process have much more complexity in dynamic behavior because of increasing of interaction between upstream and downstream units. Therefore, in the present, the landscape of the process control has changed to plantwide perspective which the interaction between those units must be taken into account in the design of the control system for any process consisting of the material recycles and energy integrations.

In recent years, plantwide control structure design has been interested and studied widely. The most importance problems of the plantwide control structure design are what variables should be selected to be controlled variables, manipulated variables, and how to pair these variables together in order to make the designed control structure handle the process under disturbed conditions, gives the process has stability, and achieve the goals of control structure mentioned in previous paragraph. A number of design procedures have been presented; however, those procedures are classified in two catagories: heuristic and mathematical-based. The most popular procedure has been presented by Luyben and his co-worker in 1997, his procedure is based on heuristics. However, his procedure does not give the exact solution and the control system designer has to have deep insight in the process to design its control system. The other popular procedure which is based on mathematics has been presented by Skogestad and his co-worker in 2000. He points out to find the controlled variables that can minimize the deviation of cost functional from its optimum, which is economic loss, when disturbances arise. Although, his procedure gives the exact solution but it is hard to understand and apply, and required a long time to design the control structure; moreover, his procedure does not give any guidance to choose the controlled variables so lots of variables have to be considered. In recent years, Wongsri has proposed the plantwide control structure design procedure in 2009; the objective of his procedure is to design the control structure that makes most disturbed variables smallest deviate from its norm. The advantages of his procedure are it is based on mathematics, this means that the control system designer does not have much more process insight, and this procedure can apply easily with short time consuming. However, his procedure does not give any insights about the designed control structure; moreover, pairing between controlled and manipulated variables may not be appropriate because physical reliability does not be taken into account. In 2011, Wongsri has presented the new control structure design procedure; his new procedure is based on heuristics with simulation framework; therefore, the control structure designer does need to have process insights to design the physically reliable control structure.

Nowadays, the requirement in fuel has been increasing but the amount of fossil fuel is decreasing continuously, so we need to find alternative fuels much and more. In many years ago, biodiesel has been the most popular alternative fuel because it can be used instead of diesel fuel and pollutes less. Moreover, biodiesel can be produced from many crops that are grown in Thailand, this makes the cost of raw material in biodiesel production be cheap. In fact, vegetable oils can be used directly as biodiesel fuel, but the direct use in diesel engine is not appropriate because of its high viscosity. The most popular method to produce biodiesel is transesterification catalyzed by alkali, which is a reaction between triglyceride and alcohol, as shown in figure 1.1.

Normally, methanol is preferred over the others because it is not expensive and gives fast reaction rate in the reaction.

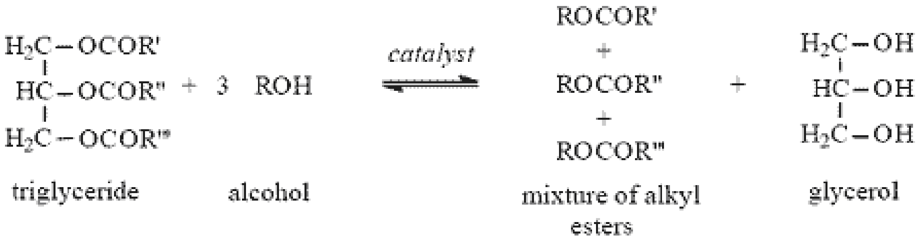


Figure 1.1: Transesterification Reaction

From mentioned reasons, in this research, the biodiesel production process from the pure triglyceride and the methanol catalyzed by sodium hydroxide, which is alkali, is designed and optimized; then, the new Wongsri's plantwide control structure design procedure is performed to design the control structure of the process. The obtained plantwide control structure has to make the process operate without constraints for control violation and reject considered setpoint change and disturbance; besides, the control performances of designed plantwide control structures are shown in term of weighted integral absolute errors.

1.2 Research Objectives

The objectives of this research are:

(1) To design a new biodiesel production process.

(2) To optimize the process in term of total equivalent energy.

(3) To design control structures of the process using the new Wongsri's plantwide control structure design procedure.

(4) To evaluate performances of the new designed control structures in term of the weighted integral absolute error.

1.3 Scopes of Research

The scopes of this research are shown as follows:

(1) The new biodiesel production process is designed.

(2) Steady-state operation of the process is simulated using commercial process simulator.

(3) The process is optimized to minimize the total equivalent energy.

(4) New control structures of the biodiesel production process are designed using new Wongsri’s plantwide control structure design procedure.

(5) Dynamic simulations of the process controlled by new designed control structures are performed using commercial process simulator.

(6) Performances of new control structures are evaluated in term of the weighted integral absolute error.

1.4 Contributions of Research

The contributions of this research are:

(1) The new biodiesel production process has been simulated in both steady-state and dynamics.

(2) The new plantwide control structures of the process are designed using new Wongsri’s plantwide control structure design procedure.

(3) The control performances in term of the weighted integral absolute error of the new designed control structures are evaluated.

1.5 Research Procedure

Procedure plans of this research are:

(1) Study the plantwide control theory, the biodiesel production process, and concerned information.

(2) Design the new biodiesel production process.

(3) Optimize the process to minimize the total equivalent energy.

(4) Simulate steady-state operation of the process.

(5) Study new Wongsri’s plantwide control structure design procedure.

(6) Design new plantwide control structures of the biodiesel production process followed new Wongsri’s plantwide control structure design procedure.

(7) Simulate the dynamic operation of the biodiesel production process controlled by the new control structures.

(8) Evaluate the performance of the new control structures based on the process throughput and disturbances.

(9) Analyze and discuss the results.

(10) Conclude the research.

1.6 Research Framework

This research has six chapters; the details of each chapter are shown below:

Chapter I: introduces to the importance of process control, plantwide control, and biodiesel, objectives, scopes, contributions, procedures, and framework of this research.

Chapter II: reviews the previous works concerned with the plantwide control history, and the development of plantwide control design procedure.

Chapter III: mentions to the basic of control, plantwide control theory, procedures to design the plantwide control structure introduced by Luyben and Wongsri.

Chapter IV: describes transesterification, which is reaction to produce the biodiesel fuel, saponification side-reaction, and the biodiesel specification.

Chapter V: shows designed biodiesel production process and its description, steady-state simulation of the process in both optimal and backed-off conditions, the new control structures of the process designed using new Wongsri's plantwide control structure design procedure, dynamic responses of the process controlled by designed control structures when the considered setpoint change and the disturbance arise, and evaluated dynamic performance index of the new control structures.

Chapter IV: presents the conclusion of this research and recommendations for future work.

This is following by:

Appendix A: Basic control modes.

Appendix B: Process and equipment data.

Appendix C: Controller type and tuning parameters.

Appendix D: Dynamic simulation response.

CHAPTER II

LITERATURE REVIEW

Many years ago, plantwide control has been being widely addressed by many researchers. A number of plantwide control design procedures are presented, both heuristic based and mathematical based, several procedures has applied on many processes and can give workable plantwide control structure. However, designing control structure for complete any chemical processes are challenging to achieve the most effective and robust control structure although the ultimate solution may not be so intuitively obvious. This chapter is presented the reviews of the previous works concerned with the development of plantwide control and design procedure.

2.1 Plantwide Control

In the past, control system design had followed the *unit operation approach* (Stephanopoulos, 1983). The control system was designed for each unit individually, and then these were connected together into an entire plant. Therefore any conflicts due to the combination of individual parts had to be reconciled. An important assumption of this unit based approach was that the combination could give the effective plant's control system. However, this approach is suitable for any processes without material recycles and/or energy integrations, because they lead to a path of disturbance propagation and an integrating effect which profoundly changes the dynamic behavior of an entire plant, or any processes contained many surge tanks to buffer disturbances, and to minimize interaction between units.

In 1970s, there was an economic force to improve capital productivity. Many surge tanks were eliminated to reduce both capital investment and its operating costs; then, material recycling and energy integration were further utilized to save raw material and utility costs. Therefore, the unit operation approach had not been befitted to use in the control system design for most existing chemical processes. Denn and Lavie (1982) stated that recycles and energy integrations need special attention while designing plantwide control structure for any processes to prevent the disturbance propagations and the integrating effects.

Buckley (1964) proposed a control design procedure which plantwide control problems were divided into two parts. The first sub-problem is material balance control; the control structure is designed to cope with low-frequency disturbances which effects on the vessel inventory levels. The second is product quality control affected high-frequency disturbances. However, his work does not obviously discuss the energy management, does not address the recycle stream issues, and does not deal with component balance.

Foss (1973) defined the control structure design as *structural decisions* including five tasks: selection of controlled variables, selection of manipulated variables, selection of measurements, selection of control configuration, and selection of controller type. Stephanopoulos (1983) summarized the creativity concerned with control strategy synthesis, but he gives a little advice about plantwide control system design. Price and Georgakis (1993) used a tired framework, following Buckley (1964), to obtain a *self-consistent* control structure that minimizes the disturbance propagation. Narraway and Perkins (1993) formulated an optimization problem to study the effect of disturbances on economics for various control structures. Wolf and Skogestad (1994) presented many advices to find the control scheme for integrated plants.

In 1994, Luyben performed a mathematical analysis on the two binary first-order systems with reaction $A \rightarrow B$ controlled by two different control structure. The results show that snowball effect can be prevented by fixing of the recycle flow rate. Luyben (1996) showed that the number of controlled variables equal to the number of *degree of freedom for control* which can be calculated by counting the number of manipulated variables in the entire process. Luyben (1997) had presented a symmetric procedure to design the plantwide control structure based upon heuristic that account for unique features of plantwide control. He gave an important philosophy that "*it is always best to utilize the simplest control system that will achieve the desired objectives*". He showed that his procedure gave workable plantwide control structures for three industrial processes involving reaction and separation sections: vinyl acetate monomer process, Eastman process, and HDA process. However, his procedure does not clear; the control system designers must have experiences and process insights for their effective usage. Moreover, heuristics cannot be relied because best solution can sometimes be unconventional. Later, Konda *et al.* (2005) had improved the heuristic procedure of Luyben (1998) by addressing associated limitations, for example, more specific and generic guidelines were included to facilitate the decision for throughput and inventory control. However, they thought that heuristics cannot always be relied, so the improved heuristic procedure was integrated with simulation. The integrated framework which synergizes the powers of both heuristics and simulation was applied on HDA process; the viable control structure was achieved.

Larsson and Skogestad (2000) introduced the new plantwide control design procedure mainly following mathematically oriented approach. His procedure divided into two parts: top-down analysis and bottom-up design. The main objective of top-down analysis which requires steady-state model, constraints, and operational objective is to select the primary controlled variables based on idea of self-optimizing control. For bottom-up design, its objectives are to obtain control configuration which has stability for regulatory control layer, to obtain the local control loops for local disturbance rejection, and to obtain configuration of supervisory control layer with real-time optimization. Skogestad (2004) published a new paper which extends his plantwide control design procedure in Larsson and Skogestad (2000). However, both versions of his procedure had not considered about precedence of each controlled variable which most disturbed variable should be controlled before each other in order to minimize effects of disturbances on it and disturbance propagation, and had not explicitly discussed how to select the appropriate manipulated variable for each controlled variable. Vasudevan *et al.* (2009) compared three different control structures of styrene monomer process. The control structures were designed by using Luyben's heuristic procedure (1997), integrated framework (Konda *et al.*, 2005), and self-optimizing control procedure (Skogestad, 2004). The results indicated that even if all procedures gave workable control structures, control structures designed by using the integrated framework and the self-optimizing control procedure had more robustness than control structure designed by heuristic procedure.

Mahajanam, Zheng, and Douglas (2001) presented a method to select the controlled variables without consideration of optimization problems. All of possible options that affect the steady state economics were ranked, and then poor choices were eliminated. Castro and Doyle (2004) had presented two plantwide control structures for pulp mill process, one was the decentralized control, and other was the combination of decentralized control and model predictive control. Simulation results showed that both control structures could be able to control the process, in addition response for the most outputs of model predictive control structure had a higher overshoot than response of decentralized control structure although the model predictive control structure had a smaller integral absolute error or IAE.

Suntisrikomol (2008) had proposed a method known as *Fixture Point Theorem* to select set of controlled variables; this task is always one of the most important in the plantwide control structure design. The fixture point theorem states that variable that is most disturbed from the disturbance effects should be controlled first by appropriated manipulated variable, because the most disturbed variable is the major cause of disturbance propagation. To find the most disturbed controlled variable, selectivity of each variables to disturbances are evaluated, the variable that is most sensitive is also most disturbed. Selecting and pairing the controlled variables with manipulated variables are performed by considering the maximum gain (or scaled gain). Suntisrikomol applied the fixture point theorem on hydro-alkylation process to design its best control structure, five control structures were designed and its performances are compared with control structure designed by Luyben (1998) by considering its integral absolute error. The results showed that one of the new designed control structures had faster responses and more performance than the control structure designed by Luyben. However, Suntisrikomol did not propose the plantwide control design procedure using the fixture point theorem; it was proposed by Detjareansri (2009). Detjareansri presented the new plantwide design procedure of Wongsri using fixture point theorem based on heuristics and mathematical analysis. He applied this design procedure on alkylation process, the results in term of performance index were compared with work of Luyben (2002) by considering two types disturbances: flow and thermal disturbances. The results showed that Wongsri’s design procedure can give the good performance control structure through the highly energy integrated process; it can cope with the effects of disturbances and also maintain the product quality close to their setpoints.

CHAPTER III

PLANTWIDE CONTROL THEORY

Chemical process consists of a lot of unit operations connected into system, the primary objective of process control is to maintain those units operating at desired condition. However, the presence of material recycle and energy integration causes the increase of interactions between those units, this problem makes the control system of individual unit cannot achieve its goal. Therefore the landscape of the process control has changed to plantwide perspective which the interaction between those units must be considered before disigning the control system. In the chapter, the basic control theory, plantwide control theory, plantwide control design procedures, and advantages of heat integration are presented.

3.1 Integrated Process

Three basic features of integrated chemical process must be taken into account to consider the entire plant's control system:

1. The effect of material recycles.
2. The effect of energy integrations.
3. The need to account for chemical component inventories.

These features make the plantwide control problem have more complexity. However, there are many fundamental reasons why each of these exists in all real processes.

3.1.1 Material Recycle

Material is recycled for six important reasons as follows:

(1) *Increase Conversion:* If chemical processes involve reversible reactions, conversion of reactants to products is limited by thermodynamic equilibrium constraints. Therefore reactor effluent must contain both reactants and products. Separation and recycle of reactants are necessary to make process be economically viable.

(2) *Improve Economics:* Generally, it is simply cheaper to use a reactor with incomplete conversion and recycle reactants than use of one reactor or multiple

reactors in series reaches the desired conversion. A reactor followed by a stripping column with recycle is cheaper than one large reactor or reactors in series.

(3) *Improve Yields:* In reaction systems such as A → B → C, where B is the desired product. Therefore the concentration of B should be kept as low as possible in the reactor and a large recycle of A is required for minimizing the per-pass conversion of A to avoid the formation of the undesirable product C.

(4) *Provide Thermal Sink:* In adiabatic reactors with highly exothermic reaction, it is often necessary to feed excess a reactant or one product to the reactor to prevent large amount of temperature increase. High temperature can create several desired events: it can lead to thermal runaways, catalyst deactivation, undesirable side reactions, mechanical failure of equipment, etc. So the heat of reaction is absorbed by the sensible heat required to raise the temperature of the excess material in the stream flowing through the reactor.

(5) *Prevent side reactions:* A large excess of a reactant is often used so that the concentration of the other is kept low. If this limiting reactant is not kept in low concentration, it could react to produce large amount of undesirable products. Therefore the excess reactant must be separated from the product and recycled back to the reactor.

(6) *Control properties:* In many polymerization reactors, the conversion of monomer is limited to achieve the desired polymer properties such as average molecular weight, molecular weight distribution, degree of branching, particle size, viscosity of polymer solution, etc.

3.1.2 Energy Integration

The reason for the use of energy integration is to improve the thermodynamic efficiency. This leads to reduce utility cost. For energy-intensive processes, the savings can be quite significant. However, the energy integration creates nontrivial control issues, this highlights why we cannot combine the control systems of individual unit operations in such processes.

3.1.3 Chemical Component Inventories

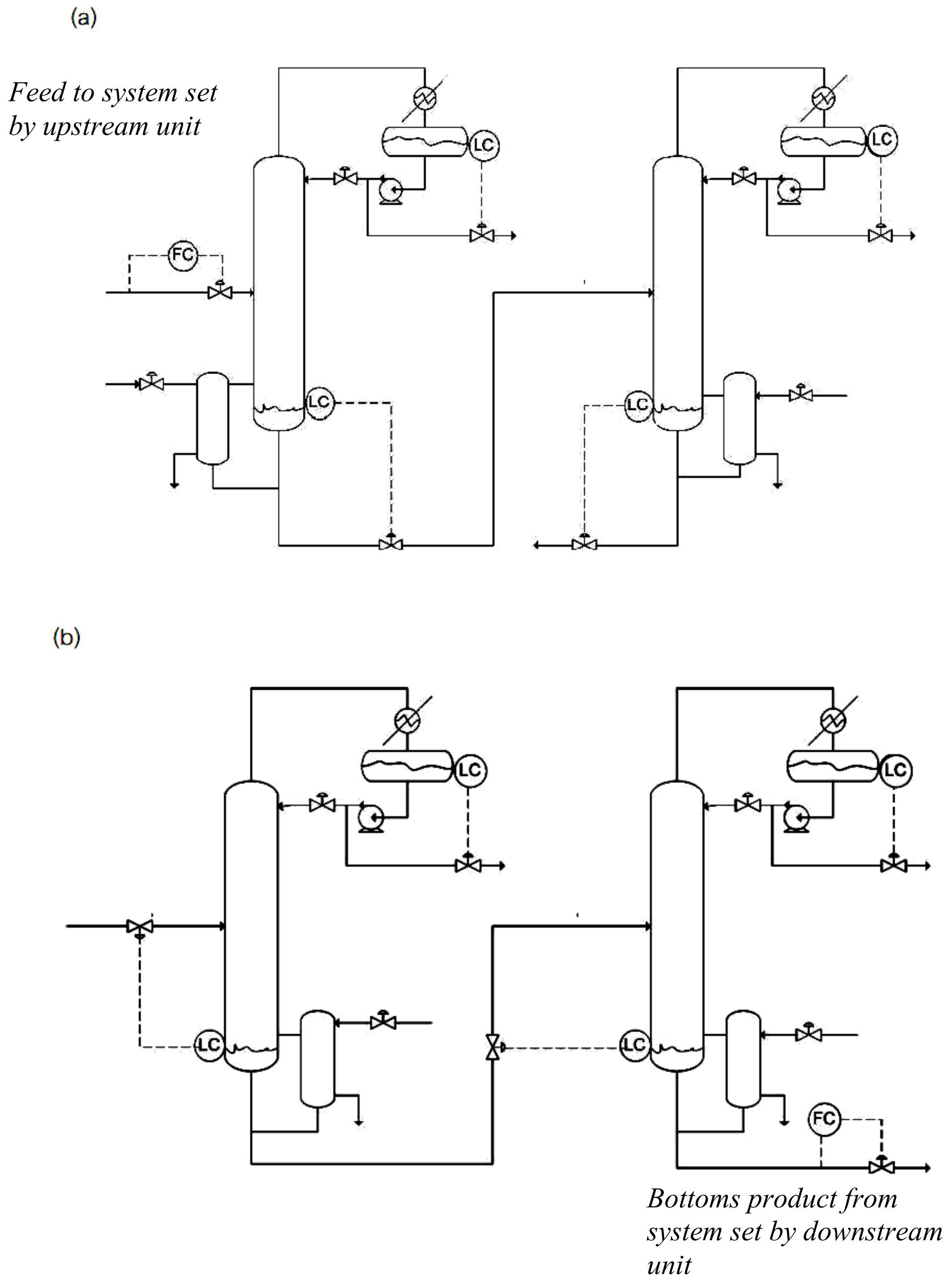
Chemical species in a process can be characterized into three types: reactants, products, and inerts. A material balance for all components must be satisfied, but problems usually encounter when we consider reactants and account for their inventories within the entire process. We want to minimize reactants leaving from the process as impurity or purge because of their value, so we must ensure that every mole of reactant fed to the process is consumed by the reactions, this represents a yield penalty. From the viewpoint of individual units, chemical component balancing is not a problem because exit streams form the units automatically adjust their flows and compositions. However, when we connect units together with recycle streams, the entire system behaves almost like a pure integrator in terms of the reactants. If additional reactant is fed into the system without changing reactor conditions to consume the reactant, this component will build up gradually within the plant because it has no place to leave the system.

3.2 Plantwide Control Problem

3.2.1 Units in Series

If units in entire process are arranged in a series configuration and there is no material and energy recycles, the plantwide control problem is greatly simplified and the issues discussed in the previous section are not had to pay a lot of attention. We can simply configure the control scheme on each individual unit to handle load disturbances.

If production rate is set at the front end of the process, the load disturbances to each unit will come from its upstream neighbor only. If the production rate depends on demand and is set at the terminus, changes in throughput will propagate back through the process. So any individual unit will see load disturbances coming from both its upstream and its downstream neighbors. Figure 3.1 shows these two possible configurations; production rate is set at the front end and terminus of the process.



Feed to system set by upstream unit

Bottoms product from system set by downstream unit

Figure 3.1: Units in series (a) Level control in direction of flow

(b) Level control in direction opposite flow

3.2.2 Effects of Recycle

Most process contained recycle streams, so the plantwide control problem becomes much more complex. The presence of recycles makes both steady-state and dynamic behaviors of the process alter. There are two basic effects of recycle:

(1) Recycle has an impact on the overall time constant in dynamic operations, any change in recycle streams can take a long time to return to steady-state. Moreover, the recycle effects are so slow, it is hard to recognize when there is any problem in the process, Intermediate vessel inventories may overfill or go empty, this makes the last column in separation section see the ramp load disturbance and product quality controller be difficult to maintain the product quality at the setpoint.

(2) Recycle leads to the "snowball" effect which is a steady-state phenomenon, but it implicates to dynamics for disturbance propagation and for inventory control. Cause of the snowball effect is the high sensitivity of recycle flow rates to small disturbances. These disturbances can lead to even larger dynamic changes in flow, which propagate around the recycle loop. It is important to select the good plantwide control structure to avoid this effect.

3.2.3 Reaction/Separation Section Interaction

Different control structures produce different behavior in handling disturbances. To understand the interaction between reaction and separation section, the two control structures of the same process are considered as shown in figures 3.2 and 3.3.

In the first shown in figure 3.2, only the separation section must absorb almost all of changes. To increase production rate of product which is in the bottom stream of distillation column by 20 percent, the overall reaction rate must increase by 20 percent. Because of both reactor temperature and reactor holdup fixed constant, only the composition in the reactor must increase 20 percent. This is a significant change in the composition of the feed stream to the distillation column. The load on the separation section changes significantly producing large variations in recycle flow rates.

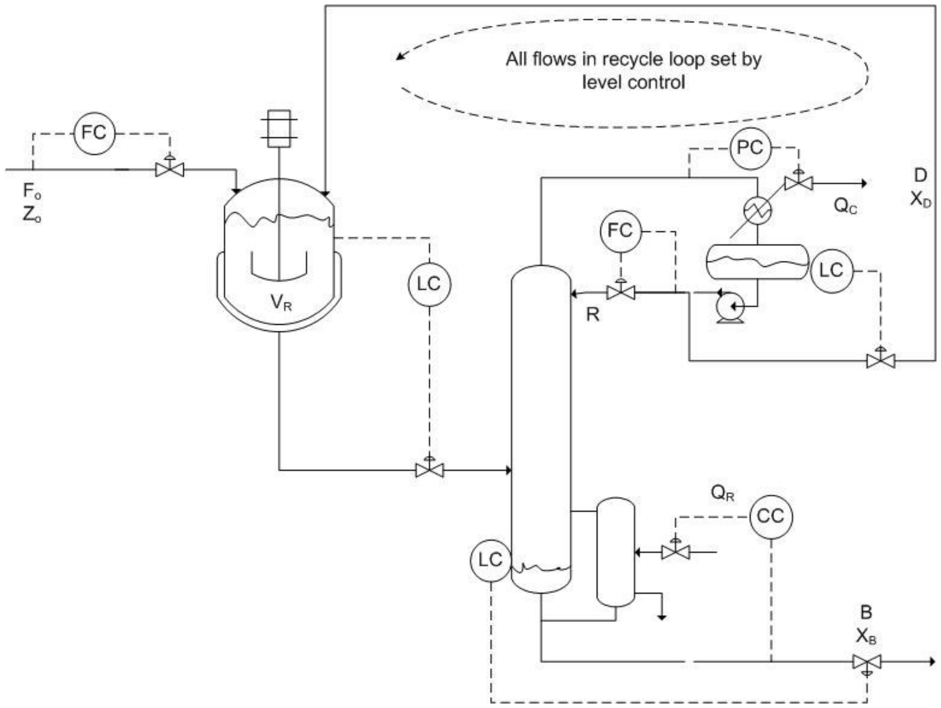


Figure 3.2: Conventional Control Structure with Fixed Reactor Holdup

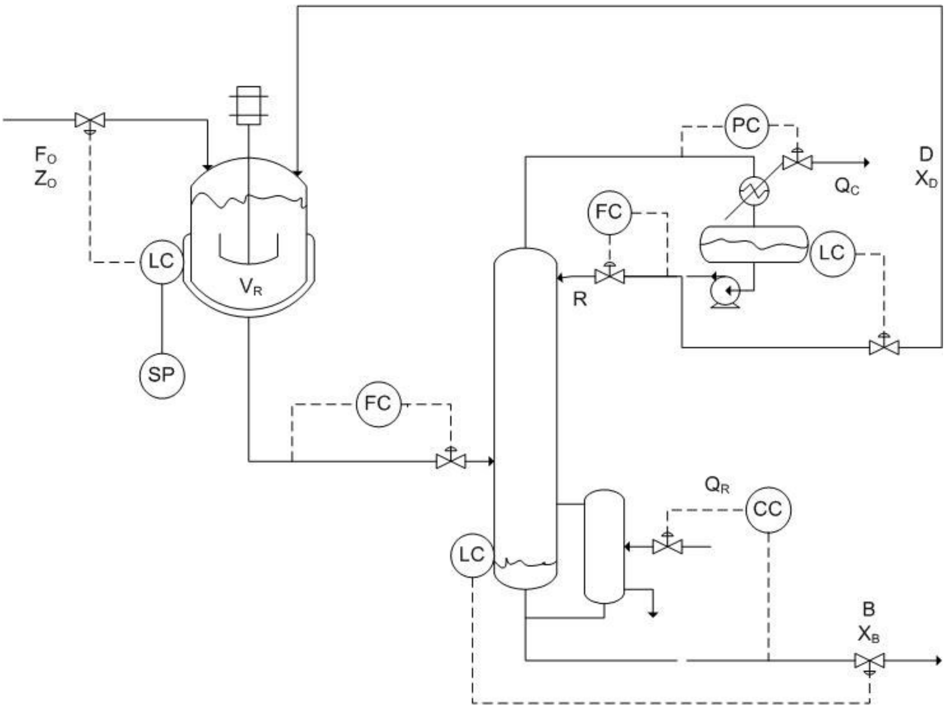


Figure 3.3: Control Structure with Variable Reactor Holdup

In the second structure shown in figure 3.3, both reactor holdup and reactor composition can change, so the separation section sees a smaller load disturbance. The effects of the disturbance can be distributed between the reaction and separation sections that results a smaller change in recycle flow.

3.3 Basic Concepts of Plantwide Control

3.3.1 Buckley basics

Page Buckley (1964) had suggested the idea to separate the plantwide control problem into two parts: material balance and product quality control. In the first step, the inventory control system, both levels and pressures, is established by setting up *hydraulic* control structure using the gas and liquid flow rates as manipulated variables, but controller tuning and inventory sizing do not yet performed in this step.

The second step is to establish the product quality control by choosing the appropriate manipulated variables. Moreover, he tries to make the time constants of the product quality control loops as small as possible to achieve the tight control structure and good control performance. However, the limitation of the achievable performance is imposed by the stability constraints.

In the final step, the inventory control loops are reconsidered to estimate the time constants of liquid level loops which are usually used proportional-only controllers for flow smoothing. The time constants of these loops are usually a factor of 10 larger than the time constants of the product quality control loops. The cause of separation in time constants is to tune material balance and product quality control loops independently.

3.3.2 Douglas doctrines

Jim Douglas (1988) has pointed out that the raw material costs and the product values are usually much greater than the capital and utilities costs; this idea leads to the *two Douglas doctrines*:

1. Minimize losses of reactants and products.
2. Maximize flow rates through gas recycle system.

The first doctrine implies that tight control of the composition of the stream leaving form the process is needed to minimize the losses of reactants and products. And the second doctrine is that the gas recycle flow rates should be maximized. However, the economic improvement should usually be compared with the additional energy costs due to the use of the recycle gas compressor.

3.3.3 Downs drill

Jim Dows (1992) had pointed out the importance of component balances around the process and checking that the control structure can handles these component balances effectively. The concepts of overall component balances go back to our first course in chemical engineering, where they learned how to apply mass and energy balances to system, microscopic or macroscopic. They did these balances for individual unit operations, for section of a plant, and for entire processes. He must ensure that all components (reactants, products, and inerts) have a way to leave or be consumed within the process.

3.3.4 Luyben laws

Luyben (1998) had presented three laws which are useful in the control systems design for many processes:

(1) The flow rates of all recycles should be controlled to prevent the snowball effect.

(2) Fresh reactant feed flow rates should not be controlled excepted to complete one-pass conversion of a reactant. If consecutive reactions such as $A + B \rightarrow M + C$ and $M + B \rightarrow D + C$ exist, the flow rates of a reactant fed to the process can be controlled to make the reaction shift to the desired product. If reactant A is excess, the reactions favor to produce more M and less D. In the other hand, excess of B makes the reactions result in the production of more D and less M.

(3) If the distillate from a distillation column is the final product, the stream fed to the column should be liquid. In the other hand, if the final product comes out the bottom of a distillation column, the feed stream to the column should be vapor (Cantrell et al., 1995). Reason of these suggestions is that changes in flow

rate or composition of liquid feed stream have less a dynamic effect on distillate composition than they do on bottoms composition, and vice versa. Therefore, the dynamic implications of feed thermal conditions in the design of a distillation column should be considered to achieve the tight product quality control loops.

3.3.5 Richardson rule

Richardson (1995) had suggested that a stream that has largest flow rate should be selected as a manipulated variable to control the liquid level in a vessel, because it can be adjusted in widely range to achieve the desired control objective.

3.3.6 Shinskey schemes

Greg Shinskey (1988) had proposed a number of *advanced* control structures such as ratio control, cascade control, override control, and valve-position control, etc. These control structures can be used to improve the dynamic performance effectively and implemented easily in basic control instrumentation.

3.4 Luyben's Plantwide Control Design Procedure

Luyben et al., (1997) introduced his plantwide control design procedure in 9 steps as follows:

***Step 1:* Establish control objectives**

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

This may be the most important step because the control structure which will be designed depends on the control objectives. These objectives include yields, production rate, product purities, safety and environment constraints. We must keep in mind that the "best" control structure depends upon the design and control criteria established.

***Step 2:* Determine control degree of freedom**

"Count the number of control valves available."

We must remember that this is not the number of design degrees of freedom but it is the number of degrees of freedom for control. This number refers to the number of variables that can be controlled to setpoint. Sometime, the control valves location can improve the dynamic performance, but often we cannot choose its location.

Most of these valves are used to achieve the control objective and basic regulatory control: (1) set production rate, (2) control product qualities, (3) maintain inventories, and (4) avoid the constraints. The other valves are used to improve either steady-state economics or dynamic controllability.

Step 3: **Establish energy management system**

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

Energy management is used in two functions: (1) the energy management is to provide a control system used to remove exothermic heats of reaction because it may lead to thermal runaways. Moreover, these heats must not be recycled and must be dissipated to utilities. (2) If there are heat integrations between process streams, the energy management is used to provide a control system used to prevent thermal disturbances propagation. Notice that the process-to-process heat exchangers and heat-integrated unit operations must be analyzed to determine that control degrees of freedom are sufficient.

Use of process-to-process heat exchangers to transfer energy between two process streams can create significant interaction. Reactor feed/effluent heat exchanger can draw down positive feedback and instability of the process. The effects of partial condensation or partial vaporization in process-to-process heat exchangers can lead to amplification of disturbances. For example, if a stream is vaporized partially in a process-to-process heat exchanger before fed to a distillation column, small changes in its composition can lead to large changes in component fractions and flow rates in vapor and liquid phases of the preheated stream, the resulting of these variations can produce severe upsets.

To reduce the energy requirement of processes, heat integration between a distillation column with other columns or with reactors is often used. Although steady-state economics favor these designs, but they can make the processes have more complex dynamic behavior and poor performance due to the recycling of disturbances. To prevent these, trim heaters/coolers or heat exchanger with bypass line must be added.

Step 4: **Set production rate**

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

Only way to increase production rate is the changes in reactor condition for increasing the overall reaction rate. This can be accomplished by raising reactor temperature, increasing reactant concentrations, increasing reactor holdup for liquid-phase reactions, or increasing reactor pressure for gas-phase reactions. The variable which is selected must be dominant for the reactor.

Generally, the dominant variable is kept at its setpoint which is adjustable to achieve the desired production rate, in addition to satisfying other economic control objectives. We should choose the variable that provide smooth and stable production rate transitions and can reject disturbances be the dominant variable. Moreover, the good dominant variable should have the least effect on the separation section and has a rapid and direct effect on the reaction rate without hitting an operational constraint.

Step 5: **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."

Because of economic and operational reasons, the product qualities must be strictly controlled. We should select the manipulated variables such that the dynamic relationships between the controlled and manipulated variables feature small time constants and deadtimes for small closed-loop time constants, and large steady-state gains to prevent the saturation of the manipulated variables.

Step 6: **Fix a flow in every recycle loop and control inventories.**

"Fix a flow every recycle loop and then select the best manipulated variables to control inventories."

The large changes in recycle flows can occur if all flows in recycle loop are controlled by levels. To prevent this problem, a flow controller should be used in all liquid recycle loops. From a steady-state viewpoint, this control strategy helps the separation section does not operate at significantly different loads. From a dynamic viewpoint, this control strategy can be used to prevent dynamic excursions of the recycles that can occur when all flows in recycle loops are controlled by levels. In a recycle loop, all controllers see load disturbances coming from the upstream unit. When load disturbances arises in a recycle loop, inventory control systems in this loop attempt to control the vessel liquid levels by changing the flow rates of streams that flow to its downstream neighbor causing the load disturbance propagation around the recycle loop.

For gas recycle loops, the circulation rates should be maximized to achieve the maximum yields.

After the flows in each recycle loop are fixed, we then determine what variable should be used to control each inventory level. An inventory variable should be controlled by variable that has the largest effect on it within that unit.

Step 7: **Check component balances.**

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

We must identify the specific mechanism or control loop to guarantee that there will be no uncontrollable buildup of any chemical component within the process. As mentioned above, we do not want reactant components to leave in the product streams because of the yield loss and the desired product purity specifications. Hence we are limited to the use of two methods to ensure that the overall component balances for all chemical species are satisfied at steady-state: consuming the reactants by reaction or adjusting their fresh feed flow. Product and inert components all must have an exit path from the system. In many systems inerts are removed by purging off a small fraction of the recycle stream. The purge rate is adjusted to control the inert composition in the recycle stream so that an economic balance is maintained between capital and operating costs.

Step 8: **Control individual unit operations**

"Establish the control loops necessary to operate each of the individual unit operations."

Step 9: **Optimize economics or improve dynamic controllability**

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

After satisfying all regulatory requirements, we usually have additional degrees of freedom involving control valves that have not been used and setpoints of some controllers that can be adjusted. These can be utilized either to optimize steady-state economics or to improve dynamic response.

3.5 Wongsri's Plantwide Control Structure Design Procedure (2009)

In recent years, Wongsri had proposed the new procedure to design the plantwide control structure based on mathematical analysis. In his procedure, the precedence of the importance of the controlled variables is established by using the fixture point theorem. The most important controlled variable is paired with the appropriate manipulated variable. The major disturbances are directed or managed explicitly to achieve the minimal interaction between the control loops by using the extended (thermal) disturbance propagation method (Wongsri, 1990) to cover the material disturbances.

3.5.1 Steps of Wongsri's design procedure (2009)

The steps of the plantwide control design procedure of Wongsri (2009) are shown below:

Step 1: Define control objectives.

Step 2: Select controlled variables using fixture point theorem.

Step 3: Select manipulated variables and measurements, the number of manipulated variables can be found using degree of freedom (DOF) analysis.

Step 4: Minimize energy requirement via heat exchanger networks.

Step 5: Pair controlled variables and manipulated variables together using heuristics.

Step 6: Check the component balances.

Step 7: Select controller types such as proportional, proportional-integral, or proportional-integral-derivative controllers.

Step 8: Validate control structure via dynamic simulation.

3.5.2 Fixture point theorem analysis

Wongsri (2008) proposed fixture point theorem in order to rank the sensitivity of variables, and then variables that is high sensitivity are selected as controlled variables. The steps of this theorem are shown following:

(1) Process is simulated in dynamics without control system until it reaches to steady-state responses.

(2) Step change in a manipulated variable is performed to identify sensitivity of each controlled variable. In this step, all available manipulated variables must be changed to determine whether controlled variable has been affected most by each manipulated variable by considering magnitude of integral absolute error, IAE.

(3) Prioritize the controlled variables via its integral absolute error; variable that has more sensitivity should be controlled before each other.

3.6 New Wongsri's Plantwide Control Structure Design Procedure

In 2012, Wongsri has presented the new control structure design procedure consisting 8 steps; his new procedure based on the heuristic approach with simulation framework which makes use of chemical engineering and process knowledge and heuristics. The design of plantwide control structure should be viewed as a whole; taking into consideration of the whole plant. The design step called plantwide level design is the decision of how to regulate the whole plant albeit a single entity as smoothly as possible; then, the designs of control loops that locally function are handled at the unit level design.

In the plantwide level control, a fixture plant is established for creating a material-balanced process plant. The establishing a fixture plant can be done by: keeping the raw materials entered to the reactor fixed, adjusting the flow of exit material streams (products, by-products, and inert) according to their accumulations, and locating the quantifiers for the rest of the components to design the control loops to regulate their inventories. For operating a plant as smoothly as possible, the disturbance effects can be diminished by designing the control loops to reject or direct the disturbances taking a whole plant into consideration. The design of the control loops at each step follows the material balance of the whole plant and the fast and efficient rejection of disturbances though out the process. In the other words, the selection of control loops is not done in unitwise manner instead they are designed by looking from perspective of a whole plant. The heat disturbance management principle is used to design the control loops for rejection the effects of any thermal disturbances that de and do not directly relate to the product quality to the environment; while the material pathway analysis is used to design control loops to direct the material disturbances.

In the unit level control, the control loops are designed at the units that are not level-, pressure-, or flow-controlled, e.g., pumps and compressors.

The design procedure is carried out in eight steps as follow:

Step 1: Gather relevant plant information and control objective including constraints for control. Before initiating work on the control structure design, it is necessary to obtain all information relevant to process control. The process objective and control constraints determine the lower/upper bounds of the controlled variables as well as setpoints on the controlled variables.

Step 2: List manipulated variables (control degree of freedom, CDOF).

The manipulated variables can be obtained using the guideline given in table 3.1 and the guideline for pairing the controlled variables with the manipulated variables is presented in table 3.2.

Table 3.1: Degree of freedom for simple units

Unit	DOF / unit
Independent stream	1
Heater, cooler, pump, and compressor	1
Process-to-process heat exchanger with a by-pass stream	1
Adiabatic reactor*	1
Non-adiabatic reactor*	2
Adiabatic flash separator	2
Simple distillation column	5
Simple distillation column with partial condenser	6

* Add one degree of freedom for pressure control

Table 3.2: Guideline pairing of manipulated and controlled variables

NO.	Guideline
1.	A control and manipulated variables must have strong causal relationship (high gain).
2.	The manipulated variables should not be far from the control variables (zero or minimal dead time).
3.	The time constant of the quality loops should be short and the time constant of the inventory loops should be last.
4.	The manipulated variables should not be saturated for the whole range of the disturbances.
5.	The manipulation of the manipulated variables should not have or have less effects on others variables (low gains with the remainder of the variables).

Step 3: Establish the fixture plant.

The principal idea of establishing a fixture plant is to have an entire plant fluid-filled and a material-balanced. This idea is similar to creating *hydraulic control structure* proposed by Buckley.

(3.1) Keep the materials entered combined with reentered fixed. However, if the composition of the recycle stream differs from the fresh feed significantly, each stream should be flow-controlled separately. In this settlement, the flow of the recycle cannot be used to regulate the inventory of its upstream unit, e.g., the level of the reflux drum or the column pressure.

(3.2) Adjust the flow of exit material streams (products, by-products, and inerts) according to their accumulations.

(3.3) Locate quantifiers, i.e., the indicators of the representative accumulation, for the rest of the components and design the control loops to regulate their inventories in the plant. The quantifier can be volume (mass), pressure, or flow rate.

Step 4: Handling the disturbances.

(4.1) Any thermal disturbances are divided into 2 categories. Heat Disturbance Category 1 (HDC1) is the heat disturbances that do not directly affect product qualities, such as heat disturbance in a process stream toward to a heater, a cooler, or a process-to-process heat exchanger. Heat Disturbance Category 2 (HDC2) is the heat disturbances affected the product qualities, such as heat disturbance in a process stream toward to a reactor or a separator.

(4.1.1) Direct the thermal disturbances that are not directly related to the product quality to the environment via the next and nearest exit points, usually heaters or coolers, to keep the thermal conditions of process stream fixed. The thermal condition of process stream is changed along the process plant, usually by heater, cooler, or process to process heat exchanger.

(4.1.2) Manage the thermal disturbances that related to the product quality in order to maintain the quality within its specification.

(4.2) Material disturbances.

The configuration of the control loops depend on the desired material pathways. The pathways can be obtained by analyzing the results of the material disturbance tests. The test is suggested to be done on the changing of composition, total flow, and component flow. The material disturbances can be generated at reactors and separators, besides coming with feeds and recycle streams. So, if the feeds combined with the recycle streams are fixed, the only places that change the material (total or component) flow rates are the reactors and the separators. At reactor, its inlet temperature is adjusted in order to keep a component flow rate or a composition in the reactor product stream. The decision of whether how to choose to control the component flow or the composition or not to control depends on the profit maximization, the smooth operation, or other control policies.

