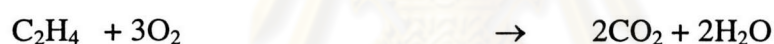
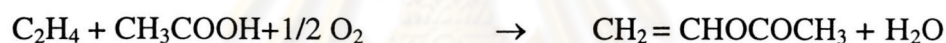


CHAPTER IV

Vinyl Acetate Process and Control Structures

4.1 Vinyl Acetate Process

The industrial process for the vapor-phase manufacture of vinyl acetate monomer is quite common and utilizes widely available raw materials. Vinyl acetate is used chiefly as a monomer to make polyvinyl acetate and other copolymers. Figure 4.1 shows the eleven basic unit operations in the process. Three raw materials, ethylene (C_2H_4), oxygen (O_2), and acetic acid (HAc), are converted into the vinyl acetate (VAc) product. Water and carbondioxide are by products. An inert component, ethane, enters with the fresh ethylene feed stream. Two reactions are considered the following:



The exothermic reactions occur in a reactor and heat is removed from the reactor by generating steam on the shell side of the tubes. Water flows to the reactor from steam drum, to which makeup water (boiler feeder water; BFW) is supplied. The steam leaves the drum as saturated vapor. The reactions are irreversible and the reaction rates have an Arrhenius-type dependence on temperature.

The following rate expressions were derived from the experimental kinetic data in Samanos et al. (1971).

$$r_1 = 0.1036 \exp(-3674/T) \frac{p_O p_E p_A (1 + 1.7 p_W)}{[1 + 0.583 p_O (1 + 1.7 p_W)] (1 + 6.8 p_A)} \quad 4.1$$

$$r_2 = 1.9365 \times 10^5 \exp(-10116/T) \frac{p_O (1 + 0.68 p_W)}{1 + 0.76 p_O (1 + 0.68 p_W)} \quad 4.2$$

Where r_1 has units of moles of vinyl acetate produced per minute per gram of catalyst and r_2 has units of moles of ethylene consumed per minute per gram of catalyst. T is the absolute temperature in Kelvin and p_i is the partial pressure of component i (O is oxygen, E is ethylene, A is acetic acid, and W is water) in psia.

The ideal-gas standard state heat of reaction is -42.1 kcal/mol of vinyl acetate for r_1 and -316 kcal/mol of vinyl acetate for r_2 . These values are calculated from ideal-gas heats of formation. Thus the reactions are quite exothermic, particularly the combustion reaction to carbon dioxide, which also is more sensitive to temperature because of the higher activation energy.

The reactor effluent flows through a process-to-process heat exchanger, where the cold stream is the gas recycle. The reactor effluent is then cooled with cooling water, and the vapor (oxygen, ethylene, carbon dioxide, and ethane) and liquid (vinyl acetate, water, and acetic acid) are separated. The vapor stream from the separator goes to the compressor and the liquid stream from the separator becomes a part of the feed to the azeotropic distillation column. The gas from the compressor enters the bottom of an absorber, where the remaining vinyl acetate is recovered. A liquid stream from the base is recirculated through a cooler and fed to the middle of the absorber. Liquid acetic acid that has been cooled is fed into the top of the absorber to provide the final scrubbing. The liquid bottoms product from the absorber combines with the liquid from the separator as the feed stream to the distillation column.

Part of the overhead gas exiting the absorber enters the carbon dioxide removal system. This could be one of several standard industrial CO_2 removal processes. This system is simplified by treating it as a component separator with a certain efficiency that is a function of rate and composition. The gas stream minus the carbon dioxide is split, with part going to the purge for removal of the ethane inert from the process. The rest combines with the large recycle gas stream and goes to the feed-effluent heat exchanger. The fresh ethylene feed stream is added. The gas recycle stream, the fresh acetic acid feed, and the recycle liquid acetic acid stream enter the vaporizer, where steam is used to vaporize the liquid. The gas stream from the vaporizer is further heated to the desired reactor inlet temperature in a trim heater using steam. Fresh oxygen is added to the gas stream from the vaporizer just prior to the reactor to keep the oxygen composition in the gas recycle loop outside the explosivity region.

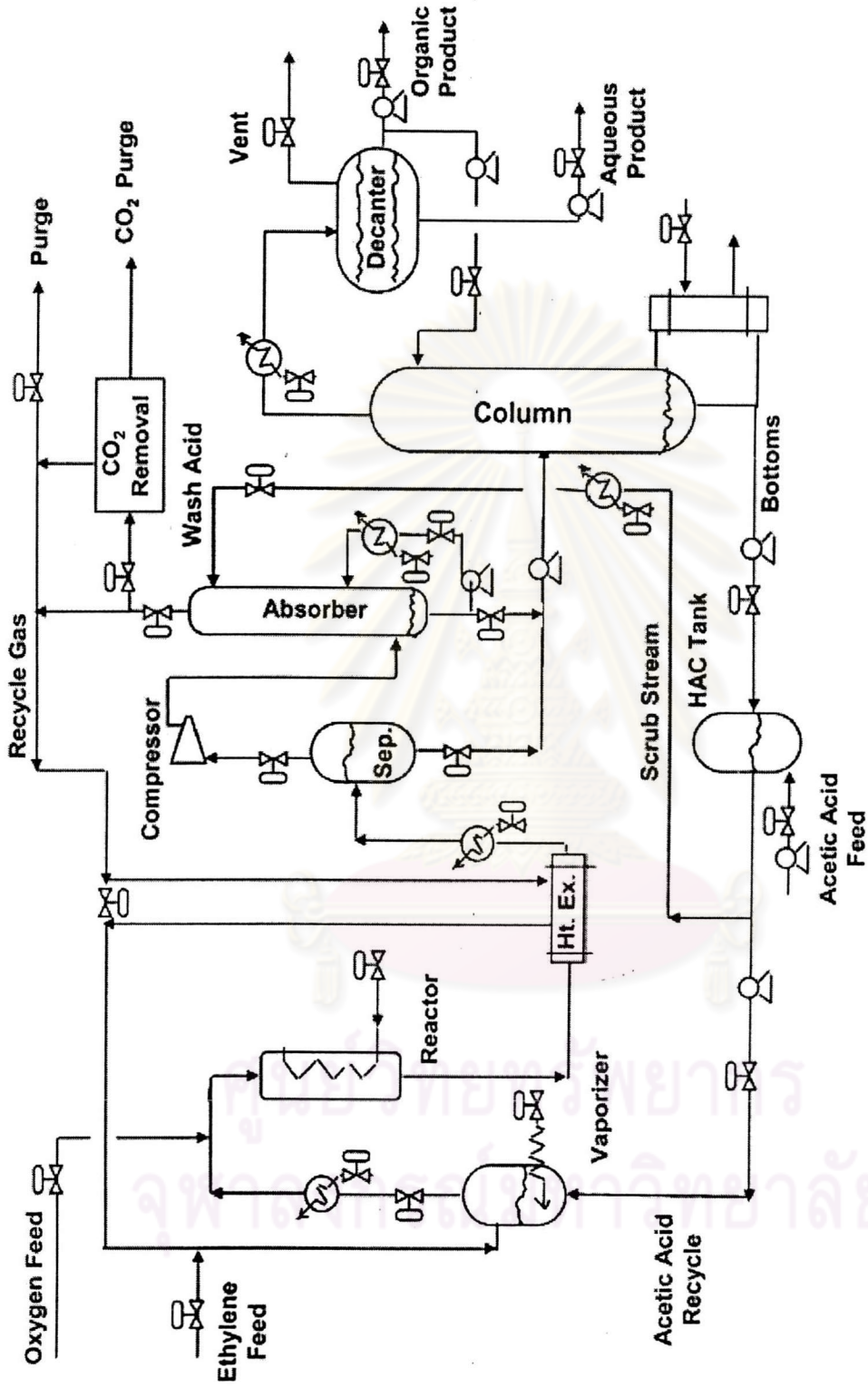


Figure 4.1 Vinyl Acetate Process flowsheet

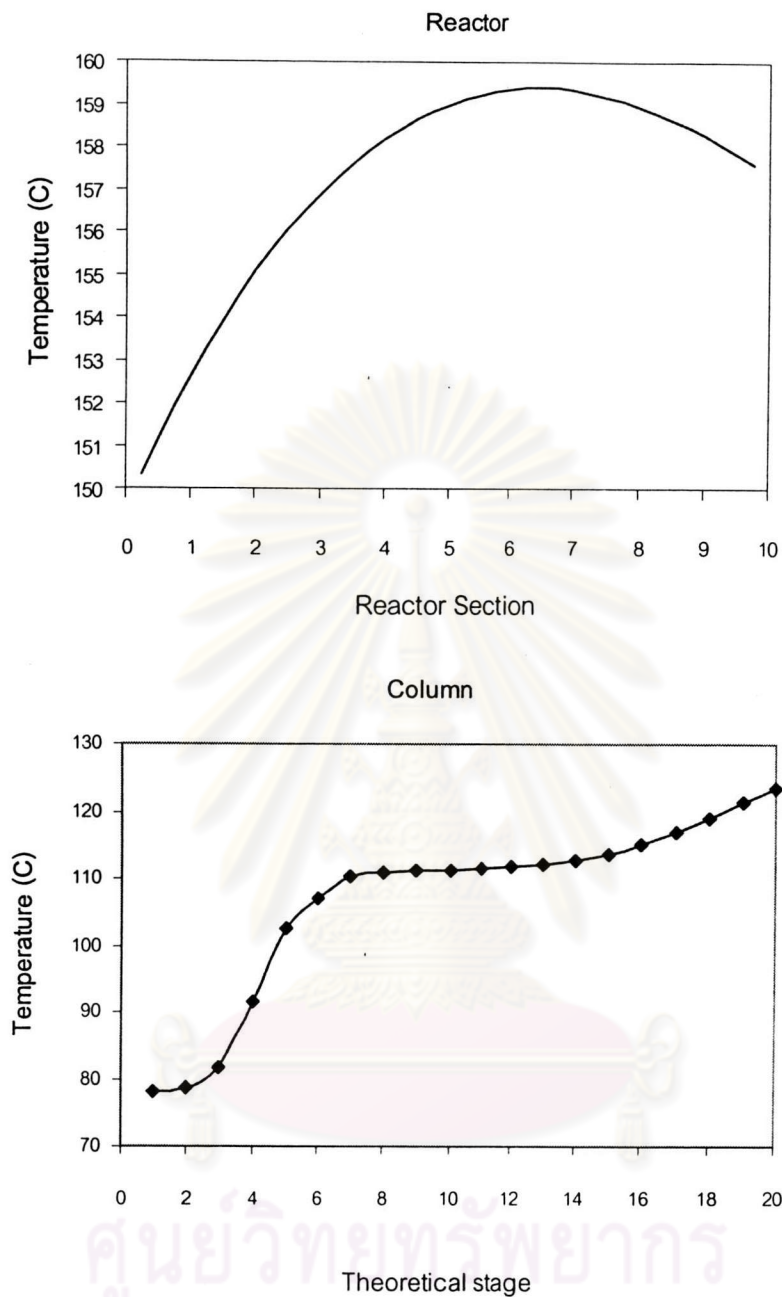


Figure 4.2 Temperature profiles (a) Azeotropic distillation column; (b) reactor

The azeotropic distillation column separates the vinyl acetate and water from the unconverted acetic acid. The overhead product is condensed with cooling water and the liquid goes to a decanter, where the vinyl acetate and water phases separate. The organic and aqueous products are sent for further refining to another distillation section. The bottoms product from the distillation column contains acetic acid, which

recycles back to the vaporizer along with fresh makeup acetic acid. Part of the bottoms stream is the wash acid used in the absorber after being cooled.

4.2 Steady state Simulation

In the steady state simulation, a large amount of information is necessary. The list of data and specifications includes:

1. Flowsheet topology (types and positioning of units)
2. Fluids package, two fluid packages are defined in the Vinyl Acetate process flowsheet. One for the main flowsheet is (Basis 1 with Wilson) and another for the decanter sub-flowsheet (Basis 2 with Van Laar). In the decanter the best property package is need for estimating the liquid-liquid equilibrium data but in the column the best physical property package for estimating the vapor-liquid equilibrium data of the vinyl acetate/ water/ acetic acid system is need. Usually two fluid packages are not the same.
3. Reactor types, sizes and conditions (temperatures, pressures, catalyst amounts, heat-transfer area, cooling or heating medium, etc.)
4. Reaction kinetic data
5. Physical property data.
6. Number of stages in all staged-operations (distillation columns, absorbers, liquid-liquid extraction columns, etc.) and operating conditions.
7. Heat-transfer rates, minimum approach temperatures, overall heat-transfer coefficients, flowrates of cooling or heating medium and areas in all heat exchangers.
8. Flowrates, temperatures, pressures and compositions of all process streams.

Some unit operation of Vinyl Acetate process from Luyben (1998) must be modified to simulate in HYSYS.

1. Vaporizer has only vapor outlet in these vessel but Hysys requires that a vessel must have both vapor and liquid exit streams. So the liquid line must be installed and place control valve that specify the valve opening to be 0%.
2. Reactor with boiler feeder water steam drum for cooling has not in Hysys so the plug flow reactor with cooling system is used and the reactor temperature profile is shown in Figure 4.2 (a).

3. An absorber has down stream unit attached to the liquid product stream, then a column sump will need to be simulated. There are several methods for simulating the column sump. A simple solution is to use a reboiled absorber, with the reboiler duty stream specified as zero in place of the absorber. Another option is to feed the liquid product stream directly into a separator, and return the separator vapor product to the bottom stage of the column.

4. Component splitter is chosen to simulate in Hysys instead CO₂ removal.

5. In azeotrope column, the reboiled absorber column is used and the three phase separator is added for decanter. Temperature profile in column is shown in Figure 4.2 (b)

4.3 Plantwide Control Strategy of Vinyl Acetate process

In this work, the three control structures are designed and compared with the reference control structure. All of schemes are generated from nine steps of plantwide control procedure. The reference control structure I (CS1) that are presented by Luyben et al. (1998). Including the designed control structures CS2, CS3 and CS4 are simulated by HYSYS simulation. After the control schemes are implemented, three setpoint disturbances are used to illustrate the dynamic behavior of them.

Step 1: Establish control objectives

For this process, the objective must be able to set the production rate of vinyl acetate while minimizing yield losses to carbon dioxide. The oxygen concentration in the gas loop must remain outside the explosivity region for ethylene and the azeotropic distillation column must produce an overhead product with essentially no acetic acid and a bottoms product with no vinyl acetate.