Since the distillation columns, usually the one-point control is common. The temperature control at upper or lower of the distillation column feed depends on the material disturbance rejection policy.

Step 5: Design the control loops for the remaining control variables and/or adding enhanced controls, i.e., cascade, feed forward controls.

Step 6: Energy management via heat exchanger networks.

If the opportunities for the process modification and/or the heat integration exist, e.g., adding process-to-process heat exchangers with the by-pass streams, it should be performed and then listed additional controlled and manipulated variables.

Step 7: Optimize economics and/or improve the control performance.

The economic optimization may be the change of the operating, or the process modification (e.g., the feed location, the sequence of separation), etc. Examples of control performance improvement are the control scheme modification at the reactor (e.g., temperature/composition sensor location for the plug flow reactor), the distillation column (e.g., using reflux-to-feed ratio control), etc. However, if the optimization is performed, the control structure design should be backtracked to the previous step as dictated.

Step 8: Validate the designed control structures by rigorous dynamic simulation, and compare the designed structure to each other.

The measures in the comparison may be costs, raw material and energy consumptions, control performances, etc. of the total plant or some selected loops.

3.7 Heat Exchanger Networks

Energy conservation is important in process design. In industrial experience, the calculation of the minimum heating and cooling requirements reveal significant energy savings. There are numerous case studies that indicate 30% to 50% energy savings compared to traditional practice. Therefore, energy integration design procedure is a very beneficial tool and is an important in determining the cost of preliminary design.

CHAPTER IV

REACTIONS AND BIODIESEL SPECIFICAIONS

Due to the increasing of demands for alternative fuels, biodiesel has been most popular in many years ago because it can be used instead of diesel fuel and pollutes less. Moreover, biodiesel can be produced from many crops that are grown in Thailand, this makes the cost of raw material in biodiesel production be cheap. This chapter is presented how to produce the biodiesel, related reactions in biodiesel production, and biodiesel specifications.

4.1 Reaction and Kinetics Descriptions

Although, the biodiesel can produced from many methods, but the most popular method is known as transesterification or alcoholysis because it is the easiest and profitable way in the biodiesel production.

$$\mathrm{TG + MeOH \xleftrightarrow{NaOH} DG + FAME}$$

$$\mathrm{DG + MeOH \xleftrightarrow{NaOH} MG + FAME}$$

$$\mathrm{MG + MeOH \xleftrightarrow{NaOH} GL + FAME}$$

Figure 4.1: Transesterification Reaction

Transesterification is the reaction that uses triglyceride and alcohol as reactants while the catalyst is generally alkali. The reaction consists of three series reversible reactions, triglyceride (TG) reacts with alcohol stepwise to form diglyceride (DG), monoglyceride (MG), and finally glycerol (GL); one mole of biodiesel is produced in each step. The triglyceride can be found in many oils such as soybean and sunflower oils, in this study, triolein which is a kind of the triglyceride and can be found a lot in the virgin soybean oil is used as a reactant because the soybean is cultivated much in Thailand, this makes the cost of raw material in biodiesel production be cheap.

Moreover, methanol (MeOH) is usually preferred to use as the alcohol reactant over the others because of its low cost and given fast reaction. If methanol is used as the alcohol reactant, the transesterification can be also called methanolysis. To simplify, soybean oil is regarded as pure triolein because the number of carbon, hydrogen atoms and its molecular weight are closed. Consequently, diolein can be used to represent the diglyceride, monoolein as the monoglyceride, and methyl-oleate as the fatty acid methyl ester (FAME) or biodiesel.

In fact, the catalyst in the transesterification can either be alkali, acid, or enzyme. However, the alkali-catalysts are usually favored for the virgin and refined waste oils because it makes the transesterification be faster than acid and enzyme catalysts. Moreover, enzyme catalysis is required very longer reaction time so it has only been carried out on laboratory scale.

Alkali-catalyzed transesterification is sensitive to the purity in reactants, both moisture and free fatty acid. Existence of triglyceride with water under basic condition causes saponification or hydrolysis of triglyceride while free fatty acid can deactivate the alkali-catalyst in water called neutralization as shown in the figure 4.2 and 4.3, both side reactions of transesterification make the salt of fatty acid known as soap formed. Soap causes the formation of emulsion that creates the difficulty to separate and purify the biodiesel and causes of slow glycerol separation via gravitation. Freedman, Pryde, and Mounts (1984) suggest that the free fatty acid content in the triglyceride should be less than 0.5 %wt, both reactants should be moisture free, and sodium hydroxide catalyst should be 1 %wt of triglyceride weight. Therefore, before the use of crude or waste cooking oil which contain a lot of water and free fatty acid to produce as reactant oil, it is necessary to pre-treat suggested by Zhang, Dube, McLean, and Kates (2003) via esterification with methanol catalyzed by sulfuric acid for removing the free fatty acid and then washing by glycerol to remove water. However, it is preferable to use acid catalysis if low-grade or waste cooking oils are used, because the fatty acid would deactivate the alkali-catalyst.

$$\mathrm{TG + 3NaOH \xrightarrow{H_2O} GL + 3Soap}$$

Figure 4.2: Triglyceride Saponification Reaction

$$\mathrm{FFA + NaOH \xrightarrow{Heat} Alcohol + 3Soap}$$

Figure 4.3: Neutralization Reaction of Free Fatty Acid

Kinetic expression for the transesterification of the triolein and the methanol catalyzed by homogenous sodium hydroxide was given by Noureddini and Zhu (1997) as shown following modified Arrhenius equation:

$$k_i = A_i \cdot T \cdot e^{-E_i/RT} \tag{4.1}$$

This model is based on the reactant concentrations and assumed that agitation rate is grate enough to mix the both reactants completely, so reaction rate becomes rate-controlled step rather than rate of diffusion between two liquid phases. The kinetic parameters for this expression are shown in table 4.1.

Table 4.1: Kinetic parameters of Transesterification of Soybean oil

Rate constant at 50 °C (l/mol min)		**Energy of Activation (cal/mol)**	
k_1	0.050	E_1	11000
k_{-1}	0.110	E_{-1}	8000
k_2	0.215	E_2	18000
k_{-2}	1.228	E_{-2}	13000
k_3	0.242	E_3	8000
k_{-3}	0.007	E_{-3}	11000

Zhang *et al.* (2003) suggest the transesterification of the pure triolein and the methanol should be performed at 60 °C and 400 kPa because of given fast reaction rate.

4.2 Biodiesel Specification

The specifications of the pure biodiesel B100 are defined by American Society for Testing and Materials (ASTM) in ASTM D-6751; the important specifications are shown in table 4.2.

Table 4.2: Biodiesel Specification

Property	**Units**	**ASTM limits**
Methanol Content	% mass	0.2 max
Water and Sediment	% volume	0.05 max
Free Glycerin	% mass	0.02 max
Total Glycerin	% mass	0.24 max

Total glycerin can be expressed as following equation:

$$Total\ Glycerin = 0.1044W_{TG} + 0.1488W_{DG} + 0.2591W_{MG} + W_{GL} \qquad (4.2)$$

where W_{TG}, W_{DG}, W_{MG}, and W_{GL} are mass fraction of triglyceride, diglyceride, monoglyceride, and glycerol, respectively.

CHAPTER V

BIODIESEL PRODUCTION PROCESS AND CONTROL STRUCTURES

Although, a lot of the biodiesel production process has been designed but, in recent years, there exists a biodiesel production process designed by Shen, Cheng, Ward, and Yu (2011) that is very interesting because the phase split and recycle in the reactor system has been introduced to decrease the alcohol raw material; however, the process has grate defects; for their given data, materials in the process are not balanced and a part of methanol in reactor should vaporize at their operating temperature of the reactors; however, the process is tried simulating but there are lots of water in the reactors caused saponification. Therefore, in this research, new biodiesel production process is designed to avoid the saponification and then optimized to minimize the energy requirement; in addition, to ensure all of units in the new process operate correctly in dynamics, control structures of the process has been designed. This chapter presents designed biodiesel production process and its optimal and backed-off conditions, the new control structures of the process designed using new Wongsri's plantwide control structure design procedure and its dynamic responses of the process.

5.1 Designed Biodiesel Production Process

New biodiesel production process that is designed to produce the biodiesel by transesterification described in chapter 4 from the 10 kmol/h of pure triolein and the methanol catalyzed by the sodium hydroxide, which should be 0.1 %wt of the triolein suggested by Freedman *et al.*, comprises five sections: reaction, methanol recovery, heat recovery, product separation, and glycerol purification sections as shown in figure 5.1.

In reaction section, 10 kmol/h fresh triolein and 1 %wt catalyst that is anhydrous sodium hydroxide are only pumped into the first reactor while the fresh methanol streams are mixed with the recycled methanol streams and then feed to both reactors. The liquid outlet stream at 60 °C and 400 kPa, which is operating condition of first and second reactors, from the first reactor are separated into two liquid phases at the first decanter; the heavy liquid, which mostly consists of methanol and glycerol, is spilt into two streams which one is backed to the first reactor and another is introduced to the distillation column at the methanol recovery section; the light liquid consisting of the unconverted reactants, intermediates, product and by-product flows to the second reactor. The second reactor and the second decanter are operated like the first reactor and the first decanter but the light stream from the second decanter is introduced into the flash tank at methanol recovery section.

The methanol recovery section comprises a vacuum distillation column without a condenser and a flash tank. The pressure at the top state of the distillation column is operated at 10 kPa like the flash tank with the 2 kPa pressure drop on each stage. There is no the condenser because methanol is the very light component and has the high relative volatility compared to each other. The vapor streams from the flash tank and the distillation column, which are almost the pure methanol, are mixed and sent to heat recovery section; the liquids form both units are also mixed and pumped into product separation section.

One compressor and three heat exchangers compose the heat recovery section. The almost pure methanol vapor at 10 kPa is drawn from the methanol recovery section to the compressor for increasing its pressure to be 400 kPa that is the operating pressure of the reactors. Afterward, the stream is split into three streams which are sent to each heat exchanger to condense and recover the energy; then, all split streams are mixed together and recycled to the reaction section. The heating streams that receive the energy from the three methanol streams are the fresh water fed to washing column, washed light stream from washing column, and washed water from washing column.

In the product separation section, the light liquid from the methanol recovery section is washed by heated water from the heat recovery section. The washed water consisting of lots of glycerol is introduced to the glycerol purification section and the

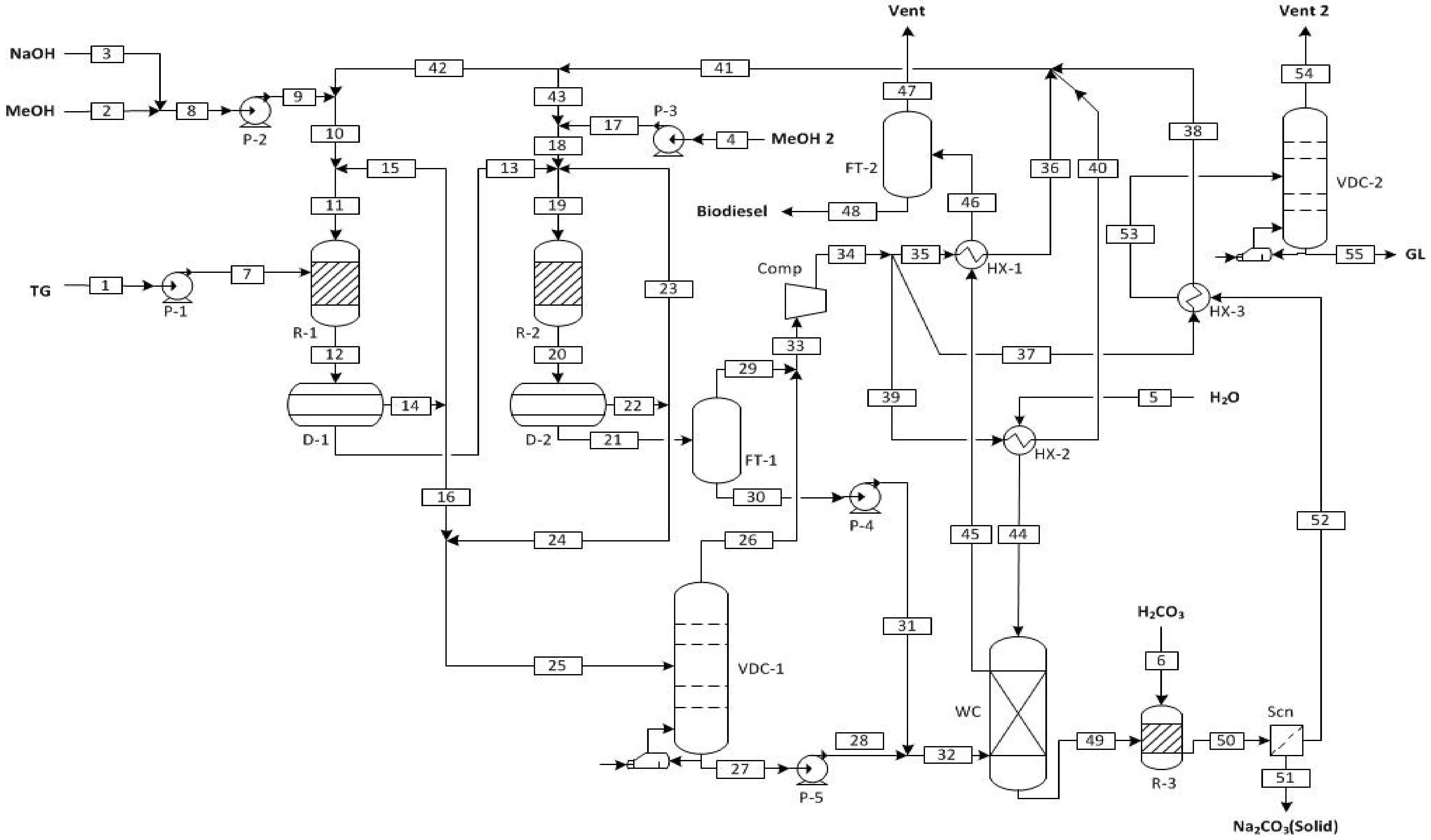


Figure 5.1: Designed Biodiesel Production Process

washed light stream is flashed to 0.1 atm at the flash tank after be heated at the heat recovery section in order to obtain biodiesel adhering to ASTM specifications. At the flash tank, the vapor is discharged to environment and the liquid phase is the biodiesel product.

For the glycerol purification section, the high grade glycerol, which consists of 92% glycerol, is preferred. Hence, after be neutralized by carbonic acid, the washed water from the product separation section is refined at the vacuum distillation column, the top stage pressure is at 10 kPa with the 2 kPa pressure drop on each stage, to obtain a high quality by-product.

5.2 Optimization and Back-off

To minimize the energy requirement of the designed biodiesel production process, the process is optimized using commercial process simulator; however, to avoid using utility prices, required energies of each unit are converted in term of equivalent thermodynamic works or equivalent energies. For units that require hot utility, heating duties (Q_H) are converted using Carnot cycle heat pump equation as shown in equation (5.1); for units that require cold utility, the cooling duties (Q_C) are converted using Carnot cycle heat engine equation as shown in equation (5.2).

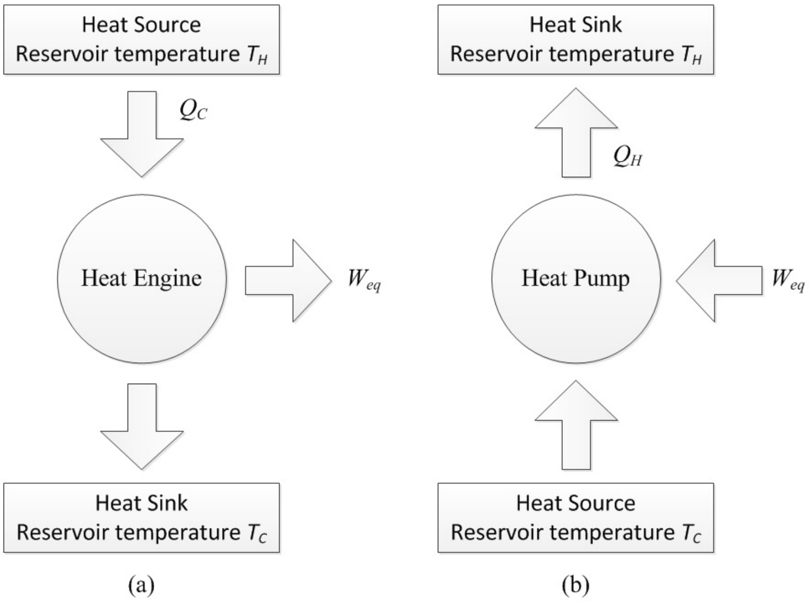


Figure 5.2: Simple Diagram of (a) Heat engine (b) Heat pump

However, the Carnot cycle heat engine and heat pump are ideal; therefore, efficiency (η) of the heat pumps and the heat engines are assumed to be 75% of Carnot efficiency. The hot temperature (T_H) of both heat pump and heat engine are utility temperature while cold temperature (T_C) is assumed to be ambient temperature, 25 °C.

$$W_{eq} = \frac{|Q_H|}{\eta}\left[1-\frac{T_C}{T_H}\right] \tag{5.1}$$

$$W_{eq} = \eta\,|Q_C|\left[1-\frac{T_C}{T_H}\right] \tag{5.2}$$

Total equivalent energy is the energy that the process requires in operation and can be calculated from equation (5.3).

$$W = \sum W_{eq} + \sum W_{pump} + W_{compressor} \tag{5.3}$$

The objective for the optimization problem of the biodiesel production process is to minimize the energy requirement in term of total equivalent energy as mentioned above. This objective can be written into the functional as shown in equation (5.4).

$$\min\ W \tag{5.4}$$

subject to 30 constraints shown is table 5.1, which are desired reaction yield and methanol recovery at first distillation column, operating condition of first and second reactor and flash point of biodiesel and degradation temperature of the glycerol suggested by Zhang *et al.* (2003), the product specifications from ASTM D6751, and desired conditions in the process design step.

Optimized conditions for the process, which is calculated for the commercial process simulation program, are shown in table 5.1 for constraints and table 5.2 for manipulated variables; the optimization result shows that 26 constraints shown in table 5.1 are active and the required total equivalent energy is 371.43 kW.

Table 5.1: Constraints for optimization

	Constraint	**Unit**	**Active**
P-1 Discharge Pressure	400	kPa	×
P-2 Discharge Pressure	400	kPa	×
P-3 Discharge Pressure	400	kPa	×
P-4 Discharge Pressure	111.325	kPa	×
P-5 Discharge Pressure	111.325	kPa	×
Compressor Pressure	400	kPa	×
FT-1 Pressure	10	kPa	×
FT-2 Pressure	0.1	atm	×
Top stage pressure of VDC-1	10	kPa	×
Top stage pressure of VDC-2	10	kPa	×
Reactor R-1 Temperature	60	°C	×
Reactor R-2 Temperature	60	°C	×
Temperature of stream 28*	≤ 150	°C	
Temperature of stream 44***	50	°C	×
Temperature of stream 46	95	°C	×
Temperature of stream 55*	≤ 150	°C	
HX-1 Temperature approach	≥ 10	°C	×
HX-2 Temperature approach	≥ 10	°C	×
HX-3 Temperature approach	≥ 10	°C	×
NaOH-TG ratio fed to R-1	1	% wt	×
NaOH flow rate in stream 50	0	mol/s	×
Vapor fraction in stream 11**	0		×
Vapor fraction in stream 19**	0		×
Reaction yield	≥ 98.5	% mol	×
MeOH recovery at VDC-1	≥ 99	% mol	×
GL Purity in stream 55	≥ 92	% mol	×
Methanol content in product	≤ 0.2	% wt	
Water content in product	≤ 0.05	% vol	×
Free glycerin in product	≤ 0.02	% wt	
Total glycerin in product	≤ 0.24	% wt	×

* *Flash point of biodiesel and Degradation temperature of glycerol are 150 °C.*

** *Transesterification is only in liquid phase.*

*** *Karaosmanogly, Cigizoglu, and Tuter suggest a way to purify the biodiesel is the washing by 50 °C hot water.*

However, the some operating conditions, which are active constraints, have to be backed-off from the optimized conditions to avoid the violation of the constraints for control while the process is operated dynamically and to make the operation of the process possible. Therefore, the temperature approach of all heat exchangers, the water content and the total glycerin in product, and the glycerol mole fraction in the by-product stream (stream 55) are backed-off to be 20 °C, 0.02 %vol, 0.2 %wt, and

0.95, respectively. Moreover, temperature of stream fed to VT-2 (stream 46) should be increased to be 121.14 °C, so the new heater is added to the process for this aim as shown in figure 5.3. However, the back-off leads to change in some manipulated variables as shown in table 5.2. Besides, the back-off introduces the optimization loss which means the required total equivalent energy increases to be 445.67 kW.

Table 5.2: Manipulated variables at optimized and backed-off conditions

Manipulated variables	Optimized	Backed-off	Unit
P-1 Work	1252.83	1252.83	watt
P-2 Work	75.37	75.38	watt
P-3 Work	97.68	100.75	watt
P-4 Work	455.07	454.78	watt
P-5 Work	33.72	33.92	watt
Compressor Work	162083.62	172256.39	watt
MeOH flow rate	14.67	14.67	kmol/h
MeOH 2 flow rate	18.82	19.41	kmol/h
NaOH flow rate	0.19	0.19	kmol/h
H_2CO_3 flow rate	0.10	0.10	kmol/h
H_2O flow rate	39.07	39.07	kmol/h
Stream 26 flow rate	17.82	18.97	kmol/h
Stream 29 flow rate	15.81	16.90	kmol/h
Stream 47 flow rate	1.55	1.67	kmol/h
Stream 54 flow rate	40.54	41.40	kmol/h
R-1 Duty	- 479797.69	- 491521.22	watt
R-2 Duty	- 24994.66	- 19715.17	watt
VDC-1 Reboiler duty	214109.48	223796.74	watt
VDC-2 Reboiler duty	300818.09	309322.71	watt
Split ratio to stream 15	0.9395	0.9395	
Split ratio to stream 23	0.9496	0.9496	
Split ratio to stream 35	0.5002	0.4943	
Split ratio to stream 39	0.0397	0.0381	
Split ratio to stream 42	0.7341	0.7341	
VDC-1 tray number	2	2	
VDC-2 tray number	2	2	
HX-1 Area	3.08	2.61	m^2
HX-2 Area	0.22	0.18	m^2
HX-3 Area	2.88	2.50	m^2
Heater Duty	-	145294.05	watt

5.3 Designed Control Structures

In this research, the plantwide control structures of the backed-off biodiesel production process in previous section are designed using new Wongsri's procedure,

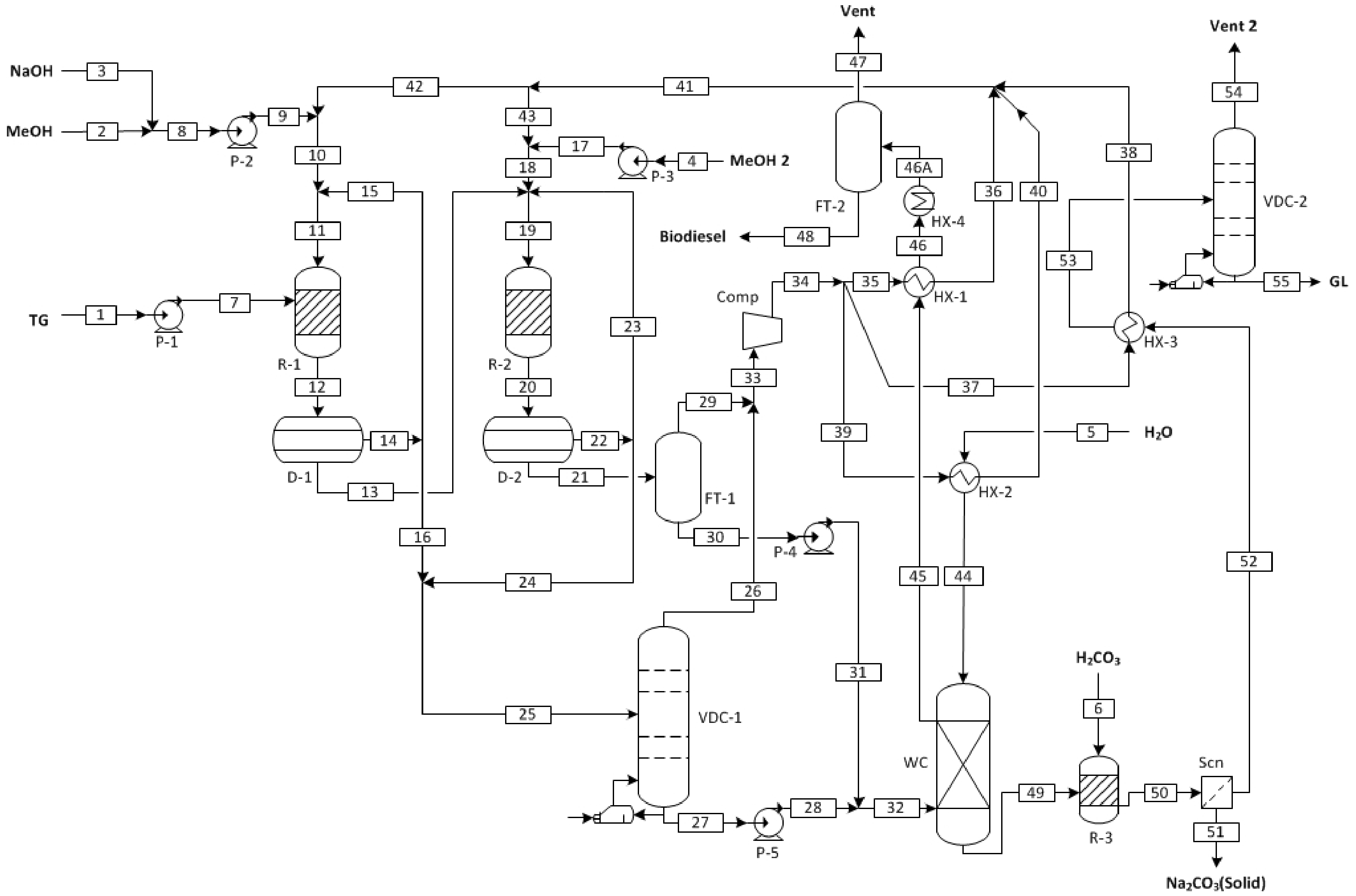


Figure 5.3: Designed Biodiesel Production Process after Back-off

which consists of 8 steps based on both heuristic and mathematic approach, described in chapter 3. Then, to find a best from many control structures designed using the procedure, control performance in term of integral absolute error of each control structure are evaluated and compared with others.

Step 1: *Gather plant information and control objective*

In this study, the designed biodiesel production process must achieve greater than or equal to 98.5% total yield of transesterification based on triglyceride limiting reactant and the designed control structure must make the process not violate constraints for control shown in table 5.3 while the process is operated.

Table 5.3: Constraints for control

	Constraint	**Unit**	**Description**
Triglyceride feed rate	9 to 11	kmol/h	Desired feed range to change the production rate
Temperature of stream 28	≤ 150	°C	Flash point of biodiesel and glycerol degradation temperature
Temperature of stream 55	≤ 150	°C	Glycerol degradation temperature
Vapor fraction in stream 11	0		Liquid phase reaction
Vapor fraction in stream 19	0		Liquid phase reaction
Glycerol purity in stream 55	≥ 92	% mol	High-grade purity
Methanol content in product	≤ 0.2	% wt	Biodiesel specification
Water content in product	≤ 0.05	% vol	Biodiesel specification
Free glycerin in product	≤ 0.02	% wt	Biodiesel specification
Total glycerin in product	≤ 0.24	% wt	Biodiesel specification

Step 2: *List all available manipulated variables (degrees of freedom for control).*

From degree of freedom analysis shown in table 5.4, the numbers of degrees of freedom for control, which equal to the number of the manipulated variables, are 36; besides, the manipulated variables in this research are also shown in the same table.

Step 3: *Establish the fixture plant.*

The principle idea of fixture plant theorem is to ensure that the process has fluid-filled and all chemicals in the entire process are material-balanced. The idea is seem to be analogous to creating *"hydraulic"* control structure proposed by Buckley in 1984; however, as description in chapter 3, this step in Wongsri's plantwide control structure design procedure is divided into 3 tasks.

Table 5.4: Control degree of freedom for each unit

Unit	Manipulated Variable	DOF per Unit	Quantity	DOF
Independent stream	Flow rate	1	6	6
Pump	Speed	1	5	5
Non-adiabatic reactor	Effluent rate Duty	2	2	4
Adiabatic reactor	Effluent rate	1	1	1
Liquid-liquid phase decanter	Heavy phase outlet flow rate Light phase outlet flow rate	2	2	4
Distillation column without condenser	Distillate rate Bottom rate Reboiler heat input	3	2	6
Flash tank	Vapor discharge rate Liquid effluent rate	2	2	4
Compressor	Power	1	1	1
Condenser	Duty	1	1	1
Two-way splitter	1 Flow rate	n-1	2	2
Three-way splitter	2 Flow rate	n-1	1	2
Total				36

** n is number of outlet streams of splitter.*
*** Splitter of stream 41 shown in figure 5.3 is not considered.*

Step 3.1: Keep each raw material entered to the reactor constant by adjusting its fresh feeds and/or recycles.

For the first reactor in this study, there exist 3 major chemicals which should be controlled: triglyceride, sodium hydroxide, and methanol. Consequently, the controller designed to keep their flow rates constant are controller FC1 for fixing the triglyceride fed to reactor and adjusting production rate of the process, FIC1 in order to keep the sodium hydroxide fed to reactor at 1 %wt of triglyceride, FIC3 to fix the methanol, which have effect on the reaction, entered to reactor as shown in figure 5.4.

At the second reactor, methanol fed to the reactor is controlled by controller FIC4 in figure 5.4 by manipulating the makeup of methanol, respectively.

Other points that should be considered are fresh feeds of water and carbonic acid. For the water feed, its flow is fixed by controller FC2. For the carbonic acid feed, controller FIC2 is designed.

Step 3.2: Adjust the flow of exit material streams according to their accumulation

To ensure the process is material-balanced and avoid the accumulation of the product, by-product, and inerts in the process, the outlet streams from the process should not be fixed. Controller LC1, LC2, PC1, and PC2 shown in fixture 5.4 are designed to attain the objective of this step.

Step 3.3: Locate the quantifiers for the rest of components and design the control loops to regulate their inventories.

Chemical inventories, which are liquid level for liquid phase and pressure for gas phase, for the rest of components, diglyceride and monoglyceride, are located; then, control loops LC3 to LC6 are designed as shown in table 5.5 to regulate it.

Table 5.5: Located quantifiers for the rest component and controller designed to control its inventory

Component	Quantifier position	Controller
Diglyceride	First and second reactors (R-1 and R-2) Light phase of first decanter D-1	LC3 to LC6
Monoglyceride	First and second reactors (R-1 and R-2) Light phase of first decanter D-1	LC3 to LC6

Step 4: *Handling the disturbances.*

This step is to design the control loops to manage both material and thermal disturbances, which the thermal disturbances is divided into 2 classes which are thermal disturbance that have and have not directly affect product quality.

Step 4.1: Heat disturbances.

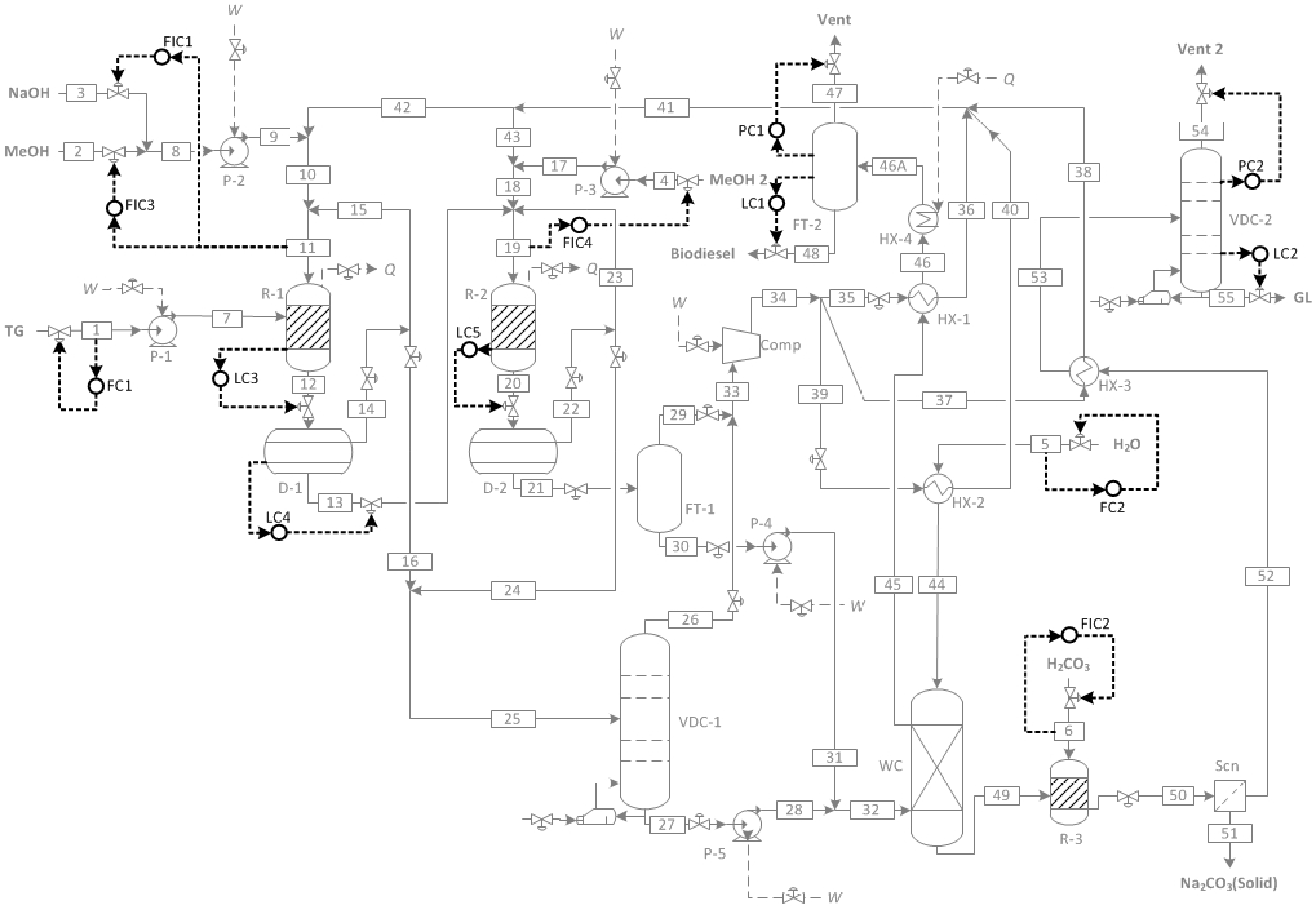


Figure 5.4: Summary of control structure designed in step 3

Step 4.1.1: Direct the heat disturbances that not directly related to quality.

Control loops are designed to reject the effect of thermal disturbances that do not directly affect product qualities to environment via the nearest downstream heater or cooler. Sometimes, the effect of the disturbances in one process stream has to shift to another process stream via process-to-process heat exchanger to meet the nearest heater or cooler.

In this study, there are 3 units can be used as heat exit points for thermal disturbances not direct effect on product qualities, which are the first and the second heat exchangers and the heater. Figure 5.5 is shown heat pathways of thermal disturbances considered in this step and its exit points. Thereupon, control loops are designed to deal the disturbances; for thermal disturbances due to product washing and in fresh water feed shown in the figure 5.5 (red and green lines, respectively), the effect of disturbance can be shifted to methanol recycle using controller TC1 and TC3 at the first and the second process-to-process heat exchangers shown in figure 5.7 by adjusting flow rates of stream 35 and 39. The effect of the thermal disturbance in the process stream fed to the heater (blue line) can be eliminated at the heater by designed controller TC2 shown in figure 5.7.

The alternative in this step is the temperature control of the washing water fed to the extraction column by adjusting the setpoint of the fresh water feed controller FC2 designed in the third step (TC3 in the CS6 shown in figure 5.41).

Step 4.1.2: Manage the heat disturbance that related to quality in order to maintain the product constraints.

This step is to design the control loop to manage the thermal disturbances that have the direct effects on the product qualities before it reach to the reactor in order to maintain the operating constraints and/or the product specifications. It is quite different between this step and the previous step; this step is to indirectly control the product qualities by controlling the temperature because generally composition sensor is not preferable because of existence of deadtime in its measurement and given slow corrective action. This step can be done by controlling the temperature of the feed stream to the reactor by adjusting the appropriate upstream heater/cooler duty or using

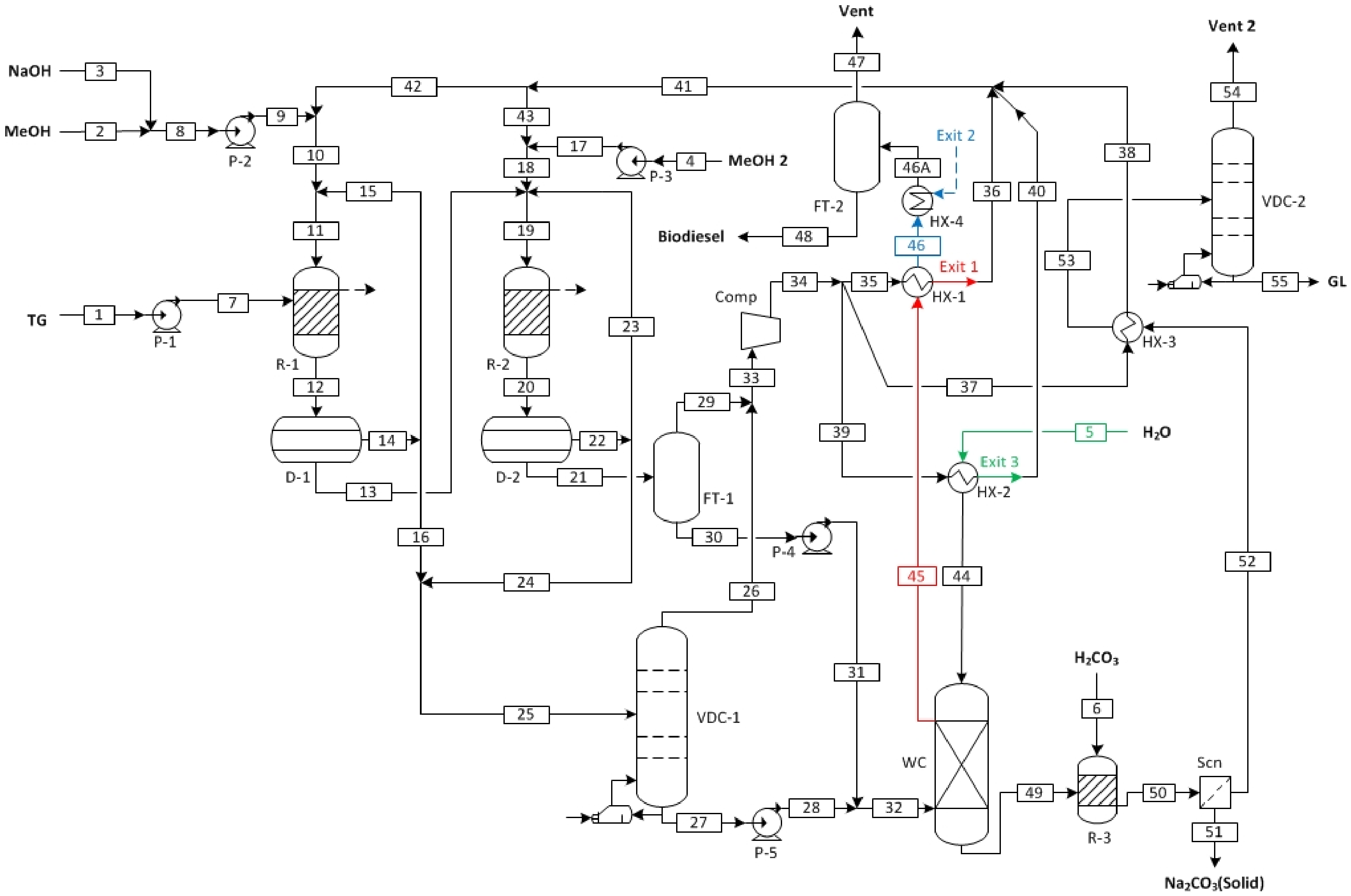


Figure 5.5: Heat pathways of thermal disturbances not directly effect on product qualities and its exit points

upstream process-to-process heat exchanger. In addition, if the reactor is not adiabatic; for example, there exists a heating coil or a cooler jacket on the reactor; the thermal disturbance effect can be eliminated by these pieces of equipment. Generally, the thermal disturbances considered in this step usually involve streams fed to reactor and last separation units.

Pathways of thermal disturbances that have the product quality effect are shown in figure 5.6 which its effects can be dealt or rejected using reactor cooling duties and reboiler duties at the distillation columns. To eliminate the effects, at feeds to the first and the second reactors (red line in figure 5.6), controller TC4 and TC5 shown in figure 5.7 are designed to control the reactor temperatures directly by adjusting direct duties of each reactor. Other points are temperature of streams fed to separating units, which are the first and the second distillation columns (blue and green lines in figure 5.6, respectively); to defeat the effects of disturbances at the locations, controller TC6 and TC7 are designed by manipulating the reboiler duties of the first and the second distillation columns, respectively. However, for the controllers designed to control temperature of both distillation columns, there exists many available points in each column to control. To determine which trays of distillation columns should be control its temperature, perturbation analysis, which is detection of which tray temperature deviated mostly from its norm, should be performed. However, in this research, there are bottom temperature constraints of both distillation columns, so the distillation column temperature controllers designed in this step is set to control the reboiler temperatures of the columns.

Step 4.2: Material disturbances.

Design the desired material pathways for each component, the excess of each component from its norm will be forced to its desired pathways; then, the control loops are designed to deal the material disturbances. However, in these designs, there are various feasible patterns of the reactor and the distillation column for each pathway depending on the control policy. To find the best, each pattern should be tested under the disturbed conditions. In this step, the designed control loops in the previous steps may be detached and replaced by new loops to achieve the better control loops; for example, the temperature control loop of the reactor designed in the

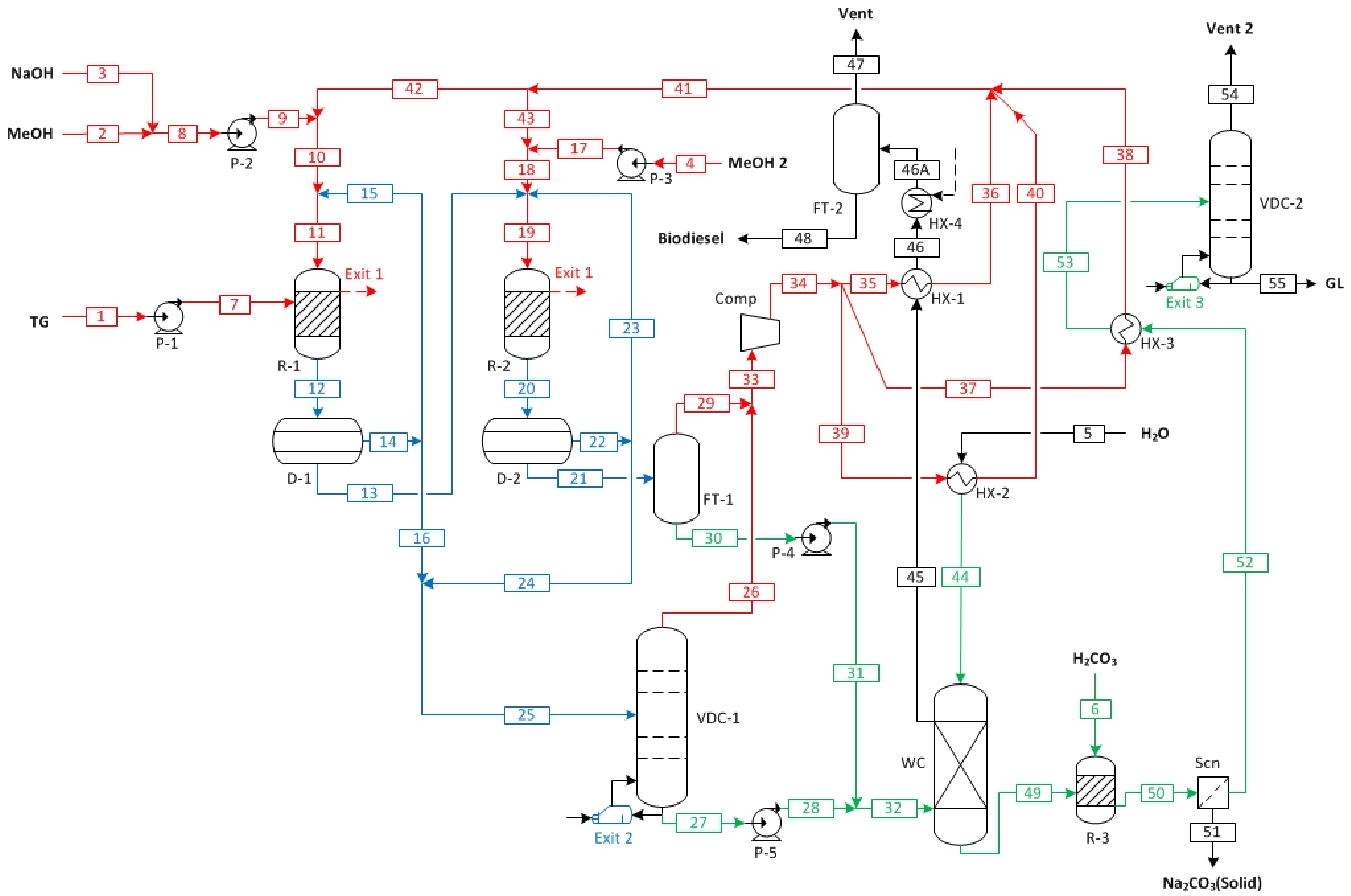


Figure 5.6: Heat pathways of thermal disturbances effect on product qualities and its exit points

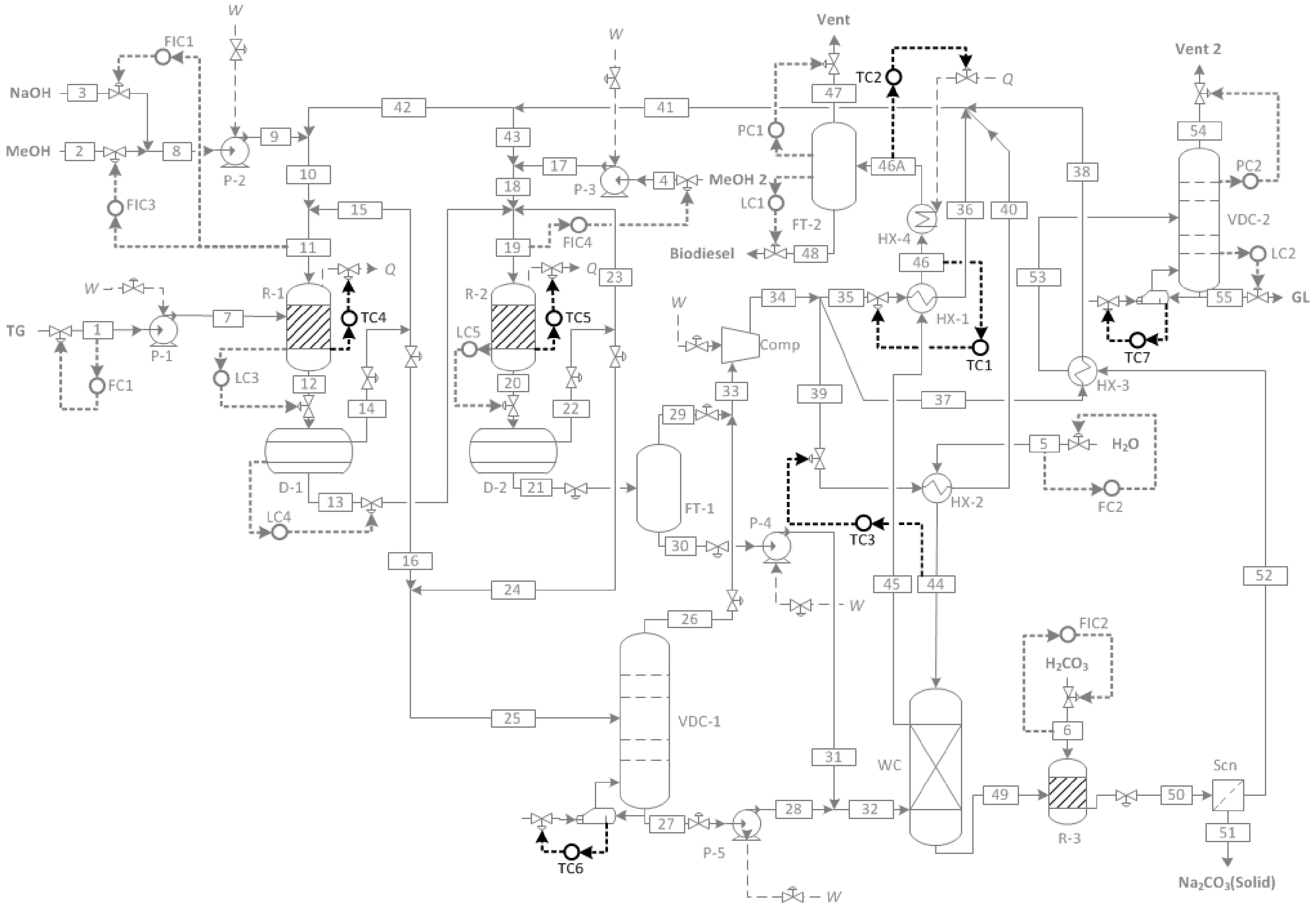


Figure 5.7: Summary of control structure designed in step 4.1

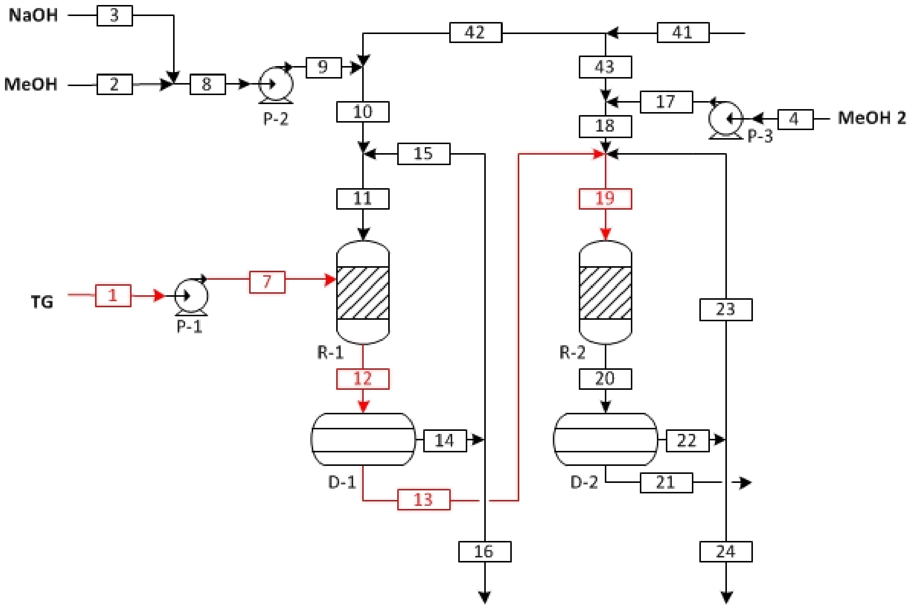


Figure 5.8: Material pathway of triglyceride

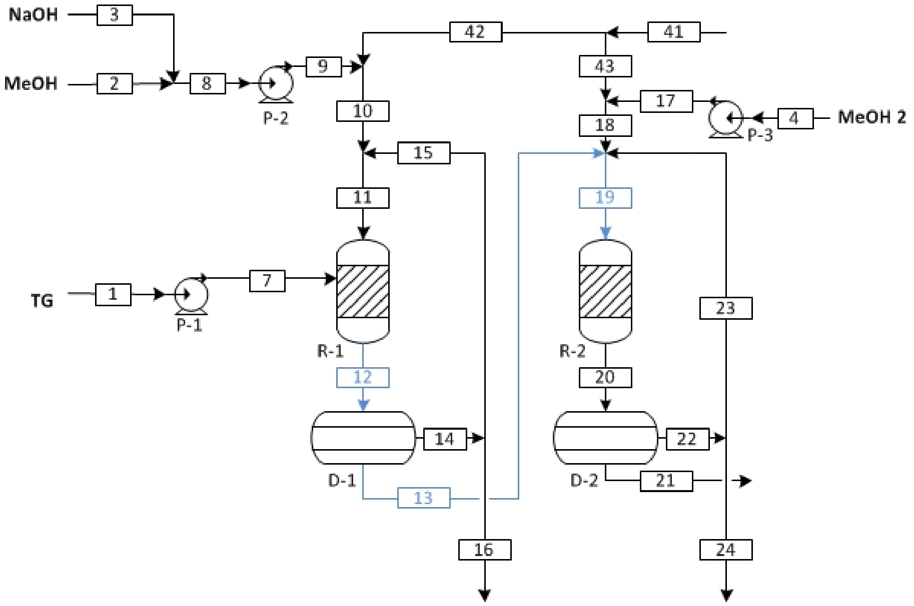


Figure 5.9: Material pathways of diglyceride and monoglyceride

previous step may be replaced by a loop that controls the product composition in the stream left from the reactor. However, cascade control may be also used to improve the control performance by reducing the effects of dead time and phase lag time; for example, the primary controller which controls the product composition in the outlet stream from the reactor can send signal to be the setpoint of the slave controller which its controlled variable is the temperature of reactor.

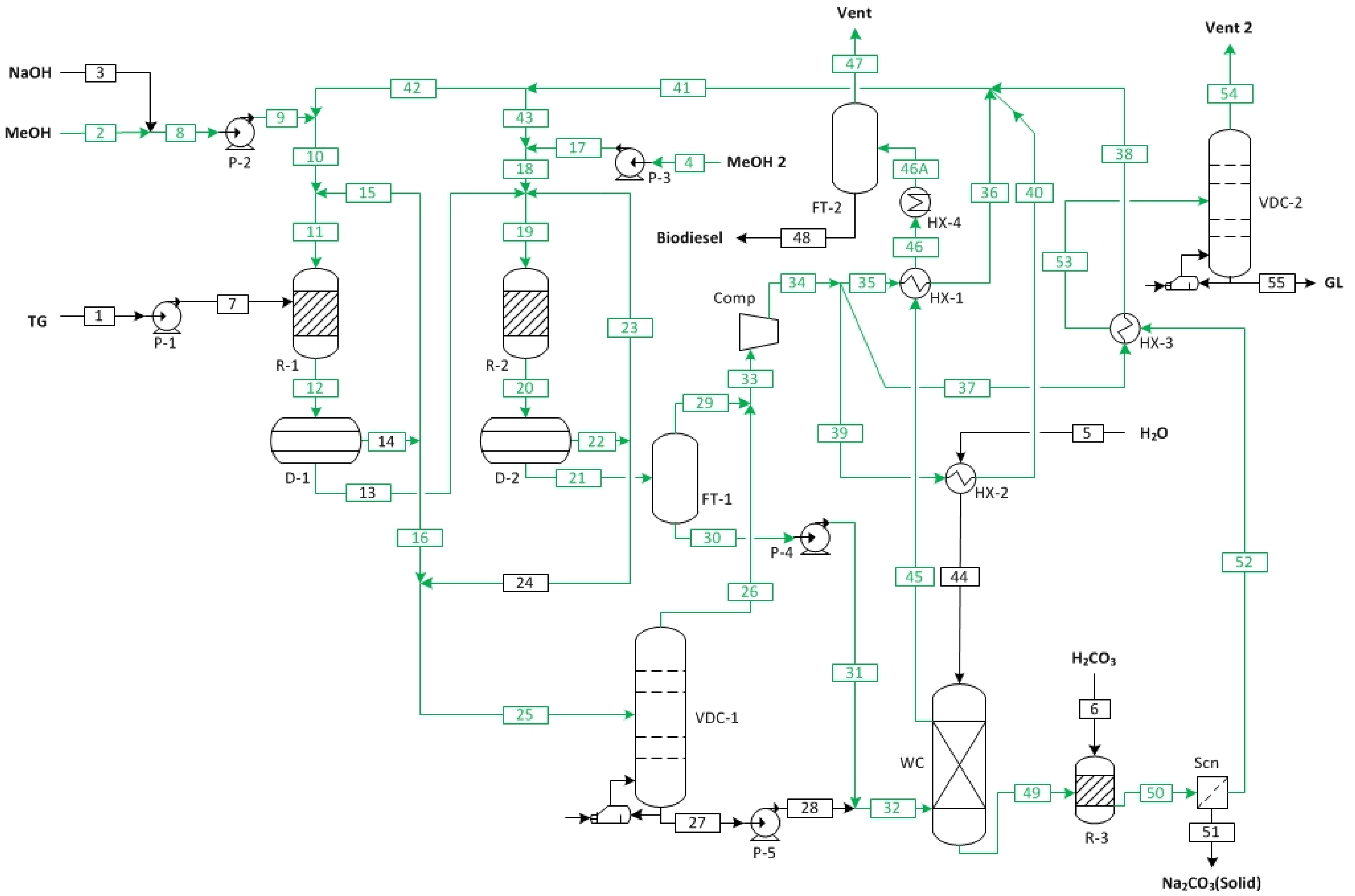


Figure 5.10: Material pathway of methanol

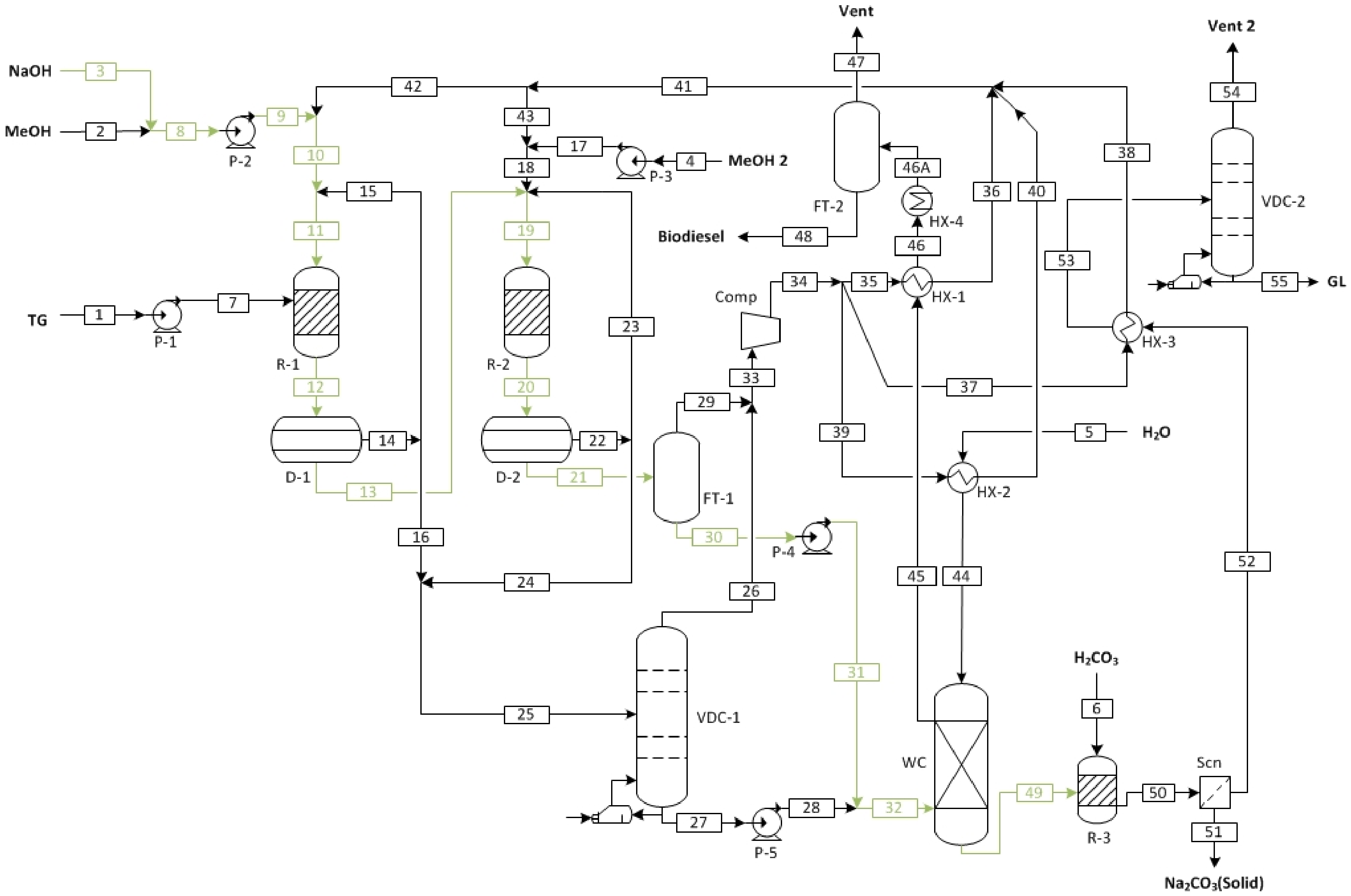


Figure 5.11: Material pathway of sodium hydroxide

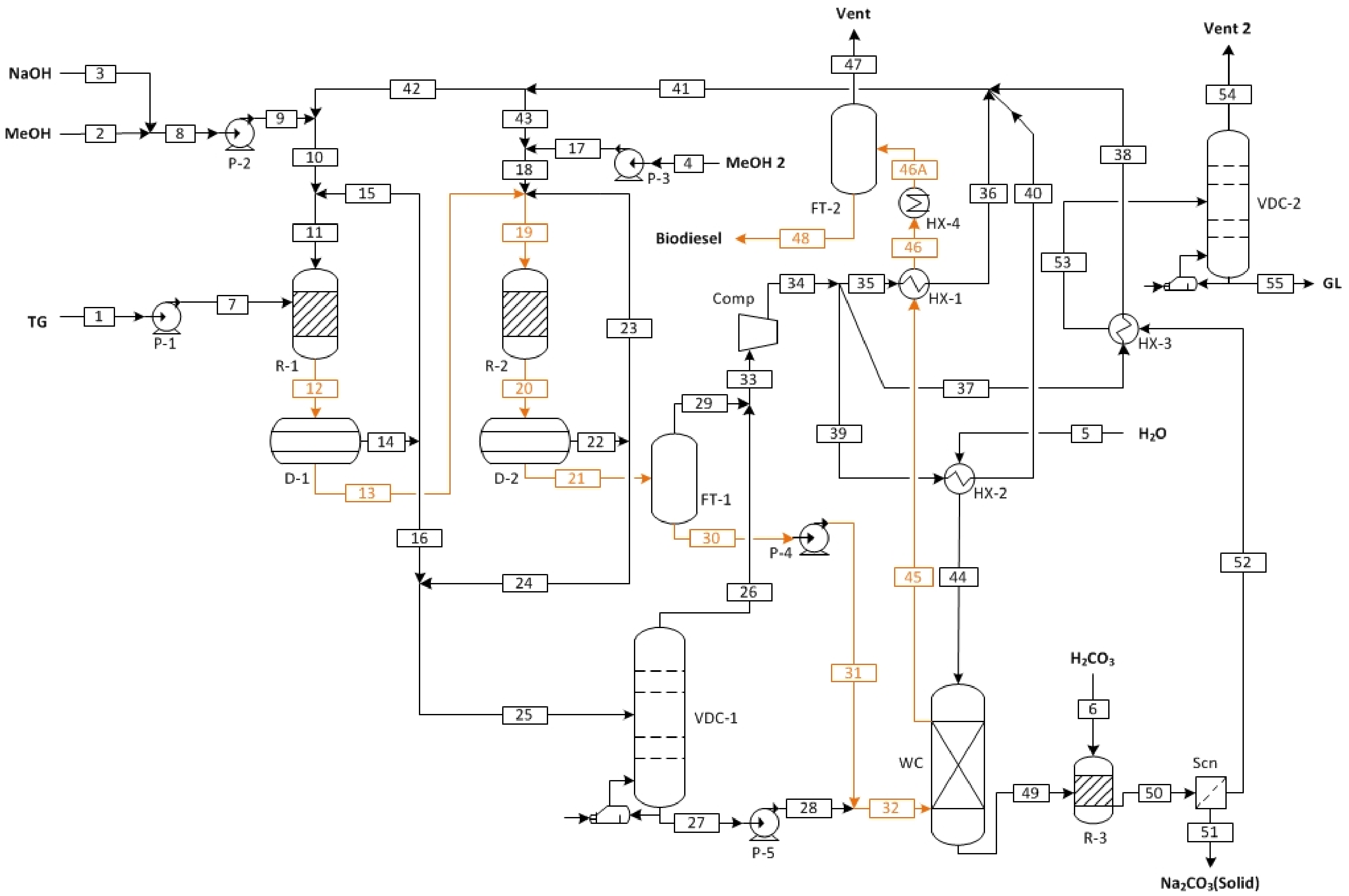


Figure 5.12: Material pathway of biodiesel

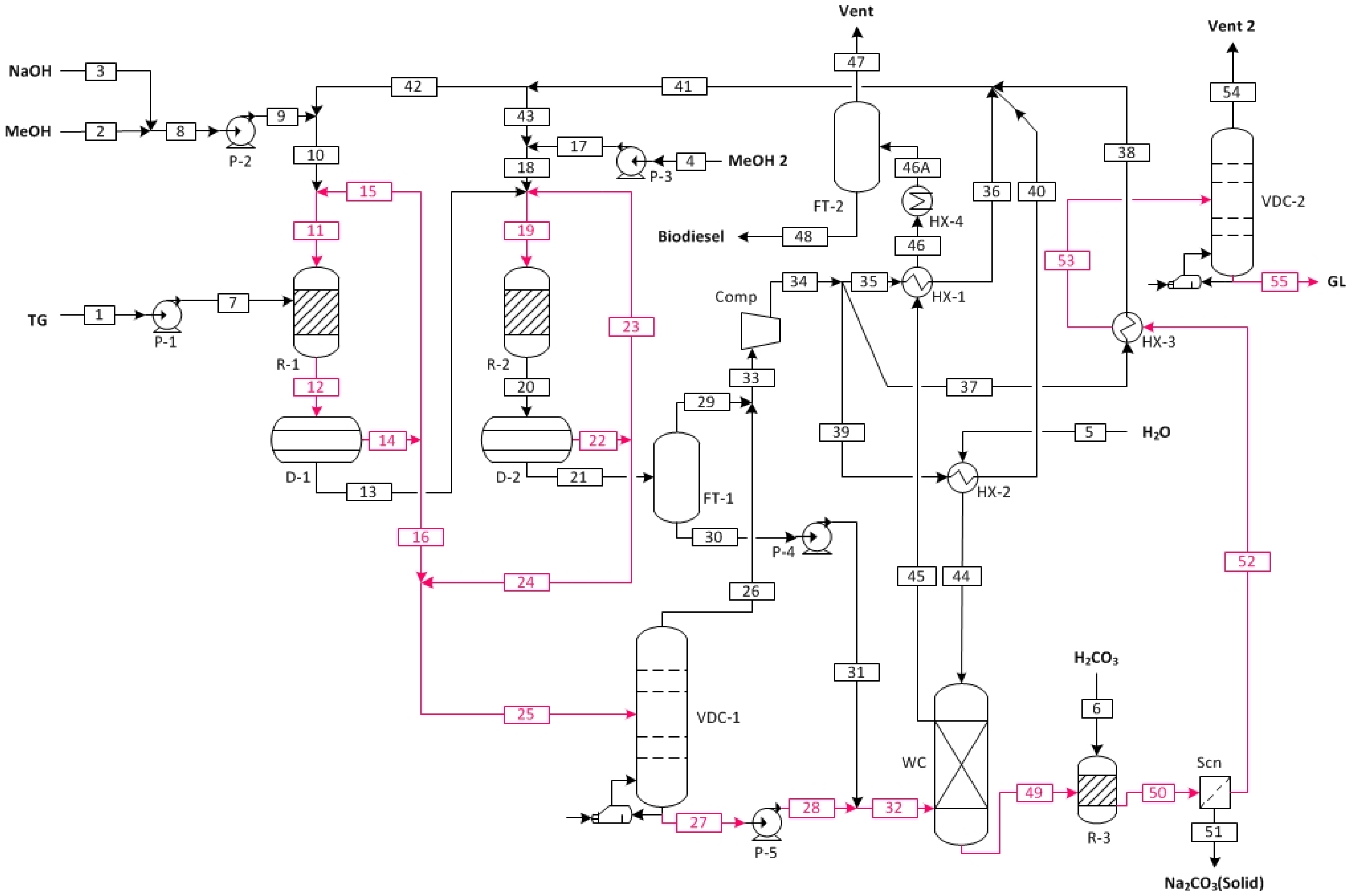


Figure 5.13: Material pathway of glycerol

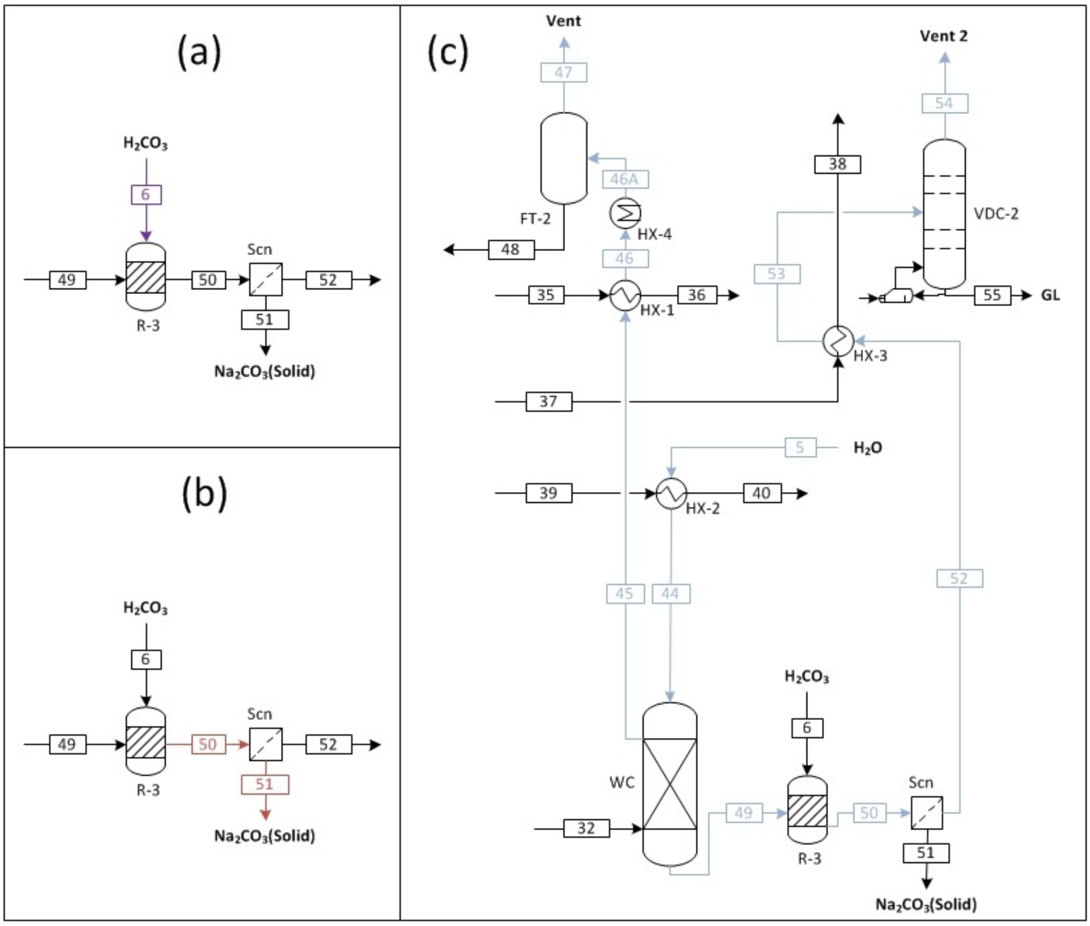


Figure 5.14: Material pathway of (a) carbonic acid,
(b) sodium carbonate, and (c) water

The desired material pathways of each chemical in this study are shown in figure 5.8 to 5.14; triglyceride limiting reactant, and diglyceride and monoglyceride which are intermediates, should be consumed completely at the reactor; while the methanol excess reactant should be recycled and leaved the process at both vent streams. The sodium hydroxide should react with the carbonic acid with complete conversion to form sodium carbonate, which is solid and quitted the process at the screening unit. For the product and by-product, which are the biodiesel and the washed glycerol, it should be flowed out the process as product and by-product streams and the washing water should be leaved at both vent streams.

Most of chemicals leave the process normally at its desired pathways except for triglyceride, diglyceride, monoglyceride and glycerol; therefore, the control loop should be designed at the reaction section to deal with this problem. For control the reactor with its considered control loops shown in figure 5.15 to 5.19 are simulated under the disturbed conditions, which are the changes of triglyceride feed rate to be 9 and 11 kmol/h.

Table 5.6: Considered control structure (additional loop) for first and second reactors

Unit	Control Structure	Controlled Variable	Manipulated Variable
First reactor	CS 1	-	-
	CS 2	Biodiesel composition of outlet stream	Setpoint of methanol fed to reactor controller
	CS 3	Biodiesel composition of outlet stream	Setpoint of temperature controller
	CS 4	Yield	Setpoint of methanol fed to reactor controller
	CS 5	Yield	Setpoint of temperature controller
Second reactor	CS 1	-	-
	CS 2	Biodiesel composition of outlet stream	Setpoint of methanol fed to reactor controller
	CS 3	Biodiesel composition of outlet stream	Setpoint of temperature controller
	CS 4	Yield	Setpoint of methanol fed to reactor controller
	CS 5	Yield	Setpoint of temperature controller
	CS 6	Total Yield	Setpoint of methanol fed to reactor controller

Note: Yield is calculated from amount of biodiesel produced in each reactor divided by the theoretical yield (summation of three times of amount of triglyceride, two times of amount of diglyceride, and amount of monoglyceride fed to the reactor). Total Yield is calculated from biodiesel produced in the reaction section divided by three times of triglyceride fed to the process.

The simulation results in local analysis of the first reactor shown that not all of available control schemes, although tuning are performed, can handle the first reactor when the fresh triglyceride feed changes; only CS1, CS4, and CS5 of the first reactor can do. Problem of CS2 is seen to be that it requires 2 actions, both direct and reverse; when the setpoint of the fresh triglyceride feed controller starts to be 9 kmol/h, this make the reaction rate and biodiesel and glycerol compositions in reactor product

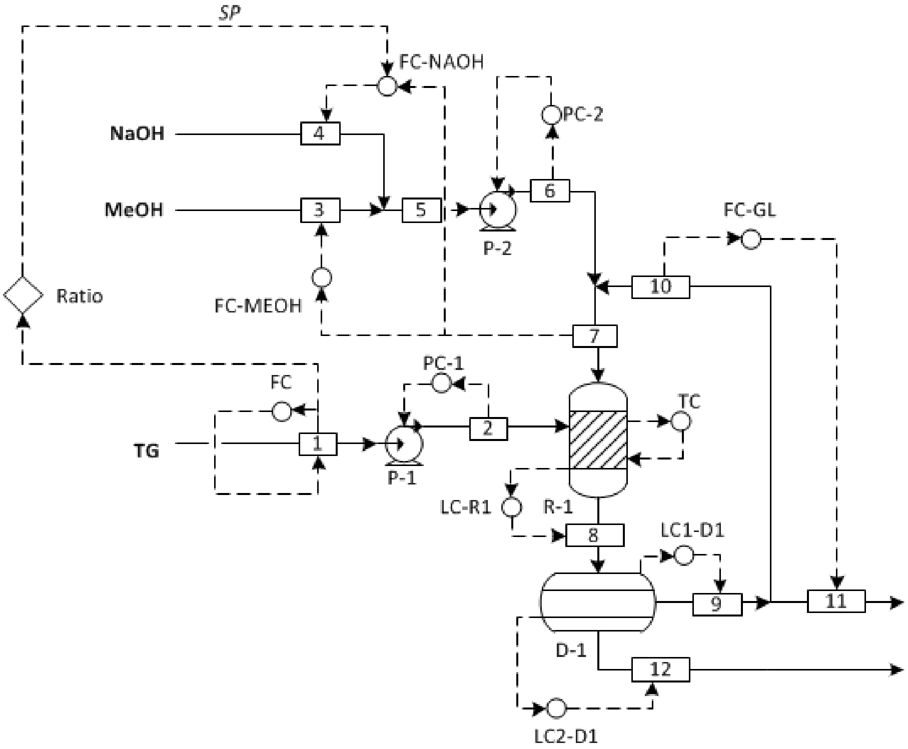


Figure 5.15: CS1 of first reactor in local analysis

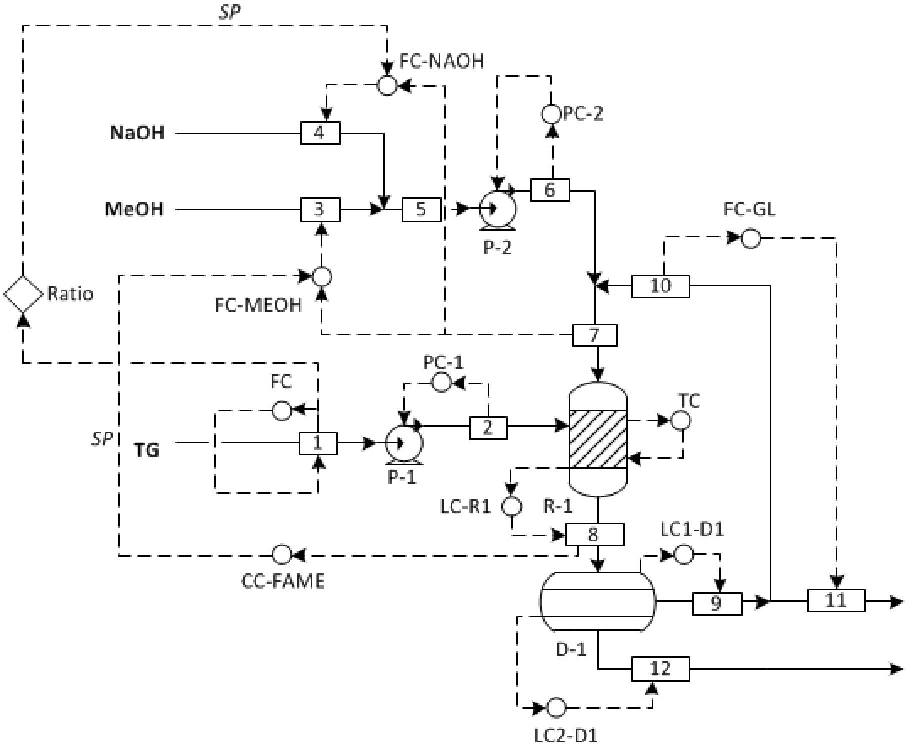


Figure 5.16: CS2 of first reactor in local analysis

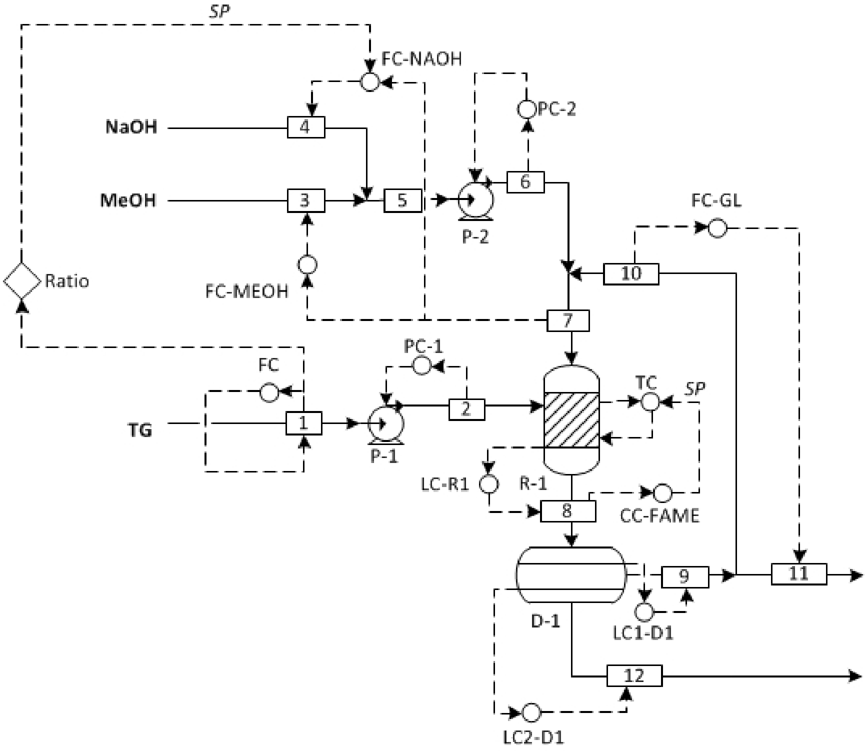


Figure 5.17: CS3 of first reactor in local analysis

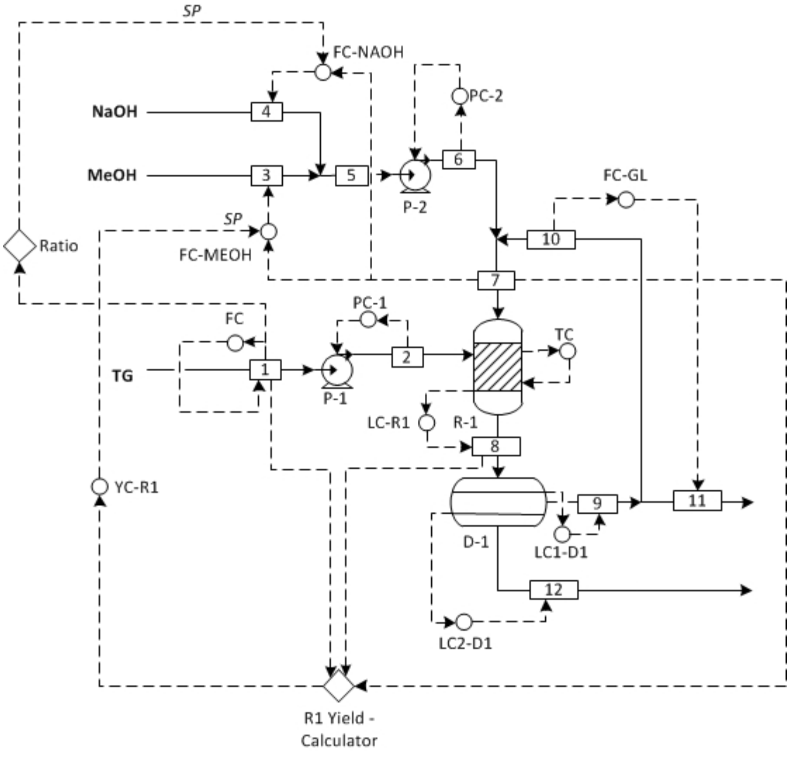


Figure 5.18: CS4 of first reactor in local analysis

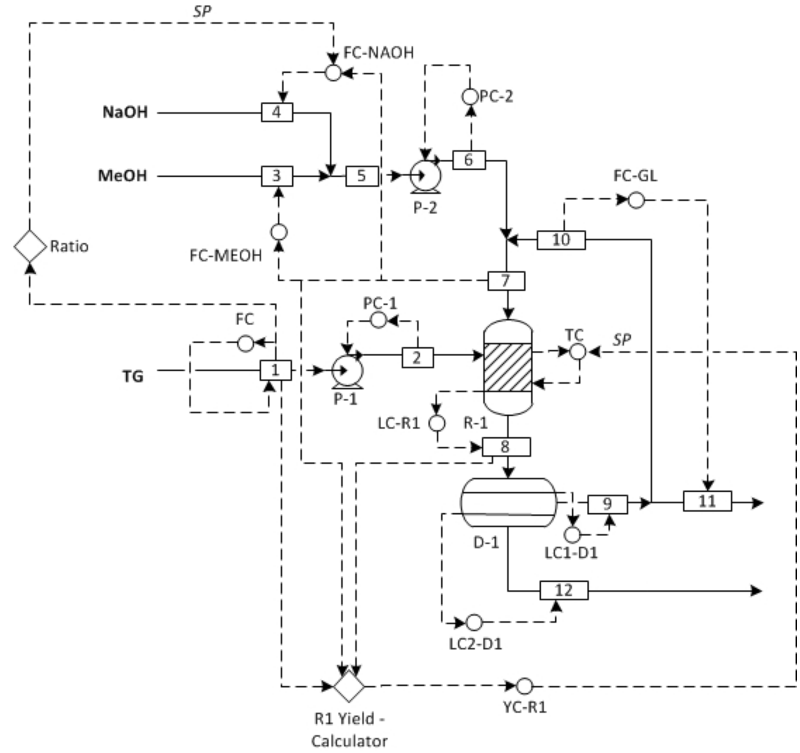


Figure 5.19: CS5 of first reactor in local analysis

stream decrease; therefore, to achieve the setpoint of biodiesel composition controller, the composition controller should be direct action to reduce the methanol, which acts as contamination in the location of composition sensor, fed to first reactor. But, when glycerol recycle stream reached to its setpoint, the biodiesel composition still less than its setpoint; thus, the methanol fed to the first reactor is still reduced by the signal from the reverse action composition controller; this makes glycerol composition in reactor feed stream increases which is opposite direction to methanol so the reaction rate decreases continuously and the composition controller never achieves its setpoint. To solve this problem, the composition controller should be set to be reverse action for increasing the forward reaction rate.