Step 2: Determine control degrees of freedom

There are 25 control degrees of freedom in this process. They include: three feed valves for oxygen, ethylene, and acetic acid; vaporizer and heater steam valves; reactor cooling water valves; vaporizer overhead valve; two coolers and absorber

cooling water valves; separator base and overhead valves; absorber overhead, base, wash acid, and liquid recirculation valves; gas valve to CO₂ removal system; gas purge valve; distillation column stream and cooling water valves; column base, reflux, and vent valves; and decanter organic and aqueous product valves

Step3: Establish energy management system

Energy management is critically important because of the highly exothermic reactions and potential for runaway. By design, heat is removed from the reactor via cooling water in the reactor. All of structures use the cooler utility valve to control the reactor exit temperature because of its direct effect. The reactor effluent stream is cooled in a process-to-process heat exchanger with the gas recycle stream. A bypass line and control valve are necessary to control one of the exchanger exit temperatures. If this exchanger is designed for only vapor flow, then the hot-side exit temperature must be controlled to a value above the dewpoint temperature by manipulating bypass valve around the exchanger on the cold side. The bypass line would not have to be added if the heat exchanger was designed to handle two-phase flow.

Step 4: Set production rate

For the CS1, CS2 and CS3 structures, ethylene and oxygen makeup feeds come from headers and the acetic acid feed is drawn from a supply tank. The vinyl acetate and water products go to downstream units. As a result, there are no design constraints that require production rate to be set either on supply or demand. Therefore, look at reactor conditions to determine how to change production rate. Because the reactor feed contains both excess ethylene and acetic acid, manipulating the partial pressure of either component would not be effective. The partial pressure of oxygen is constrained by the safety limit, and once this is reached no further adjustments could be made. Pressure is limited by the process equipment design maximum. Therefore the most direct handle for setting production rate is by changing the reactor exit temperature.

In the CS4 structure, the flowrate of fresh ethylene feed is flow-controlled, which set the production rate. The process must take whatever amount of fresh ethylene feed because it is limiting reactant in reaction.

Step 5: Control product quality and handle safety, operational, and environmental constraints

Product quality

The azeotropic distillation column does not produce the final salable vinyl acetate product. Its primary role is to recover and recycle unreacted acetic acid and to remove from the process all of the vinyl acetate and water produced. So acetic acid in the overhead should be a little amount because this represents a yield loss. Also, the bottoms stream should contain no vinyl acetate. Water is an intermediate component with a boiling point between vinyl acetate and acetic acid. It can be chosen to control the product quality in this column. The inventory of water in the process will be automatically accounted if its component is controlled.

In the CS1 and the CS4 structures, the composition of vinyl acetate is controlled by changing column steam. The temperature profile in figure 4.2a has a sharp break representing the change in vinyl acetate composition near the bottom of the column. Hence Column steam (boilup) is the appropriate choice for temperature control because of its fast response compared with reflux. The reflux flow is used to control the water composition in the bottom stream. The dynamics of the composition loop are slow because the reflux flow is far from the bottom stream. Therefore cascade control is used to achieve this quick response.

Instead, in the CS2 structure is changed to control water composition in the distillate by using reflux flow directly. Reflux has a direct effect in changing water composition in distillate so it has a fast response with no cascade controlling.

The CS3 structure is changed the control scheme that is appropriate the process has high boilup ratio (boilup ratio >4) in column. For this scheme, the bottom flow should be used to control bottom composition and heat input should control base level. One of the strengths of this system is that it is fast response in large column. For the Vinyl Acetate process, boilup ratio in azeotrope column is 4.97 so this scheme is chosen. Hence the vinyl acetate composition or temperature profile in column is controlled by changing the bottom flowrate and column base level is controlled by heat input. The reflux flow is used to control water composition in distillate similar to the CS2 structure.

Safety

The overriding safety constraint in this process involves oxygen concentration in the gas loop, which must contain below 0.1 mole to remain outside the explosivity envelope for ethylene mixtures at process conditions. The most direct manipulated variable to control oxygen composition at the reactor feed is fresh oxygen feed flow.

Step 6: Control Inventories (Pressures and Levels) and Fix a Flow in Every Recycle Loop.

Two pressures must be controlled: in the column and in the gas loop. The most direct handle to control column pressure is controlled by manipulating the vent stream from the decanter. In gas loop pressure, fresh ethylene feed flow is chosen to control gas recycle loop pressure because it has a large effect more than purge flow and flow to CO₂ removal.

In the CS4 structure, fresh ethylene feed has been previously selected to set production rate so it cannot be used to control pressure. There are three vessels (vaporizer, separator, absorber) within the gas loop. The overhead vapor stream valves from vessels can be used to manipulate gas pressure loop instead of fresh ethylene feed. The vapor stream from the separator is selected because its flow is largest.

Six liquid levels are in the process: vaporizer, separator, absorber, column base, and two decanter layers. Control of the decanter levels is straightforward. The organic product flow controls the organic phase inventory and the aqueous product flow controls the aqueous phase inventory.

The most direct way to control the remaining levels would be with the exit valves. However, All of the flows around the liquid recycle loop would be set on the basis of levels, which would lead to undesirable propagation of disturbances. So a flow somewhere in this loop should be controlled. Acetic acid is the main component in the liquid recycle loop. Recycle and fresh acetic acid feed determine the component's composition is the reactor feed. A reasonable choice at this point is to control the total acetic acid feed stream flow into the vaporizer. This means the fresh acetic acid feed stream can be used to control column base level, since this is an indication of the acetic acid inventory in the process. Vaporizer level is then

controlled with the vaporizer steam flow and separator and absorber levels can be controlled with the liquid exit valves from the units.

In the CS2 and the CS3 structures is changed the manipulator of column base level. The liquid recycle loop has a HAc tank that can reduce an undesirable propagation of disturbances and snowball effect. Thus column base level can be controlled by column bottom exit valve and fresh acetic acid feed is used to manipulate the total acetic acid feed stream flow into the vaporizer.

Step 7: Check Component Balances.

Table 4.1 summarize the component balance control strategy.

- Ethane is an inert component that enters with the ethylene feed. It can be removed from the process only via the gas purge stream, so purge flow is used to control ethane composition.
- Carbon dioxide is an unwanted by-product that leaves in the CO₂ removal system. As long as the amount of carbon dioxide removed is proportion in some way to the CO₂ removal system feed, this valve is used to control carbon dioxide composition.
- Oxygen inventory is accounted for via composition control with fresh oxygen feed.
- Inventory of ethylene can be controlled to maintain gas loop pressure, since ethylene composes the bulk of the gas recycle. In CS4 structure, ethylene inventory is regulated by using flow control of reactor feed.
- Acetic acid inventory is regulated by using the fresh acetic acid feed to control base level in the distillation column. In CS2 and CS3 structures, acetic acid inventory is regulated by using the fresh acetic acid feed to control total acetic feed in vaporizer.
- The temperature control loop in the distillation column achieves vinyl acetate composition control.

- The inventory of water in the process for via water composition control in azeotrope column.

Table 4.1 Component Material Balance

Component	Input	+ Generation	- Output	- Consumption	= Accumulation Inventory controlled by
O ₂	Fresh feed	0	0	$V_R(0.5r_1+3r_2)$	Composition control of reactor feed
CO ₂	0	$2V_Rr_2$	CO ₂ removal	0	Composition control of recycle gas
C ₂ H ₄	Fresh feed	0	0	$V_R(r_1+r_2)$	Pressure control of recycle gas loop CS4: Flow control of reactor feed
C ₂ H ₆	Fresh feed	0	Purge stream	0	Composition control of recycle gas
VAc	0	V_Rr_1	Product stream	0	Temperature control in distillation column
H ₂ O	0	$V_R(r_1+2r_2)$	Product stream	0	Column composition control
HAc	Fresh feed	0	0	V_Rr_1	Level control in column base CS2,CS3: flow control of vaporizer feed

Where V_R is the reactor volume

r_1 is vinyl acetate reaction rate

r_2 is side reaction rate

Step 8: Control Individual Unit Operations

Several control valves now remain unassigned. Steam flow to the trim heater controls reactor inlet temperature. Cooling water flow to the trim cooler is used to control the exit process temperature and provide the required condensation in the reactor effluent stream. Liquid recirculation in the absorber is flow-controlled to

achieve product recovery, while the cooling water flow to the absorber cooler controls the recirculating liquid temperature. Acetic acid flow to the top of the absorber is flow-controlled to meet recovery specifications on the overhead gas stream. Cooling water flow to the cooler on this acetic acid feed to the absorber is regulated to control the stream temperature. Cooling water flow in the column condenser controls decanter temperature.

Step 9: Optimize Economics or Improve Dynamic Controllability

The basic regulatory strategy has been established. It has some remaining degree of freedom to optimize steady state economic process performance or to improve dynamic response. Based upon the heuristic established by Fisher et al. (1988) that recycle gas flows should be maximized to improve reactor yield. The separator, vaporizer, and absorber overhead valves is full opened and run the compressor full out. The CS4 structure has two remaining control valves because the separator overhead control valve has been selected to control gas loop pressure in step 7. To minimize the decanter temperature for improved organic recovery, the column condenser cooling water is set at maximum flow. Optimization of several controller setpoints can be based upon economics.

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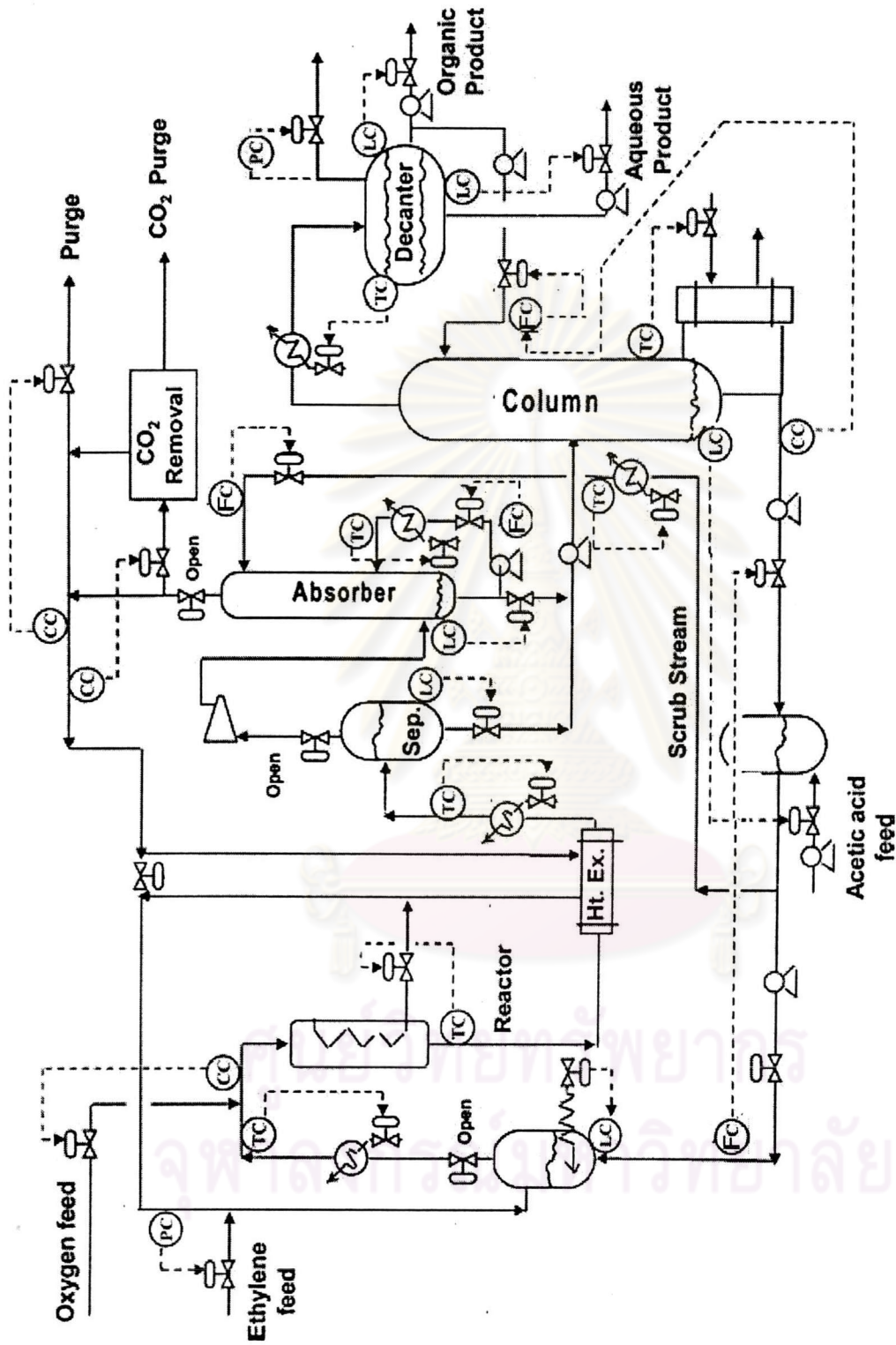