CS3 of the first reaction is unable to control the process because the biodiesel composition in the product stream reduces when the setpoint of the fresh triglyceride feed controller is 9 kmol/h; to achieve the setpoint of the biodiesel composition controller, it tries to increase the reactor temperature until the cooling duty of reactor becomes saturated. The cause of this problem is that the reactor requires heating duty to achieve the desired biodiesel composition if the fresh triglyceride feed is changed to be 9 kmol/h.

For the local analysis of second reactor, the first reactor with its controllable control structures, which are CS1, CS4, and CS5 of the first reactor, is paired with the second reactor with its considered control structure shown in table 5.6 and then simulated under the same disturbed conditions with the local analysis of the first reactor. A reason for consideration of the first reactor with its controllable control structures in the local analysis of the second reactor is the control loops of the first reactor effect controllability of the second reactor control loops. Actually, if the methanol recycle section exists, the control loops of the second reactor affect the controllability of the control loops of the first reactor; however, in this study, this case does not be taken into account, which causes many patterns of available control structures for the reaction section disappearance, because if the control structure cannot control the process not consisting the recycle including the energy integration under the considered disturbed conditions, it have low robustness in control and may not control the process consisting recycle when the recycle flow change so much.

Figure 5.20 to 5.35 are shown sixteen considered control schemes for the local analysis of the second reactor. Simulation results of the second reactor local analysis shows only five control structures can control the process under the consider disturbed conditions, which are CS1, CS2, CS6, CS7 and CS11 shown in figure 5.20, 5.21, 5.25, 5.26, and 5.30, respectively.

Problem of CS3 and CS8 is that, when triglyceride controller setpoint is changed to be 9 kmol/h, the biodiesel composition in product stream of the second reactor decreases; therefore, its controller send signal to its slave controller, which is a temperature controller at the second reactor, for reducing the cooling duty (reduces heat removal) at the second reactor; but the temperature of second reactor cannot reach to its setpoint due to saturation of the cooling duty (zero); however, the temperature of the stream fed to the second reactor increases slowly making the second reactor temperature increased continuously and slowly. If the process is operated for a very long time, the cooling duty of the second reactor may become unsaturated and the temperature of second reactor including the biodiesel composition may be reached to its setpoints. Moreover, if the composition controller is set to have

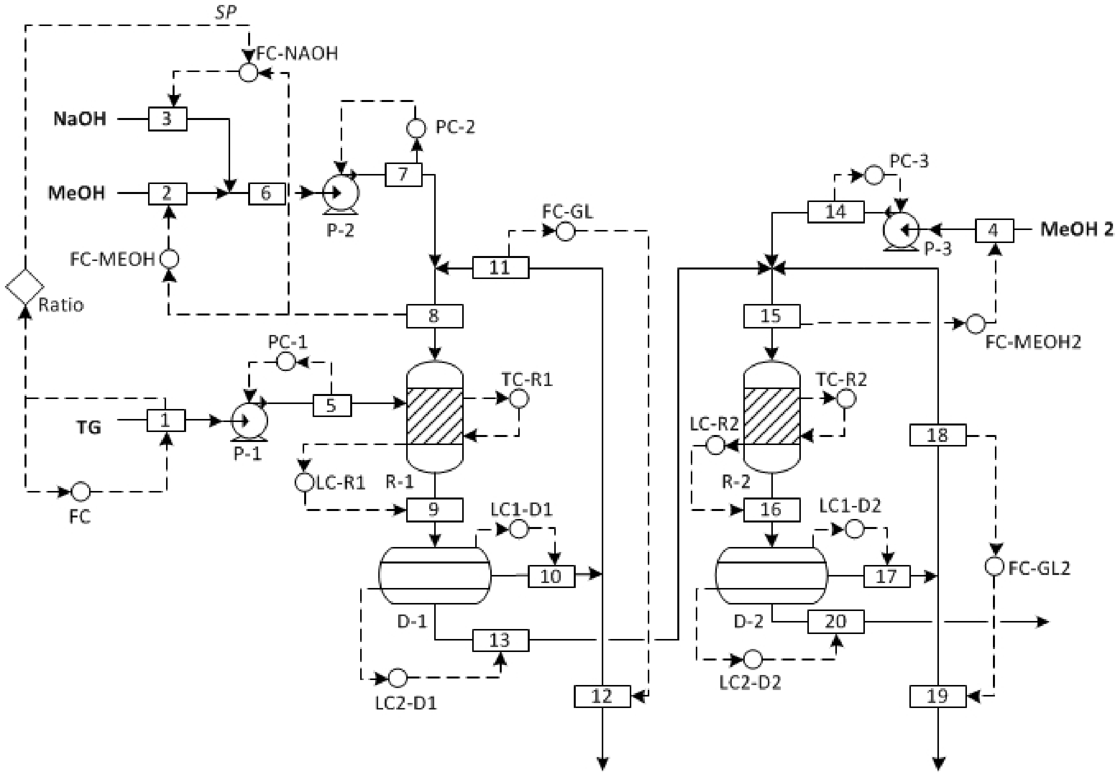


Figure 5.20: CS1 of second reactor in local analysis

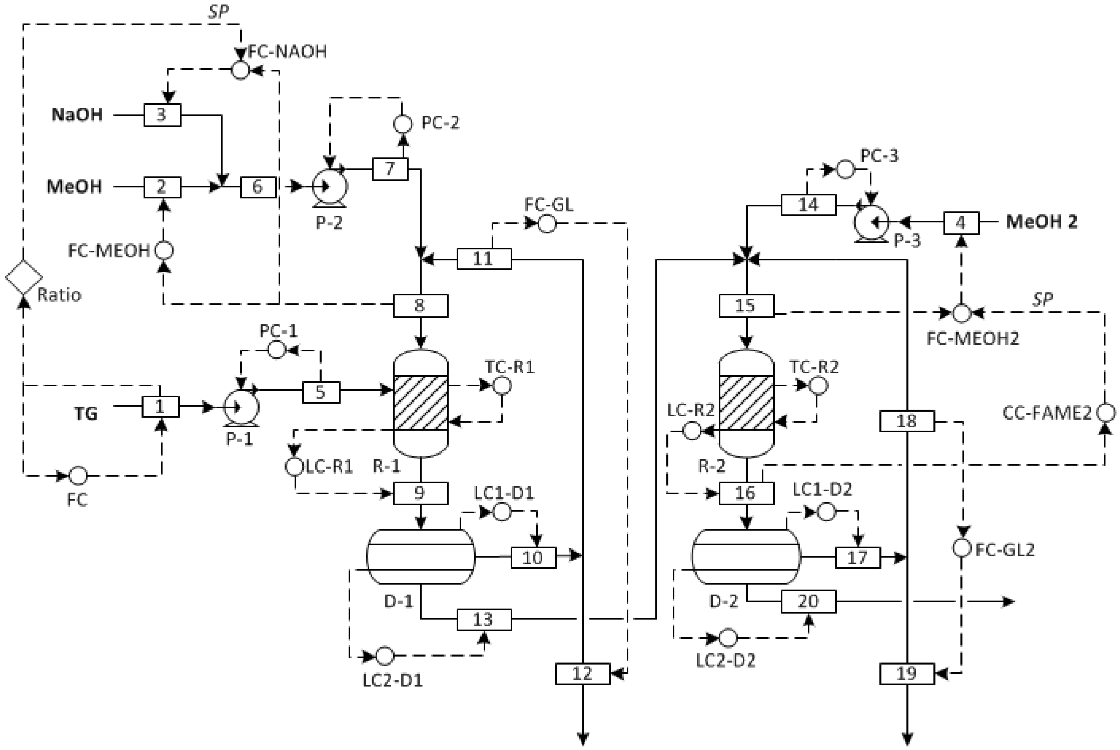


Figure 5.21: CS2 of second reactor in local analysis

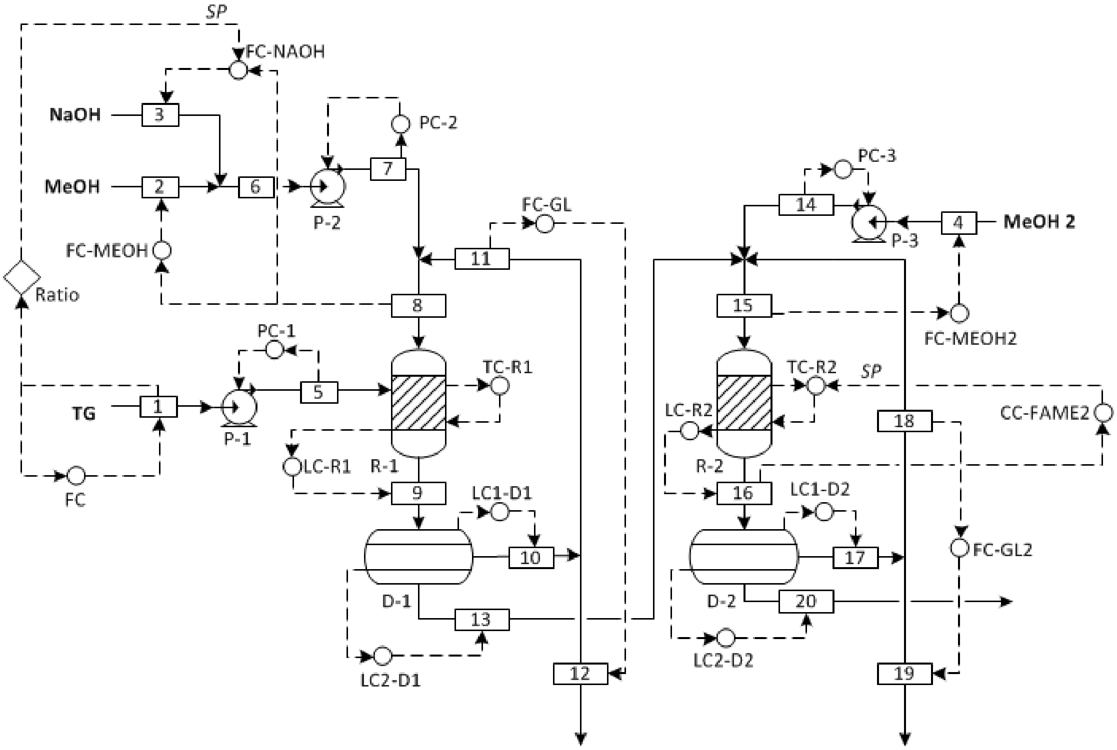


Figure 5.22: CS3 of second reactor in local analysis

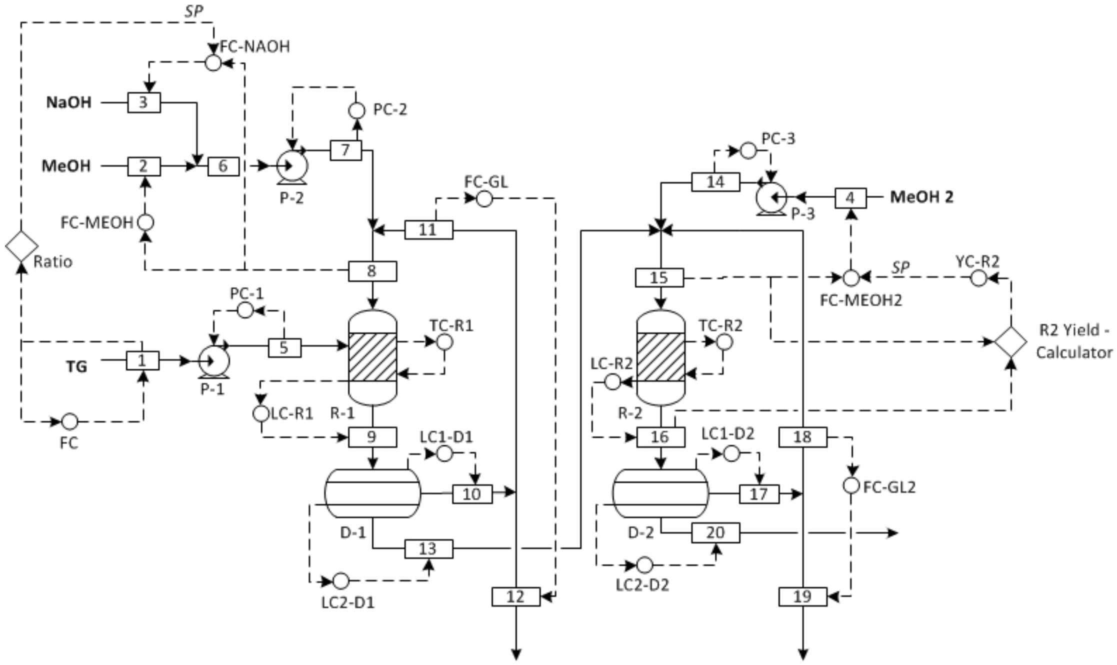


Figure 5.23: CS4 of second reactor in local analysis

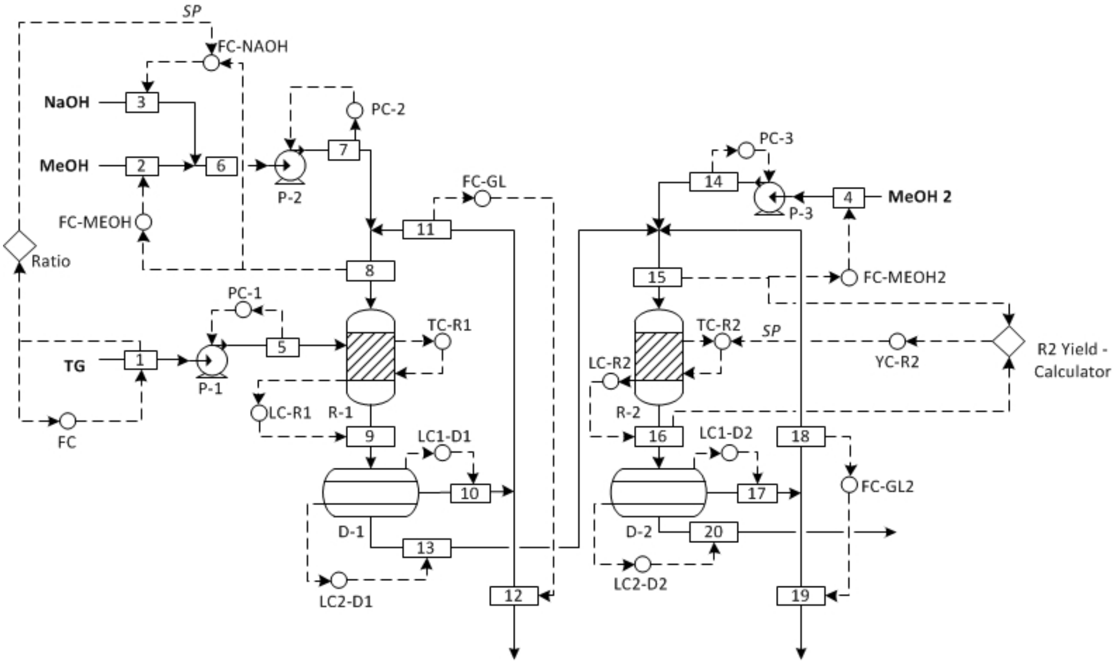


Figure 5.24: CS5 of second reactor in local analysis

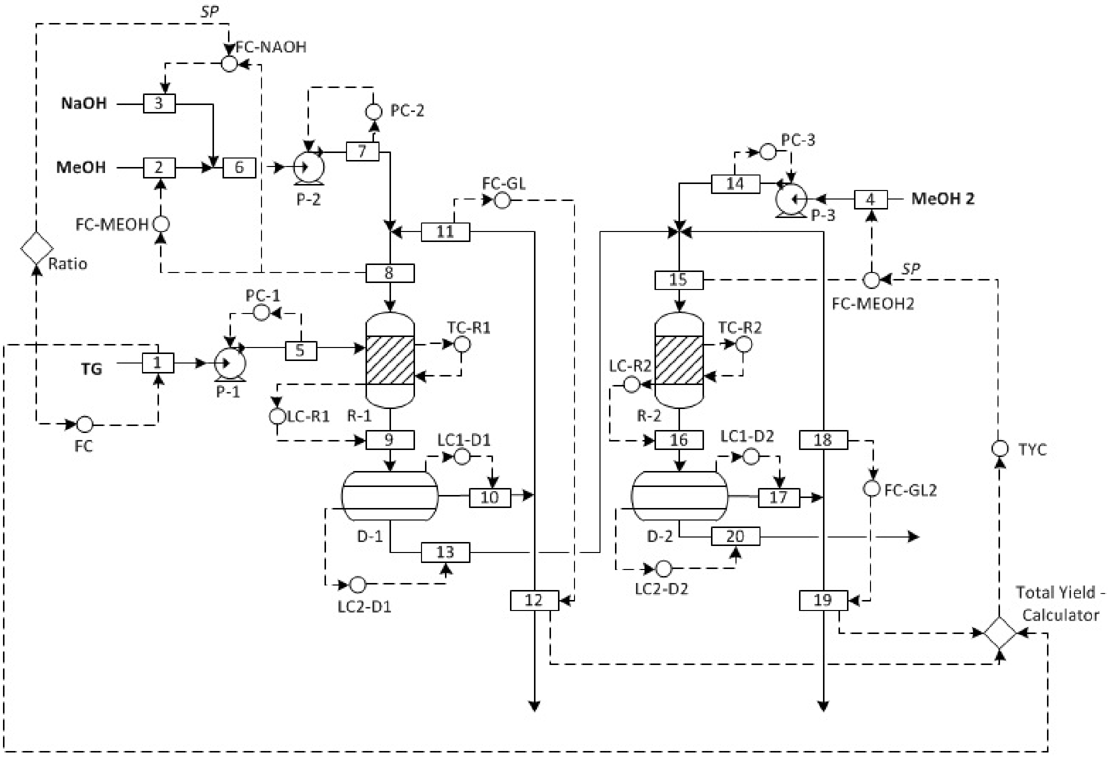


Figure 5.25: CS6 of second reactor in local analysis

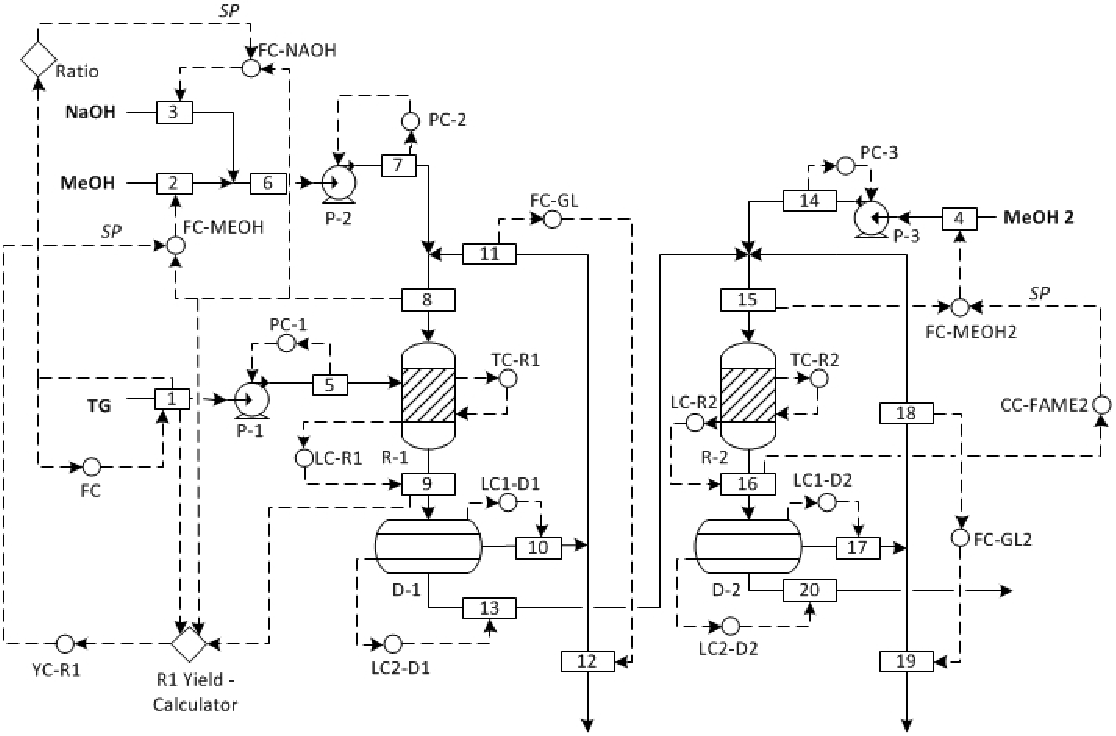


Figure 5.26: CS7 of second reactor in local analysis

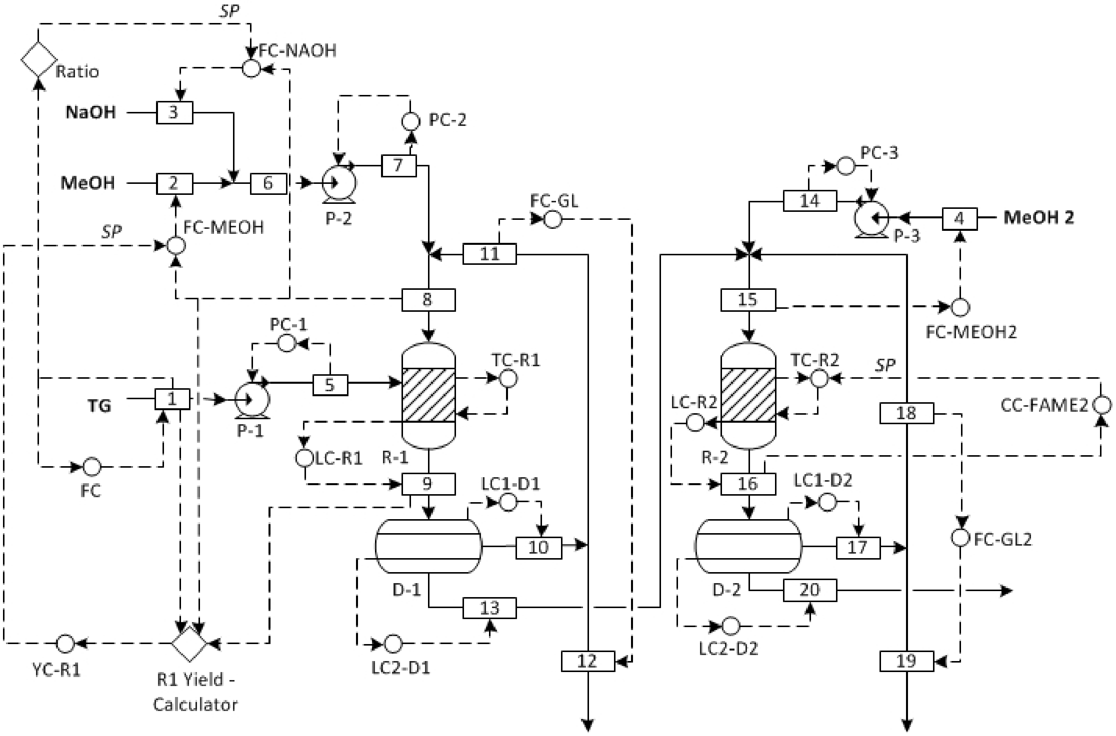


Figure 5.27: CS8 of second reactor in local analysis

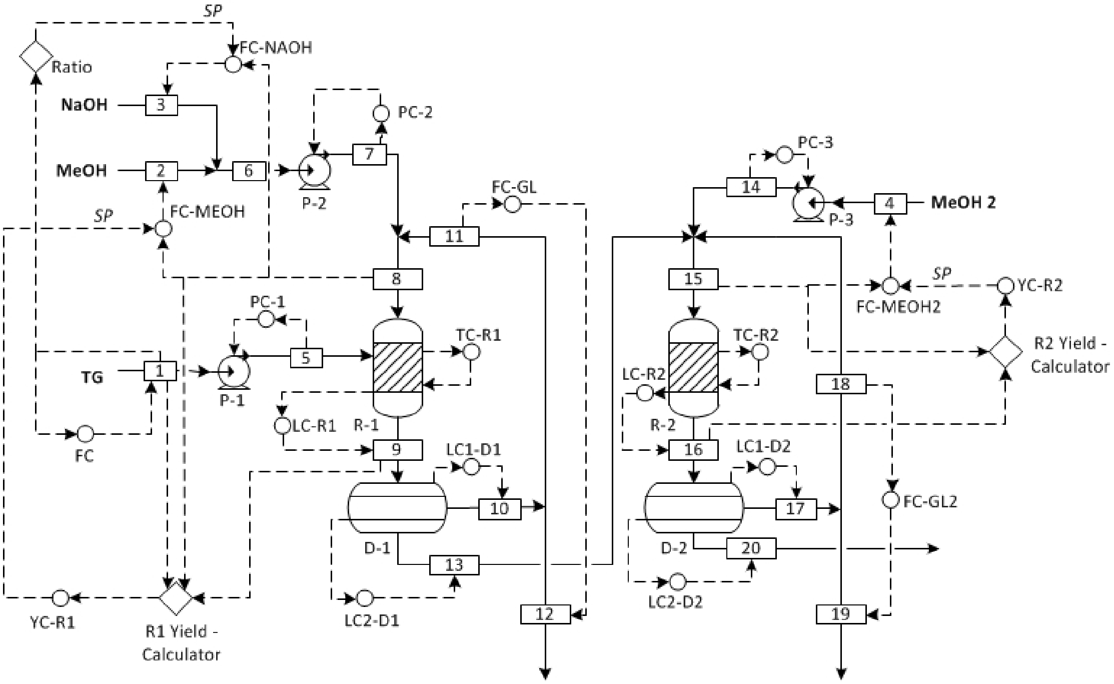


Figure 5.28: CS9 of second reactor in local analysis

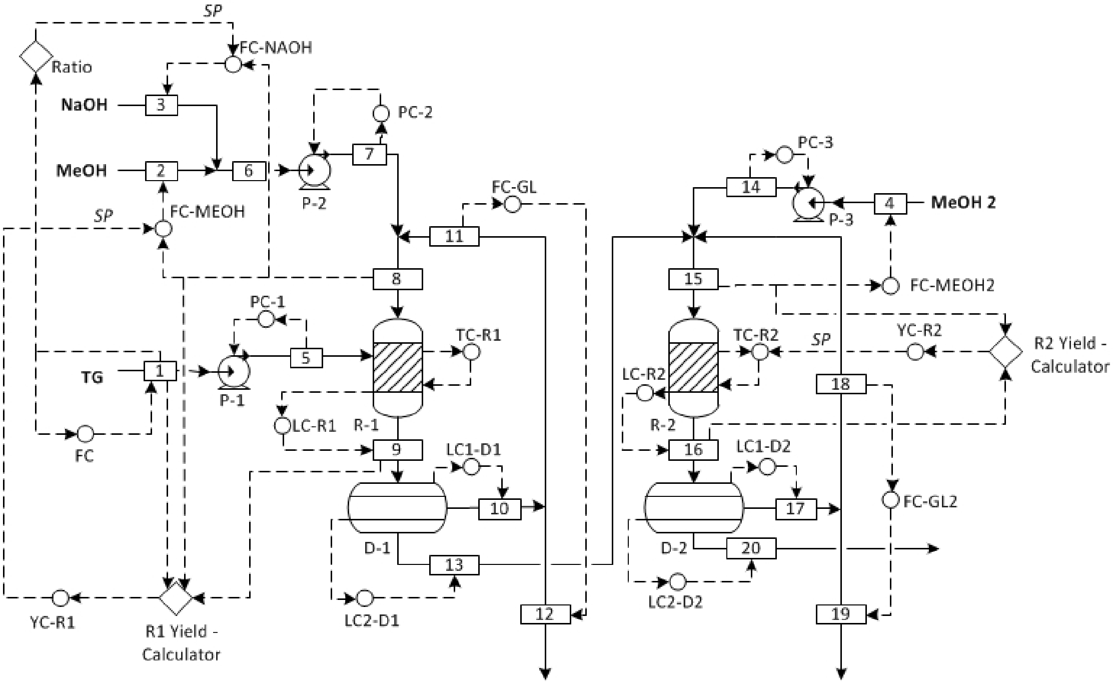


Figure 5.29: CS10 of second reactor in local analysis

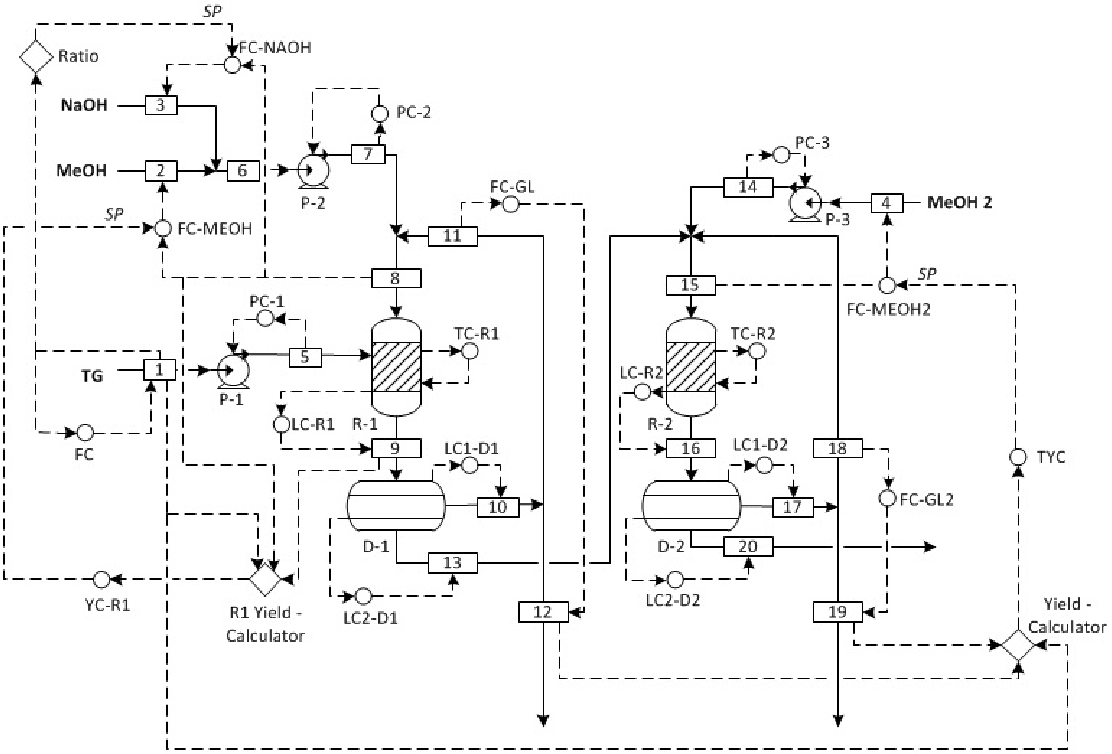


Figure 5.30: CS11 of second reactor in local analysis

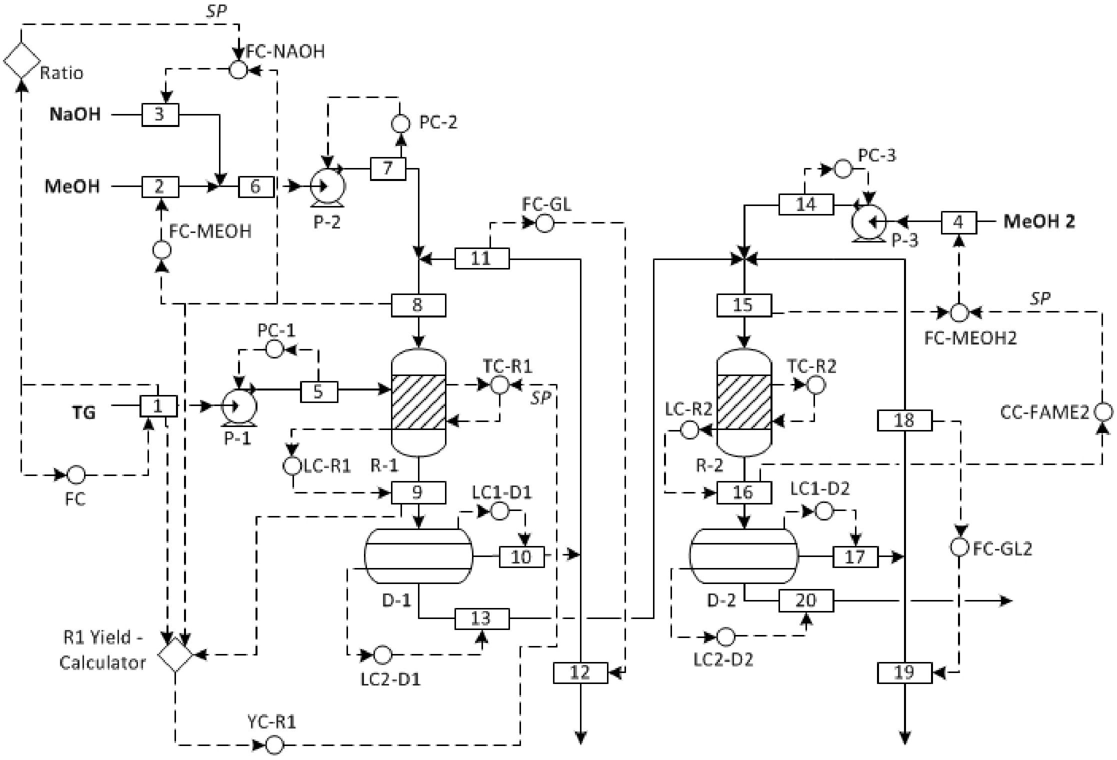


Figure 5.31: CS12 of second reactor in local analysis

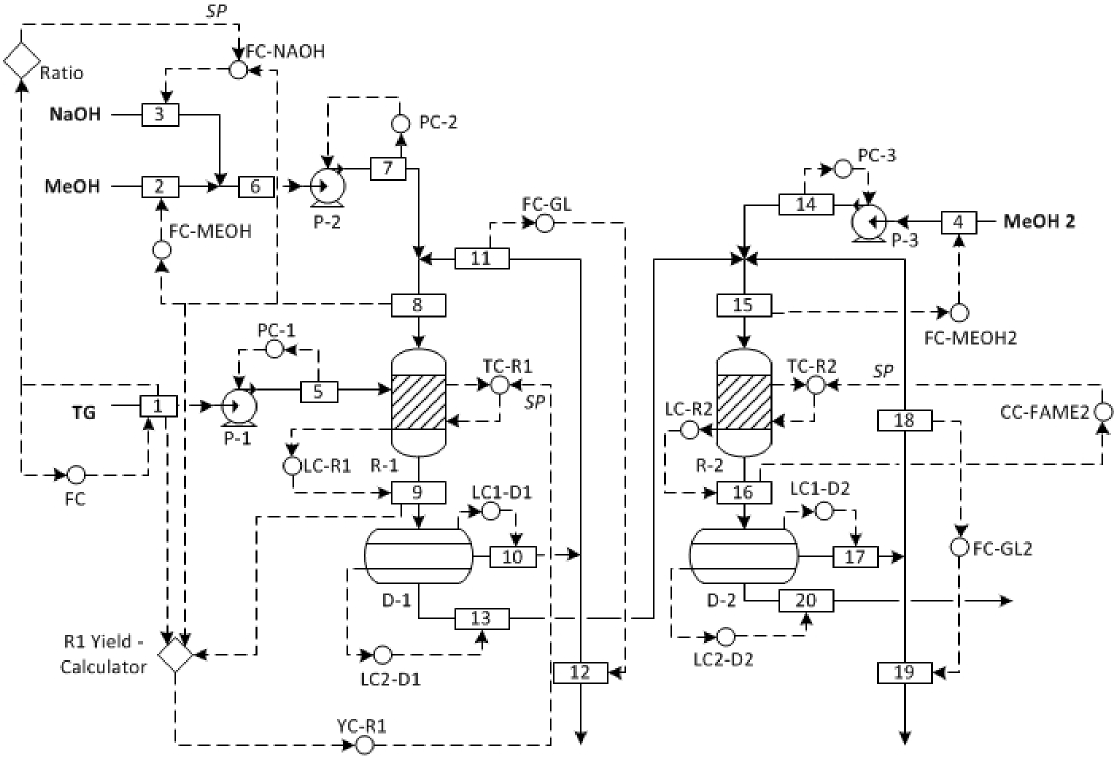


Figure 5.32: CS13 of second reactor in local analysis

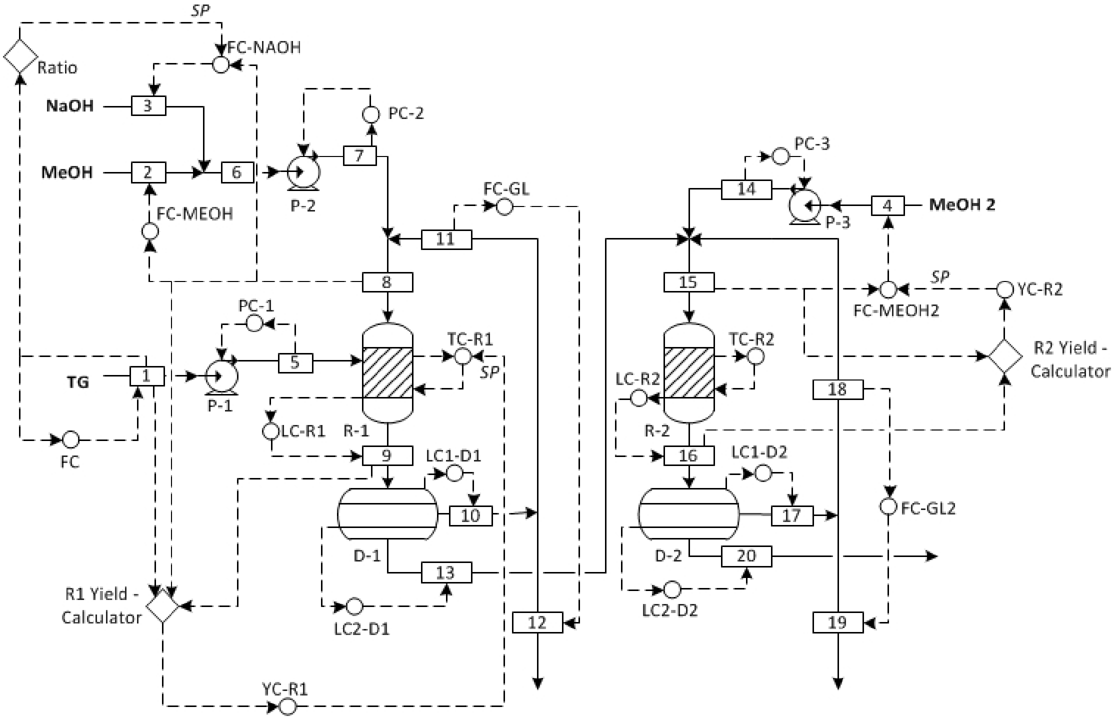


Figure 5.33: CS14 of second reactor in local analysis

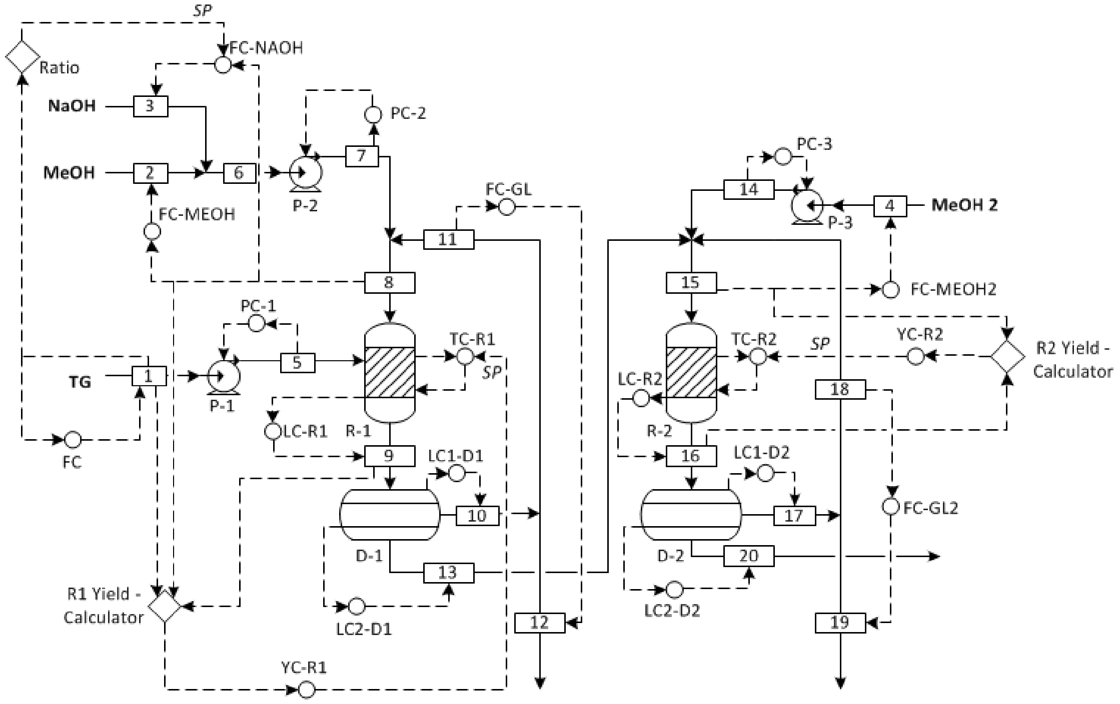


Figure 5.34: CS15 of second reactor in local analysis

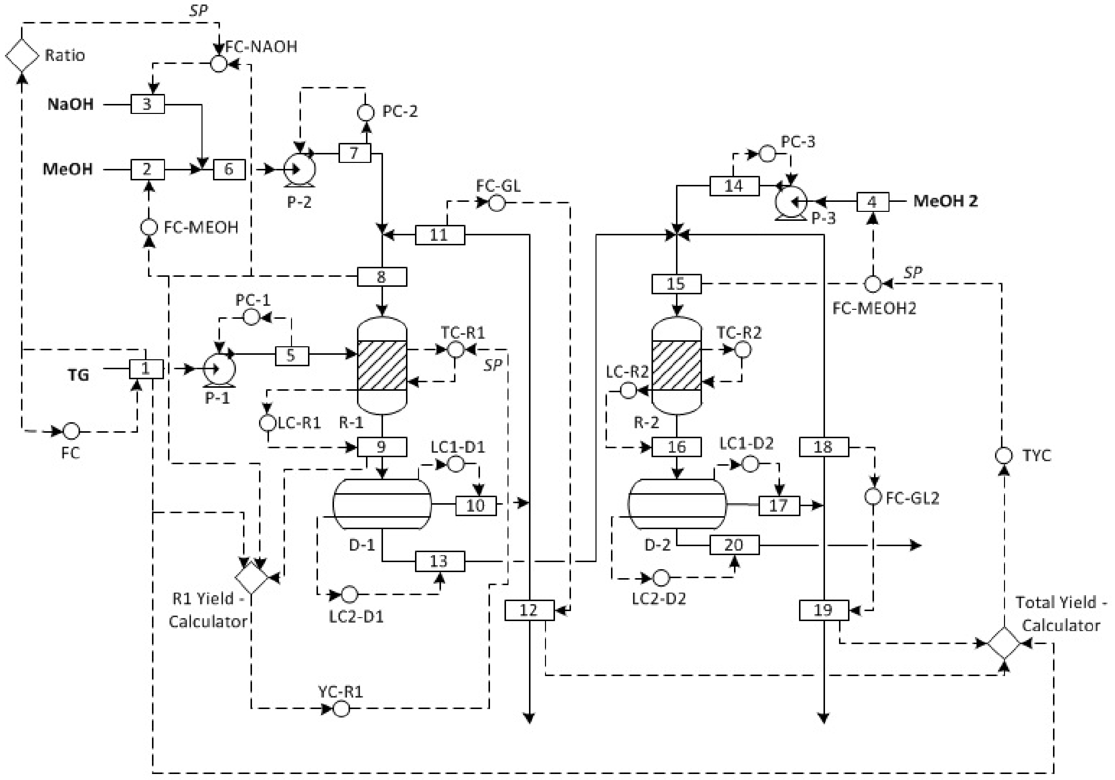


Figure 5.35: CS16 of second reactor in local analysis

extremely slow action by changing its tuning parameters, although the second reactor temperature can reached to its setpoint, the cooling duty still be saturated. Cause of this problem is the process requires heating duty instead of cooling duty at the second reactor to achieve the setpoint of the biodiesel composition controller.

For CS5, if the yield controller is tuned appropriately, all manipulated variables are not be saturated when the setpoint of the fresh triglyceride feed controller is changed to be 9 kmol/h; but the process is required a very long time, which is greater than 300 hours, to approach to a steady-state point; therefore, this structure should not be used.

When the setpoint of the triglyceride controller in CS10 and CS15 is changed to be 11 kmol/h, the manipulated variable of the temperature controller at the second reactor becomes saturated at its upper bound (zero) because of heating duty required to achieve the desired yield of the second reactor.

In case of CS12, CS13, and CS16, to achieve the setpoint of the yield controller at the first reactor when the fresh triglyceride feed is reduced to be 9 kmol/h, the temperature of the first reactor is reduced by its master controller. This makes the temperature of the second reactor cannot be reached to 60 °C although the cooling duty becomes zero. Moreover, for CS13, the biodiesel composition controller at the product stream of the second reactor is also never reached to its setpoint.

For CS4, CS9 and CS14 of the local analysis of the second reactor, the control objective, which is at least 98.5% total yield, is not achieved when the triglyceride setpoint is changed to be 11 kmol/h.

Step 5: *Design the control loops for the rest of the control variables or adding enhanced controls, i.e. cascade, feed forward controls.*

This step is to design the control loops in order to satisfy the operational limitations and the product specifications.

Additional control loops, in this study, are the pressure control for each pump and compressor (controller PC5 to PC10) and inventory control for the uncontrolled inventory vessels to ensure each unit does not overflow and dry up. In this study, the controllers installed to vessels, which their liquid levels do not be controlled, are controller LC6 to LC8 at both decanters, controller LC9 at the first flash tank, controller LC10 at the first distillation column, and controller LC11 at the third reactors; for the pressure, 2 control loops, PC3 and PC4, are designed to adapt the pressure of the first distillation column and the first flash tank. Moreover, to satisfy the some objectives in the process design which are 1%wt of sodium hydroxide to triglyceride fed to first reactor, which is the appropriate ratio between catalyst and oil reactant, and no sodium hydroxide in the stream 50 in order to hold the composition of sodium hydroxide in stream entered to second vacuum distillation column for neutralized the by-product stream, setpoints of the fresh sodium hydroxide and fresh carbonic acid feed controllers designed in step 3.1 are adjusted to be signals of flow rate sensor located at the fresh triglyceride feed and the sodium hydroxide flow rate sensor located at third reactor inlet stream, respectively. The alternative structure designed in step 4.1 is the temperature control of the washing water by adjusting the setpoint of the fresh water feed controller (TC3 in CS6 shown in figure 5.41); thus, the additional loop for this case is including the flowrate of the hot stream fed to the heat exchanger HX-2 (Stream 39) as shown in figure 5.41.

Step 6: *Energy management via heat exchanger networks.*

In this step, the heat exchanger network is designed if it does not exist to improve energy utilization efficiency. However, in this research, the heat exchanger network is designed while the process is being designed.

Step 7: *Optimize economics and improve control performance.*

This step is to optimize the process if it does not be performed and to improve the control performance by tuning the controllers. However, to make the process operate dynamically without the constraint violation, the operating condition may be backed-off from the optimized condition.

Although, the optimization is performed in previous section but the controller tuning is not. The control loops designed in each step, which are only controllable (CS1, CS4 and CS5 in the local analysis of the first reactor and CS1, CS2, CS6, CS7 and CS11 in the second rector local analysis), are combined together into control structures of the process shown in figure 5.36 to 5.40 and then each controller is tuned; the tuning parameters of each controller are shown in Appendix in note that composition sensors have deadtime for 3 minutes in detection of the composition, the

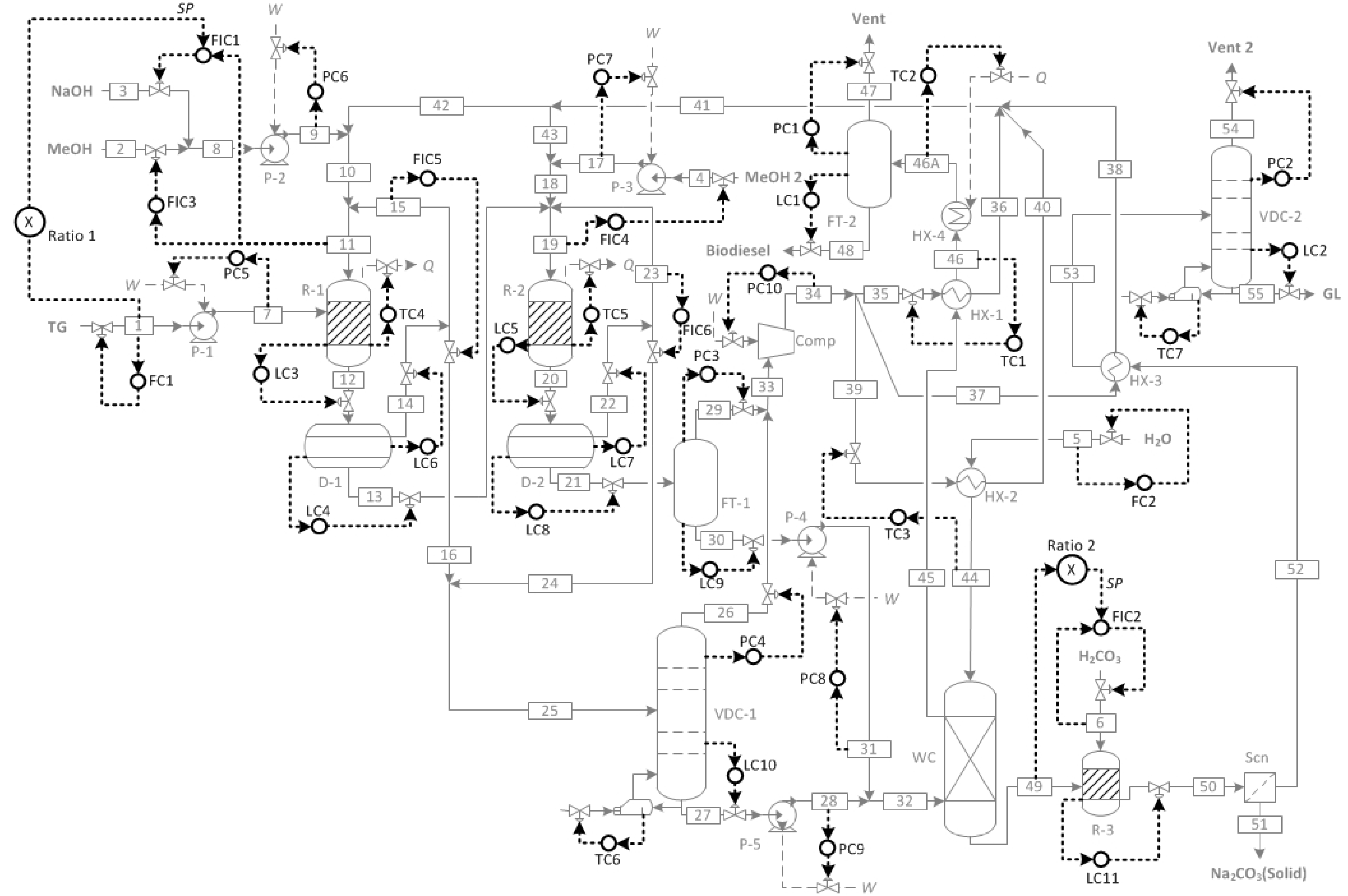


Figure 5.36: CS1 of the biodiesel production process

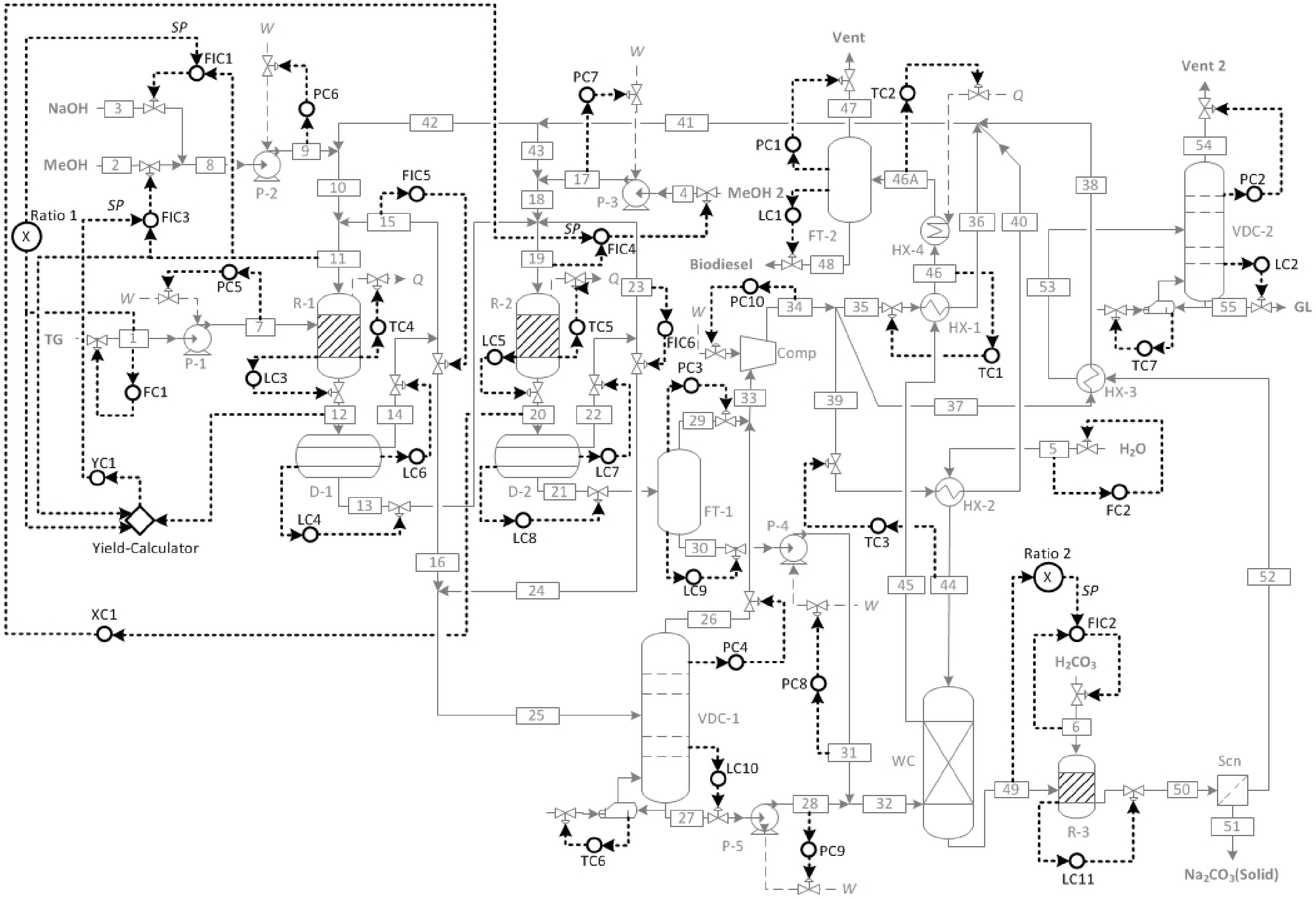


Figure 5.37: CS2 of the biodiesel production process

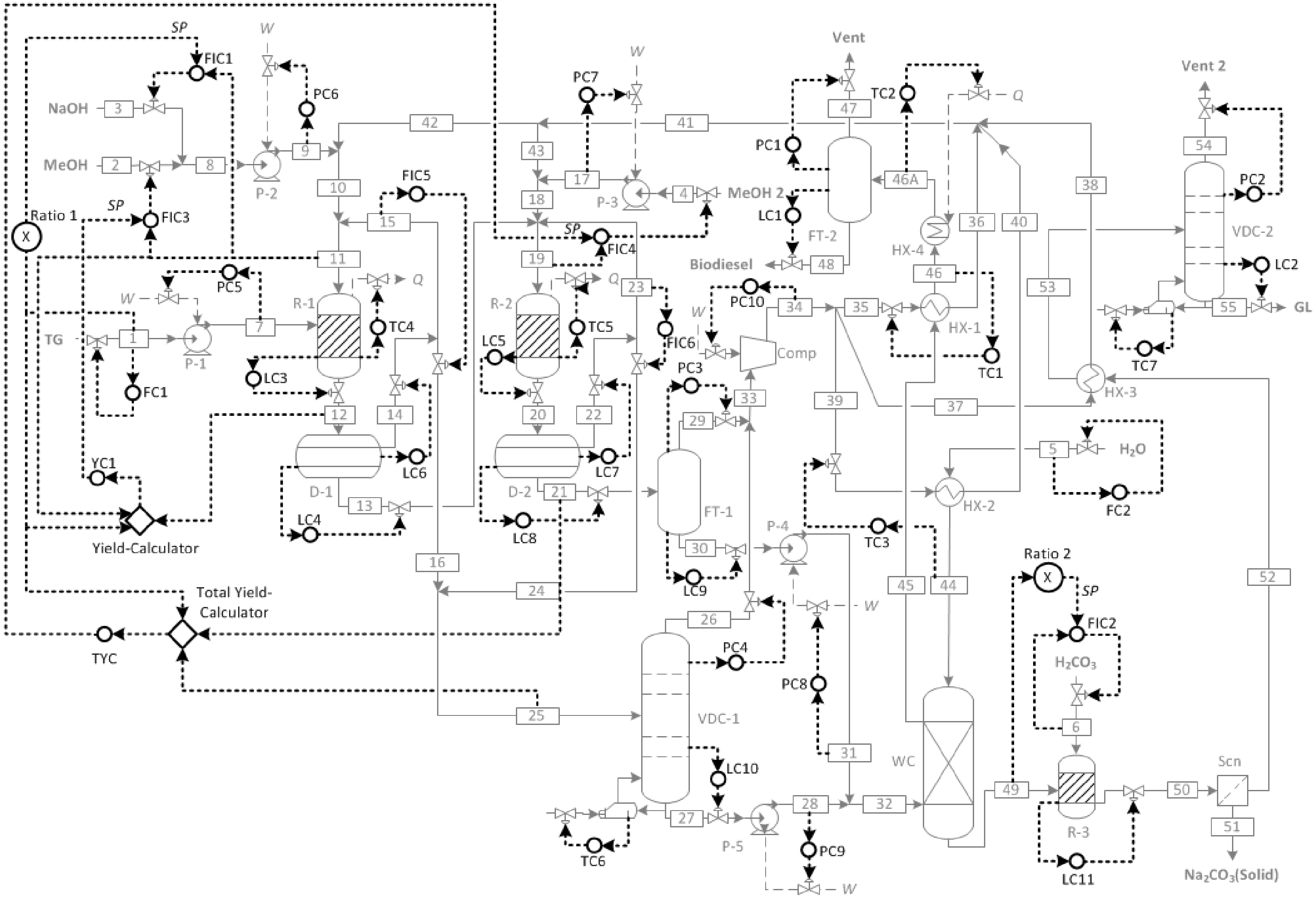


Figure 5.38: CS3 of the biodiesel production process

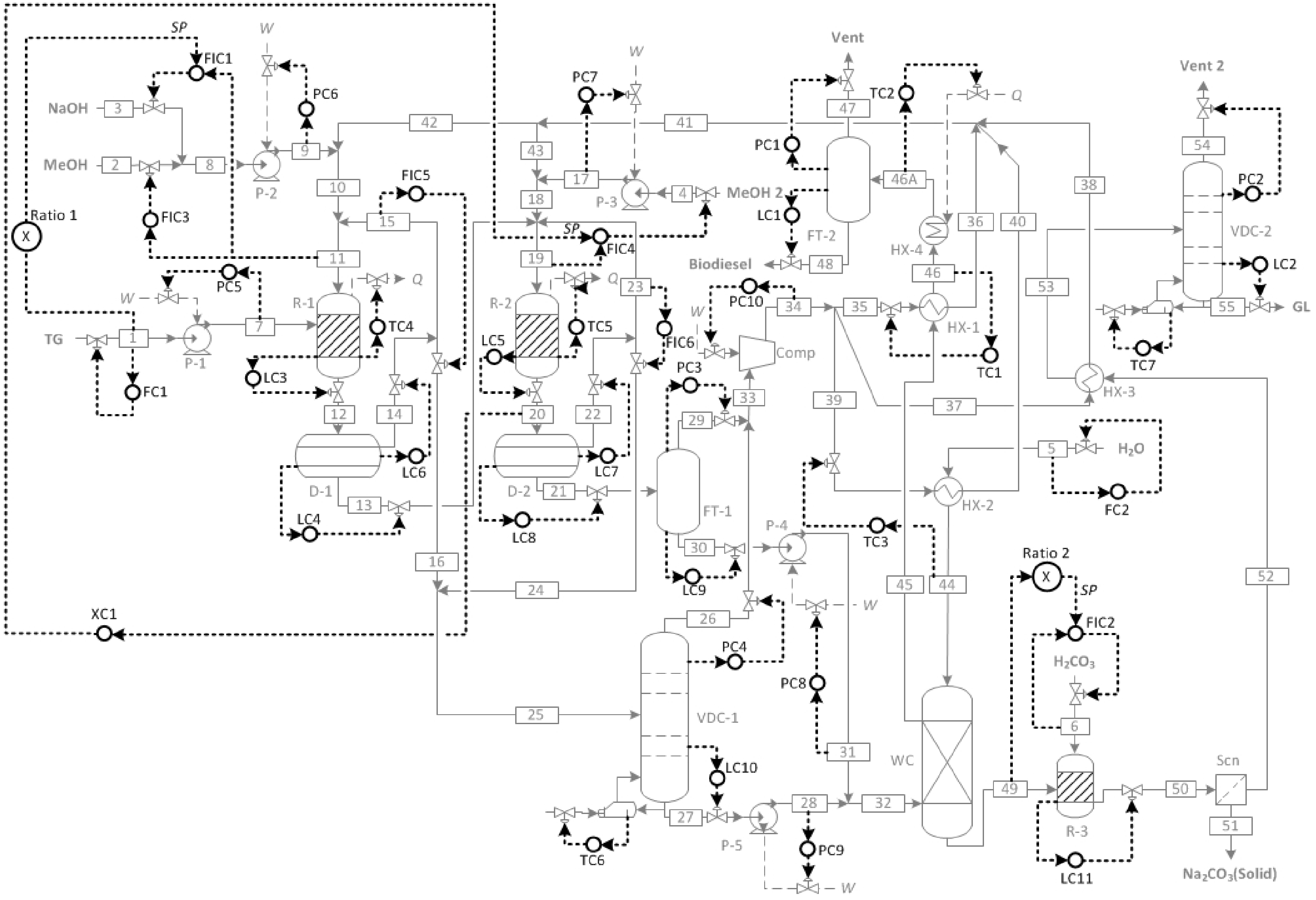


Figure 5.39: CS4 of the biodiesel production process

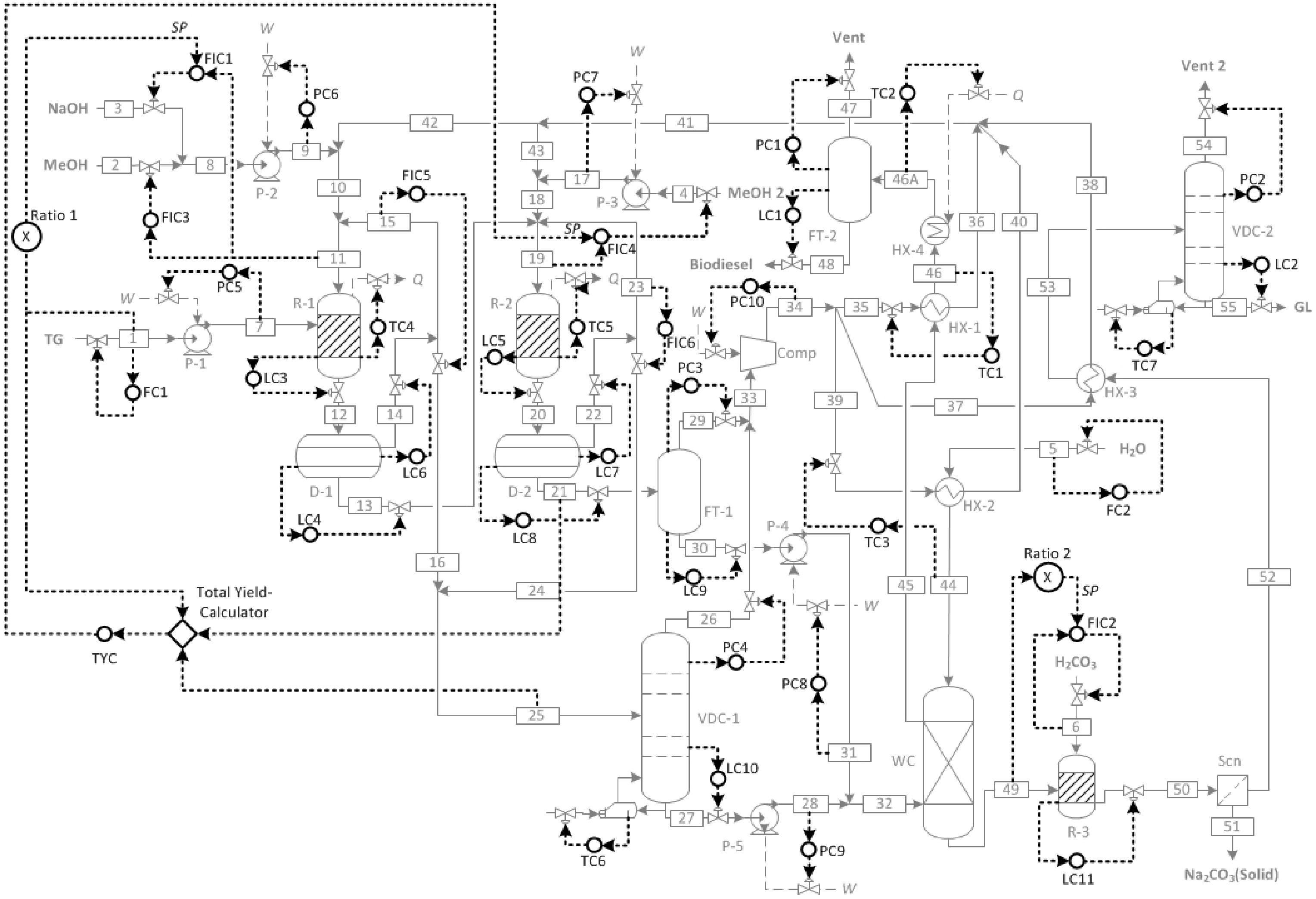


Figure 5.40: CS5 of the biodiesel production process

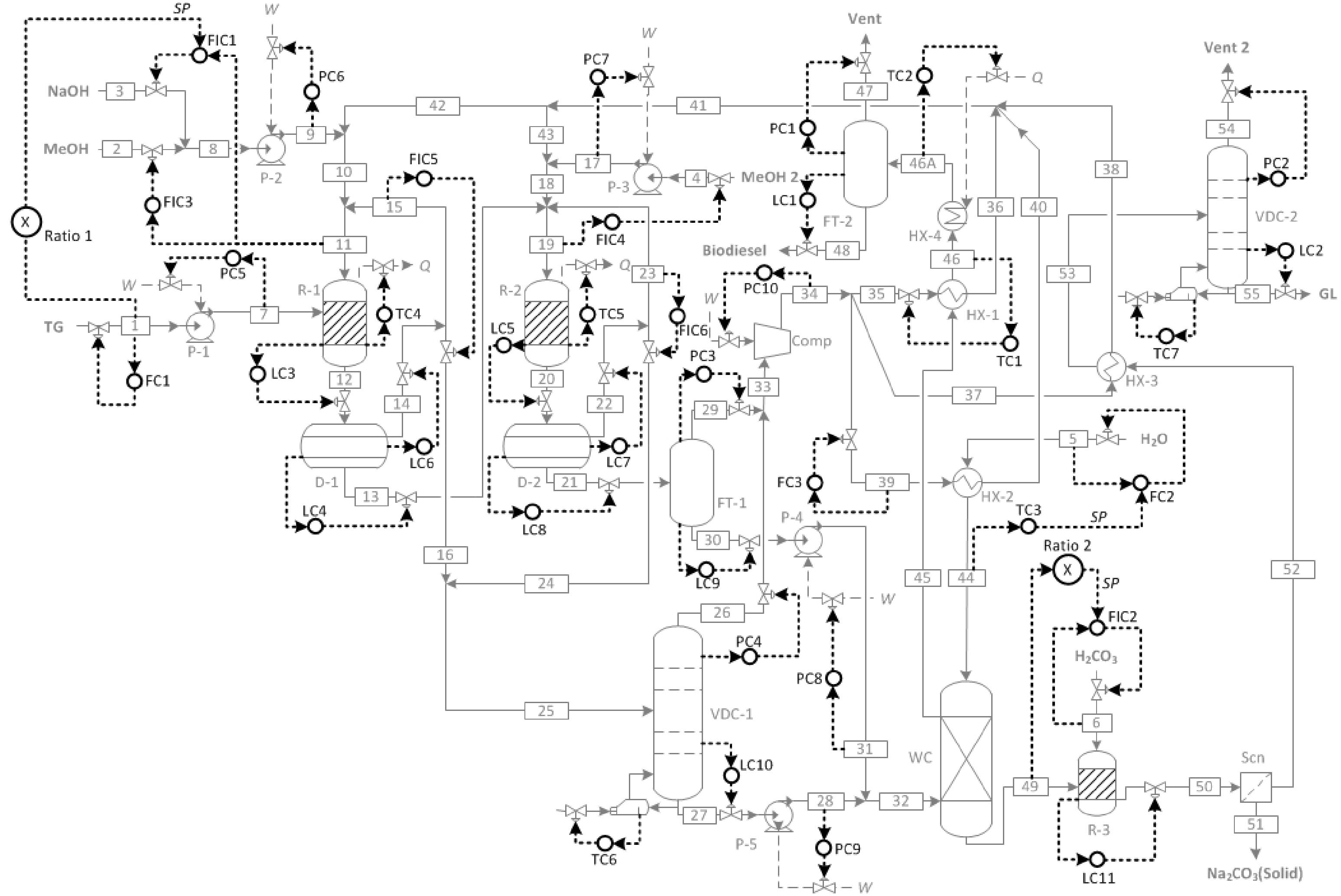


Figure 5.41: CS6 of the biodiesel production process

temperature sensors in any liquid stream have two 0.5 minutes lag in measurement, and component flow rates including the yield and the total yield, which are the process variables in many controllers, are calculated from signals of the composition and the flow rate sensors.

Step 8: *Validate designed control structures by rigorous dynamic simulation.*

The designed control structures shown in figure 5.36 to 5.41 are validated by test the setpoint changes and/or the disturbances. The setpoint changes and the disturbances considered for validation of the control structures in this research are shown in the table 5.7; the responses of the process with each control structure are shown and discussed in next section.

Table. 5.7: Setpoint change and disturbance

No.	Type	Description	Change
T1	Setpoint change	Triglyceride feed rate	± 10%
T2	Disturbance	H_2O mole fraction in fresh H_2CO_3 feed	+0.5

5.4 Dynamic Operating Responses

In order illustrate the dynamic response of each designed control structure shown in figure 5.36 to 5.41, a setpoint change and a disturbance, the triglyceride feed rate and the composition of the water in the fresh carbonic acid feed, are applied 5 hours after the beginning of each simulation run to test the process with each control structure in the dynamic simulation in note that the temperature sensors have two 0.5 minute lag in measurement; the composition sensors have deadtime for 3 minute in detection; the component flow rates are calculated from signals of the composition and the flow rate sensors. The workable control structure must make all controlled variables achieved its setpoints as smoothly and quickly as possible while all manipulated variables must not be saturated; in addition, the constraints for control and product specifications must not violate its limits. There is the note that not all of the controller responses are presented in this context, only total fresh methanol feed, production rate and purities of the product and the by-product, and transesterification yield are displayed.

5.4.1 Change in the Setpoint of Fresh triglyceride feed controller

The fresh triglyceride feed is manipulated by changing the setpoint of the fresh feed controller to 9 (-10%) and 11 (+10%) kmol/h as shown in figure 5.42 to check which designed control structures can handle the process and deal the production rate changing.

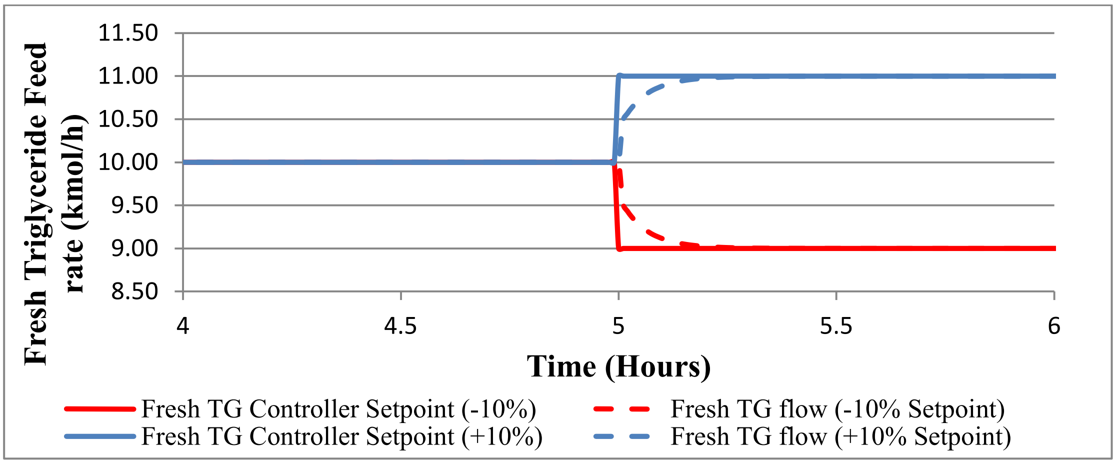


Figure 5.42: Setpoint of fresh triglyceride feed and its response

Although, as shown in figure 5.43, the methanol requirement fluctuate while the operation is in transient (shift from old steady-state condition to the new point) due to the different control policy at the reaction section, but, at the new steady-state condition, the processes with all control schemes are consumed the fresh methanol very similarly.

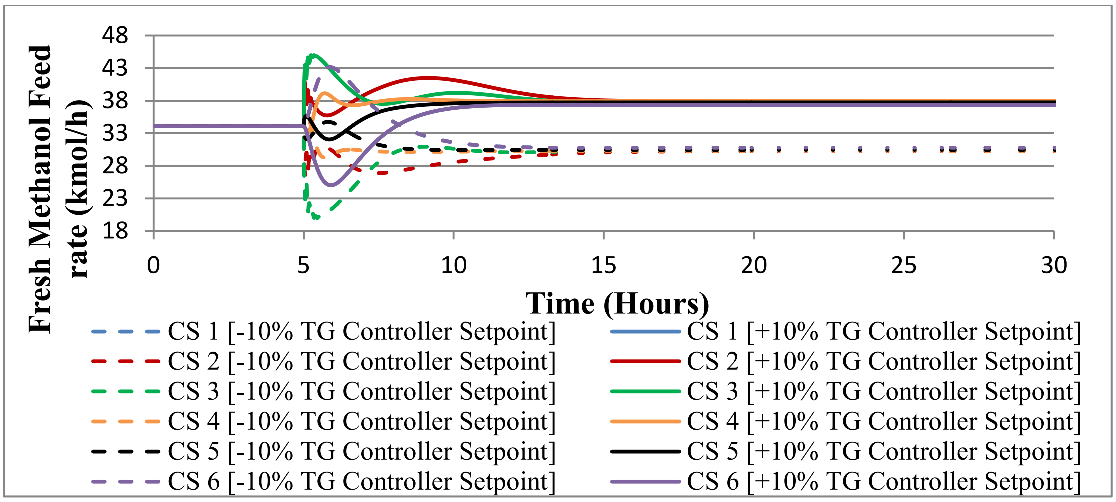


Figure 5.43: Methanol required

For the yield, the figure 5.44 is shown that the yield responses of each control structure are quite similar and can be reached to the objective in the control structure design; because the similar yield response, the production rates of the product and by-product for each control structure are also similar as shown in figure 5.45 and 5.46, respectively.

It can be seen from figure 5.47 to 5.50 that, at new steady-state point, the product impurities except the biodiesel mass fraction for each control structure are quite similar and do not change significantly from the initial points. The reason is that