Figure 4.3 Reference control structure (CS1) of Vinyl Acetate Process

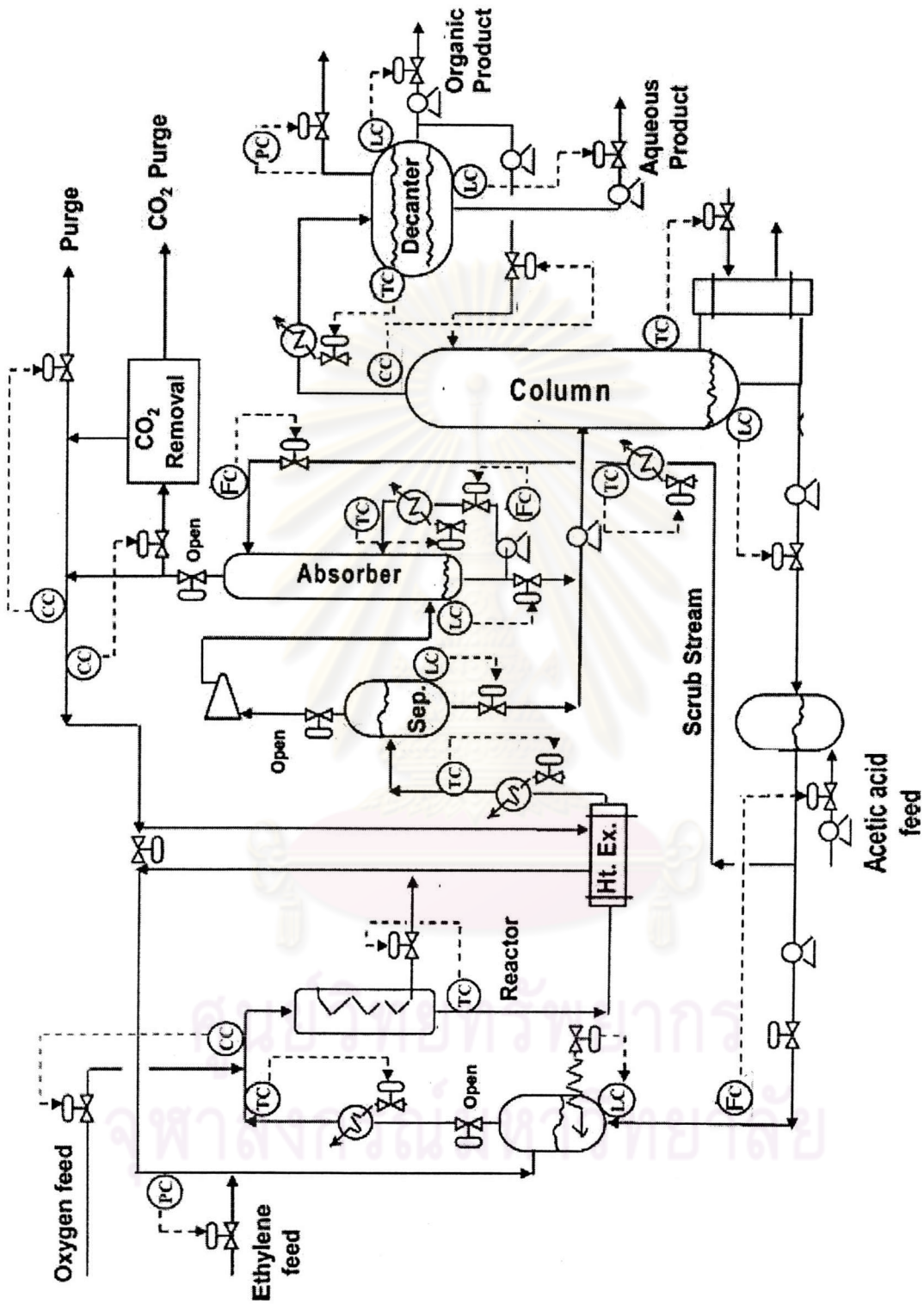


Figure 4.4 Designed control structure I (CS2) of Vinyl Acetate Process

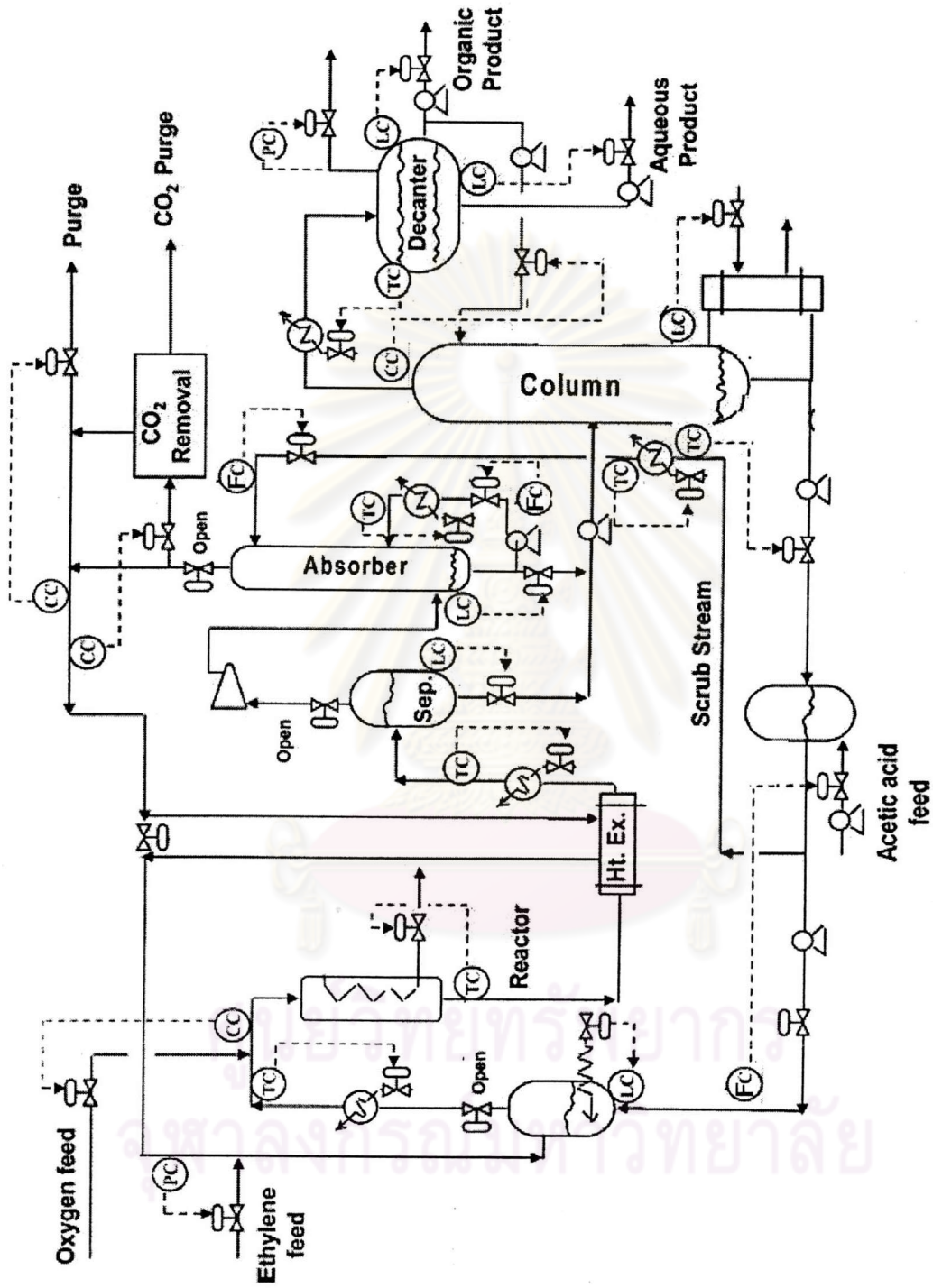


Figure 4.5 Designed control structure II (CS3) of Vinyl Acetate Process

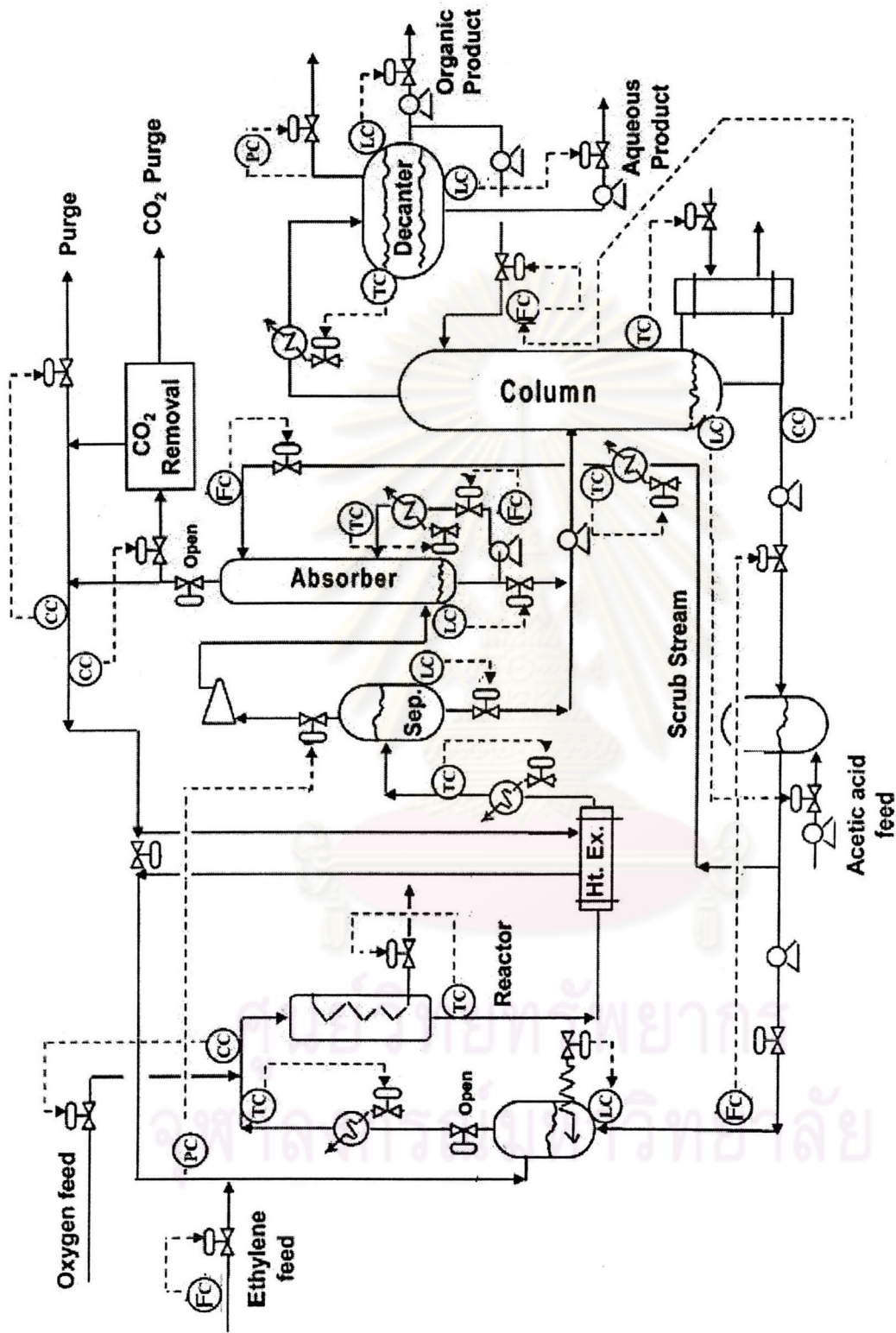


Figure 4.6 Designed control structure III (CS4) of Vinyl Acetate Process

4.4 Control structure Alternatives

The four alternative control structures that are designed in this work are shown in figure 4.3-4.6. In all of these schemes, the following loops are used:

1. The reactor exit temperature is controlled by the reactor cooler utility valve.
2. The oxygen composition at the reactor feed is controlled by the fresh oxygen feed flow.
3. The column pressure is controlled by manipulating the vent stream from the decanter.
4. The organic product flow is manipulated to control the organic level of decanter.
5. The aqueous product flow is manipulated to control the aqueous level of decanter.
6. The vaporizer level is controlled by the vaporizer steam flow.
7. The separator level can be controlled with the liquid exit valves from its units.
8. The absorber level can be controlled with the liquid exit valves from its units.
9. The purge flow is used to control the ethane composition.
10. The feed flow to the CO₂ removal system is used to control the CO₂ composition.
11. The steam flow to the trim heater is used to control the reactor inlet temperature.
12. The separator coolant flow is used to control separator temperature.
13. The liquid recirculation in the absorber is flow-controlled.
14. The absorber scrub flowrate from the bottom of column is flow-controlled.
15. The circulation stream temperature is controlled by the cooling water flow to the absorber cooler.
16. The absorber scrub temperature is controlled by the cooling water flow to the cooler.
17. The cooling water flow in the column condenser is used to control decanter temperature.

The four alternative control structures had some different control loops. The following loops are used:

1. In the CS1, CS2 and CS3, the gas recycle loop pressure is controlled by changing the fresh ethylene feed but in the CS4, the gas recycle loop pressure is controlled by manipulating the separator overhead flow.
2. The CS1 and CS4 use the reboiler duty to control the column temperature and use the reflux flow to control the water composition by cascade control. In the CS2 the water composition control loop is changed. The overhead water composition in column is controlled by reflux flow directly instead of using the cascade control. The CS3 is modified from the CS2, the reboiler duty is used to control the column base level and the bottom is used to control the tray temperature in column.
3. In the CS1 and CS4, the fresh acetic acid feed valve is used to control column base level and the total acetic acid flowrate is controlled by manipulating the bottom flow from azeotrope column. However, in the CS2 and CS3, the fresh acetic acid feed is used to control the total acetic feed to vaporizer but the column base level had the different manipulator.

4.5 Reference structure (CS1)

The reference structure of Vinyl acetate process is presented by Luyben (1998) in figure 4.3. This structure is used fresh acetic feed valve to control base level of azeotrope column and used fresh ethylene feed valve to control gas recycle loop pressure. In azeotrope column, the water in bottoms is controlled by using cascade control to manipulate the reflux flow.