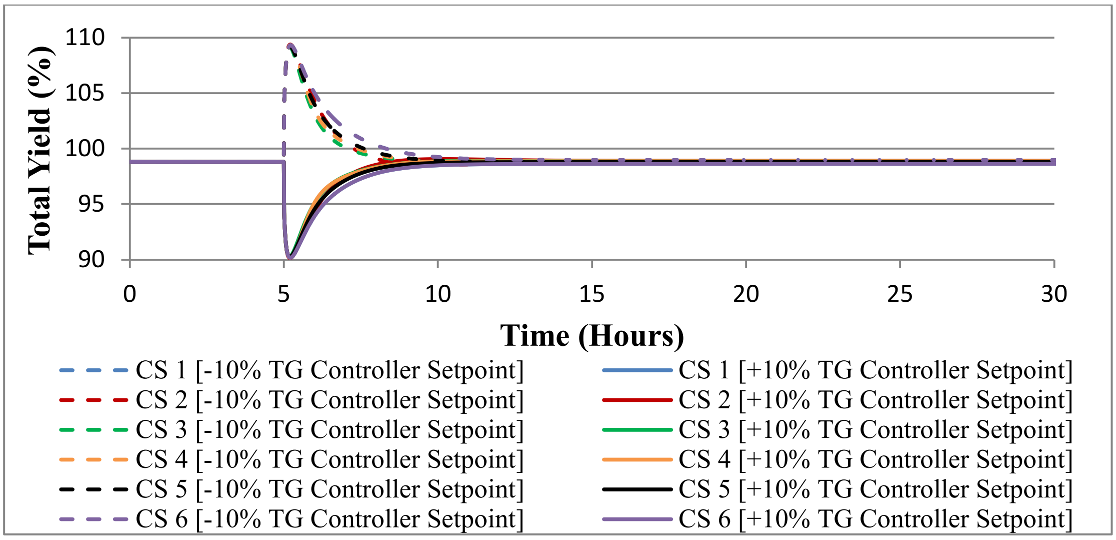


Figure 5.44: Yield

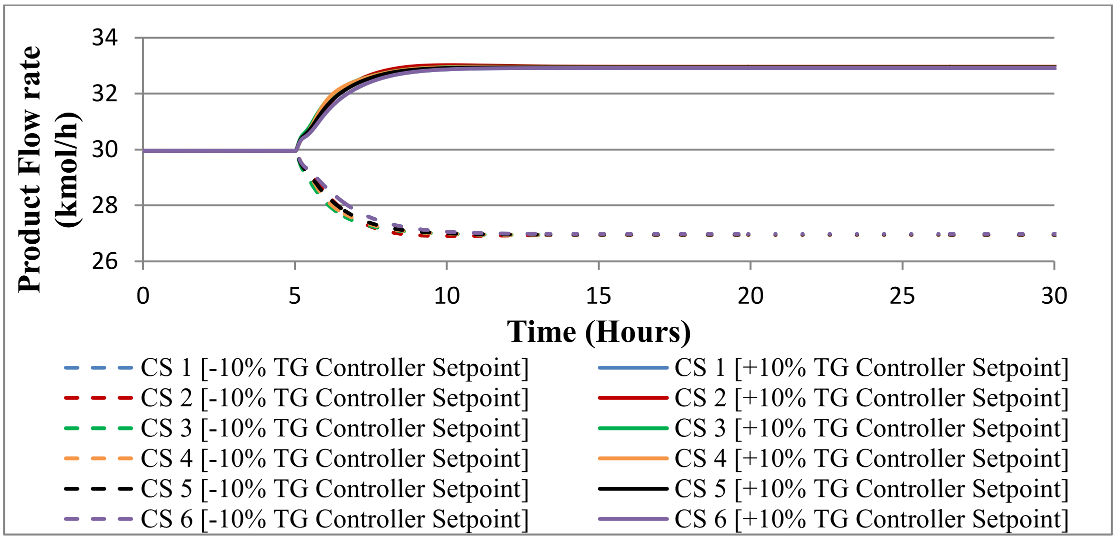


Figure 5.45: Product rate

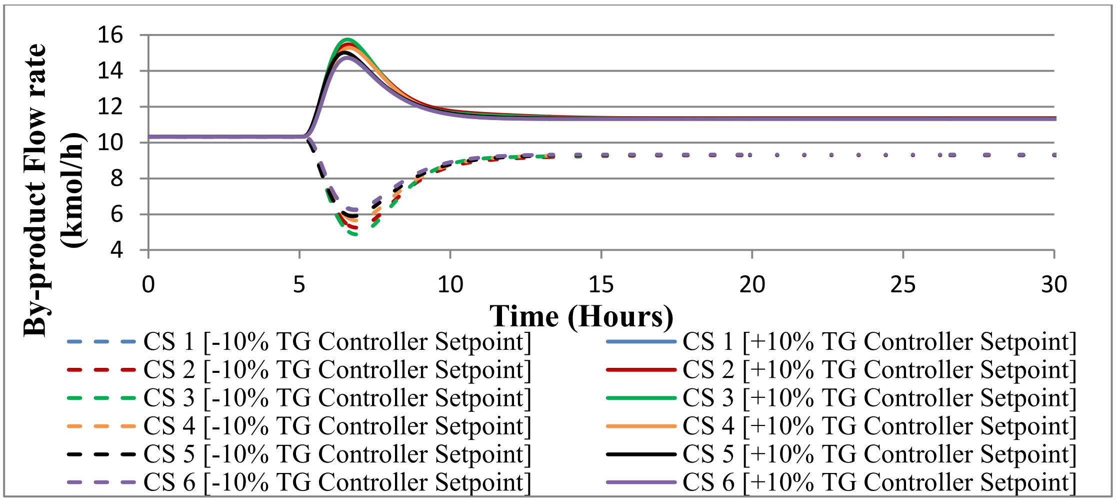


Figure 5.46: By-product rate

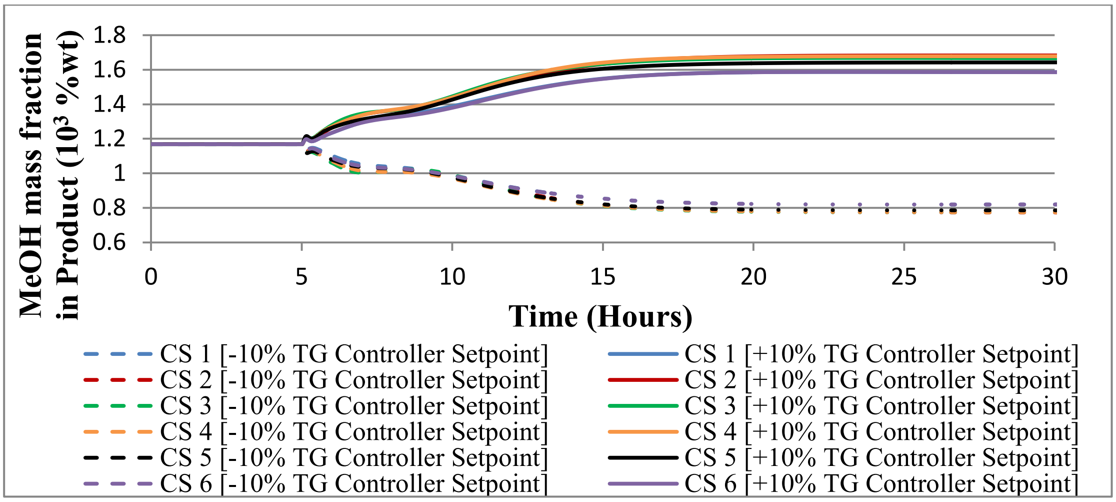


Figure 5.47: Methanol content in the product

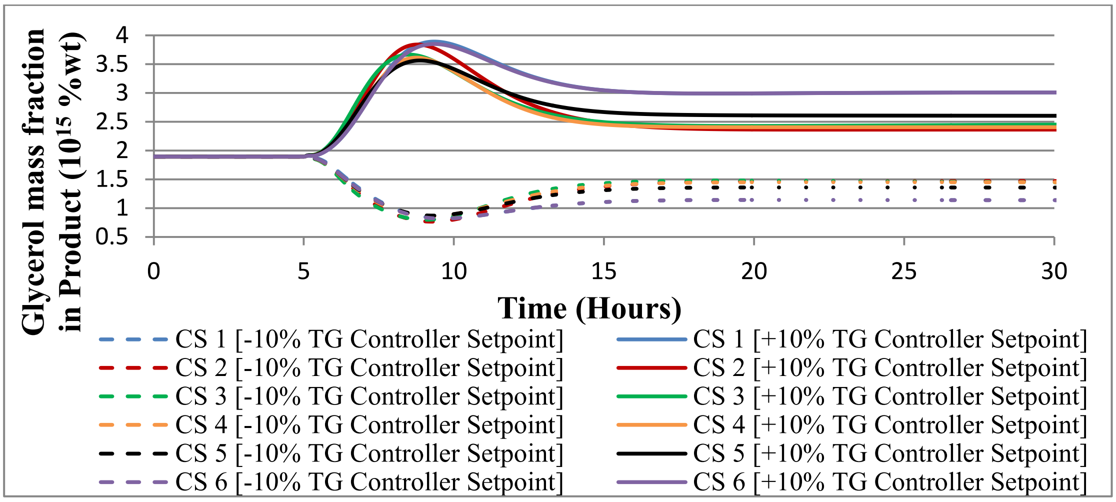


Figure 5.48: Glycerol mass fraction in the product

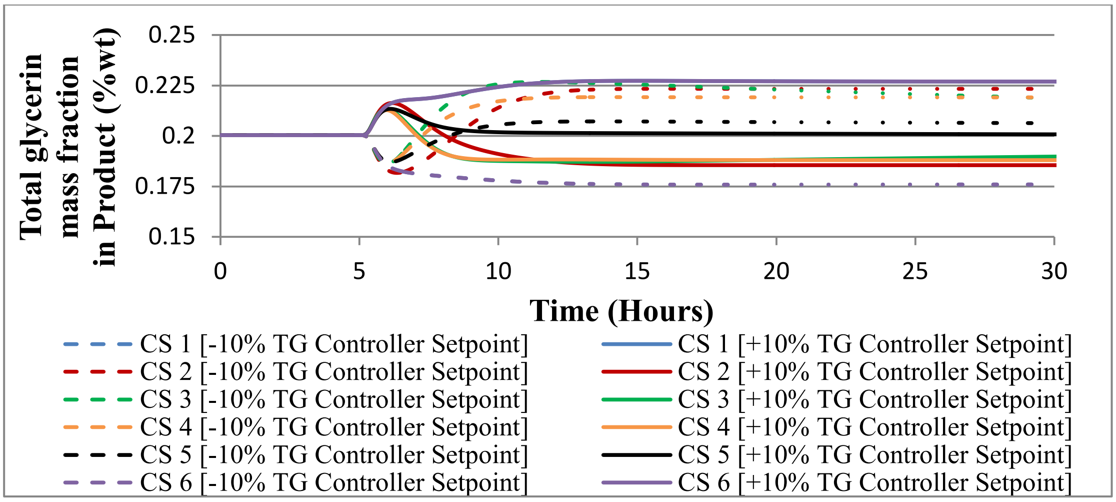


Figure 5.49: Total glycerin in the product

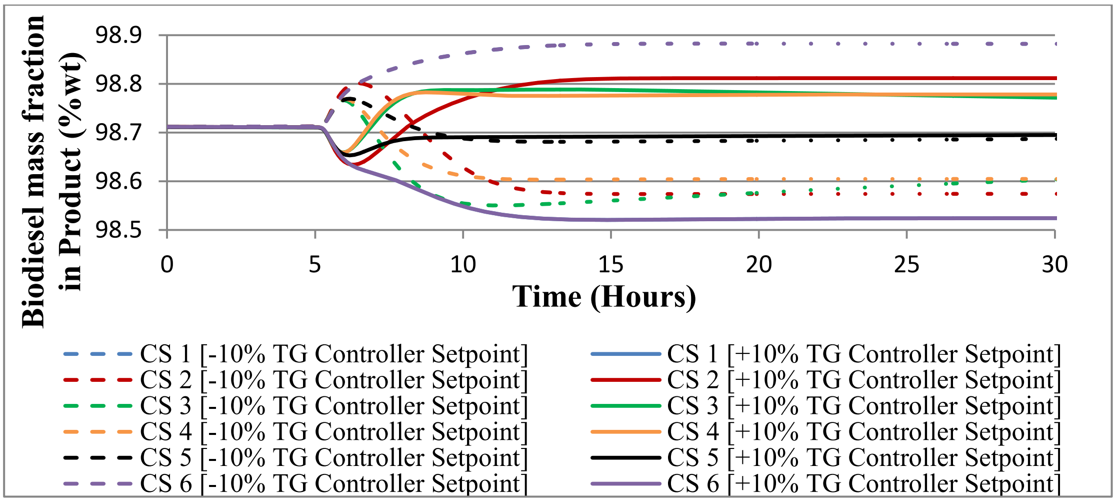


Figure 5.50: Biodiesel purity in the product

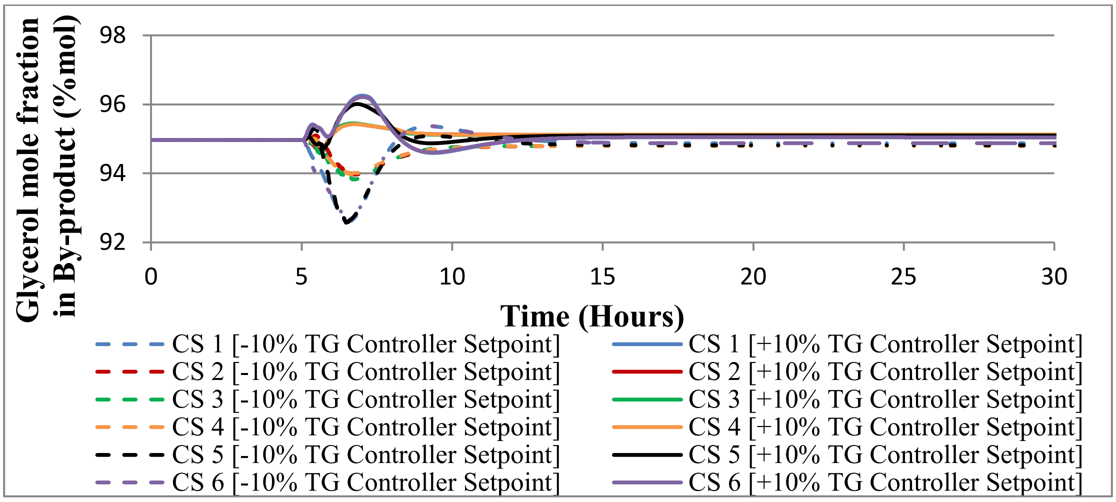


Figure 5.51: By-product purity

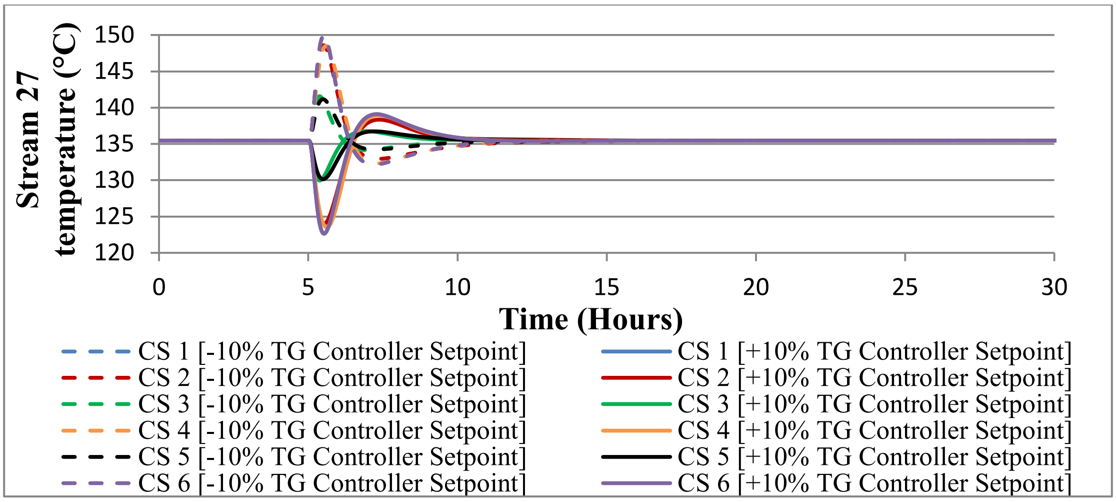


Figure 5.52: Stream 27 temperature

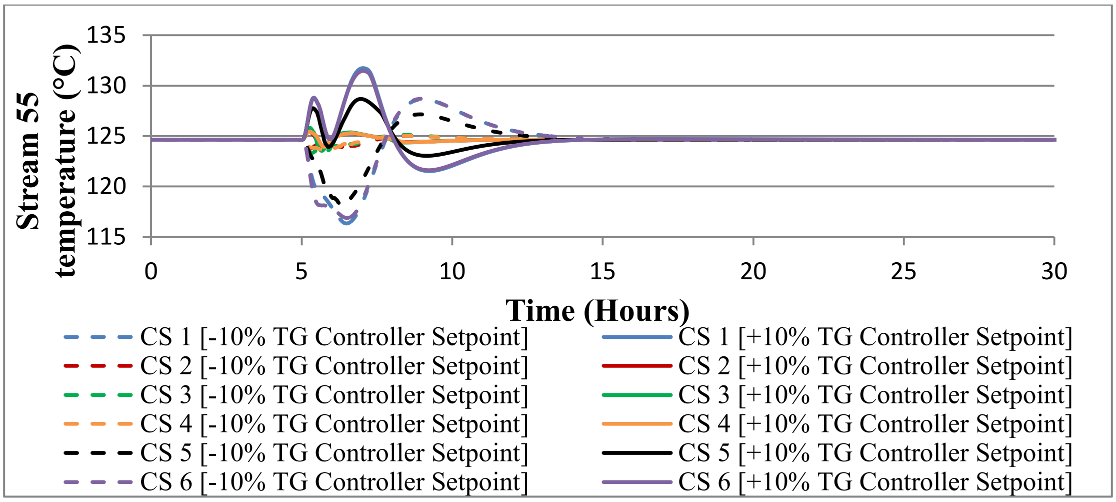


Figure 5.53: Stream 55 temperature

the boiling points of the methanol and the water contaminates are extremely different with the biodiesel boiling point so it is easy to separate most of both components from the biodiesel at the second flash tank, the water is the very good solvent using to extract the glycerol from the biodiesel. For the biodiesel mass fraction, the responses of each control scheme are little different expect for CS1 and CS6; the cause of this is that, although the achieved yields for CS1 and CS6 is not different so much from each other, it give the large effect on the biodiesel purity because the conversions of the triglyceride including the diglyceride and the monoglyceride at the reaction section are quite different. It is seem that CS5 can deal with the biodiesel purity problem excellently; although, in the transition to new steady-state, CS3 and CS4 can handle the biodiesel purity better. However, all control structure can be control the product within the specifications. For by-product, all control structure can handle the by-product purity in the limitation as shown if figure 5.51, but the purity response for CS1, CS5 and CS6 overshoot in transition to new steady-state due to the poor temperature control at the second vacuum distillation column.

Figure 5.52 and 5.53 are shown that the temperatures of both columns, which one is the maximum in the process, do not exceed the glycerol degradation temperature 150 °C; but the temperature responses of CS1, CS2, CS4 and CS6 for the first distillation column and CS1, CS5 and CS6 for the second distillation column oscillate highly; however, there are many causes of this; for example, the response of other temperature controller, or the flow rate fed to the unit.

5.4.2 Water composition Disturbance in Fresh Carbonic acid feed

The mole fraction of the water in the fresh carbonic acid feed is suddenly changed from 0 to 0.5 (+50%) as shown in figure 5.54 to check whether the by-product quality (mole fraction of glyceride) is in its specification.

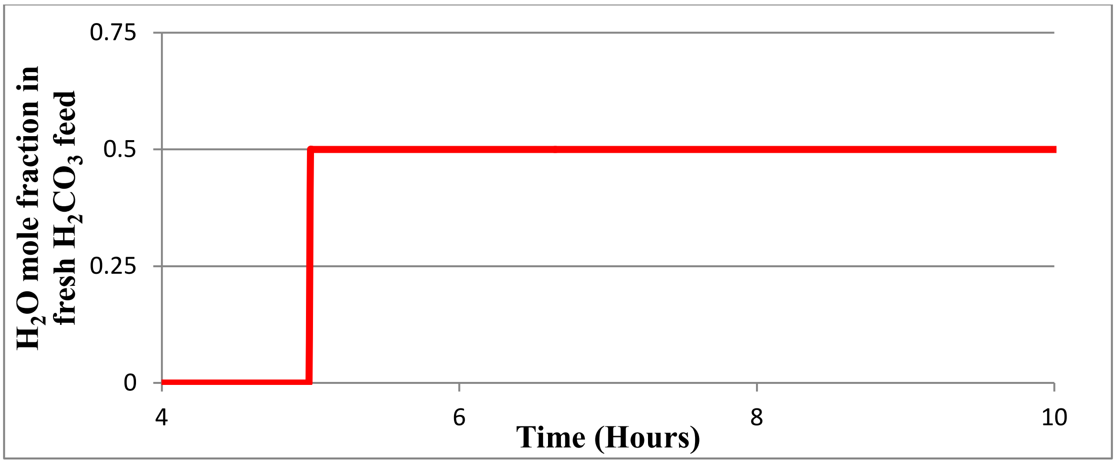


Figure 5.54: Mole fraction of water in fresh carbonic acid feed

The simulation result shows that the by-product purity decreases slightly as shown in figure 5.55 while the by-product flow rate shown in figure 5.56 increases insignificantly, since, at the second vacuum distillation column, the reboiler temperature is controlled instead of glycerol composition in by-product stream so the by-product purity including its flow rate may sometimes be returned to its initial values.

For the temperature constraint at the second distillation column, the temperature of the stream 55 is also slightly changed and controlled back to its temperature setpoint readily as shown in figure 5.57.

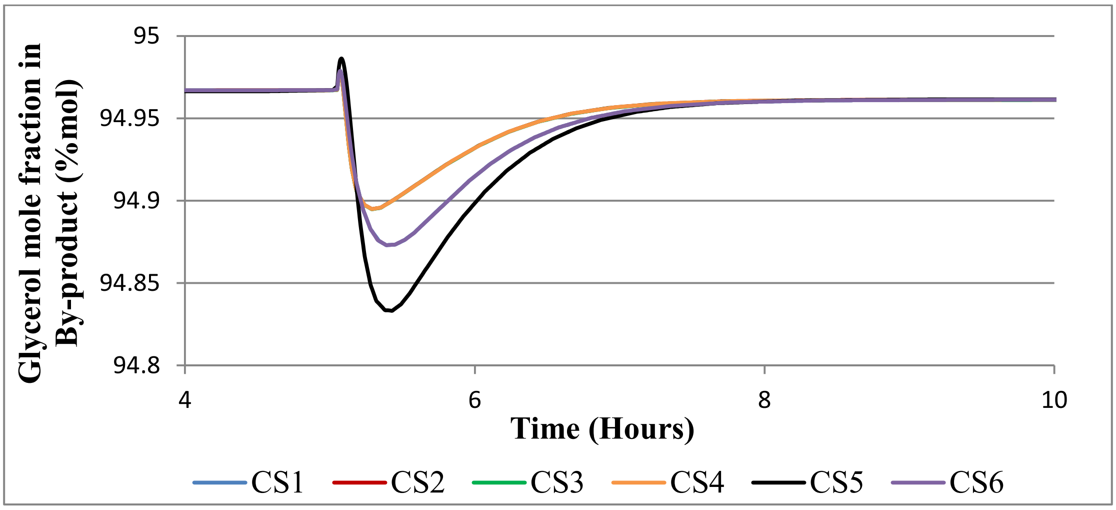


Figure 5.55: By-product purity (when water mole fraction in fresh carbonic feed is changed to 0.5)

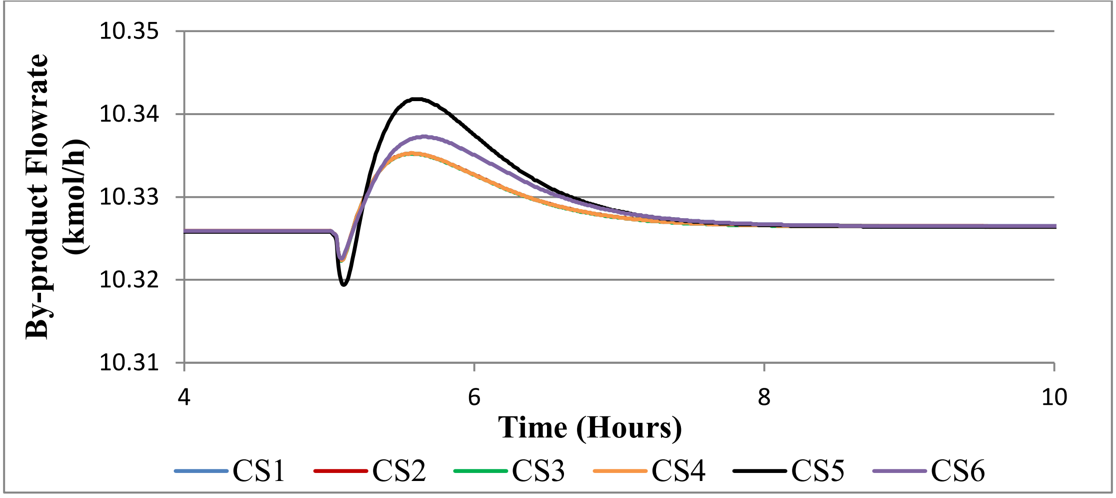


Figure 5.56: By-product rate (when water mole fraction in fresh carbonic feed is changed to 0.5)

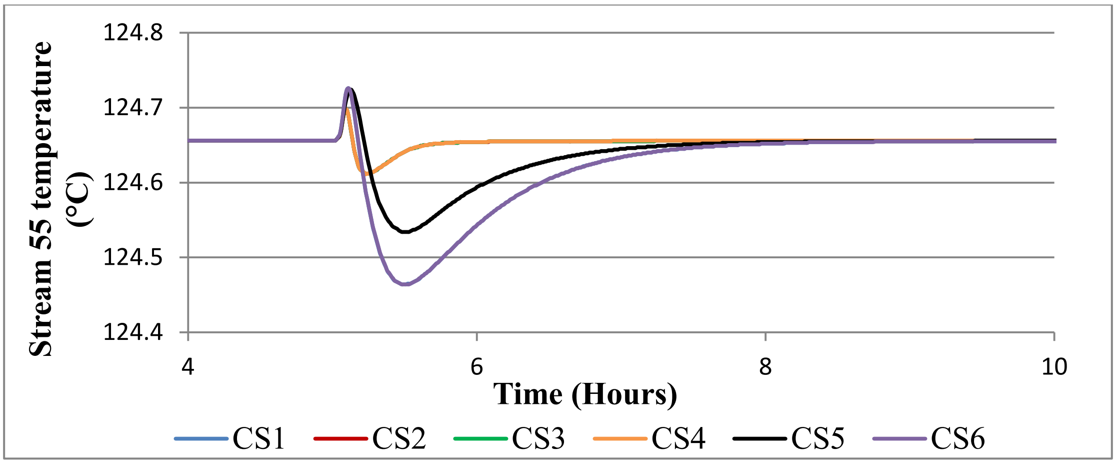


Figure 5.57: Stream 55 temperature (when water mole fraction in fresh carbonic feed is changed to 0.5)

5.5 Control structure Performance Evaluation

An important of a controller is to reduce the difference between its controlled variable and its setpoint or the error to zero as quickly as possible; the most common criterion used to determine how fast of the controlled variable converged to its setpoint over the whole range of time is integral error. One of the three most popular integral error criterions (IAE, ISE, and ITAE) is integral absolute error (IAE) which a quantity has shown the performance of a controller in minimizing the error. However, in the plantwide control, there exist a number of controllers in the plant combining into a control structure which each controller has different importance, i.e., a controller related to the safety in the operation should be emphasized mostly; to compare the control performance of each control structure, in this research, the weighted (and also normalized) integral absolute error (WIAE) for each structure is determined and compared to the others.

To calculate the WIAE for each structure, all controllers are classified into six types: flow-, pressure-, level-, temperature-, composition-, and yield- (including total yield) controls because IAE of the same type controller (same dimension) should be normalized and weighted by the same value. Then, the IAE of each controller under each considered disturbed condition is calculated from equation (5.5) given following:

$$IAE = \int |e(t)| dt \tag{5.5}$$

Then, as said above, the IAEs of the same type controller should be normalized by the same value because it is sometimes not appropriate if the IAE of each controller is normalized by the range of its process variable. For example, in case of 2 composition controllers which one have the large IAE and the huge process variable range (difference between maximum and minimum value of the controlled variable when the setpoint changes and/or disturbances arise) and the other have small in both values; there is nothing to ensure the normalized IAE of the first controller will be larger than it of the other. In this research, the number using for IAE normalization, which can be found from the simulation results, is the difference between maximum and minimum value of the process variables in the controller group. Because of the same value used for normalization of the IAE in the same controller group, the summation of normalized IAEs for each controller group can be calculated easily from equation (5.6):

$$(normalized\ IAE)_i = \frac{\sum IAE_i}{(n_i)(PV_{max,i} - PV_{min,i})} \tag{5.6}$$

In the summation of normalized IAEs calculation, a number of the controller in the considered controller group is also taken into account because each control structure may have the number of the controller in a controller group unequally. For example, there are the five and the ten flow-controllers in the first and the second control structures, respectively; therefore, if the number of the controller in the flow control group is not considered in the calculation of the summation of the normalized IAEs, the summation of the normalized IAEs of the second control structure may often be huger than it of the other although all controllers in the second control structure have tightly control (small IAE) than those in the other.

Table 5.8: Weighed factor for each controller group.

Control loop	Weighted factor	Control loop	Weighted factor
Flow-control	0.05	Level-control	0
Pressure-control	0.35	Composition-control	0.05
Temperature-control	0.35	Yield-control	0.2

Moreover, the importance of each control loop is different, i.e., the safety (temperature and pressure) control loop should be mostly emphasized; therefore, in the calculation of the WIAE using equation (5.7), the summation of the normalized IAEs of each controller group are weighted by the different weighted factors shown in table 5.8.

$$WIAE = \sum (W_i \times normalized\ \ IAE_i) \tag{5.7}$$

To compare the designed control structure without prejudice, the control structure having the same control groups should be compared together because, for example, if the CS1 and CS2 in this research, which have 4 and 6 controller groups respectively, are compared directly, the weighted IAE of the control structure (CS2) having higher a number of the control groups is often greater than it of the other (CS1) which have lower a number of the control groups. In this research, the weighed IAEs, which are calculated from the normalized IAEs of the flow-, pressure-, temperature-, level-, and composition-control loops, of the CS2 and the CS4 are compared firstly. Figure 5.58 shows that the control of the CS4 is tighter than CS2.

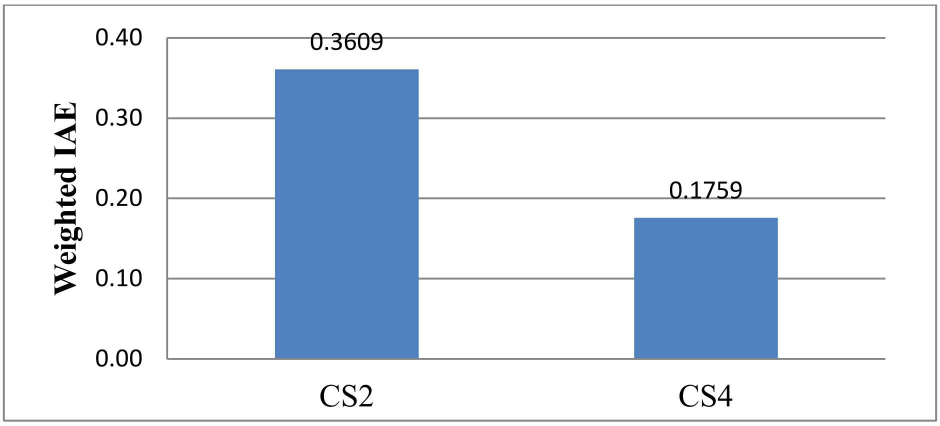


Figure 5.58: WIAE of CS2 and CS4 (Calculated from normalized IAEs of the flow-, pressure-, temperature-, level-, and composition-control loops)

The weighed IAEs, which are calculated from the normalized IAEs of the flow-, pressure-, temperature-, level-, and yield-control loops, of the CS2, CS3, and CS5 are then compared. The result shown in figure 5.59 indicates that the CS3 have the tight control over the others.

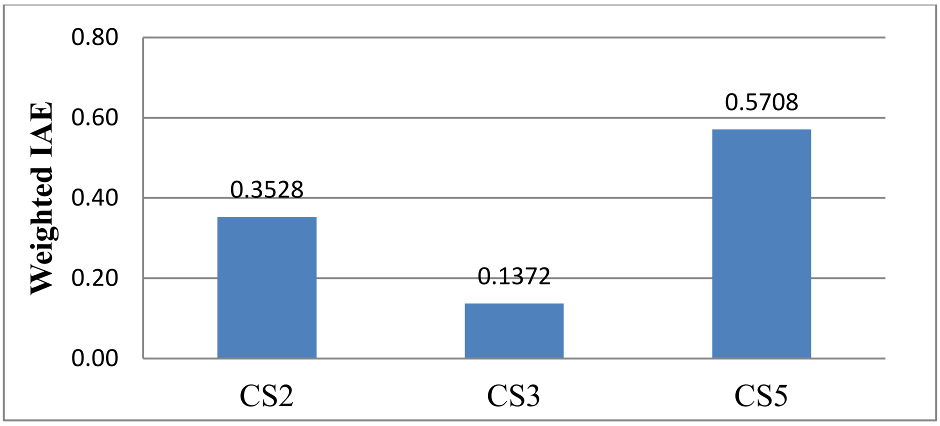


Figure 5.59: WIAE of CS2, CS3 and CS5 (Calculated from normalized IAEs of the flow-, pressure-, temperature-, level-, and yield-control loops)

Finally, the weighed IAE, which is calculated from the normalized IAEs of the flow-, pressure-, temperature-, and level-control loops, of the CS3 is then compared with it of the CS1, CS4 and CS6. As shown in figure 5.60, the CS3 is the control structure that has the maximum control performance.

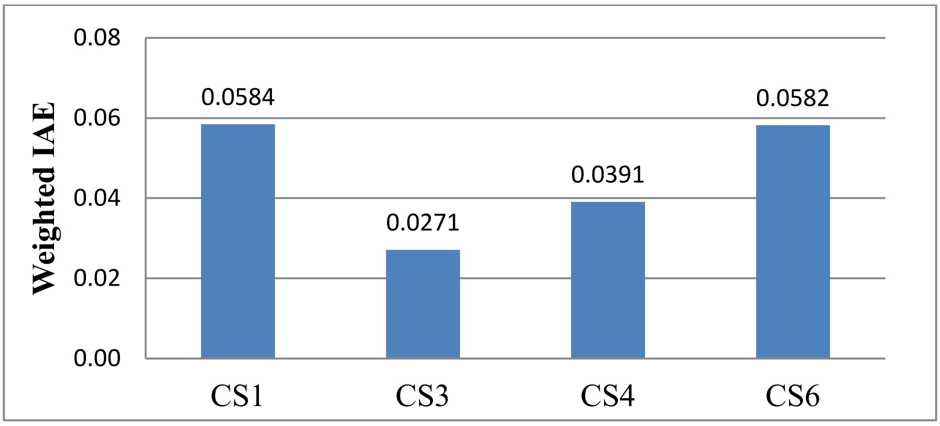


Figure 5.60: WIAE of CS1, CS3 and CS4 (Calculated from normalized IAEs of the flow-, pressure-, temperature-, and level-control loops)

5.6 Utility Requirement

In this section, the real-time required utilities in term of the total equivalent energies for each control structure are shown. Figure 5.61 shows the total equivalent energies when the biodiesel processes with its control structure are under the fresh triglyceride feed changes; and the figure 5.62 shows the required energy when the considered disturbance, the water composition in the fresh carbonic acid feed, arises.

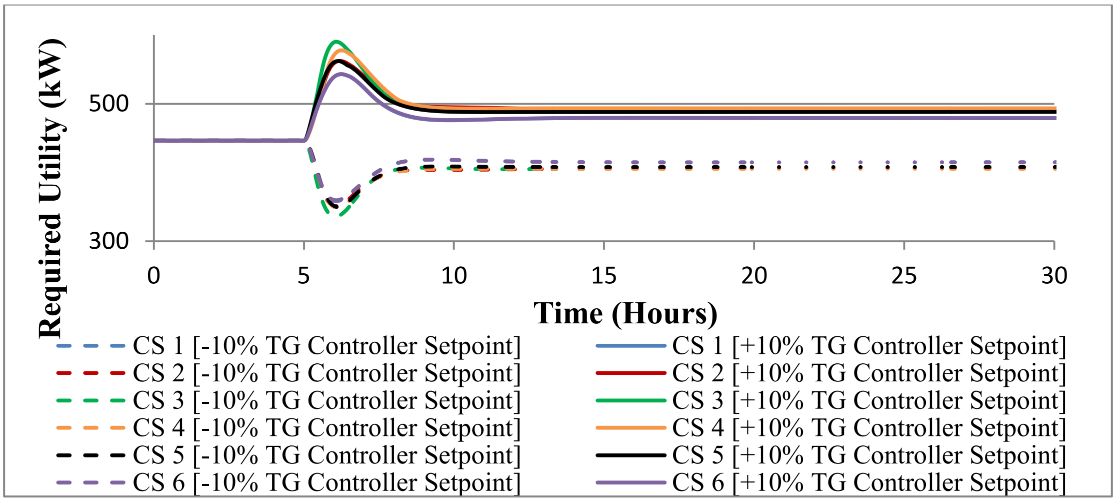


Figure 5.61: Utility requirement in case of triglyceride feed change

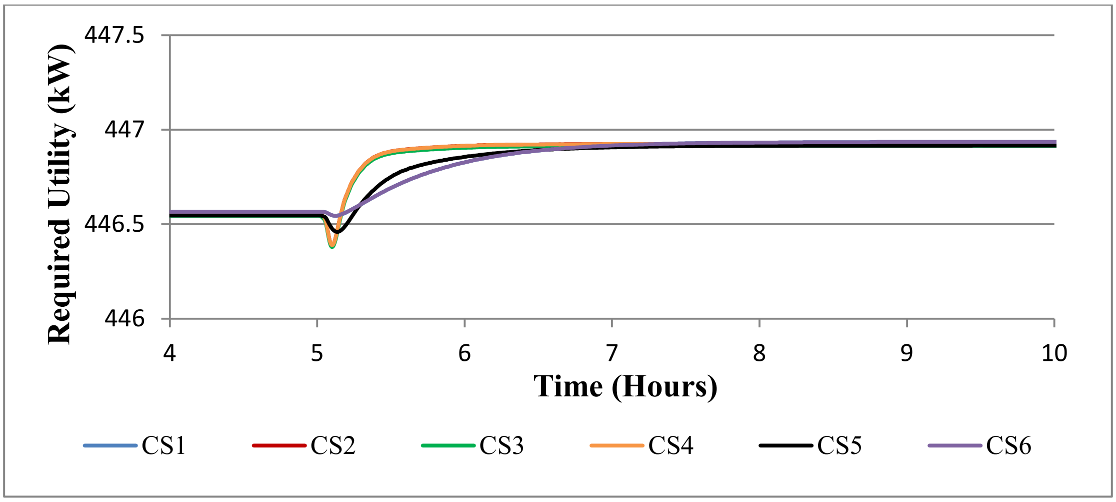


Figure 5.62: Utility requirement in case of water composition in carbonic acid change

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusion

Wongsri's plantwide control structure design procedure consisting of 8-step based on heuristics has been applied to design the plantwide control structure for the new optimized biodiesel production process with back-off to avoid the constraint violation. The Wongsri's procedure can apply easily and be reliable physically; therefore, the control structure designer does not need to have deep process insights to design the plantwide control structure for any process.

Five control structures designed using the procedure of the process can be used to handle the considered setpoint changes and the considered disturbance. Although, the simulation results show all control structure can achieve the objective in the control structure design and make the process not violated the constraints for control, to find the best control structure from those, the weighted integral absolute error for each control structure are calculated and compared to it of the others.

The results of the weighed integral absolute error comparison show the control structure that has the maximum control performance is CS3 which can maintain the process at the desired condition, can reduce the difference between its controlled variable and its setpoint or the error to zero quickly, and can make the process operate safely and efficiently while the product qualities are satisfied within the product specifications.

6.2 Recommendation

The recommendation for further study can be done by focusing on the other available control structures, which are not considered in this research and can be applied to the process; for example, at the step 3.1, fixture plant can also be achieved by the composition-controls (one fresh feed uses to control the flow rate of the stream fed to the reactor, and other fresh feeds including glycerol recycle use to control the composition of the location) instead of the flow-control for each components.

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APPENDICES

APPENDIX A
BASIC CONTROL MODES

In this subject, three basic control modes of feedback control (i.e., proportional, integral, and derivative) are considered.

A.1 Proportional Control

In feedback control, the objective is to reduce the error signal to zero:

$$\boldsymbol{e(t) = y_{sp}(t) - y_m(t)} \tag{A.1}$$

and

$\boldsymbol{e(t)}$ = error signal

$\boldsymbol{y_{sp}(t)}$ = setpoint

$\boldsymbol{y_m(t)}$ = measured value of controlled variable

Although the set point can be varied with time but it is kept constant for long time in many process control problems.

For proportional control, the controller output is proportional to the error signal:

$$\boldsymbol{p(t) = \bar{p} - K_c e(t)} \tag{A.2}$$

where

$\boldsymbol{p(t)}$ = controller output

$\boldsymbol{\bar{p}}$ = bias or steady-state value

$\boldsymbol{K_c}$ = controller output

Main concepts for proportional control are: (1) the controller gain can be adjusted to change the sensitivity of controller output to error signal; (2) the sign of the controller gain can be chosen to make the controller has direct or reverse action. For example, if the controller gain is positive, the controller output is directly proportional to the error signal.

The bias can be adjustable referred to manual reset. If bias is adjusted equal to the controller output when the error is zero, so the controller output and the manipulated variable are at their nominal steady-state values. In fact, the controller can saturate when its output reaches to a physical limit, either maximum or minimum limits.

If controller gain is dimensionless, proportional band setting can be used instead of a controller gain which is defined as:

$$PB \triangleq \frac{100\%}{K_c} \tag{A.3}$$

A disadvantage of the proportional controller is that an offset (or steady-state error) encounters after the setpoint change or sustained disturbance, which can be eliminated by manually reseting either setpoint or bias after an offset appears. Therefore, the proportional control is usually used for applications that offset can be accepted such as liquid level control.

A.2 Integral and Proportional-Integral Control

For integral control, the controller output depends on the integral of error over time:

$$p(t) = \bar{p} - \frac{1}{\tau_i}\int_0^t e(t^*)dt^* \tag{A.4}$$

where an adjustable parameter, τ_i, is also known as the integral time or reset time. Important advantage or integral control is the elimination of offset because of existence of integral term in the controller algorithm.

Normally, the integral control is not usually used by itself because it takes a little control action until the error exists for a long time. Therefore, the proportional-integral control is usually used instead of the integral-only control because proportional control can take a corrective action immediately as soon as the error is detected. An equation which describes the corrective action of proportional –integral control shows below:

$$p(t) = \bar{p} - K_c\left(e(t) + \frac{1}{\tau_i}\int_0^t e(t^*)dt^*\right) \tag{A.5}$$

Important disadvantages of the integral control are that it leads to: (1) oscillation of response because there is too much integral action, (2) reducing the stability of feedback control system, and (3) phenomenon that is known as reset windup, buildup of the integral term in the equations (A.4) and (A.5) becomes quite large while the controller is saturated, this phenomenon usually occurs when PI or PID encounters a large sustained disturbance. Nowadays, commercial controllers provide antireset windup to prevent this phenomenon by temporarily halting the integral control action whenever the controller output saturates.

A.3 Derivative and Proportional-Integral-Derivative Control

Main function of derivative control is to reduce the oscillation in response of PI controller by anticipating behavior of the error by considering its rate of change. Controller output of derivative control is proportional to the rate of change in the error:

$$p(t) = \bar{p} - \tau_d\frac{de(t)}{dt} \tag{A.6}$$

where a parameter, τ_d, is derivative time. Derivative control is never used alone, because there is no corrective action although the error is not equal to zero.

The derivative mode tends to stabilize the control process, thus it is often used to counteract the destabilization tendency of the integral mode.

Although the derivative mode takes action to improve the dynamic response by reducing the process setting time, the time takes the process to reach steady-state, but it is very sensitive to noise in measurement which make the derivative action amplifies the noise. Consequently, derivative mode is seldom used for flow controller because flow control loops respond quickly and flow measurements tend to be noisy.

For proportional-integral-derivative control, the algorithm of the parallel form which is the most popular form of the PID controller is given by:

$$\boldsymbol{p(t) = \bar{p} - K_c\left(e(t) + \frac{1}{\tau_i}\int_0^t e(t^*)dt^* + \tau_d\frac{de(t)}{dt}\right)} \qquad \text{(A.7)}$$

A disadvantage of PID controller is that a sudden change in the *setpoint* causes the derivative term momentarily to become very large and thus provide a *derivative kick* to the final control element. The algorithm of PID controller is modified to eliminate the derivative kick as shown:

$$\boldsymbol{p(t) = \bar{p} - K_c\left(e(t) + \frac{1}{\tau_i}\int_0^t e(t^*)dt^* - \tau_d\frac{dy_m(t)}{dt}\right)} \qquad \text{(A.8)}$$

For more flexibility, the algorithm can be obtained by weighting the setpoint in the proportional term, as well as in the derivative term. This modification eliminates the proportional kick that also occurs after step change in setpoint. The modified algorithm shows below:

$$\boldsymbol{p(t) = \bar{p} - K_c\left(e_p(t) + \frac{1}{\tau_i}\int_0^t e(t^*)dt^* + \tau_d\frac{de_d(t)}{dt}\right)} \qquad \text{(A.9)}$$

with:

$$\boldsymbol{e_p(t) \triangleq \beta y_{sp}(t) - y_m(t)}$$

$$\boldsymbol{e(t) \triangleq y_{sp}(t) - y_m(t)}$$

$$\boldsymbol{e_d(t) \triangleq \gamma y_{sp}(t) - y_m(t)}$$

where $\boldsymbol{\beta}$ and $\boldsymbol{\gamma}$ are nonnegative constant. This algorithm is known as the parallel PID controller with proportional and derivative mode weighting, or beta-gamma controller. To eliminate the proportional kick, $\boldsymbol{\beta}$ is set to zero; to eliminate the derivative kick, $\boldsymbol{\gamma}$ is set to zero.

APPENDIX B

PROCESS AND EQUIPMENT DATA

Table B.1: Equipment Data

Units/Properties		Dimension
Compressor (Comp)		
Polytopic Efficiency	0.90	
Mechanical Efficiency	0.85	
Network Required	172256.39	Watt
Discharge Pressure	400.00	kPa
Decanter (D-1, D-2)		
Configuration	Horizontal	
Length	5.40	meter
Diameter	1.80	meter
Pressure	400.00	kPa
Extraction Column (WC)		
Top stage pressure	101.33	kPa
Number of stages	5	
Stream 44 feed stage	1	
Stream 32 feed stage	5	
Pressure Drop	2.00	kPa per stage
Heat Exchanger (HX1, HX-2, HX-3)		
Flow Direction	Counter-Current	
Minimum Temperature Approach	10.00	°C
Correction Factor	0.80	
Overall Heat Transfer Coefficient	1419.57	Watt/m^2K
Heat exchanged		
HX-1	237313.06	Watt
HX-2	19203.59	Watt
HX-3	220553.59	Watt
Area		
HX-1	2.61	m^2
HX-2	0.18	m^2
HX-3	2.50	m^2
Heater (HX-4)		
Heat exchanged	145294.05	Watt
Pump (P-1 to P-5)		
Pump Efficiency	0.76	
Driver Efficiency	0.85	
Discharge Pressure		
P-1, P-2, P-3	400.00	kPa
P-4, P-5	111.33	kPa

Table B.1 (Continue): Equipment Data

Units/Properties		Dimension
Pump (P-1 to P-5)		
Net work required		
P-1	1252.83	Watt
P-2	75.38	Watt
P-3	100.75	Watt
P-4	454.78	Watt
P-5	33.92	Watt
Reactor (R-1, R-2)		
Type	CSTR	
Configuration	Vertical	
Pressure	400.00	kPa
Temperature	60.00	°C
Residence Time	1.00	hour
Duty		
R-1	-491521.22	Watt
R-2	-19715.17	Watt
Length		
R-1	3.25	meter
R-2	3.1	meter
Diameter		
R-1	5.49	meter
R-2	5.18	meter
Reactor (R-3)		
Type	CSTR	
Configuration	Vertical	
Pressure	101.33	kPa
Length	0.8	meter
Diameter	1.6	meter
Flash Tank (FT-1)		
Configuration	Vertical	
Pressure	10.00	kPa
Length	2.00	meter
Diameter	0.50	meter
Flash Tank (FT-2)		
Configuration	Vertical	
Pressure	10.13	kPa
Length	0.60	meter
Diameter	0.15	meter
Distillation Column (VDC-1, VDC-2)		
Number of Trays	2	
Feed Tray	1	
Top Stage Pressure	10.00	kPa
Pressure Drop	2.00	kPa per stage
Tray Spacing	0.61	meter

Table B.1 (Continue): Equipment Data

Units/Properties		**Dimension**
Distillation Column (VDC-1, VDC-2)		
Diameter		
VDC-1	0.60	meter
VDC-2	0.70	meter
Sump Height		
VDC-1	1.20	meter
VDC-2	1.40	meter
Tray Type	Bubble Cap	
Reboiler Duty		
VDC-1	223796.74	Watt
VDC-2	309322.71	Watt

Note that the polytropic and the mechanic efficiencies of the axial compressor are assumed to be 90% and 85% which are general efficiencies for the most compressors. The overall heat transfer coefficients for each process-to-process heat exchanger are reasonable estimated values used for preliminary heat exchanger design suggested by Douglas (1988). Most of vessels contained only liquid are designed to have 0.25 hours in resident time except for reactors. The sizes of flash tanks, which contain both liquid and vapor, are calculated from its maximum vapor velocity with 5 minutes for liquid inventory using Souders-Brown equation. For sizing of the distillation columns, Douglas (1988) suggested the vapor velocity should be 60% of flooding velocity; therefore, the diameters of both columns are calculated from their flooding velocities and 0.61 meters in tray spacing. The efficiencies for the most pumps are generally 76% for pump and 85% for its motor.