4.5.1 Change in reactor temperature

- decrease temperature

The starting conditions are the base-case design where reactor exit temperature is 159.5 °C. The reactor temperature controller is tuned at this operating point. Step changes of 8 °C decreasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

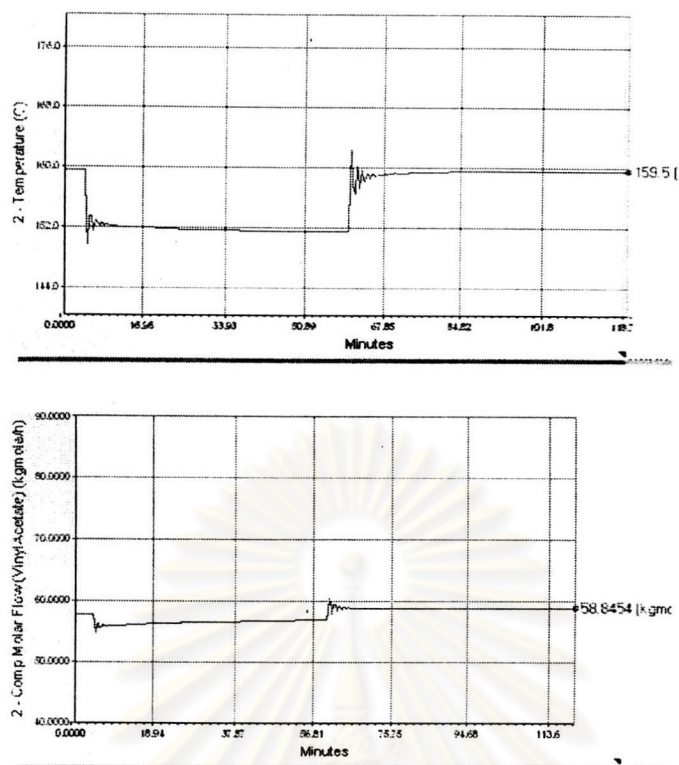


Figure 4.7(a) Dynamic response of base case to 8°C decrease in reactor temperature of CS1 structure

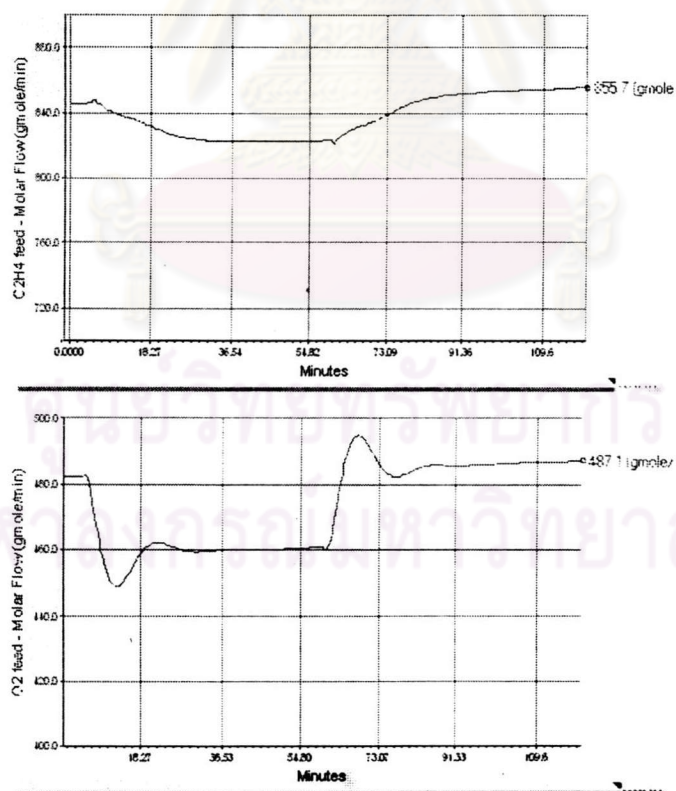


Figure 4.7 (b) Dynamic response of base case to 8°C decrease in reactor temperature of CS1 structure

Figure 4.7 (a) gives simulation result for decreasing the temperature. The reactor temperature decrease, the rate of production of vinyl acetate decrease because of decreasing temperature can reduce the reaction rate. The temperature response is oscillatory and it comes close to new setpoint value within 40 minute. When reaction rate decrease, a large amount of ethylene, oxygen and acetic acid remain in reactor so the fresh feed of them is decrease.

Figure 4.7(b) and (c) show the fresh feed of ethylene, oxygen and total acetic acid feed in process and oxygen composition in gas loop responses. The fresh feed ethylene and oxygen decrease fairly quickly because the process has a gas-phase reactor. The flow controller of total acetic feed has a small changing and can recovers in about 25 minutes. At the beginning oxygen composition reactor inlet increase because but it remain within about 0.1 mol % of setpoint.

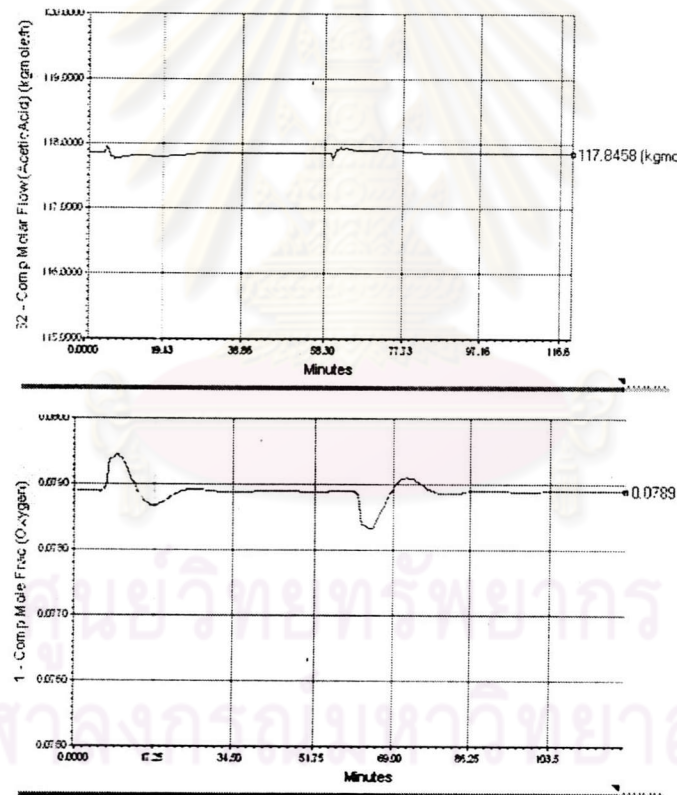


Figure 4.7 (c) Dynamic response of base case to 8°C decrease in reactor temperature of CS1 structure

The separation section does not see the changing in temperature disturbance load because the large time constant in liquid recycle loop and the structure has the controlling of total acetic acid flow to the reactor.

- *increase temperature*

Step changes of 6 °C decreasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

Figure 4.8 (a) and (b) give result for step increase in the reactor temperature controller setpoint from 159.5 to 165.5 °C. The step increase in reactor temperature response is slightly more oscillatory than the decrease because this reactor is highly nonlinear. It comes close to new setpoint value within 30 minute. Increasing the reactor temperature setpoint increases the production rate of vinyl acetate, so there must ultimately be net increases in all three fresh reactant feed streams. Oxygen and ethylene flow respond fairly quickly within about 50 minutes. Figure 4.8 (c) shows the response of total acetic feed and oxygen composition. The total acetic acid is slightly decrease and recovers in about 30 minutes. The response of oxygen composition decrease and return to the setpoint at 30 minutes and it stands the safety constraint.

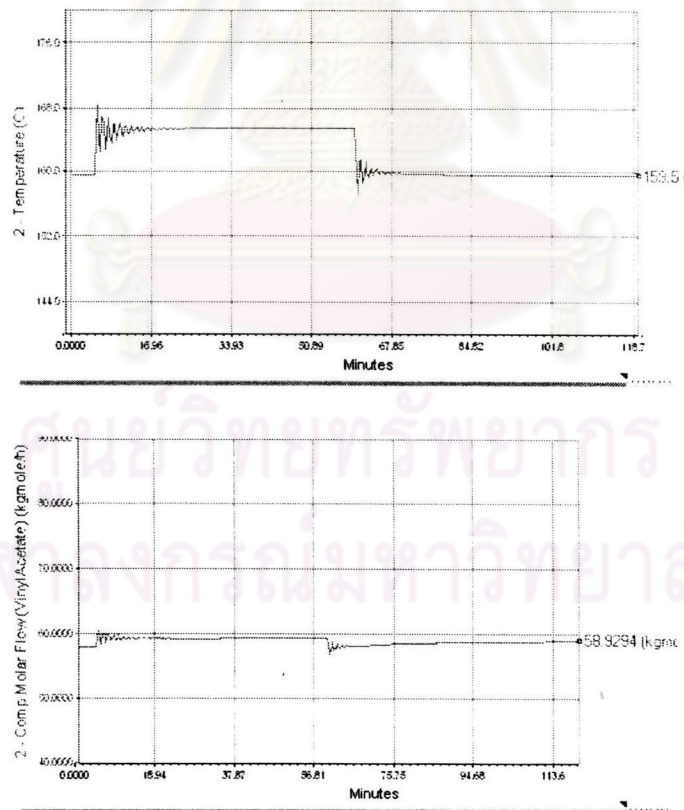


Figure 4.8 (a) Dynamic response of base case to 6°C increase in reactor temperature of CS1 structure

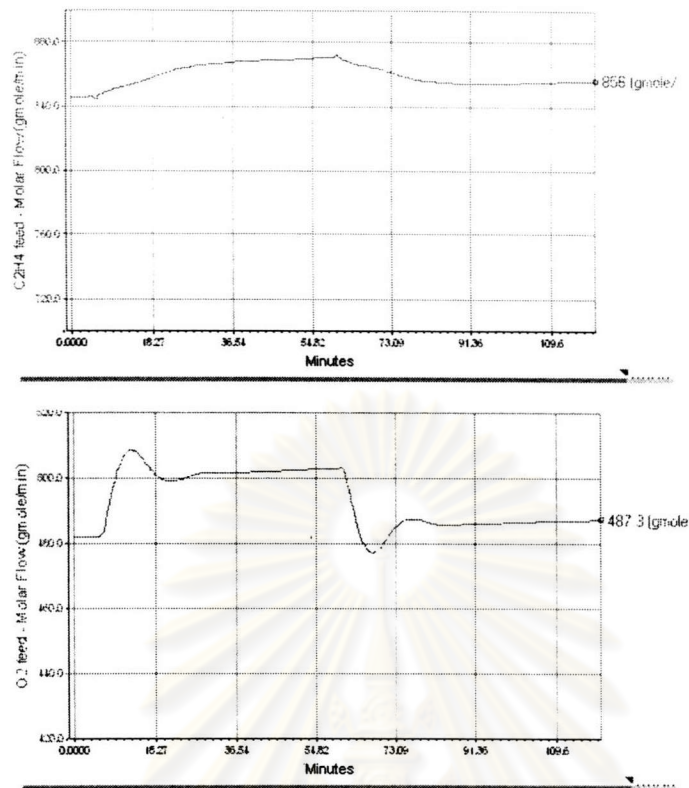


Figure 4.8 (b) Dynamic response of base case to 6°C increase in reactor temperature of CS1 structure

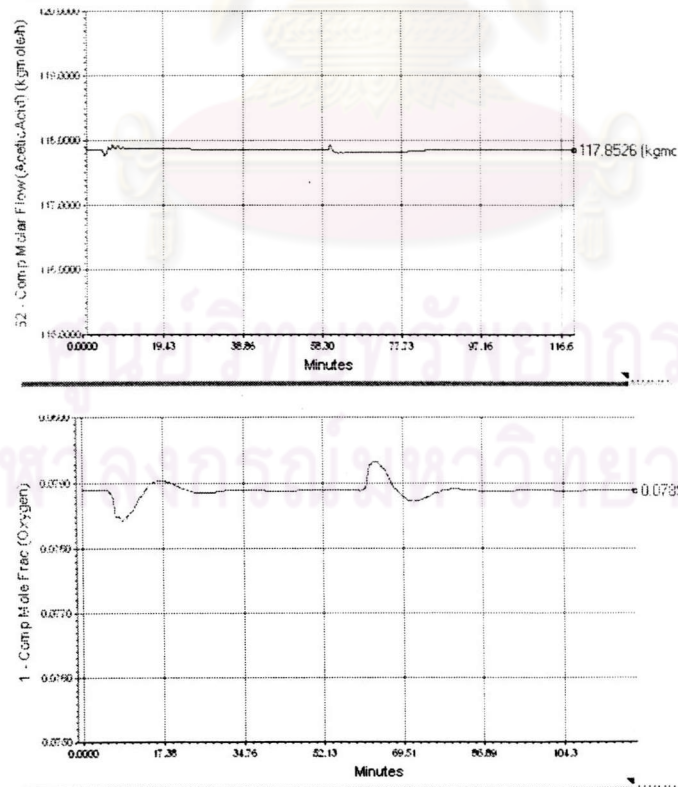


Figure 4.8 (c) Dynamic response of base case to 6°C increase in reactor temperature of CS1 structure

4.5.2 Change in acetic acid recycle flowrate

Step changes of 20 percent at time 5 minutes and 100 minutes are made in the setpoint of the total acetic acid flow controller

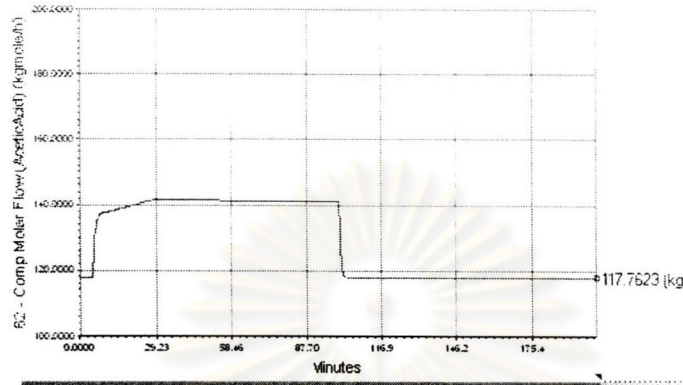


Figure 4.9 (a) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS1 structure

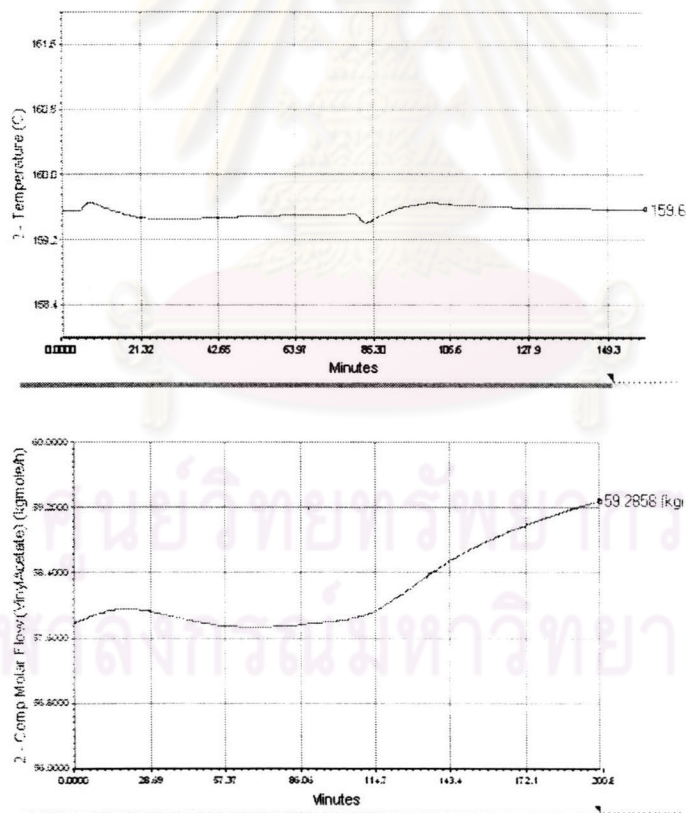


Figure 4.9 (b) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS1 structure

Figure 4.9 gives the dynamic responses of the process for a 20 percent increase in the flowrate of total acetic acid to the vaporizer. The flow reaches new

setpoint after 55 minutes in figure 4.9 (a). The large step increase in recycle flowrate causes a drop in reactor exit temperature. Increasing recycle flow of acetic acid almost instantaneous effect of decreasing reactor temperature because more material in flowing and another reason is due to the unusual reaction kinetics in equation 4.2. After temperature drop, the reactor temperature controller is changed duty to return back to its initial condition. The increase reactor temperature results in a decrease in the reaction rate so the production rate of vinyl acetate decreases in figure 4.9 (b).

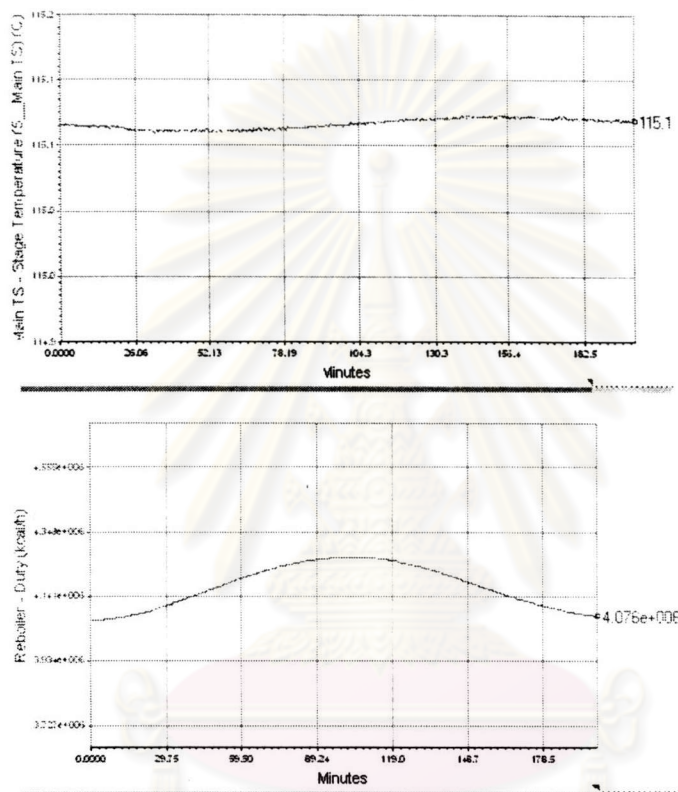


Figure 4.9 (c) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS1 structure

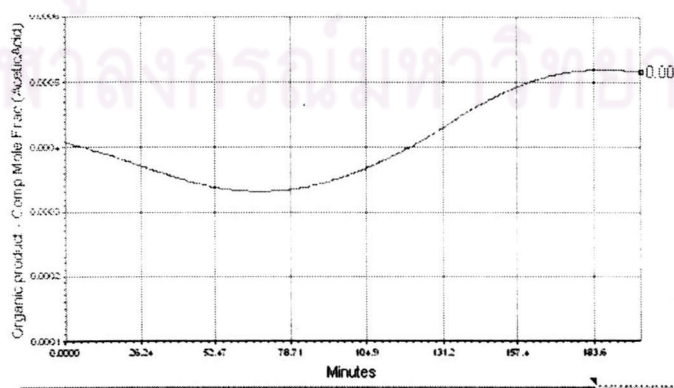


Figure 4.9 (d) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS1 structure

The increase of the total acetic flowrate causes the increase in the column feed. When the feed flow of column increase, it will affect the decrease in tray temperature because of more material inflowing. Therefore, the reboiler heat input increase to manipulate the tray temperature return to the setpoint in figure 4.9 (c).

Figure 4.9 (d) show the response of acetic acid composition in organic product. When the feed in column rises, the temperature in column drop so heavy key component (acetic acid) in the overhead of column decrease. While the heat input manipulate the tray temperature in column increase to the setpoint, acetic acid composition increases.

4.6 Designed Control structure I (CS2)

This scheme that is shown in figure 4.4 is changed to control base column level by manipulating bottom flow of column and control total acetic feed in vaporizer by manipulating the fresh acetic feed. The dynamic response flow control loop is faster than the reference structure (CS1). Including, the water composition control loop is changed. The overhead water composition in column is controlled by reflux flow directly instead of using the cascade control. The cascade control, two controllers are used: a primary controller and secondary controller. The setpoint of the reflux flow controller comes from the water in bottom composition controller. Although the cascade control gives the good dynamic responses, its tuning is more difficult. An advantage of this scheme is the fast response in total acetic acid flow controlled loop.

4.6.1 Change in reactor temperature

- *decrease temperature*

The starting conditions are the base-case design where reactor exit temperature is 159.5 °C. The reactor temperature controller is tuned at this operating point. Step changes of 8 °C decreasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

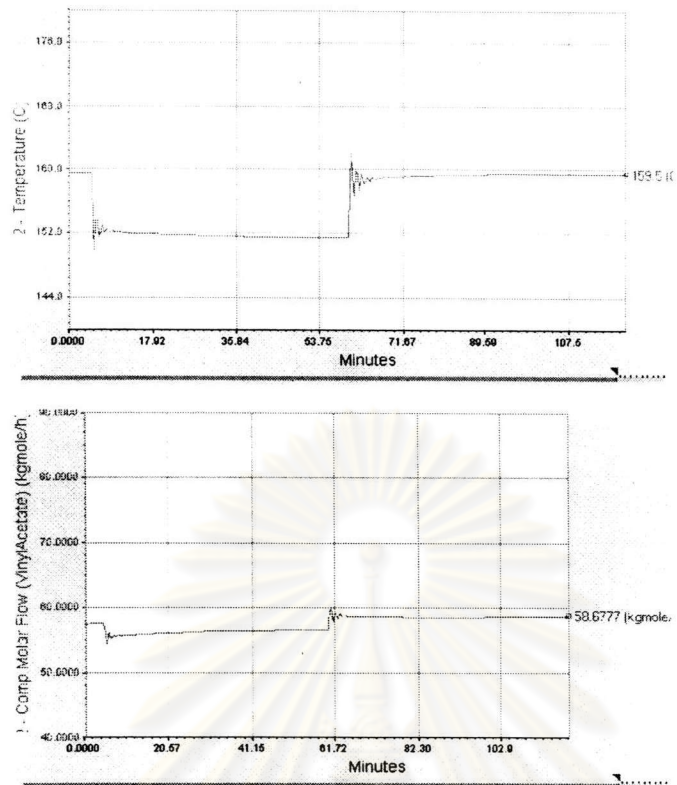


Figure 4.10(a) Dynamic response of base case to 8°C decrease in reactor temperature of CS2 structure

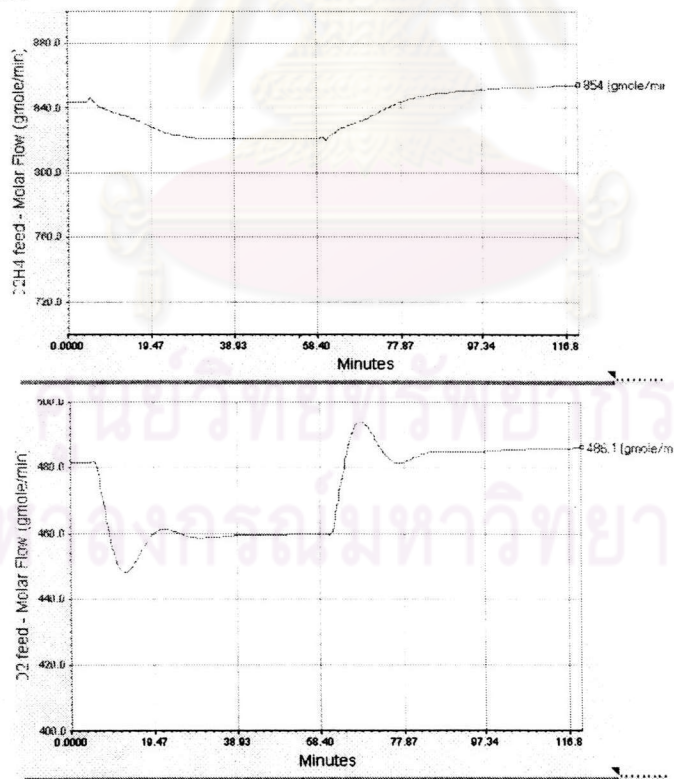


Figure 4.10(b) Dynamic response of base case to 8°C decrease in reactor temperature of CS2 structure

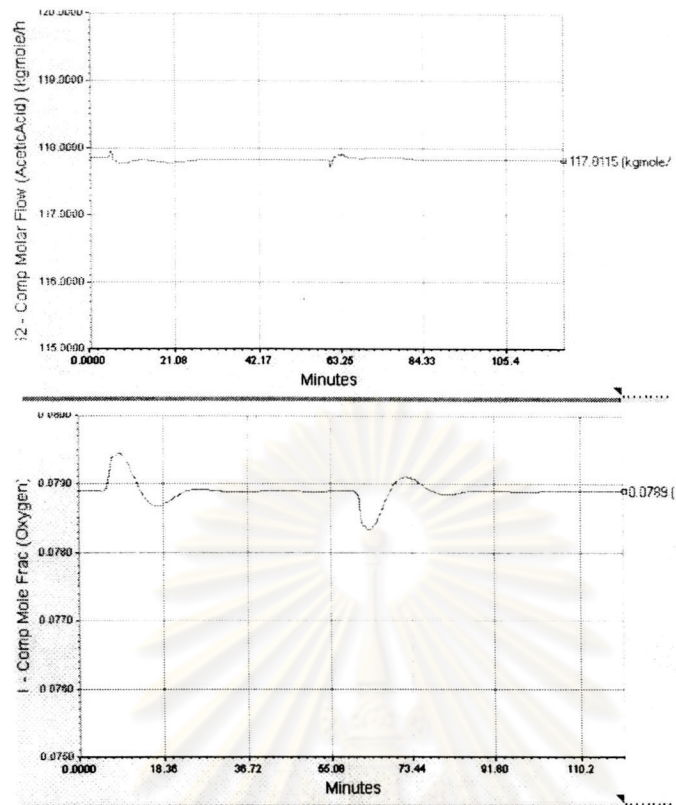


Figure 4.10(c) Dynamic response of base case to 8°C decrease in reactor temperature of CS2 structure

Figure 4.10 give result for step increase in the reactor temperature controller setpoint from 159.5 to 151.5°C . The response of this scheme is similar to CS1. The decreasing of reactor temperature results the production of vinyl acetate decreases so the fresh feed of oxygen and ethylene decrease. The response of oxygen composition loop remains the safety constraint. The reactor temperature reaches the new setpoint at 35 minute.

- *increase temperature*

The starting conditions are the base-case design where reactor exit temperature is 159.5°C . The reactor temperature controller is tuned at this operating point. Step changes of 6°C increasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

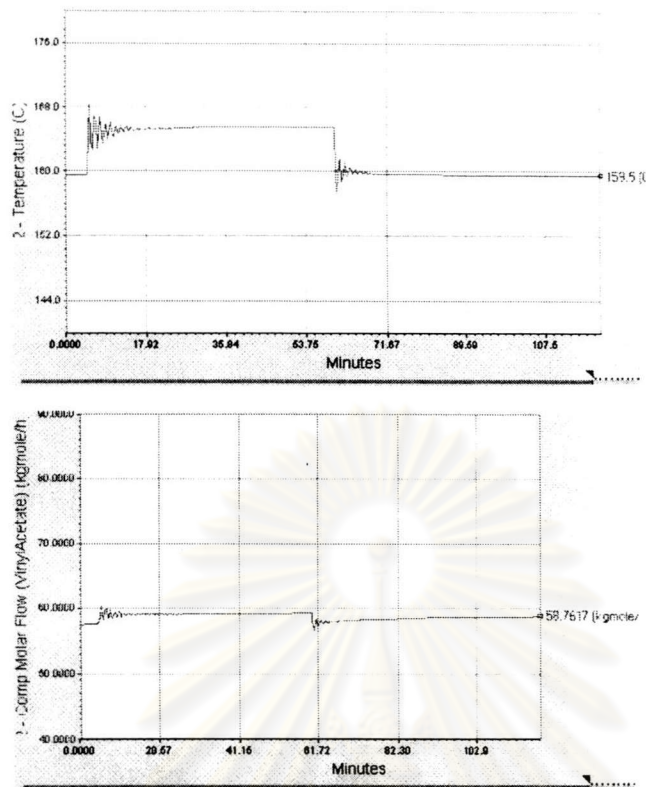


Figure 4.11 (a) Dynamic response of base case to 6°C increase in reactor temperature of CS2 structure

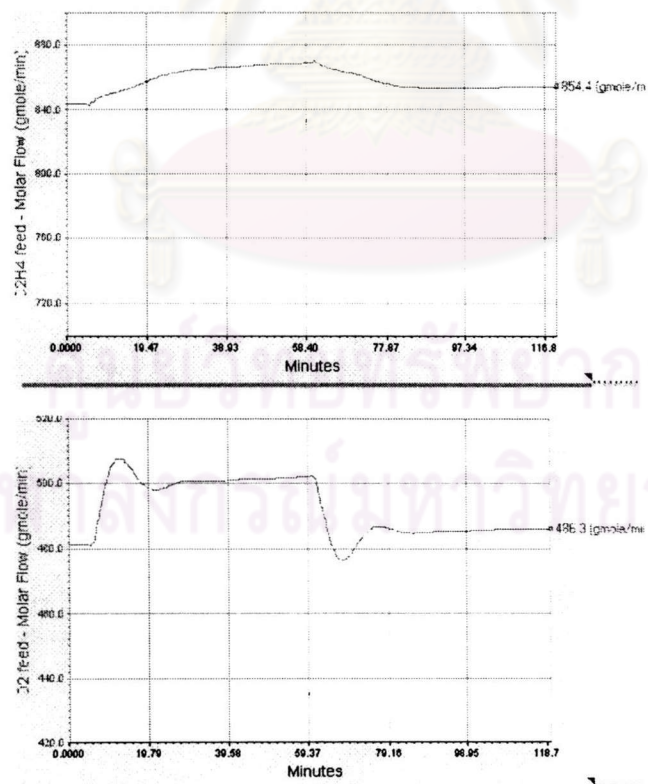


Figure 4.11(b) Dynamic response of base case to 6°C increase in reactor temperature of CS2 structure