Table B.2: Stream Information for Optimized Process

Name	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Total Flow (kmol/hr)	10.00	14.67	0.19	18.82	39.07	0.10	10.00	14.86	14.86	39.54
Total Flow (kg/hr)	8854.49	470.06	7.71	603.10	703.88	5.98	8854.49	477.77	477.77	1268.59
Total Flow (m^3/hr)	9.76	0.59	0.00	0.76	0.71	0.01	9.76	0.59	0.59	1.64
Temperature (°C)	25.00	25.00	25.00	25.00	25.00	25.00	25.12	24.96	25.04	48.24
Pressure (atm)	1.00	1.00	1.00	1.00	1.00	1.00	3.95	1.00	3.95	3.95
Vapor Fraction	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mole Fraction										
TG	1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000
DG	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MG	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
GL	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
FAME	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MEOH	0.0000	1.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.9870	0.9870	0.9951
NAOH	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	0.0130	0.0130	0.0049
H2O	0.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.2 (Continue): Stream Information for Optimized Process

Name	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**
Total Flow (kmol/hr)	280.18	290.18	34.05	256.13	240.64	15.50	18.82	27.76	296.58	296.58
Total Flow (kg/hr)	16348.3	25202.8	9152.15	16050.8	15079.7	971.12	603.10	889.61	19982.3	19982.3
Total Flow (m^3/hr)	15.03	26.49	11.20	14.36	13.49	0.87	0.76	1.14	23.87	23.36
Temperature (°C)	61.83	60.00	63.23	63.23	63.23	63.23	25.08	37.09	58.23	60.00
Pressure (atm)	3.95	3.95	3.95	3.95	3.95	3.95	3.95	3.95	3.95	3.95
Vapor Fraction	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mole Fraction										
TG	0.0000	0.0024	0.0201	0.0000	0.0000	0.0000	0.0000	0.0000	0.0023	0.0001
DG	0.0000	0.0030	0.0252	0.0000	0.0000	0.0000	0.0000	0.0000	0.0029	0.0004
MG	0.0009	0.0031	0.0185	0.0011	0.0011	0.0011	0.0000	0.0000	0.0025	0.0006
GL	0.4311	0.4431	0.0010	0.5019	0.5019	0.5019	0.0000	0.0000	0.1251	0.1317
FAME	0.0002	0.0884	0.7514	0.0002	0.0002	0.0002	0.0000	0.0000	0.0881	0.1016
MEOH	0.5599	0.4524	0.1820	0.4884	0.4884	0.4884	1.0000	1.0000	0.7771	0.7636
NAOH	0.0079	0.0076	0.0018	0.0084	0.0084	0.0084	0.0000	0.0000	0.0020	0.0020
H2O	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.2 (Continue): Stream Information for Optimized Process

Name	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
Total Flow (kmol/hr)	49.37	247.22	234.76	12.46	27.95	17.81	10.14	10.14	15.80	33.56
Total Flow (kg/hr)	9514.74	10467.5	9940.14	527.45	1498.57	570.88	927.69	927.69	506.46	9008.28
Total Flow (m^3/hr)	11.33	11.56	10.97	0.58	1.44	4527.76	0.80	0.79	4033.41	10.44
Temperature (°C)	62.42	62.42	62.42	62.42	63.07	33.07	137.85	138.04	34.32	34.32
Pressure (atm)	3.95	3.95	3.95	3.95	3.95	0.10	0.12	1.10	0.10	0.10
Vapor Fraction	0.00	0.00	0.00	0.00	0.00	1.00	0.00	0.00	1.00	0.00
Mole Fraction										
TG	0.0008	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0012
DG	0.0023	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0034
MG	0.0010	0.0005	0.0005	0.0005	0.0008	0.0000	0.0023	0.0023	0.0000	0.0015
GL	0.0005	0.1579	0.1579	0.1579	0.3486	0.0000	0.9611	0.9611	0.0000	0.0008
FAME	0.5984	0.0024	0.0024	0.0024	0.0012	0.0000	0.0033	0.0033	0.0000	0.8801
MEOH	0.3962	0.8370	0.8370	0.8370	0.6437	1.0000	0.0177	0.0177	1.0000	0.1119
NAOH	0.0007	0.0022	0.0022	0.0022	0.0057	0.0000	0.0156	0.0156	0.0000	0.0010
H2O	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.2 (Continue): Stream Information for Optimized Process

Name	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**
Total Flow (kmol/hr)	33.56	43.70	33.62	33.62	16.82	16.82	15.47	15.47	1.34	1.34
Total Flow (kg/hr)	9008.28	9935.97	1077.34	1077.34	538.84	538.84	495.68	495.68	42.82	42.82
Total Flow (m^3/hr)	10.45	11.51	8561.17	398.70	199.41	0.72	183.44	0.67	15.85	0.05
Temperature (°C)	34.36	42.37	33.66	304.29	304.29	58.69	304.29	66.63	304.29	35.10
Pressure (atm)	1.10	1.10	0.10	3.95	3.95	3.95	3.95	3.95	3.95	3.95
Vapor Fraction	0.00	0.00	1.00	1.00	1.00	0.00	1.00	0.00	1.00	0.00
Mole Fraction										
TG	0.0012	0.0010	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
DG	0.0034	0.0026	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MG	0.0015	0.0017	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
GL	0.0008	0.2236	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
FAME	0.8801	0.6767	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MEOH	0.1119	0.0901	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
NAOH	0.0010	0.0044	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2O	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.2 (Continue): Stream Information for Optimized Process

Name	**41**	**42**	**43**	**44**	**45**	**46**	**47**	**48**	**49**	**50**
Total Flow (kmol/hr)	33.62	24.68	8.94	39.07	31.62	31.62	1.55	30.07	51.16	51.16
Total Flow (kg/hr)	1077.34	790.82	286.52	703.88	8935.20	8935.20	28.30	8906.91	1704.64	1700.41
Total Flow (m^3/hr)	1.44	1.06	0.38	0.73	10.50	10.91	464.13	10.83	1.54	1.55
Temperature (°C)	61.45	61.45	61.45	50.00	48.58	95.00	92.07	92.07	48.20	56.62
Pressure (atm)	3.95	3.95	3.95	1.00	1.00	1.00	0.10	0.10	1.10	1.00
Vapor Fraction	0.00	0.00	0.00	0.00	0.00	0.00	1.00	0.00	0.00	0.00
Mole Fraction										
TG	0.0000	0.0000	0.0000	0.0000	0.0013	0.0013	0.0000	0.0014	0.0000	0.0000
DG	0.0000	0.0000	0.0000	0.0000	0.0036	0.0036	0.0000	0.0038	0.0000	0.0000
MG	0.0000	0.0000	0.0000	0.0000	0.0023	0.0023	0.0000	0.0025	0.0000	0.0000
GL	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.1910	0.1910
FAME	0.0000	0.0000	0.0000	0.0000	0.9354	0.9354	0.0001	0.9836	0.0000	0.0000
MEOH	1.0000	1.0000	1.0000	0.0000	0.0008	0.0008	0.0134	0.0002	0.0764	0.0764
NAOH	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0038	0.0000
H2O	0.0000	0.0000	0.0000	1.0000	0.0565	0.0565	0.9864	0.0086	0.7288	0.7326
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.2 (Continue): Stream Information for Optimized Process

Name	**51**	**52**	**53**	**54**	**55**
Total Flow (kmol/hr)	0.10	51.16	51.16	40.54	10.62
Total Flow (kg/hr)	10.21	1700.41	1700.41	784.93	915.48
Total Flow (m^3/hr)	0.0041	1.55	375.66	10735.3	0.77
Temperature (°C)	56.62	56.62	104.24	45.71	110.35
Pressure (atm)	1.00	1.00	1.00	0.10	0.12
Vapor Fraction	Solid	0.00	0.24	1.00	0.00
Mole Fraction					
TG	0.0000	0.0000	0.0000	0.0000	0.0000
DG	0.0000	0.0000	0.0000	0.0000	0.0000
MG	0.0000	0.0000	0.0000	0.0000	0.0000
GL	0.0000	0.1910	0.1910	0.0000	0.9200
FAME	0.0000	0.0000	0.0000	0.0000	0.0000
MEOH	0.0000	0.0764	0.0764	0.0959	0.0020
NAOH	0.0000	0.0000	0.0000	0.0000	0.0000
H2O	0.0000	0.7326	0.7326	0.9041	0.0780
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	1.0000	0.0000	0.0000	0.0000	0.0000

Table B.3: Stream Information for Backed-off Process

Name	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Total Flow (kmol/hr)	10.00	14.67	0.19	19.41	39.07	0.09	10.00	14.86	14.86	41.19
Total Flow (kg/hr)	8854.49	470.06	7.59	622.01	703.88	5.88	8854.49	477.64	477.64	1321.23
Total Flow (m^3/hr)	9.76	0.59	0.00	0.78	0.71	0.01	9.76	0.59	0.59	1.72
Temperature (°C)	25.00	25.00	25.00	25.00	25.00	25.00	25.12	24.96	25.04	54.25
Pressure (atm)	1.00	1.00	1.00	1.00	1.00	1.00	3.95	1.00	3.95	3.95
Vapor Fraction	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mole Fraction										
TG	1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000
DG	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MG	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
GL	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
FAME	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MEOH	0.0000	1.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.9872	0.9872	0.9954
NAOH	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	0.0128	0.0128	0.0046
H2O	0.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.3 (Continue): Stream Information for Backed-off Process

Name	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**
Total Flow (kmol/hr)	298.50	308.50	34.62	273.89	257.32	16.57	19.41	28.95	300.72	300.72
Total Flow (kg/hr)	17167.9	26022.4	9155.27	16867.2	15846.7	1020.51	622.01	927.64	19895.5	19895.5
Total Flow (m^3/hr)	15.89	27.26	11.16	15.20	14.28	0.92	0.78	1.20	23.89	23.45
Temperature (°C)	62.21	60.00	63.00	63.00	63.00	63.00	25.08	40.24	58.23	60.00
Pressure (atm)	3.95	3.95	3.95	3.95	3.95	3.95	3.95	3.95	3.95	3.95
Vapor Fraction	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mole Fraction										
TG	0.0000	0.0019	0.0174	0.0000	0.0000	0.0000	0.0000	0.0000	0.0020	0.0001
DG	0.0000	0.0024	0.0218	0.0000	0.0000	0.0000	0.0000	0.0000	0.0025	0.0003
MG	0.0009	0.0028	0.0166	0.0011	0.0011	0.0011	0.0000	0.0000	0.0023	0.0005
GL	0.4172	0.4298	0.0010	0.4840	0.4840	0.4840	0.0000	0.0000	0.1102	0.1161
FAME	0.0002	0.0848	0.7537	0.0003	0.0003	0.0003	0.0000	0.0000	0.0891	0.1009
MEOH	0.5742	0.4710	0.1879	0.5068	0.5068	0.5068	1.0000	1.0000	0.7921	0.7803
NAOH	0.0074	0.0072	0.0017	0.0079	0.0079	0.0079	0.0000	0.0000	0.0018	0.0018
H2O	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.3 (Continue): Stream Information for Backed-off Process

Name	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
Total Flow (kmol/hr)	50.98	249.74	237.16	12.58	29.16	18.97	10.19	10.19	16.90	34.08
Total Flow (kg/hr)	9561.79	10333.7	9813.07	520.71	1541.22	607.76	933.46	933.46	541.46	9020.34
Total Flow (m^3/hr)	11.39	11.61	11.03	0.59	1.49	4809.39	0.80	0.79	4281.68	10.44
Temperature (°C)	62.25	62.25	62.25	62.25	62.86	32.38	135.45	135.67	32.15	32.15
Pressure (atm)	3.95	3.95	3.95	3.95	3.95	0.10	0.12	1.10	0.10	0.10
Vapor Fraction	0.00	0.00	0.00	0.00	0.00	1.00	0.00	0.00	1.00	0.00
Mole Fraction										
TG	0.0007	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0010
DG	0.0019	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0028
MG	0.0008	0.0005	0.0005	0.0005	0.0008	0.0000	0.0023	0.0023	0.0000	0.0011
GL	0.0005	0.1396	0.1396	0.1396	0.3353	0.0000	0.9595	0.9595	0.0000	0.0008
FAME	0.5807	0.0029	0.0029	0.0029	0.0014	0.0000	0.0040	0.0040	0.0000	0.8686
MEOH	0.4148	0.8549	0.8549	0.8549	0.6571	1.0000	0.0188	0.0188	1.0000	0.1247
NAOH	0.0007	0.0020	0.0020	0.0020	0.0053	0.0000	0.0153	0.0153	0.0000	0.0010
H2O	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.3 (Continue): Stream Information for Backed-off Process

Name	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**
Total Flow (kmol/hr)	34.08	44.27	35.86	35.86	17.73	17.73	16.77	16.77	1.37	1.37
Total Flow (kg/hr)	9020.34	9953.80	1149.21	1149.21	568.08	568.08	537.34	537.34	43.80	43.80
Total Flow (m^3/hr)	10.44	11.51	9091.07	423.96	209.57	0.77	198.23	0.74	16.16	0.06
Temperature (°C)	32.19	40.33	32.27	302.53	302.53	66.99	302.53	74.50	302.53	45.00
Pressure (atm)	1.10	1.10	0.10	3.95	3.95	3.95	3.95	3.95	3.95	3.95
Vapor Fraction	0.00	0.00	1.00	1.00	1.00	0.00	1.00	0.00	1.00	0.00
Mole Fraction										
TG	0.0010	0.0008	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
DG	0.0028	0.0022	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MG	0.0011	0.0014	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
GL	0.0008	0.2215	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
FAME	0.8686	0.6696	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MEOH	0.1247	0.1003	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
NAOH	0.0010	0.0043	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2O	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.3 (Continue): Stream Information for Backed-off Process

Name	**41**	**42**	**43**	**44**	**45**	**46**	**47**	**48**	**49**	**50**
Total Flow (kmol/hr)	35.86	26.33	9.54	39.07	31.62	31.62	1.67	29.95	51.72	51.72
Total Flow (kg/hr)	1149.21	843.58	305.63	703.88	8933.55	8933.55	31.10	8902.46	1724.12	1719.95
Total Flow (m^3/hr)	1.56	1.14	0.41	0.73	10.49	10.91	538.66	11.08	1.56	1.57
Temperature (°C)	69.70	69.70	69.70	50.00	46.99	95.00	119.29	119.29	46.27	54.50
Pressure (atm)	3.95	3.95	3.95	1.00	1.00	1.00	0.10	0.10	1.10	1.00
Vapor Fraction	0.00	0.00	0.00	0.00	0.00	0.00	1.00	0.00	0.00	0.00
Mole Fraction										
TG	0.0000	0.0000	0.0000	0.0000	0.0011	0.0011	0.0000	0.0012	0.0000	0.0000
DG	0.0000	0.0000	0.0000	0.0000	0.0030	0.0030	0.0000	0.0032	0.0000	0.0000
MG	0.0000	0.0000	0.0000	0.0000	0.0020	0.0020	0.0000	0.0021	0.0000	0.0000
GL	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.1896	0.1896
FAME	0.0000	0.0000	0.0000	0.0000	0.9373	0.9373	0.0012	0.9896	0.0000	0.0000
MEOH	1.0000	1.0000	1.0000	0.0000	0.0009	0.0009	0.0147	0.0001	0.0853	0.0853
NAOH	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0037	0.0000
H2O	0.0000	0.0000	0.0000	1.0000	0.0557	0.0557	0.9840	0.0038	0.7214	0.7251
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table B.3 (Continue): Stream Information for Backed-off Process

Name	**51**	**52**	**53**	**54**	**55**
Total Flow (kmol/hr)	0.09	51.72	51.72	41.40	10.32
Total Flow (kg/hr)	10.05	1719.95	1719.95	807.52	912.43
Total Flow (m^3/hr)	0.0040	1.57	401.21	10958.9	0.77
Temperature (°C)	54.50	54.50	104.05	45.61	124.66
Pressure (atm)	1.00	1.00	1.00	0.10	0.12
Vapor Fraction	Solid	0.00	0.25	1.00	0.00
Mole Fraction					
TG	0.0000	0.0000	0.0000	0.0000	0.0000
DG	0.0000	0.0000	0.0000	0.0000	0.0000
MG	0.0000	0.0000	0.0000	0.0000	0.0000
GL	0.0000	0.1896	0.1896	0.0000	0.9500
FAME	0.0000	0.0000	0.0000	0.0000	0.0000
MEOH	0.0000	0.0853	0.0853	0.1062	0.0014
NAOH	0.0000	0.0000	0.0000	0.0000	0.0000
H2O	0.0000	0.7251	0.7251	0.8938	0.0486
H2CO3	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3	1.0000	0.0000	0.0000	0.0000	0.0000

APPENDIX C

CONTROLLER TYPE AND TUNING PARAMETERS

Note that the temperature sensors have two 0.5 minute lag in measurement, the composition sensors have deadtime for 3 minute in detection and the component flow rate of each component is calculated from signals of the composition analyzer and the flow rate sensors.

Table C.1: Controller type and Tuning parameters for CS1

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
FC1	Total feed flow rate of Triglyceride	Feed flow rate of stream 1	Reverse	PI	0.59	1.21
FC2	Total feed flow rate of Water	Feed flow rate of stream 5	Reverse	PI	0.59	1.21
FIC1	Sodium hydroxide flow rate	Feed flow rate of stream 3	Reverse	PI	3.54	21.12
FIC2	Carbonic acid (Stream 6) flow rate	Feed flow rate of stream 6	Reverse	PI	0.81	0.26
FIC3	Methanol (Stream 11) flow rate	Feed flow rate of stream 2	Reverse	PI	9.64	0.26
FIC4	Methanol (Stream 19) flow rate	Feed flow rate of stream 4	Reverse	PI	7.94	0.26
FIC5	Glycerol (Stream 15) flow rate	Flow rate of stream 16	Direct	PI	16.48	0.26
FIC6	Glycerol (Stream 23) flow rate	Flow rate of stream 24	Direct	PI	8.11	0.26
LC1	Level of flash tank FT-2	Liquid outlet flow rate (Stream 48)	Direct	P	2.00	-
LC2	Column VDC-2 Sump Level	Bottom flow rate (Stream 55)	Direct	P	2.00	-
LC3	Reactor R-1 Level	Liquid outlet flow rate (Stream 12)	Direct	P	10.00	-
LC4	Light liquid Level of Decanter D-1	Light phase outlet flow rate (Stream 13)	Direct	P	2.00	-
LC5	Reactor R-2 Level	Liquid outlet flow rate (Stream 20)	Direct	P	10.00	-
LC6	Heavy liquid Level of Decanter D-1	Heavy phase outlet flow rate (Stream 14)	Direct	P	2.00	-
LC7	Heavy liquid Level of Decanter D-2	Heavy phase outlet flow rate (Stream 22)	Direct	P	2.00	-
LC8	Light liquid Level of Decanter D-2	Light phase outlet flow rate (Stream 21)	Direct	P	2.00	-
LC9	Level of flash tank FT-1	Liquid outlet flow rate (Stream 30)	Direct	P	2.00	-
LC10	Column VDC-1 Sump Level	Bottom flow rate (Stream 27)	Direct	P	2.00	-

Table C.1 (Continue): Controller type and Tuning parameters for CS1

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
LC11	Reactor R-3 Level	Liquid outlet flow rate (Stream 50)	Direct	P	10.00	-
PC1	Pressure of flash tank FT-2	Vapor discharge rate (Stream 47)	Direct	PI	0.06	1.20
PC2	Top stage pressure of VDC-2	Vapor discharge rate (Stream 54)	Direct	PI	0.27	5.42
PC3	Pressure of flash tank FT-1	Vapor discharge rate (Stream 29)	Direct	PI	0.80	2.32
PC4	Top stage pressure of VDC-1	Vapor discharge rate (Stream 26)	Direct	PI	0.52	16.38
PC5	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.50
PC6	Discharge Pressure	Shaft speed	Reverse	PI	0.56	0.12
PC7	Discharge Pressure	Shaft speed	Reverse	PI	0.35	0.15
PC8	Discharge Pressure	Shaft speed	Reverse	PI	0.22	1.20
PC9	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.20
PC10	Discharge Pressure	Compressor work	Reverse	PI	0.83	0.15
TC1	Stream 46 Temperature	Stream 35 flow rate	Reverse	PI	2.66	2.31
TC2	Stream 46A Temperature	Heater HX-4 Duty	Reverse	PI	2.85	1.20
TC3	Stream 44 Temperature	Stream 39 flow rate	Reverse	PI	1.64	2.53
TC4	Reactor R-1 Temperature	Reactor R-1 Duty	Reverse	PI	2.77	208.08
TC5	Reactor R-2 Temperature	Reactor R-2 Duty	Reverse	PI	25.31	168.20
TC6	Reboiler temperature of VDC-1	Reboiler Duty at VDC-1	Reverse	PI	1.80	16.47
TC7	Reboiler temperature of VDC-2	Reboiler Duty at VDC-2	Reverse	PI	1.20	29.84

Table C.2: Controller type and Tuning parameters for CS2

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
FC1	Total feed flow rate of Triglyceride	Feed flow rate of stream 1	Reverse	PI	0.59	1.21
FC2	Total feed flow rate of Water	Feed flow rate of stream 5	Reverse	PI	0.59	1.21
FIC1	Sodium hydroxide flow rate	Feed flow rate of stream 3	Reverse	PI	3.54	21.12
FIC2	Carbonic acid (Stream 6) flow rate	Feed flow rate of stream 6	Reverse	PI	0.81	0.26
FIC3	Methanol (Stream 11) flow rate	Feed flow rate of stream 2	Reverse	PI	9.64	0.26
FIC4	Methanol (Stream 19) flow rate	Feed flow rate of stream 4	Reverse	PI	7.94	0.26
FIC5	Glycerol (Stream 15) flow rate	Flow rate of stream 16	Direct	PI	16.48	0.26
FIC6	Glycerol (Stream 23) flow rate	Flow rate of stream 24	Direct	PI	8.11	0.26
LC1	Level of flash tank FT-2	Liquid outlet flow rate (Stream 48)	Direct	P	2.00	-
LC2	Column VDC-2 Sump Level	Bottom flow rate (Stream 55)	Direct	P	2.00	-
LC3	Reactor R-1 Level	Liquid outlet flow rate (Stream 12)	Direct	P	10.00	-
LC4	Light liquid Level of Decanter D-1	Light phase outlet flow rate (Stream 13)	Direct	P	2.00	-
LC5	Reactor R-2 Level	Liquid outlet flow rate (Stream 20)	Direct	P	10.00	-
LC6	Heavy liquid Level of Decanter D-1	Heavy phase outlet flow rate (Stream 14)	Direct	P	2.00	-
LC7	Heavy liquid Level of Decanter D-2	Heavy phase outlet flow rate (Stream 22)	Direct	P	2.00	-
LC8	Light liquid Level of Decanter D-2	Light phase outlet flow rate (Stream 21)	Direct	P	2.00	-
LC9	Level of flash tank FT-1	Liquid outlet flow rate (Stream 30)	Direct	P	2.00	-
LC10	Column VDC-1 Sump Level	Bottom flow rate (Stream 27)	Direct	P	2.00	-

Table C.2 (Continue): Controller type and Tuning parameters for CS2

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
LC11	Reactor R-3 Level	Liquid outlet flow rate (Stream 50)	Direct	P	10.00	-
PC1	Pressure of flash tank FT-2	Vapor discharge rate (Stream 47)	Direct	PI	0.06	1.20
PC2	Top stage pressure of VDC-2	Vapor discharge rate (Stream 54)	Direct	PI	0.27	5.42
PC3	Pressure of flash tank FT-1	Vapor discharge rate (Stream 29)	Direct	PI	0.80	2.32
PC4	Top stage pressure of VDC-1	Vapor discharge rate (Stream 26)	Direct	PI	0.52	16.38
PC5	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.50
PC6	Discharge Pressure	Shaft speed	Reverse	PI	0.56	0.12
PC7	Discharge Pressure	Shaft speed	Reverse	PI	0.35	0.15
PC8	Discharge Pressure	Shaft speed	Reverse	PI	0.22	1.20
PC9	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.20
PC10	Discharge Pressure	Compressor work	Reverse	PI	0.83	0.15
TC1	Stream 46 Temperature	Stream 35 flow rate	Reverse	PI	2.66	2.31
TC2	Stream 46A Temperature	Heater HX-4 Duty	Reverse	PI	1.00	20.00
TC3	Stream 44 Temperature	Stream 39 flow rate	Reverse	PI	1.64	2.53
TC4	Reactor R-1 Temperature	Reactor R-1 Duty	Reverse	PI	2.77	208.08
TC5	Reactor R-2 Temperature	Reactor R-2 Duty	Reverse	PI	25.31	168.20
TC6	Reboiler temperature of VDC-1	Reboiler Duty at VDC-1	Reverse	PI	2.40	17.35
TC7	Reboiler temperature of VDC-2	Reboiler Duty at VDC-2	Reverse	PI	4.24	6.32
XC1	Biodiesel mole fraction in Stream 20	Setpoint of FIC4	Direct	PI	2.74	26.90
YC1	Yield of Reactor R-1	Setpoint of FIC3	Reverse	PI	1.34	9.78

Table C.3: Controller type and Tuning parameters for CS3

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
FC1	Total feed flow rate of Triglyceride	Feed flow rate of stream 1	Reverse	PI	0.59	1.21
FC2	Total feed flow rate of Water	Feed flow rate of stream 5	Reverse	PI	0.59	1.21
FIC1	Sodium hydroxide flow rate	Feed flow rate of stream 3	Reverse	PI	3.54	21.12
FIC2	Carbonic acid (Stream 6) flow rate	Feed flow rate of stream 6	Reverse	PI	0.81	0.26
FIC3	Methanol (Stream 11) flow rate	Feed flow rate of stream 2	Reverse	PI	9.64	0.26
FIC4	Methanol (Stream 19) flow rate	Feed flow rate of stream 4	Reverse	PI	7.94	0.26
FIC5	Glycerol (Stream 15) flow rate	Flow rate of stream 16	Direct	PI	16.48	0.26
FIC6	Glycerol (Stream 23) flow rate	Flow rate of stream 24	Direct	PI	8.11	0.26
LC1	Level of flash tank FT-2	Liquid outlet flow rate (Stream 48)	Direct	P	2.00	-
LC2	Column VDC-2 Sump Level	Bottom flow rate (Stream 55)	Direct	P	2.00	-
LC3	Reactor R-1 Level	Liquid outlet flow rate (Stream 12)	Direct	P	10.00	-
LC4	Light liquid Level of Decanter D-1	Light phase outlet flow rate (Stream 13)	Direct	P	2.00	-
LC5	Reactor R-2 Level	Liquid outlet flow rate (Stream 20)	Direct	P	10.00	-
LC6	Heavy liquid Level of Decanter D-1	Heavy phase outlet flow rate (Stream 14)	Direct	P	2.00	-
LC7	Heavy liquid Level of Decanter D-2	Heavy phase outlet flow rate (Stream 22)	Direct	P	2.00	-
LC8	Light liquid Level of Decanter D-2	Light phase outlet flow rate (Stream 21)	Direct	P	2.00	-
LC9	Level of flash tank FT-1	Liquid outlet flow rate (Stream 30)	Direct	P	2.00	-
LC10	Column VDC-1 Sump Level	Bottom flow rate (Stream 27)	Direct	P	2.00	-

Table C.3 (Continue): Controller type and Tuning parameters for CS3

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
LC11	Reactor R-3 Level	Liquid outlet flow rate (Stream 50)	Direct	P	10.00	-
PC1	Pressure of flash tank FT-2	Vapor discharge rate (Stream 47)	Direct	PI	0.06	1.20
PC2	Top stage pressure of VDC-2	Vapor discharge rate (Stream 54)	Direct	PI	0.27	5.42
PC3	Pressure of flash tank FT-1	Vapor discharge rate (Stream 29)	Direct	PI	0.80	2.32
PC4	Top stage pressure of VDC-1	Vapor discharge rate (Stream 26)	Direct	PI	0.52	16.38
PC5	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.50
PC6	Discharge Pressure	Shaft speed	Reverse	PI	0.56	0.12
PC7	Discharge Pressure	Shaft speed	Reverse	PI	0.35	0.15
PC8	Discharge Pressure	Shaft speed	Reverse	PI	0.22	1.20
PC9	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.20
PC10	Discharge Pressure	Compressor work	Reverse	PI	0.83	0.15
TC1	Stream 46 Temperature	Stream 35 flow rate	Reverse	PI	2.66	2.31
TC2	Stream 46A Temperature	Heater HX-4 Duty	Reverse	PI	1.00	20.00
TC3	Stream 44 Temperature	Stream 39 flow rate	Reverse	PI	1.64	2.53
TC4	Reactor R-1 Temperature	Reactor R-1 Duty	Reverse	PI	2.77	208.08
TC5	Reactor R-2 Temperature	Reactor R-2 Duty	Reverse	PI	50.65	163.75
TC6	Reboiler temperature of VDC-1	Reboiler Duty at VDC-1	Reverse	PI	3.33	6.99
TC7	Reboiler temperature of VDC-2	Reboiler Duty at VDC-2	Reverse	PI	4.24	6.32
TYC	Total Yield	Setpoint of FIC4	Reverse	PI	0.12	6.00
YC1	Yield of Reactor R-1	Setpoint of FIC3	Reverse	PI	1.34	9.78

Table C.4: Controller type and Tuning parameters for CS4

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
FC1	Total feed flow rate of Triglyceride	Feed flow rate of stream 1	Reverse	PI	0.59	1.21
FC2	Total feed flow rate of Water	Feed flow rate of stream 5	Reverse	PI	0.59	1.21
FIC1	Sodium hydroxide flow rate	Feed flow rate of stream 3	Reverse	PI	3.54	21.12
FIC2	Carbonic acid (Stream 6) flow rate	Feed flow rate of stream 6	Reverse	PI	0.81	0.26
FIC3	Methanol (Stream 11) flow rate	Feed flow rate of stream 2	Reverse	PI	9.64	0.26
FIC4	Methanol (Stream 19) flow rate	Feed flow rate of stream 4	Reverse	PI	7.94	0.26
FIC5	Glycerol (Stream 15) flow rate	Flow rate of stream 16	Direct	PI	16.48	0.26
FIC6	Glycerol (Stream 23) flow rate	Flow rate of stream 24	Direct	PI	8.11	0.26
LC1	Level of flash tank FT-2	Liquid outlet flow rate (Stream 48)	Direct	P	2.00	-
LC2	Column VDC-2 Sump Level	Bottom flow rate (Stream 55)	Direct	P	2.00	-
LC3	Reactor R-1 Level	Liquid outlet flow rate (Stream 12)	Direct	P	10.00	-
LC4	Light liquid Level of Decanter D-1	Light phase outlet flow rate (Stream 13)	Direct	P	2.00	-
LC5	Reactor R-2 Level	Liquid outlet flow rate (Stream 20)	Direct	P	10.00	-
LC6	Heavy liquid Level of Decanter D-1	Heavy phase outlet flow rate (Stream 14)	Direct	P	2.00	-
LC7	Heavy liquid Level of Decanter D-2	Heavy phase outlet flow rate (Stream 22)	Direct	P	2.00	-
LC8	Light liquid Level of Decanter D-2	Light phase outlet flow rate (Stream 21)	Direct	P	2.00	-
LC9	Level of flash tank FT-1	Liquid outlet flow rate (Stream 30)	Direct	P	2.00	-
LC10	Column VDC-1 Sump Level	Bottom flow rate (Stream 27)	Direct	P	2.00	-

Table C.4 (Continue): Controller type and Tuning parameters for CS4

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
LC11	Reactor R-3 Level	Liquid outlet flow rate (Stream 50)	Direct	P	10.00	-
PC1	Pressure of flash tank FT-2	Vapor discharge rate (Stream 47)	Direct	PI	0.06	1.20
PC2	Top stage pressure of VDC-2	Vapor discharge rate (Stream 54)	Direct	PI	0.27	5.42
PC3	Pressure of flash tank FT-1	Vapor discharge rate (Stream 29)	Direct	PI	0.80	2.32
PC4	Top stage pressure of VDC-1	Vapor discharge rate (Stream 26)	Direct	PI	0.52	16.38
PC5	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.50
PC6	Discharge Pressure	Shaft speed	Reverse	PI	0.56	0.12
PC7	Discharge Pressure	Shaft speed	Reverse	PI	0.35	0.15
PC8	Discharge Pressure	Shaft speed	Reverse	PI	0.22	1.20
PC9	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.20
PC10	Discharge Pressure	Compressor work	Reverse	PI	0.83	0.15
TC1	Stream 46 Temperature	Stream 35 flow rate	Reverse	PI	2.66	2.31
TC2	Stream 46A Temperature	Heater HX-4 Duty	Reverse	PI	1.00	20.00
TC3	Stream 44 Temperature	Stream 39 flow rate	Reverse	PI	1.64	2.53
TC4	Reactor R-1 Temperature	Reactor R-1 Duty	Reverse	PI	2.77	208.08
TC5	Reactor R-2 Temperature	Reactor R-2 Duty	Reverse	PI	25.31	168.20
TC6	Reboiler temperature of VDC-1	Reboiler Duty at VDC-1	Reverse	PI	2.40	17.35
TC7	Reboiler temperature of VDC-2	Reboiler Duty at VDC-2	Reverse	PI	4.24	6.32
XC1	Biodiesel mole fraction in Stream 20	Setpoint of FIC4	Direct	PI	49.20	26.80

Table C.5: Controller type and Tuning parameters for CS5

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
FC1	Total feed flow rate of Triglyceride	Feed flow rate of stream 1	Reverse	PI	0.59	1.21
FC2	Total feed flow rate of Water	Feed flow rate of stream 5	Reverse	PI	0.59	1.21
FIC1	Sodium hydroxide flow rate	Feed flow rate of stream 3	Reverse	PI	3.54	21.12
FIC2	Carbonic acid (Stream 6) flow rate	Feed flow rate of stream 6	Reverse	PI	0.81	0.26
FIC3	Methanol (Stream 11) flow rate	Feed flow rate of stream 2	Reverse	PI	9.64	0.26
FIC4	Methanol (Stream 19) flow rate	Feed flow rate of stream 4	Reverse	PI	7.94	0.26
FIC5	Glycerol (Stream 15) flow rate	Flow rate of stream 16	Direct	PI	16.48	0.26
FIC6	Glycerol (Stream 23) flow rate	Flow rate of stream 24	Direct	PI	8.11	0.26
LC1	Level of flash tank FT-2	Liquid outlet flow rate (Stream 48)	Direct	P	2.00	-
LC2	Column VDC-2 Sump Level	Bottom flow rate (Stream 55)	Direct	P	2.00	-
LC3	Reactor R-1 Level	Liquid outlet flow rate (Stream 12)	Direct	P	10.00	-
LC4	Light liquid Level of Decanter D-1	Light phase outlet flow rate (Stream 13)	Direct	P	2.00	-
LC5	Reactor R-2 Level	Liquid outlet flow rate (Stream 20)	Direct	P	10.00	-
LC6	Heavy liquid Level of Decanter D-1	Heavy phase outlet flow rate (Stream 14)	Direct	P	2.00	-
LC7	Heavy liquid Level of Decanter D-2	Heavy phase outlet flow rate (Stream 22)	Direct	P	2.00	-
LC8	Light liquid Level of Decanter D-2	Light phase outlet flow rate (Stream 21)	Direct	P	2.00	-
LC9	Level of flash tank FT-1	Liquid outlet flow rate (Stream 30)	Direct	P	2.00	-
LC10	Column VDC-1 Sump Level	Bottom flow rate (Stream 27)	Direct	P	2.00	-

Table C.5 (Continue): Controller type and Tuning parameters for CS5

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
LC11	Reactor R-3 Level	Liquid outlet flow rate (Stream 50)	Direct	P	10.00	-
PC1	Pressure of flash tank FT-2	Vapor discharge rate (Stream 47)	Direct	PI	0.06	1.20
PC2	Top stage pressure of VDC-2	Vapor discharge rate (Stream 54)	Direct	PI	0.27	5.42
PC3	Pressure of flash tank FT-1	Vapor discharge rate (Stream 29)	Direct	PI	0.80	2.32
PC4	Top stage pressure of VDC-1	Vapor discharge rate (Stream 26)	Direct	PI	0.52	16.38
PC5	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.50
PC6	Discharge Pressure	Shaft speed	Reverse	PI	0.56	0.12
PC7	Discharge Pressure	Shaft speed	Reverse	PI	0.35	0.15
PC8	Discharge Pressure	Shaft speed	Reverse	PI	0.22	1.20
PC9	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.20
PC10	Discharge Pressure	Compressor work	Reverse	PI	0.83	0.15
TC1	Stream 46 Temperature	Stream 35 flow rate	Reverse	PI	2.66	2.31
TC2	Stream 46A Temperature	Heater HX-4 Duty	Reverse	PI	2.85	1.20
TC3	Stream 44 Temperature	Stream 39 flow rate	Reverse	PI	1.64	2.53
TC4	Reactor R-1 Temperature	Reactor R-1 Duty	Reverse	PI	2.77	208.08
TC5	Reactor R-2 Temperature	Reactor R-2 Duty	Reverse	PI	50.65	163.75
TC6	Reboiler temperature of VDC-1	Reboiler Duty at VDC-1	Reverse	PI	1.80	16.47
TC7	Reboiler temperature of VDC-2	Reboiler Duty at VDC-2	Reverse	PI	1.20	29.84
TYC	Total Yield	Setpoint of FIC4	Reverse	PI	0.12	6.00

Table C.6: Controller type and Tuning parameters for CS6

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
FC1	Total feed flow rate of Triglyceride	Feed flow rate of stream 1	Reverse	PI	0.59	1.21
FC2	Total feed flow rate of Water	Feed flow rate of stream 5	Reverse	PI	0.59	1.21
FC3	Flow rate of Stream 39	Flow rate of Stream 39	Reverse	PI	0.88	0.90
FIC1	Sodium hydroxide flow rate	Feed flow rate of stream 3	Reverse	PI	3.54	21.12
FIC2	Carbonic acid (Stream 6) flow rate	Feed flow rate of stream 6	Reverse	PI	0.81	0.26
FIC3	Methanol (Stream 11) flow rate	Feed flow rate of stream 2	Reverse	PI	9.64	0.26
FIC4	Methanol (Stream 19) flow rate	Feed flow rate of stream 4	Reverse	PI	7.94	0.26
FIC5	Glycerol (Stream 15) flow rate	Flow rate of stream 16	Direct	PI	16.48	0.26
FIC6	Glycerol (Stream 23) flow rate	Flow rate of stream 24	Direct	PI	8.11	0.26
LC1	Level of flash tank FT-2	Liquid outlet flow rate (Stream 48)	Direct	P	2.00	-
LC2	Column VDC-2 Sump Level	Bottom flow rate (Stream 55)	Direct	P	2.00	-
LC3	Reactor R-1 Level	Liquid outlet flow rate (Stream 12)	Direct	P	10.00	-
LC4	Light liquid Level of Decanter D-1	Light phase outlet flow rate (Stream 13)	Direct	P	2.00	-
LC5	Reactor R-2 Level	Liquid outlet flow rate (Stream 20)	Direct	P	10.00	-
LC6	Heavy liquid Level of Decanter D-1	Heavy phase outlet flow rate (Stream 14)	Direct	P	2.00	-
LC7	Heavy liquid Level of Decanter D-2	Heavy phase outlet flow rate (Stream 22)	Direct	P	2.00	-
LC8	Light liquid Level of Decanter D-2	Light phase outlet flow rate (Stream 21)	Direct	P	2.00	-
LC9	Level of flash tank FT-1	Liquid outlet flow rate (Stream 30)	Direct	P	2.00	-

Table C.6 (Continue): Controller type and Tuning parameters for CS6

Controller	Controlled Variable	Manipulated Variable	Action	Type	Controller Gain	Integral Time (min)
LC10	Column VDC-1 Sump Level	Bottom flow rate (Stream 27)	Direct	P	2.00	-
LC11	Reactor R-3 Level	Liquid outlet flow rate (Stream 50)	Direct	P	10.00	-
PC1	Pressure of flash tank FT-2	Vapor discharge rate (Stream 47)	Direct	PI	0.06	1.20
PC2	Top stage pressure of VDC-2	Vapor discharge rate (Stream 54)	Direct	PI	0.27	5.42
PC3	Pressure of flash tank FT-1	Vapor discharge rate (Stream 29)	Direct	PI	0.80	2.32
PC4	Top stage pressure of VDC-1	Vapor discharge rate (Stream 26)	Direct	PI	0.52	16.38
PC5	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.50
PC6	Discharge Pressure	Shaft speed	Reverse	PI	0.56	0.12
PC7	Discharge Pressure	Shaft speed	Reverse	PI	0.35	0.15
PC8	Discharge Pressure	Shaft speed	Reverse	PI	0.22	1.20
PC9	Discharge Pressure	Shaft speed	Reverse	PI	0.23	1.20
PC10	Discharge Pressure	Compressor work	Reverse	PI	0.83	0.15
TC1	Stream 46 Temperature	Stream 35 flow rate	Reverse	PI	2.66	2.31
TC2	Stream 46A Temperature	Heater HX-4 Duty	Reverse	PI	2.85	1.20
TC3	Stream 44 Temperature	Setpoint of FC2	Direct	PI	37.24	3.96
TC4	Reactor R-1 Temperature	Reactor R-1 Duty	Reverse	PI	2.77	208.08
TC5	Reactor R-2 Temperature	Reactor R-2 Duty	Reverse	PI	25.31	168.20
TC6	Reboiler temperature of VDC-1	Reboiler Duty at VDC-1	Reverse	PI	1.80	16.47
TC7	Reboiler temperature of VDC-2	Reboiler Duty at VDC-2	Reverse	PI	1.20	29.84

APPENDIX D
DYNAMIC SIMULATION RESPONSES

Note that x-axis and y-axis for each graph represent simulation time (hours) and the process variable of the controller shown in Appendix C, respectively.