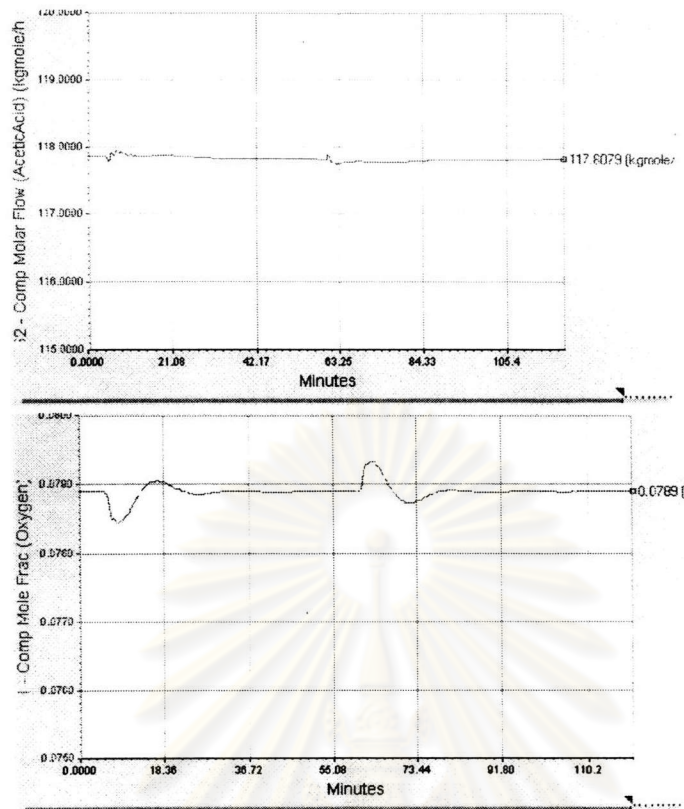


Figure 4.11(c) Dynamic response of base case to 6°C increase in reactor temperature of CS2 structure

Figure 4.11 give result for step increase in the reactor temperature controller setpoint from 159.5 to 165.5 °C. Increasing the temperature is more oscillatory than decreasing temperature but it can get the new setpoint within 30 minute that is faster than decreasing. The increasing of reactor temperature results the production of vinyl acetate increases because at the high temperature the reaction rate occurs rapidly. Therefore, the fresh feed of oxygen and ethylene flow increase. The response of oxygen composition loop remains the safety constraint.

4.6.2 Change in acetic acid recycle flowrate

Step changes of 20 percent at time 5 minutes and 100 minutes are made in the setpoint of the total acetic acid flow controller.

Figure 4.12 gives the dynamic responses of the process for a 20 percent increase in the flowrate of total acetic acid to the vaporizer. In figure 4.12 (a), the flowrate reaches new setpoint after 15 minutes that is faster than CS1 because the

fresh acetic feed valve is direct effect to control the total acetic feed more bottoms of column valve. The increase in total flowrate of acetic acid decreases the reactor temperature, and production rate of vinyl acetate. After total acetic acid flow decreases in base case, the production rate increases rapidly because of unusual reaction rate.

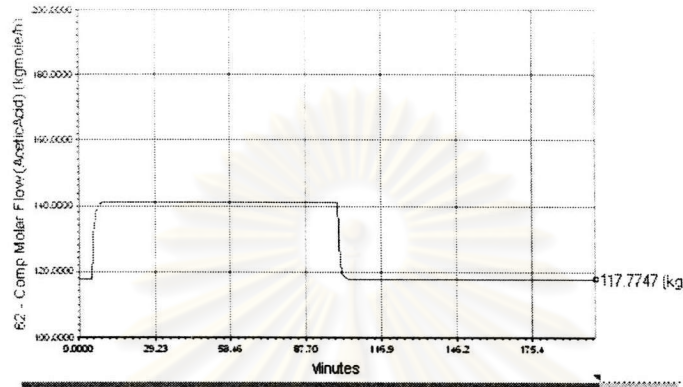


Figure 4.12 (a) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS2 structure

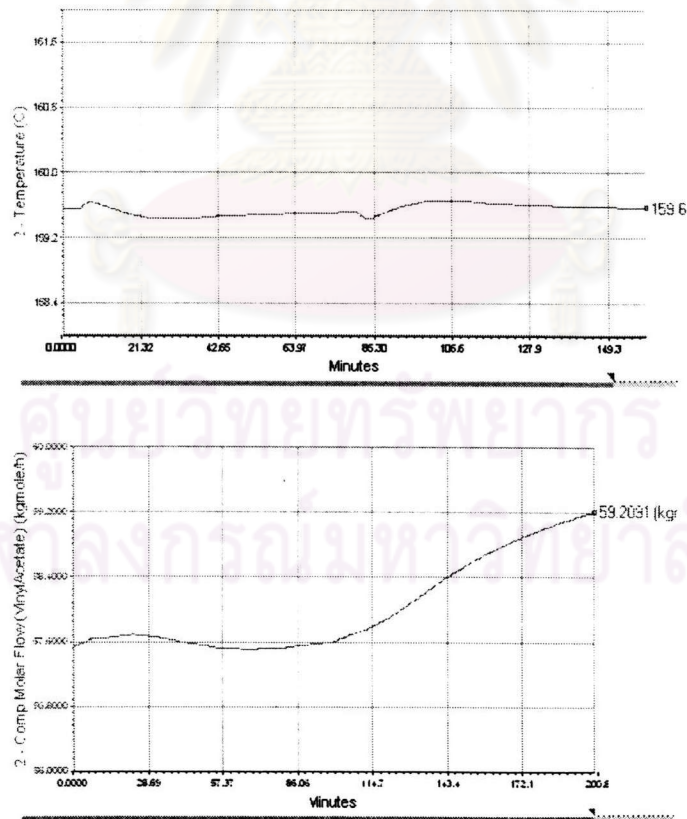


Figure 4.12 (b) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS2 structure

Figure 4.12 (b), (c) and (d) show the dynamic response in the column. When the total acetic feed increase, it is more feed in column. The reboiler level rises so the level controller changes the bottom flowrate increasing. Thus the tray 5 temperature decreases at first and then the temperature controller brings in more heat input. The tray 5 temperature can increase to the setpoint. The acetic acid composition changing of CS2 is less than CS1 because CS2 control composition in overhead of column. Hence the composition in distillate is small changing.

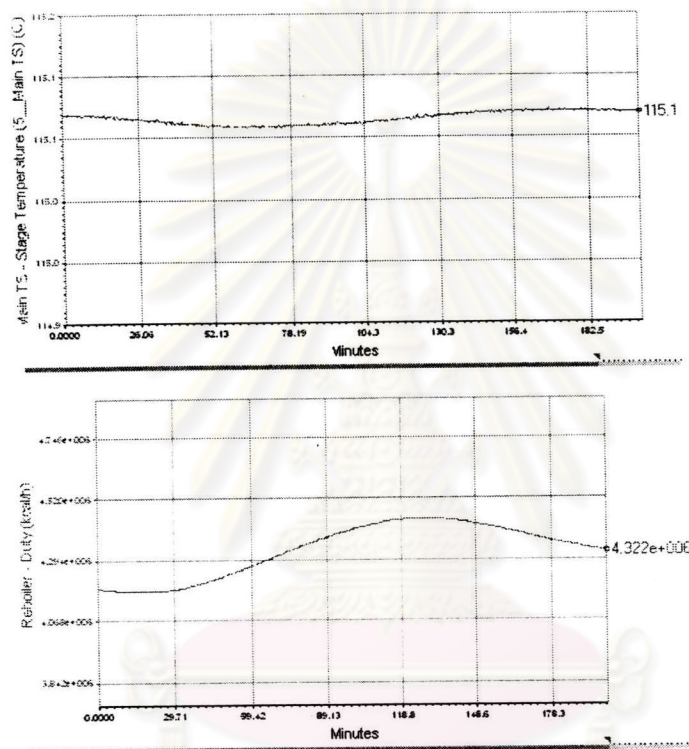


Figure 4.12 (c) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS2 structure

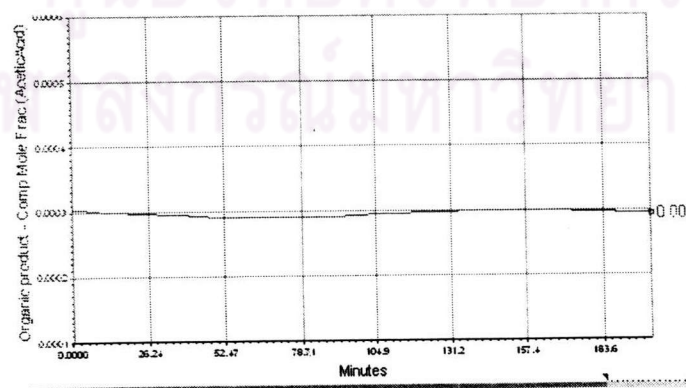


Figure 4.12 (d) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS2 structure

4.7 Designed Control structure II (CS3)

In figure 4.5 shows the CS3 structure that is different from the CS2 in composition loop and base level loop in azeotrope column. In this column, the boilup ratio is high so the other structure that appropriate for this column is designed. The bottoms flow should be used to control bottoms composition and heat input should control base level. This scheme is direct material balance control structure that using the bottom flowrate to control composition.

4.7.1 Change in reactor temperature

- decrease temperature

Step changes of 8 °C decreasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

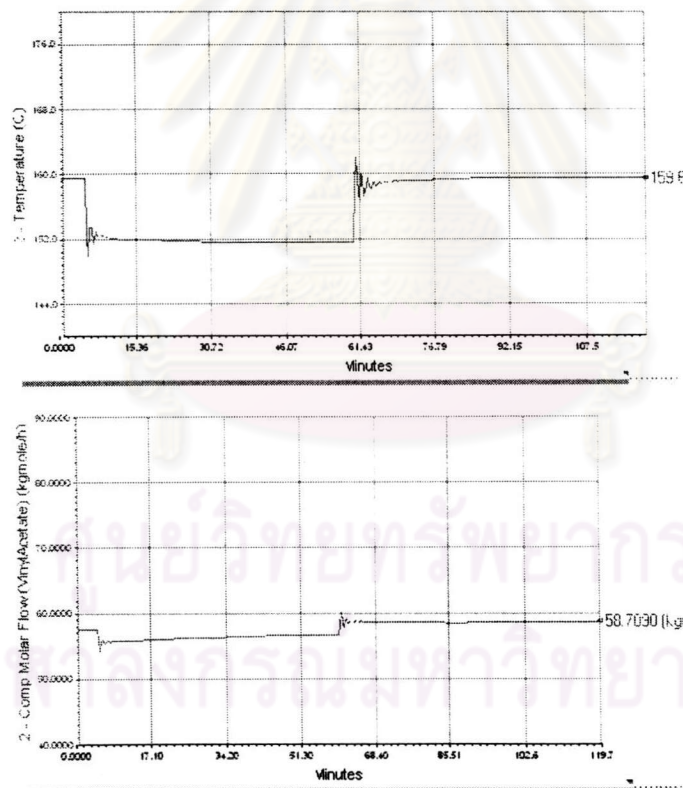


Figure 4.13(a) Dynamic response of base case to 8°C decrease in reactor temperature of CS3 structure

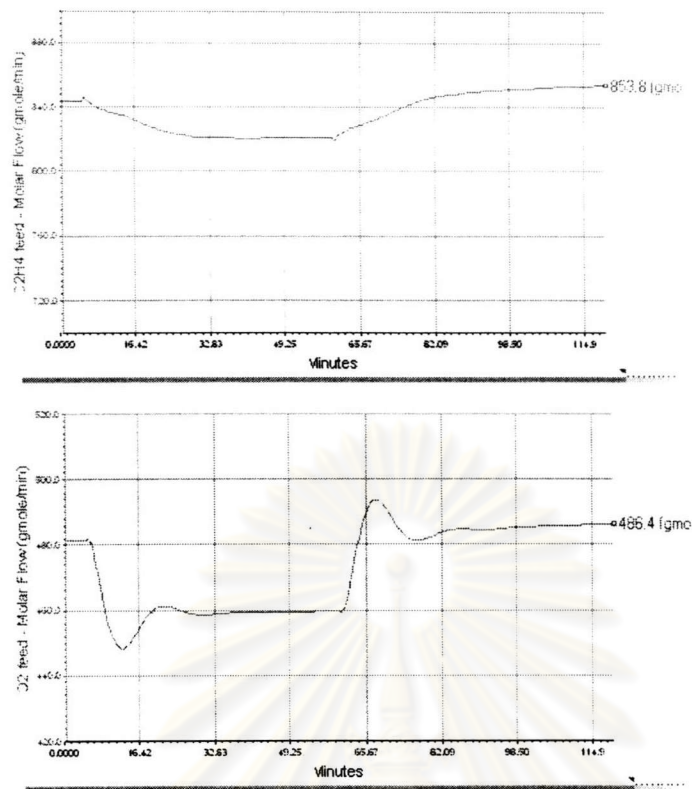


Figure 4.13(b) Dynamic response of base case to 8°C decrease in reactor temperature of CS3 structure

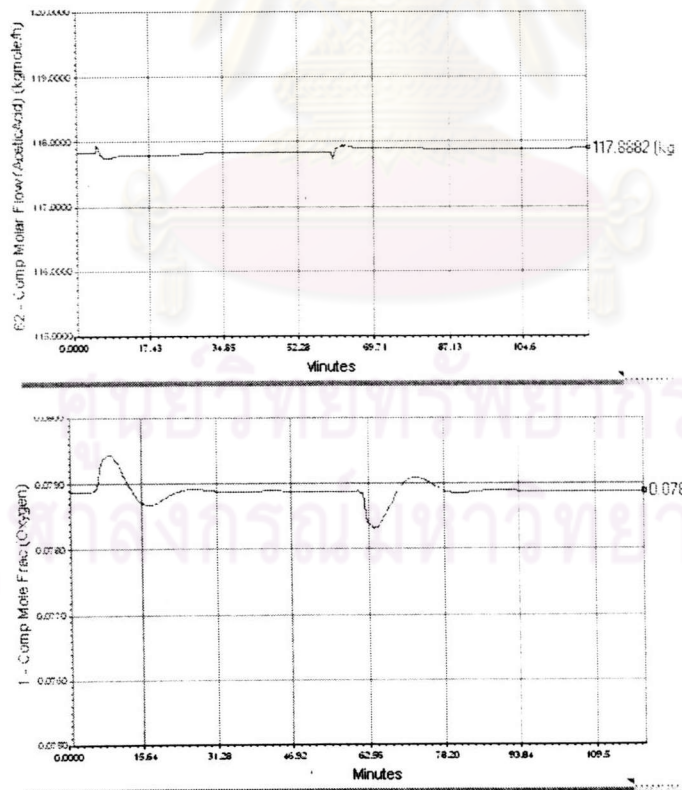


Figure 4.13(c) Dynamic response of base case to 8°C decrease in reactor temperature of CS3 structure

Figure 4.13 gives result for step increase in the reactor temperature controller setpoint from 159.5 to 151.5 °C. The decreasing of reactor temperature results the production of vinyl acetate decreases so the fresh feed of oxygen and ethylene decrease. The reactor temperature reaches the new setpoint at 35 minute. The response of oxygen composition loop remains the safety constraint.

- increase temperature

Step changes of 6 °C increasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

The results of increasing in reactor temperature are shown in figure 4.14. The setpoint of temperature increase to the new setpoint within 30 minutes and response is more oscillatory. The production of vinyl acetate increases because of the high production rate. In the gas loop has less ethylene, oxygen and acetic acid so all of fresh feed are increase. The oxygen composition in gas loop remains less than 0.1%. The response of separation unit has less changing when the energy disturbance is load in the plant.

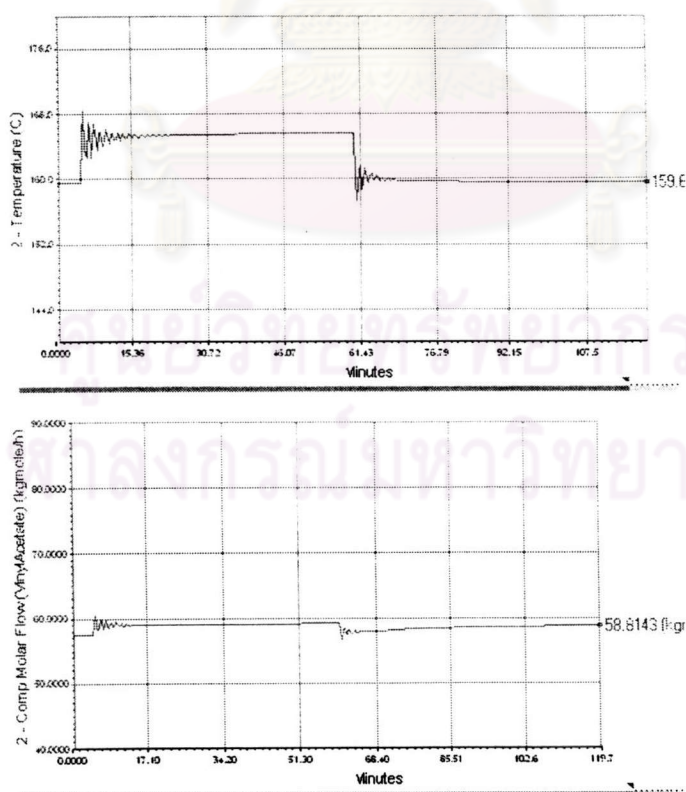


Figure 4.14 (a) Dynamic response of base case to 6°C increase in reactor temperature of CS3

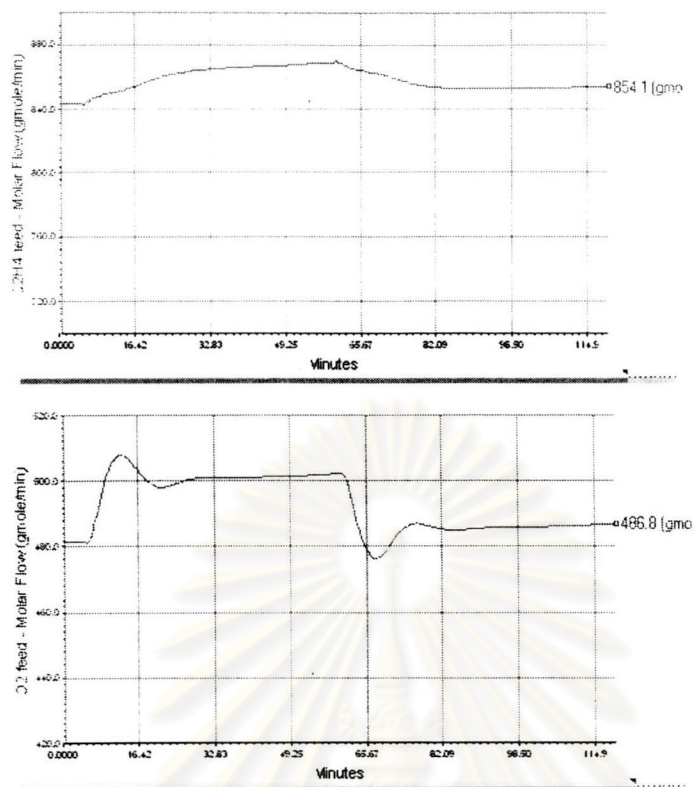


Figure 4.14 (b) Dynamic response of base case to 6°C increase in reactor temperature of CS3

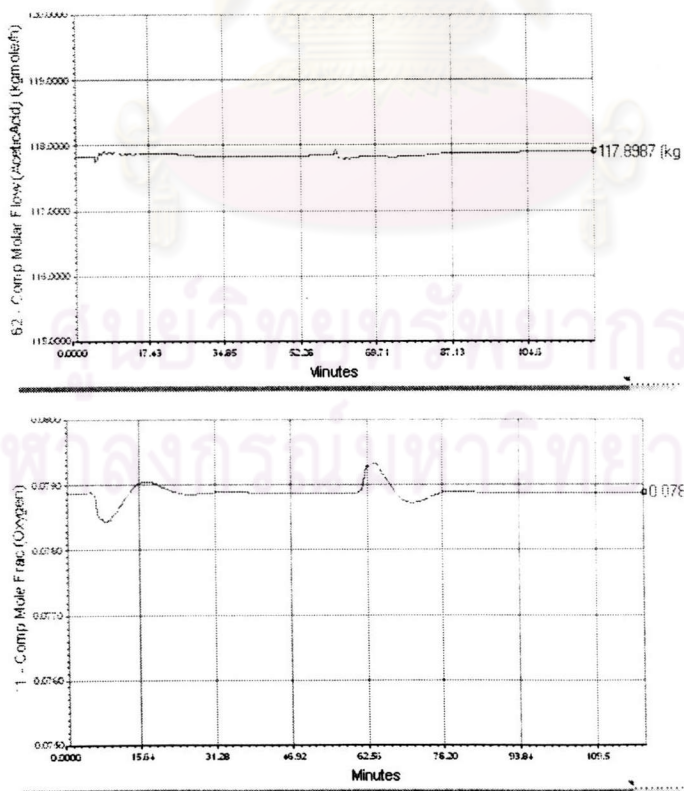


Figure 4.14(c) Dynamic response of base case to 6°C increase in reactor temperature of CS3

4.7.2 Change in acetic acid recycle flowrate

Step changes of 20 percent at time 5 minutes and 100 minutes are made in the setpoint of the total acetic acid flow controller.

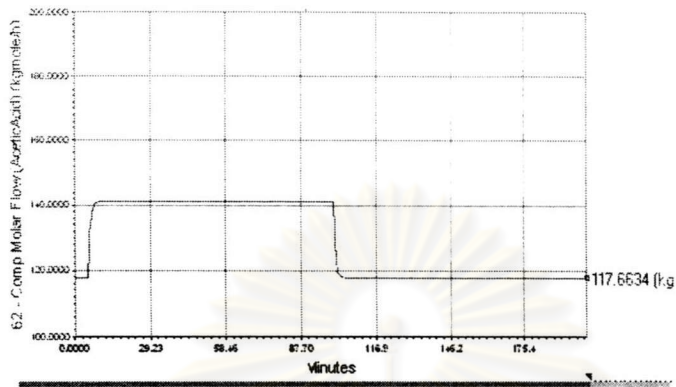


Figure 4.15 (a) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS3 structure

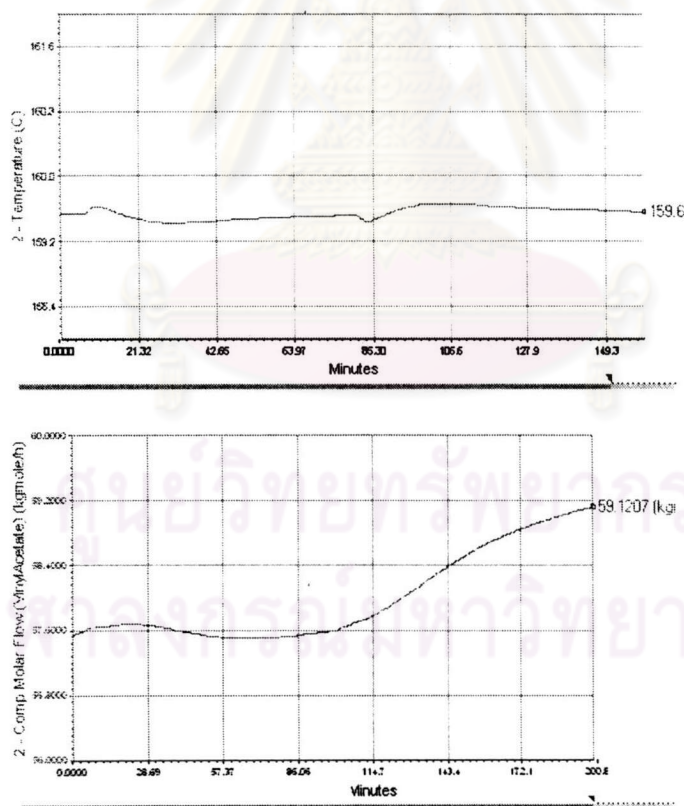


Figure 4.15 (b) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS3 structure

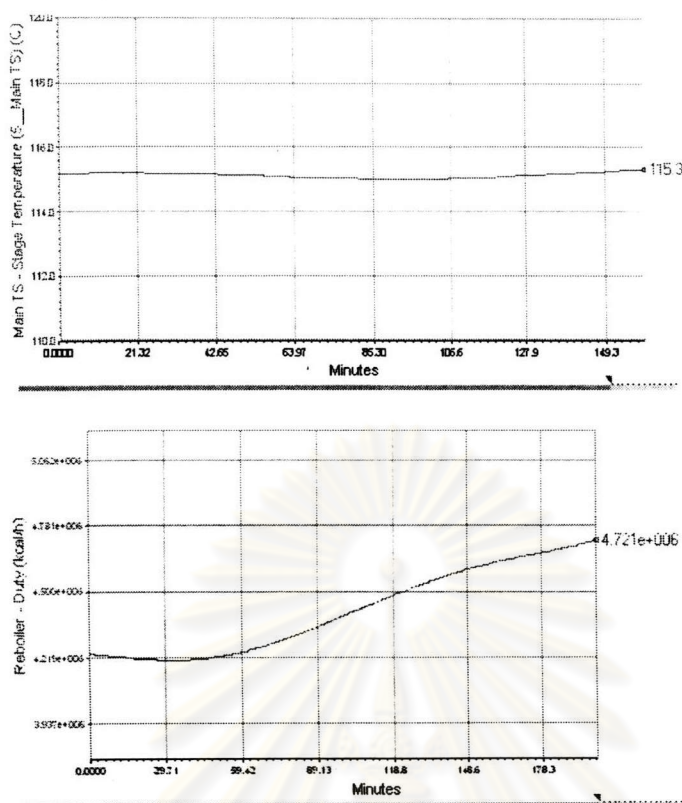


Figure 4.15 (c) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS3 structure

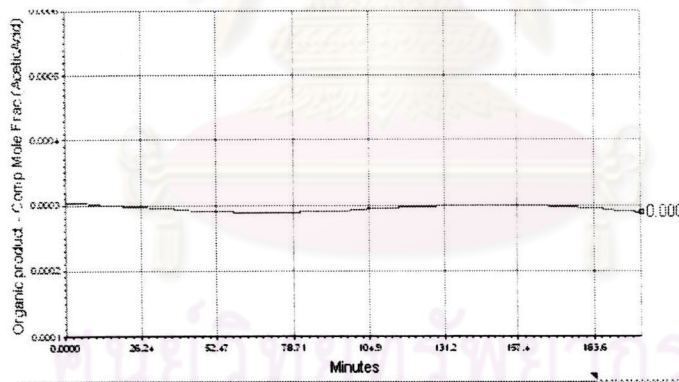


Figure 4.15 (d) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS3 structure

Figure 4.15 shows the dynamic responses for changes in inlet flowrate of total acetic acid feed. The flow controller of acetic acid gets the new setpoint within 15 minutes like the CS2 that is faster than CS1. The increase in total flowrate of acetic acid decreases the reactor temperature, and production rate of vinyl acetate. In column, the change in acetic acid recycle flow produces a large change in the column temperature. The increase of feed in column brings the base level rising so the level

controller increases the heat input to decrease base level. Thus, the temperature in column is rise. For the composition controller, the bottoms flowrate decreases when the base level decreases so the tray temperature increases. The control loop between level and composition has an interaction so the controller gain must be small. The acetic acid composition changing of CS2 is less than CS1 because CS2 control composition in overhead of column. Hence the composition in distillate is small changing.

4.8 Designed Control structure III (CS4)

In CS4 structure, the flow rate of the fresh feed of ethylene is flow controlled, which sets the production rate. The heat from the exothermic reaction is controlled by changing cooling water valve in reactor and the gas recycle loop pressure is controlled by manipulating separator overhead exit valve. This structure has a low production rate of vinyl acetate when compare with the other structures because the fresh feed ethylene is fixed. This scheme might be used when the reactant comes from upstream unit.

4.8.1 Change in reactor temperature

- decrease temperature

Step changes of 8 °C decreasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

Figure 4.16 gives simulation results for the reactor temperature decrease. The decrease in reactor temperature, which tends to drop the production rate of vinyl acetate and fresh oxygen feed. The fresh ethylene feed does not change because its flow controlled. This control structure, which has fixed flowrate of ethylene, responds to this disturbance by producing fewer products. The total acetic acid is slightly changing and the oxygen composition in gas loop in less than 0.1%. Temperature controller reaches to the setpoint at 40 minute.