Table D.1: Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 9 kmol/h (After 5 hours of the beginning of the simulation run)

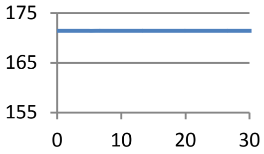
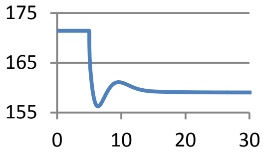
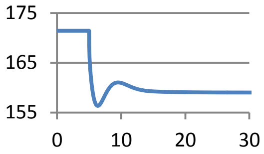
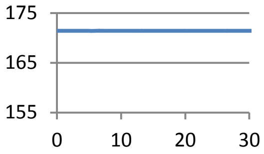
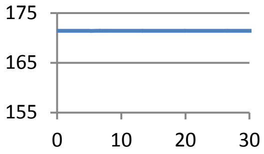
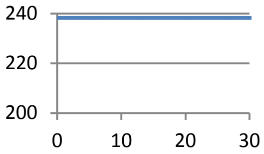
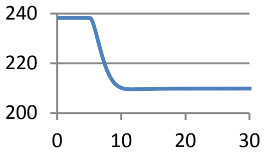
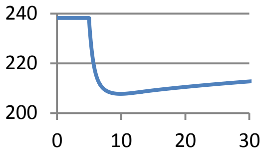
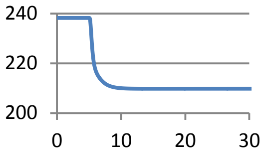
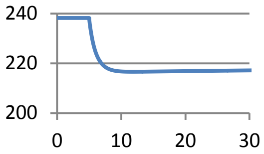
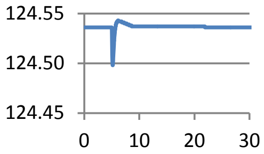
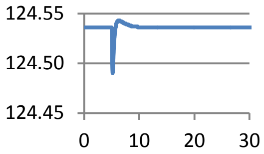
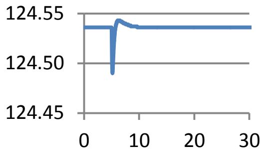
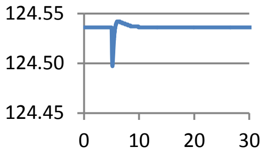
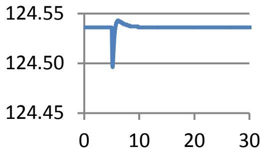
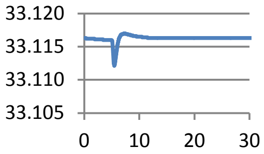
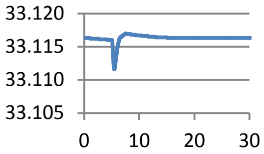
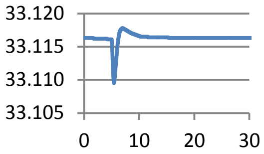
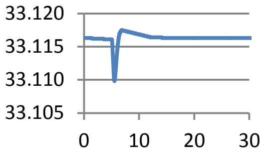
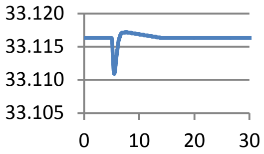
Controller	CS1	CS2	CS3	CS4	CS5
FIC3					
FIC4					
FIC5					
FIC6					

Table D.1 (Continue): Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 9 kmol/h (After 5 hours of the beginning of the simulation run)

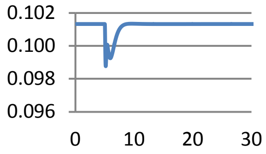
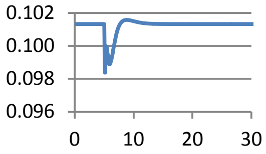
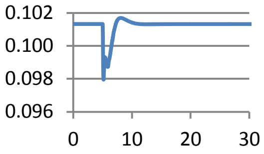
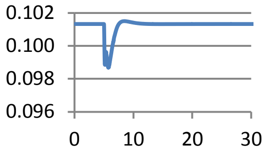
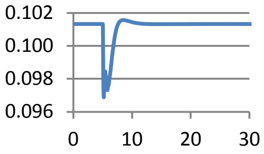
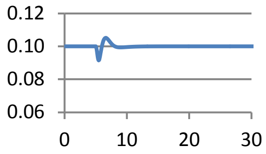
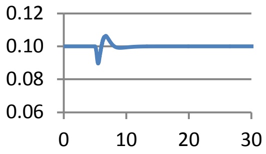
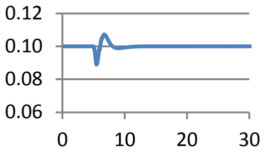
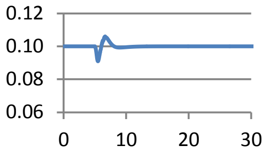
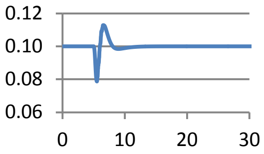
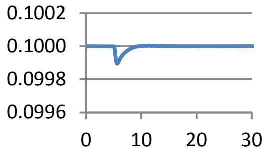
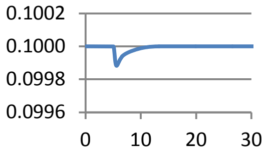
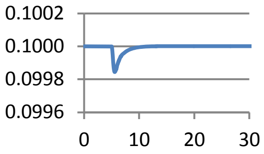
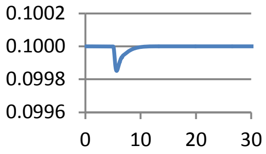
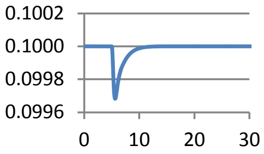
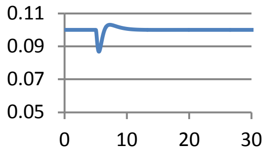
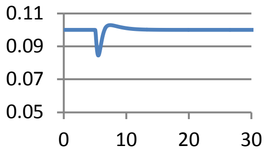
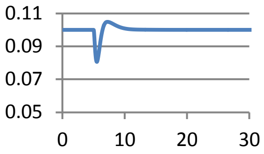
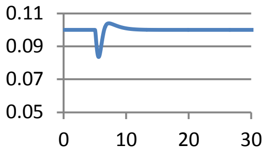
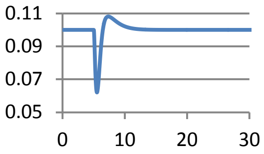
Controller	CS1	CS2	CS3	CS4	CS5
PC1					
PC2					
PC3					
PC4					

Table D.1 (Continue): Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 9 kmol/h (After 5 hours of the beginning of the simulation run)

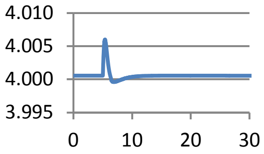
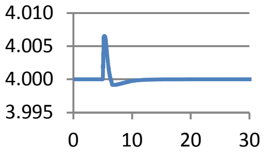
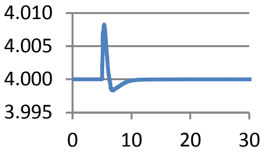
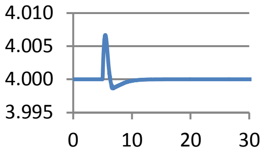
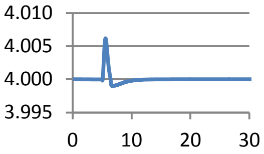
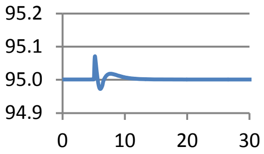
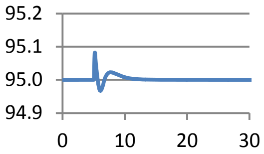
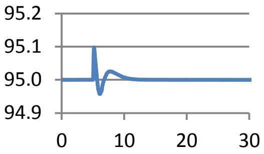
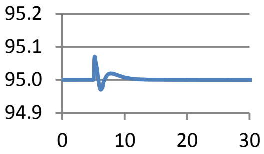
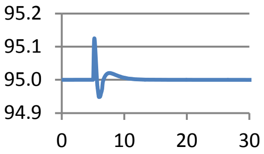
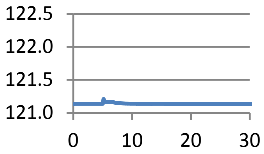
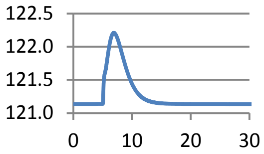
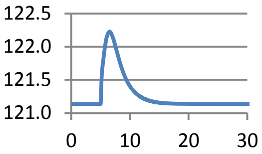
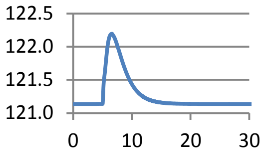
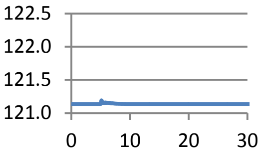
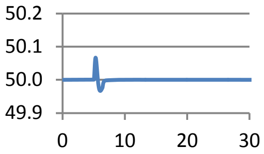
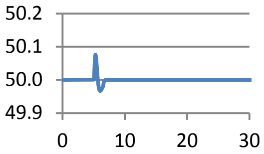
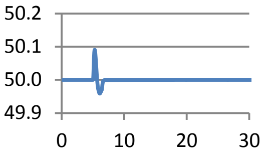
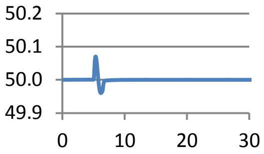
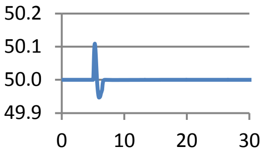
Controller	CS1	CS2	CS3	CS4	CS5
PC10					
TC1					
TC2					
TC3					

Table D.1 (Continue): Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 9 kmol/h (After 5 hours of the beginning of the simulation run)

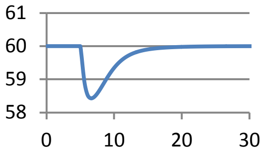
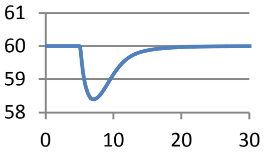
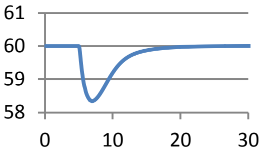
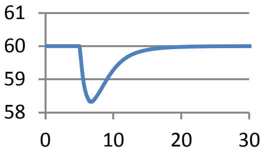
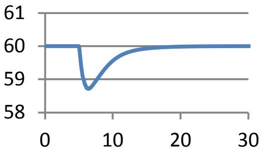
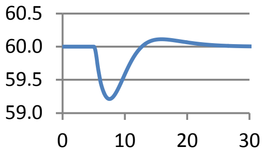
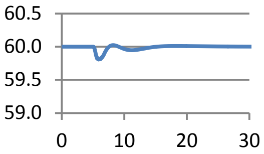
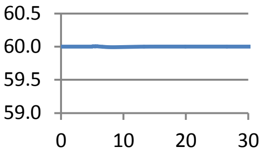
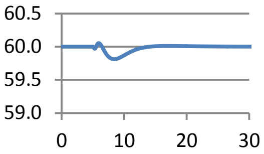
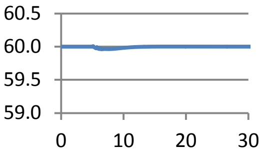
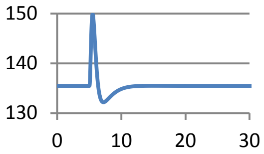
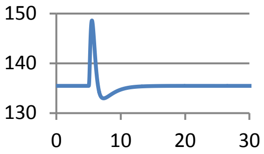
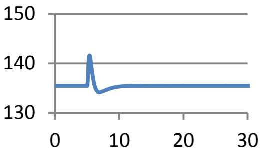
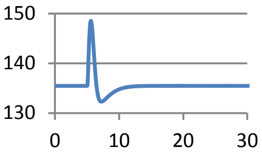
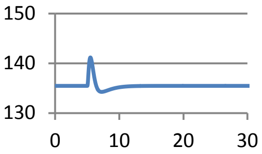
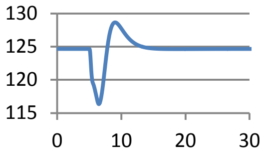
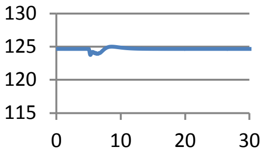
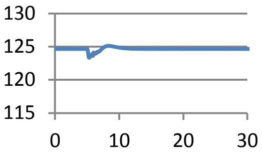
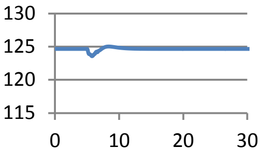
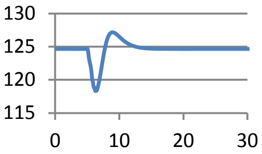
Controller	CS1	CS2	CS3	CS4	CS5
TC4					
TC5					
TC6					
TC7					

Table D.1 (Continue): Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 9 kmol/h (After 5 hours of the beginning of the simulation run)

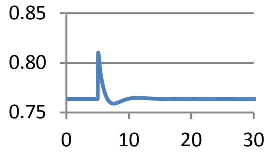
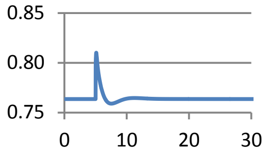
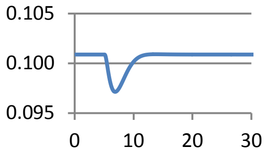
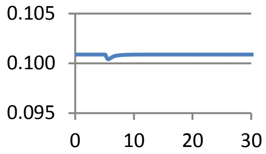
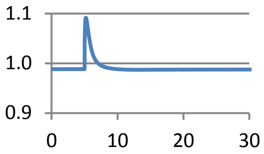
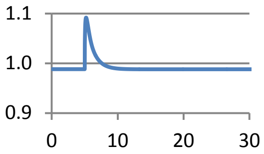
Controller	CS1	CS2	CS3	CS4	CS5
YC1	-			-	-
XC1	-		-		-
TYC	-	-		-	

Table D.2: Dynamic simulation responses for CS6 when the setpoint of the fresh triglyceride feed controller is changed to 9 kmol/h (After 5 hours of the beginning of the simulation run)

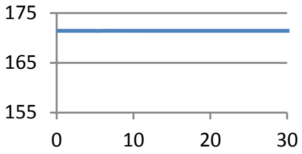
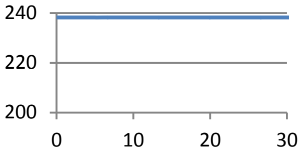
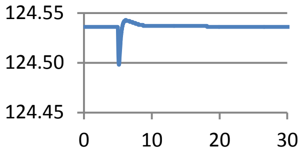
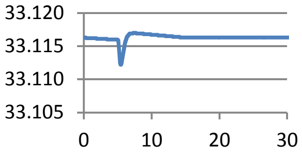
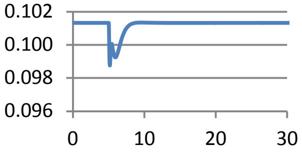
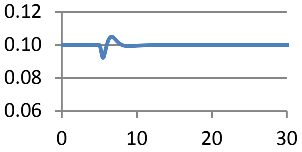
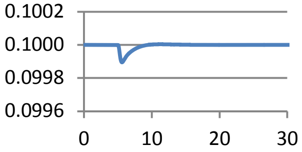
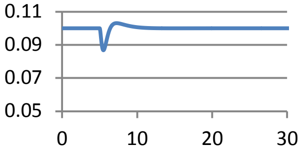
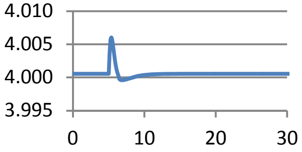
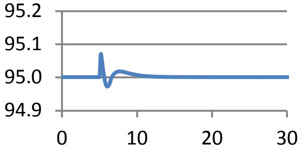
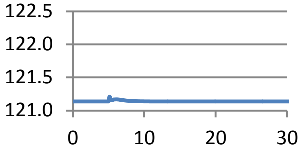
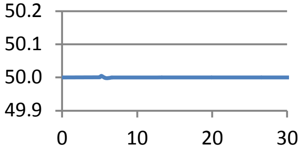
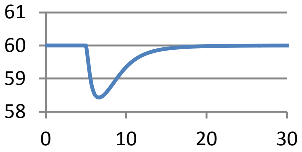
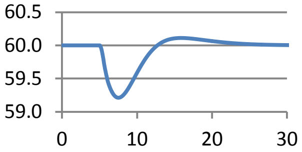
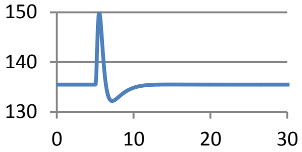
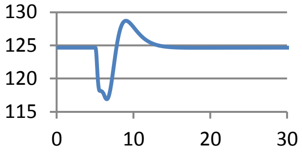
FIC3	FIC4	FIC5	FIC6
			
PC1	PC2	PC3	PC4
			
PC10	TC1	TC2	TC3
			
TC4	TC5	TC6	TC7
			

Table D.3: Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 11 kmol/h (After 5 hours of the beginning of the simulation run)

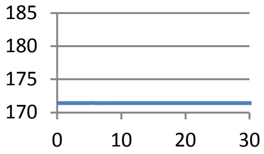
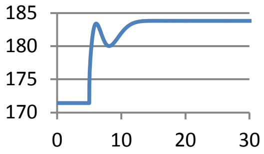
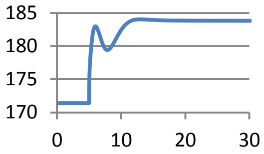
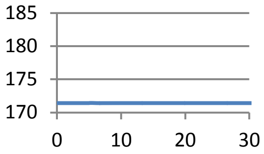
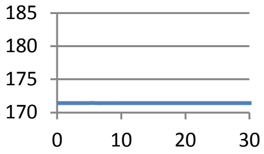
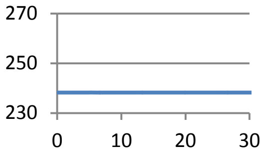
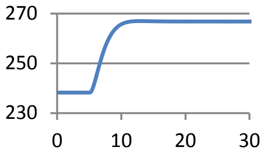
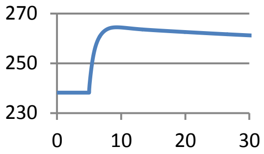
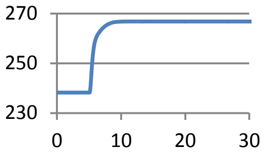
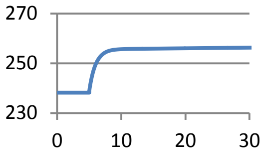
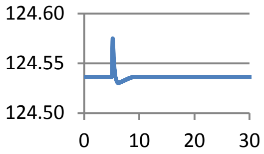
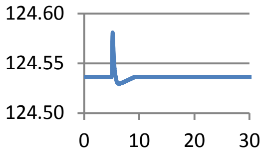
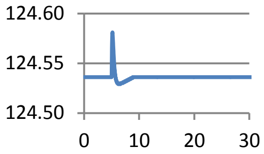
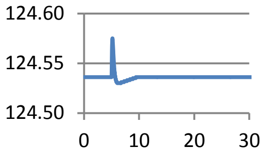
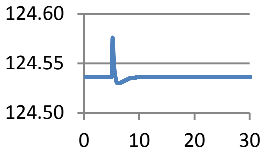
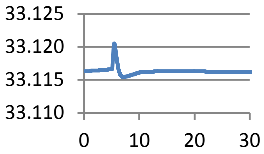
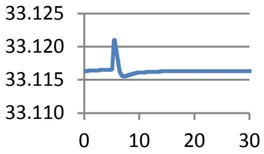
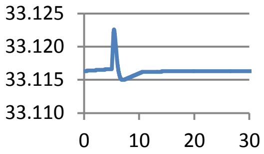
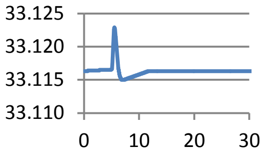
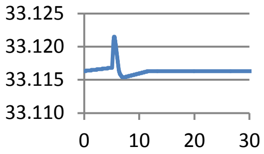
Controller	CS1	CS2	CS3	CS4	CS5
FIC3					
FIC4					
FIC5					
FIC6					

Table D.3 (Continue): Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 11 kmol/h (After 5 hours of the beginning of the simulation run)

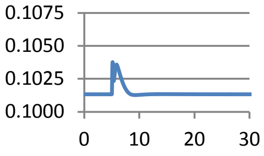
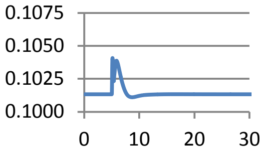
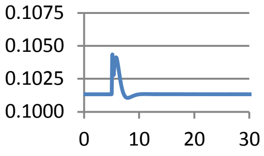
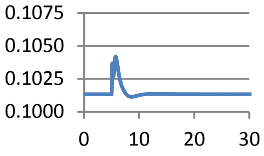
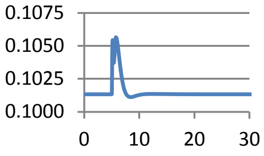
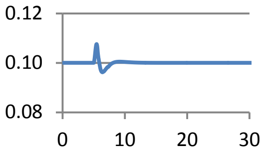
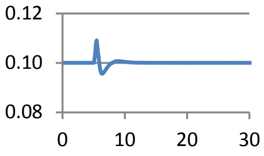
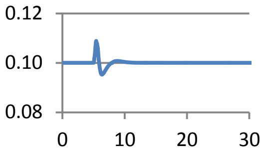
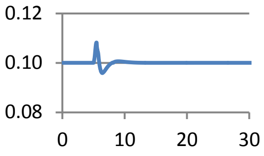
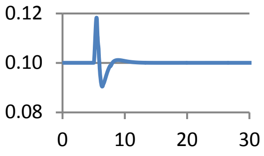
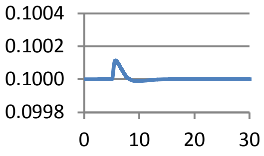
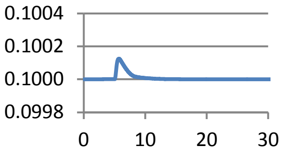
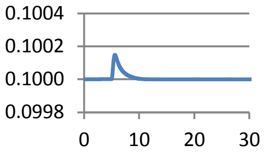
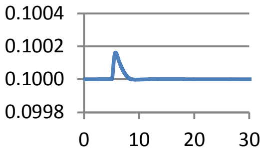
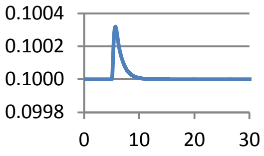
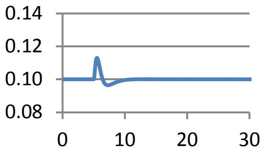
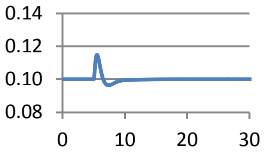
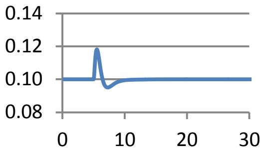
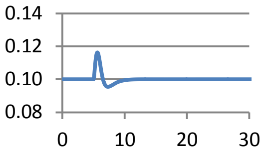
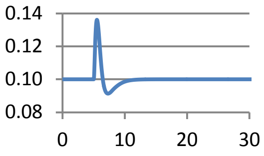
Controller	CS1	CS2	CS3	CS4	CS5
PC1					
PC2					
PC3					
PC4					

Table D.3 (Continue): Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 11 kmol/h (After 5 hours of the beginning of the simulation run)

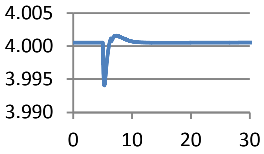
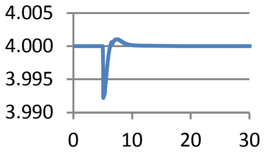
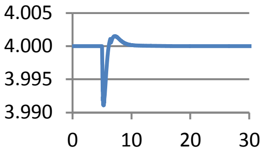
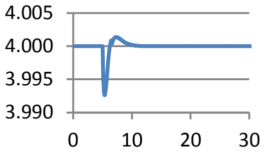
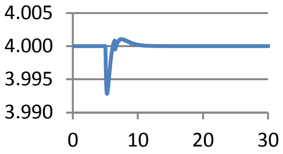
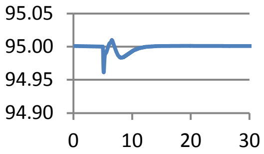
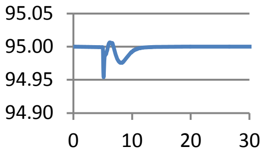
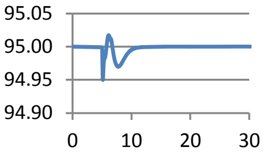
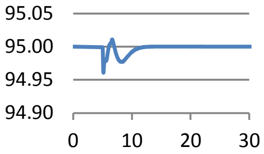
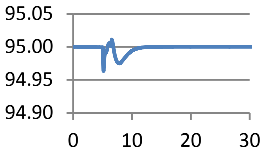
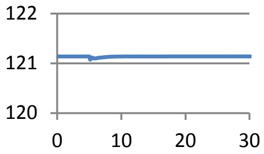
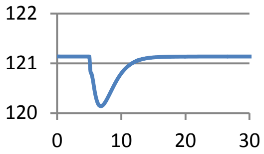
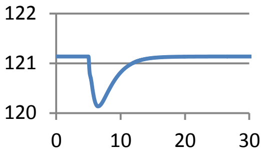
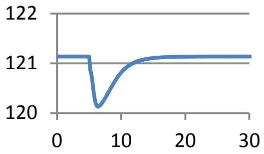
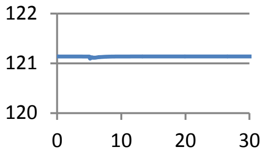
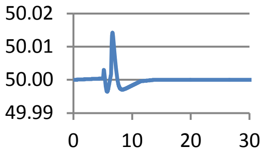
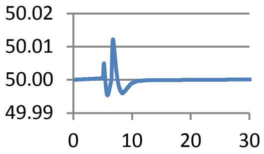
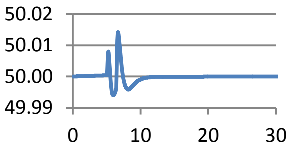
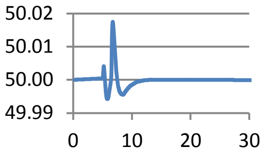
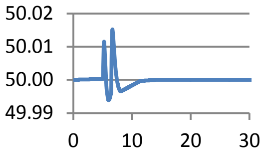
Controller	CS1	CS2	CS3	CS4	CS5
PC10					
TC1					
TC2					
TC3					

Table D.3 (Continue): Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 11 kmol/h (After 5 hours of the beginning of the simulation run)

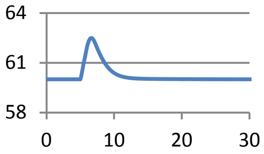
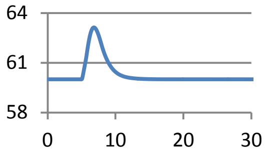
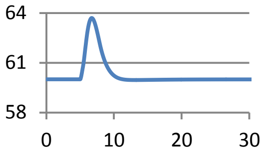
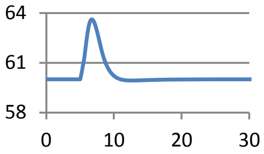
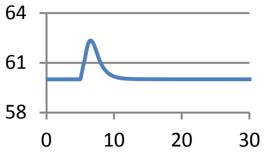
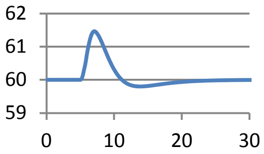
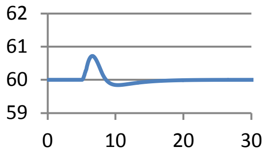
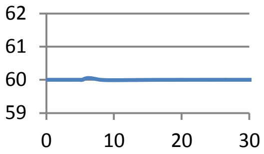
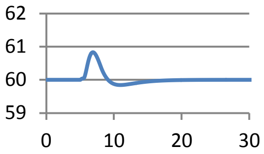
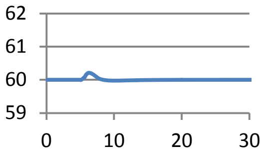
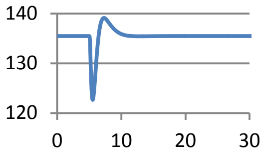
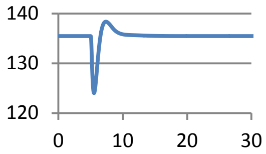
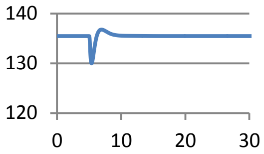
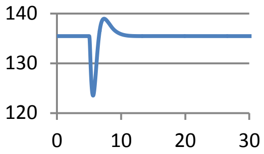
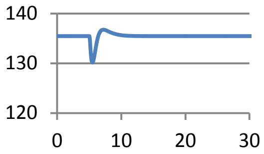
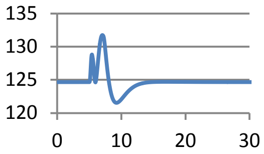
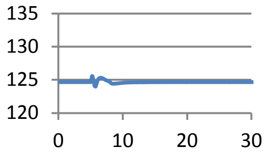
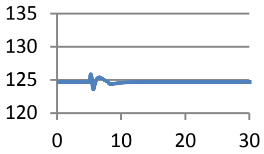
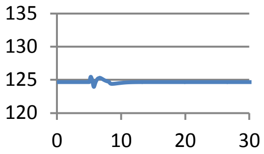
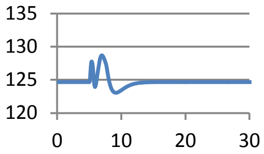
Controller	**CS1**	**CS2**	**CS3**	**CS4**	**CS5**
TC4					
TC5					
TC6					
TC7					

Table D.3 (Continue): Dynamic simulation responses for CS1-CS5 when the setpoint of the fresh triglyceride feed controller is changed to 11 kmol/h (After 5 hours of the beginning of the simulation run)

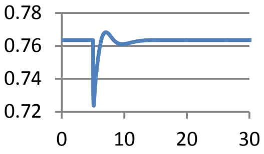
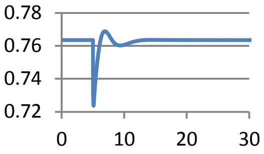
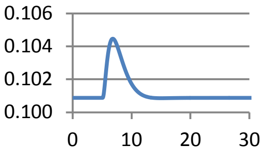
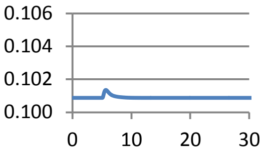
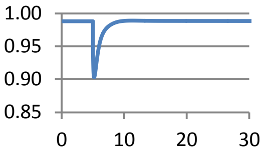
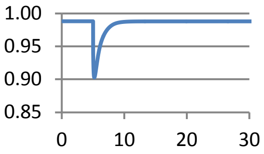
Controller	CS1	CS2	CS3	CS4	CS5
YC1	-			-	-
XC1	-		-		-
TYC	-	-		-	

Table D.4: Dynamic simulation responses for CS6 when the setpoint of the fresh triglyceride feed controller is changed to 11 kmol/h (After 5 hours of the beginning of the simulation run)

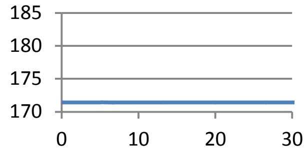
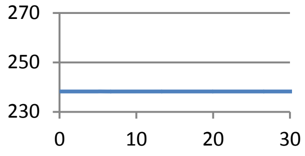
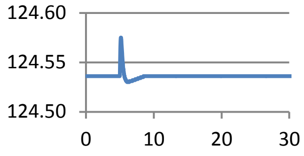
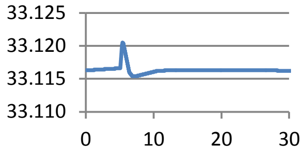
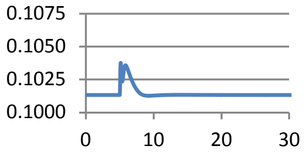
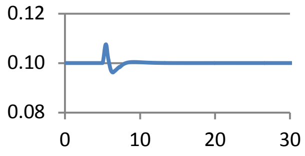
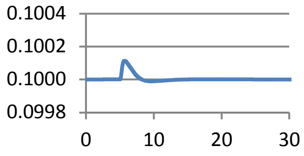
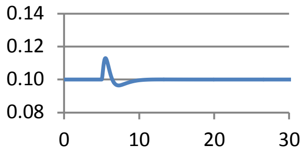
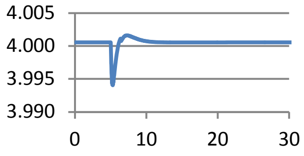
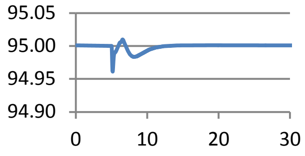
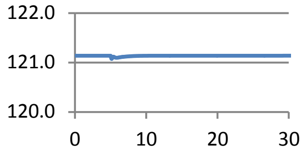
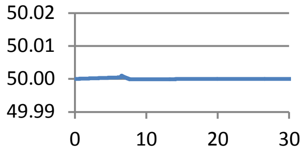
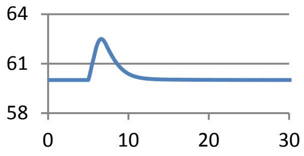
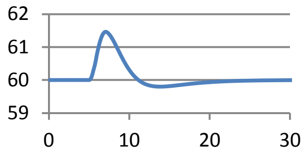
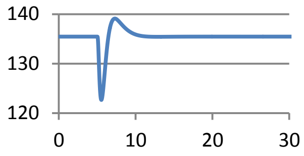
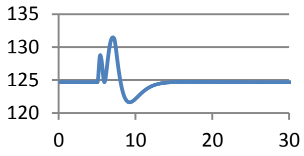
FIC3	FIC4	FIC5	FIC6
			
PC1	**PC2**	**PC3**	**PC4**
			
PC10	**TC1**	**TC2**	**TC3**
			
TC4	**TC5**	**TC6**	**TC7**
			

Table D.5: Dynamic simulation responses for CS1-CS5 when the water composition in the fresh carbonic acid feed changes to 0.5 (After 5 hours of the beginning of the simulation run)

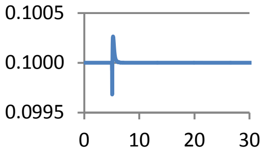
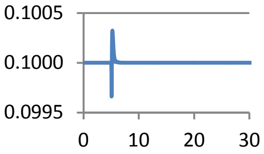
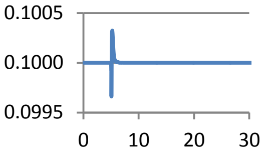
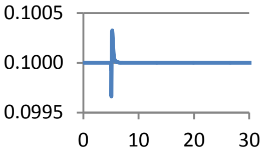
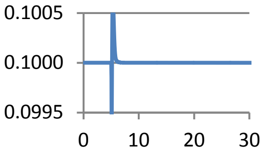
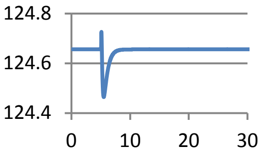
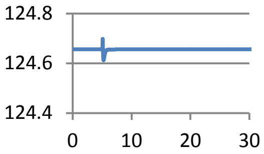
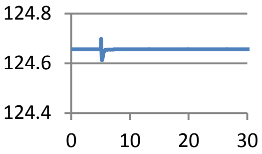
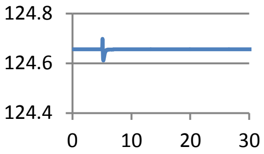
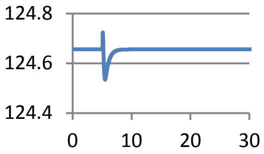
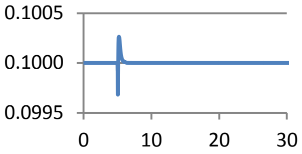
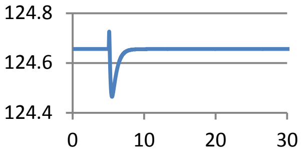
Controller	**CS1**	**CS2**	**CS3**	**CS4**	**CS5**
PC2					
TC7					

Table D.6: Dynamic simulation responses for CS6 when the water composition in the fresh carbonic acid feed changes to 0.5 (After 5 hours of the beginning of the simulation run)

PC2	TC7
	

APPENDIX E
ACTUAL YIELD

In this research, actual yields are sometimes greater than theoretical yield (100%); the question is whether it is possible. The answer of the question is that, if the amount of limiting reactant and product are measured accurately, it cannot be possible. However, the abovementioned explanation is not the cause of the measured (actual) yield in this research over the theoretical yield. The cause of this is the inventory control in the dynamic operation.

In the dynamics, the levels of any vessels, e.g., reactor and decanter, decrease when the flow of its feed stream is reduced. If the reaction is not in equilibrium (kinetics), the actual reaction yield is decreased due to the reducing of the residence time in the reactor (the discharge rate is greater than the feed rate). However, when the reactor reached to the new steady-state (the discharge rate equals to the feed rate), the actual reaction yield at the new condition will be over its initial value because of the increasing of the residence time. But, although the CSTRs with kinetic parameters of transesterification (kinetic model) are used in this research, the residence times of chemicals confined in the reactors are much more than the time required reaching the equilibrium; therefore, the change in the residence times does not affect the detected

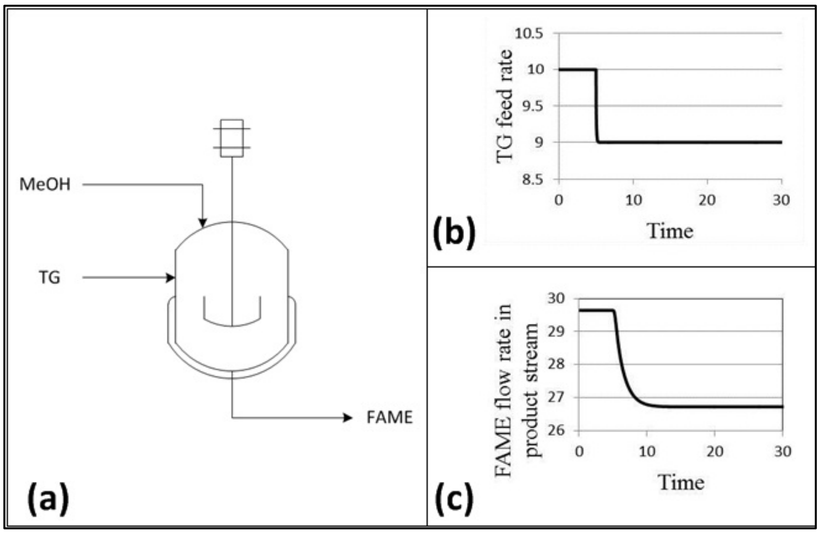


Figure E.1: Transesterification reactor (a) CSTR reactor, (b) Fresh TG feed, (c) FAME flow rate in product stream actual yield. As said above, the cause of the measured actual yield over 100% is the inventory control in the dynamic operation.

To understand more easily, a reactor which the transesterification occurs inside are considered. When the fresh triglyceride feed to the reactor are reduced suddenly, the level of the reactor reduces too. To achieve the setpoint of the level controller, the controller sends the signal to reduce the opening of the valve at the discharge stream. But the reducing of the opening of the valve is not sudden; therefore, the triglyceride feed is not consistent with the biodiesel in the product stream. The sensor of biodiesel detects that biodiesel reduces slowly from its initial value; while the triglyceride sensing shows that it reduces rapidly to its new setpoint. Therefore, the yield controller in the process can sometimes see that the measured actual yield is greater than 100% while the process transit to the new steady-state point.

Nowadays, there are analyzers which use Fourier Transform Infrared (FT-IR) or Fourier Transform Near-Infrared (FT-NIR) techniques to obtain infrared spectrums of absorption, emission, photoconductivity of many components in a solid, liquid, or gas simultaneously; therefore, only one analyzer can also detect the compositions of the many components in a stream simultaneously. In the biodiesel production process, MB3600 analyzer of ABB Corporation can detect the reactants (triglyceride and methanol), the intermediates (diglyceride and monoglyceride), the catalyst (sodium hydroxide and potassium hydroxide), and the products (methyl ester, glycerin, and water) within two minutes; if this real-time analyzer is used with the Extended Automation System 800xA of the corporation, it can be used in the real-time process control. Other company is Thermo Scientific; the analyzer from the company, which can be used as the composition sensing in the biodiesel plant, is Antaris II FT-NIR analyzer. The multi-component detection of the analyzer is also real-time, completed within 30 seconds, and can be used in-line or online to allow process adjustments using the closed-loop control strategies. Moreover, the company manufactures a multiplexing FT-NIR, Antaris MX, which have ability to measure multiple sample points simultaneously making in-line measurement more practical and economical.

VITAE

Mr. Tanapalin Yimlamai was born in Bangkok, Thailand on January 27, 1988. He graduated at King Mongkut's University of Technology Thonburi, Thailand and received the degree of Bachelor of Engineering in the field of Chemical Engineering with Second Class Honours in 2009. Next year, he entered to Graduate School of Chulalongkorn University to get the degree of Master of Engineering in Chemical Engineering.