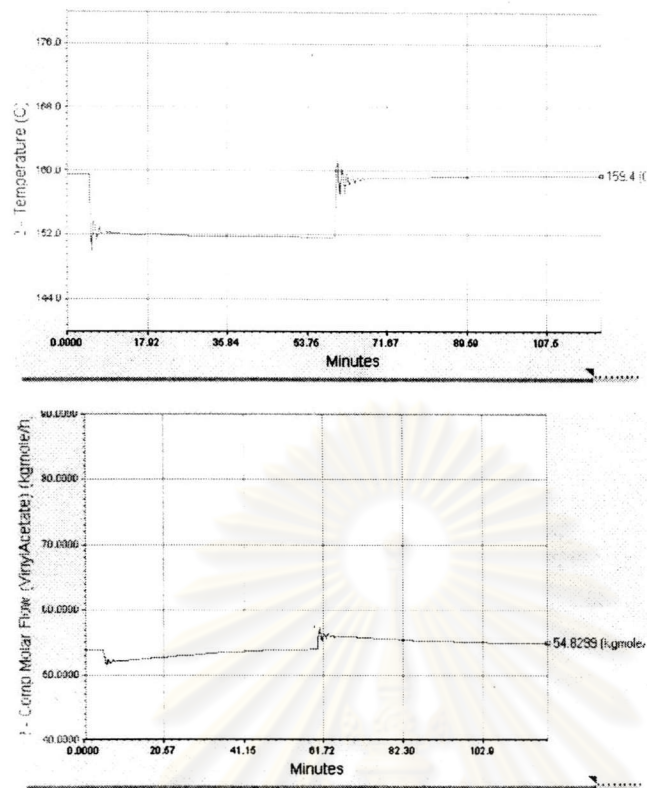


Figure 4.16(a) Dynamic response of base case to 8°C decrease in reactor temperature of CS4 structure

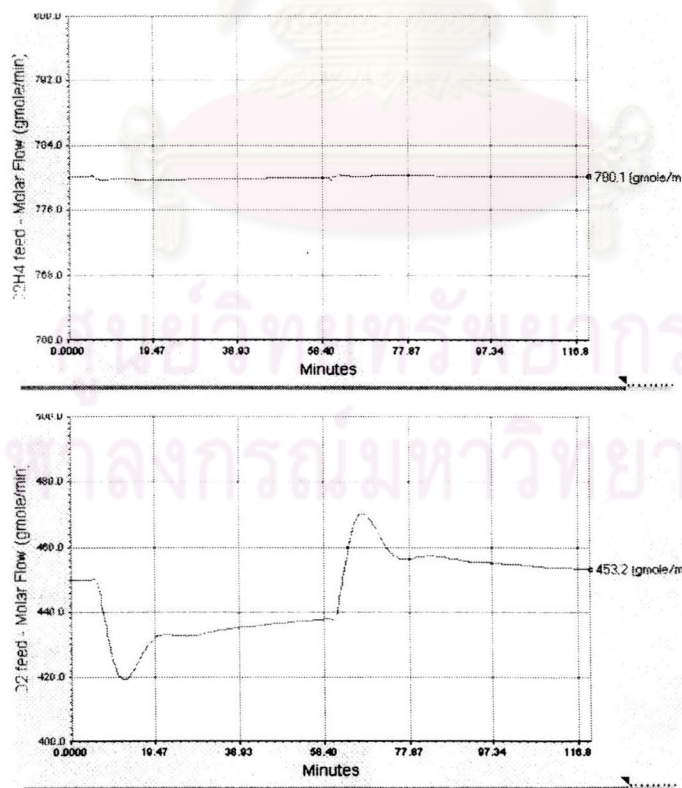


Figure 4.16(b) Dynamic response of base case to 8°C decrease in reactor temperature of CS4 structure

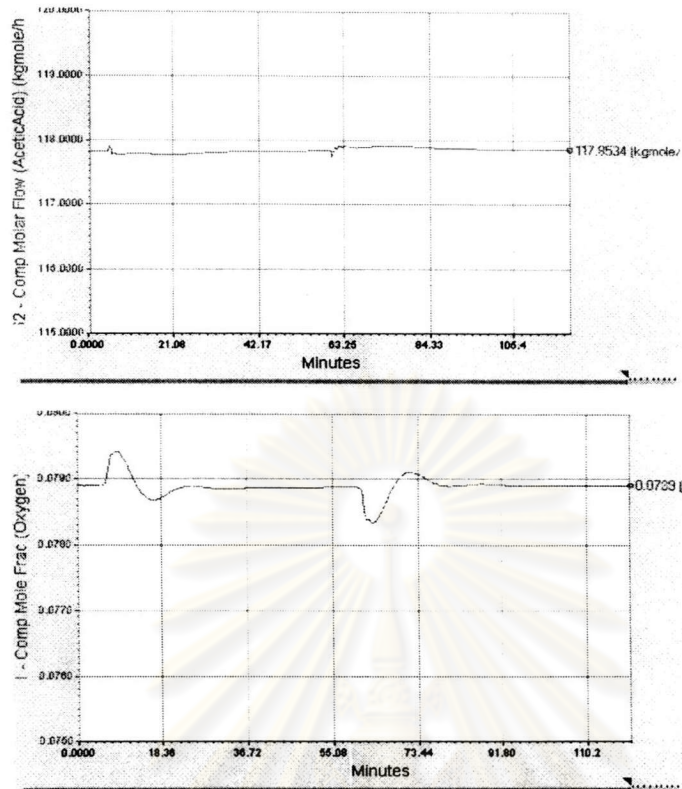


Figure 4.16(c) Dynamic response of base case to 8°C decrease in reactor temperature of CS4 structure

- *increase temperature*

Step changes of 6 °C increasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

The increase setpoint of reactor temperature controller increases the production rate and fresh oxygen feed. The production rate of vinyl acetate is less produced than another control structures because the fresh ethylene feed is fixed. The fresh ethylene feed is used to set the production rate in this control structure. This scheme can handled temperature changes and can operate in safety constraint. The reactor temperature controller can achieve the new setpoint within 30 minutes.

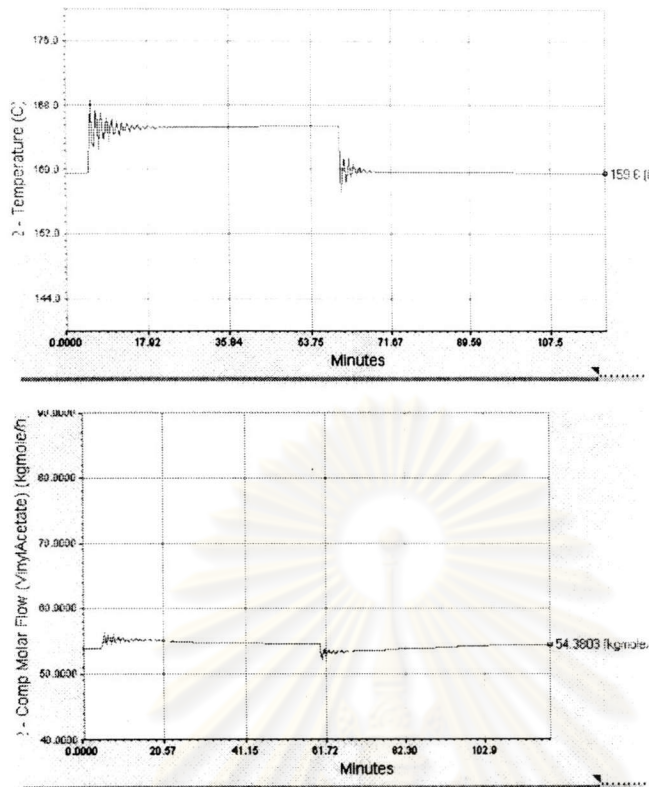


Figure 4.17 (a) Dynamic response of base case to 6°C increase in reactor temperature of CS4 structure

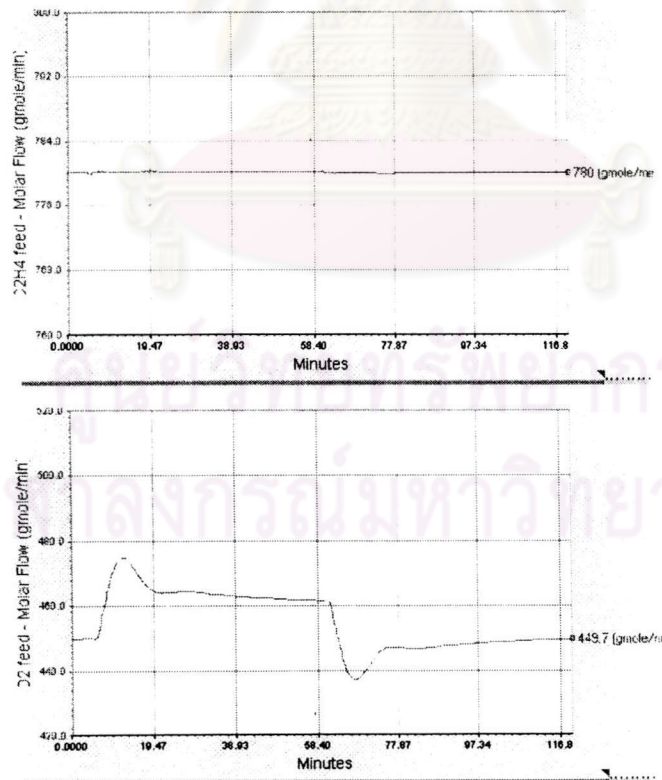


Figure 4.17 (b) Dynamic response of base case to 6°C increase in reactor temperature of CS4 structure

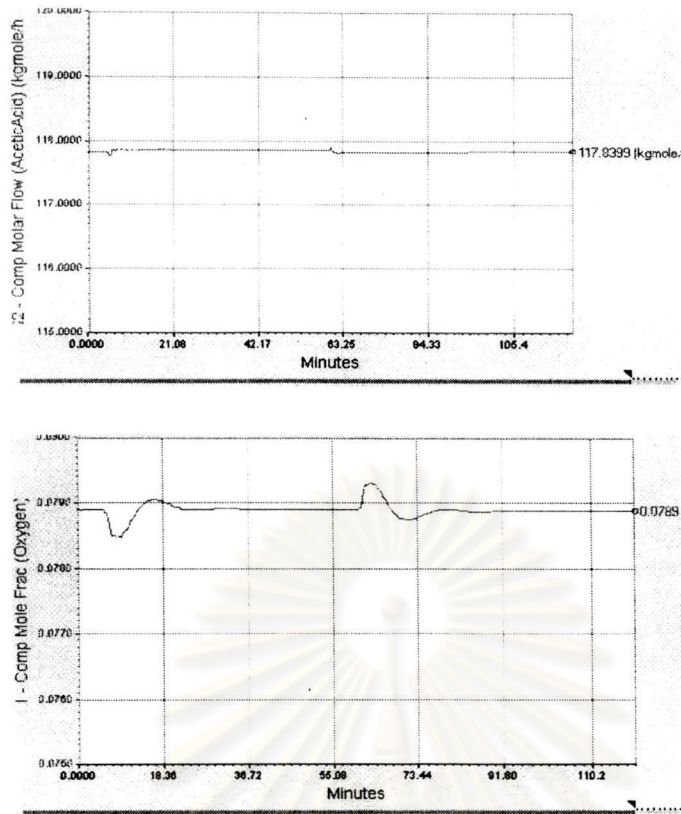


Figure 4.17 (c) Dynamic response of base case to 6°C increase in reactor temperature of CS4 structure

4.8.2 Change in acetic acid recycle flowrate

Step changes of 20 percent at time 5 minutes and 100 minutes are made in the setpoint of the total acetic acid flow controller.

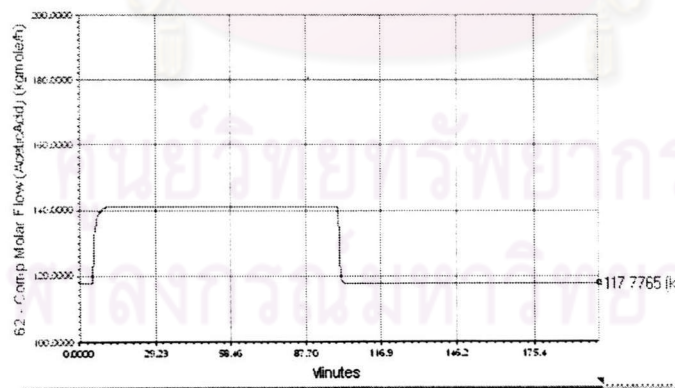


Figure 4.18 (a) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS4 structure

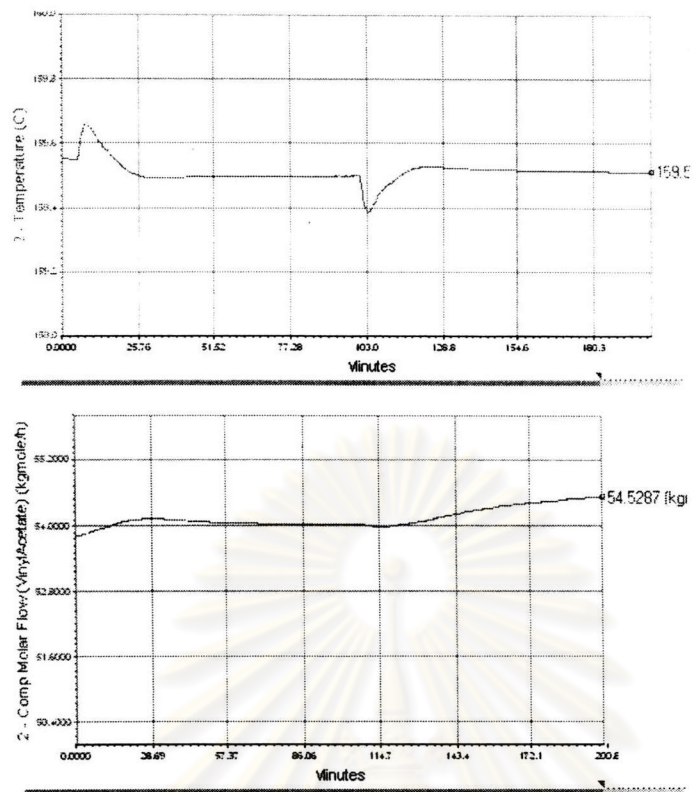


Figure 4.18 (b) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS4 structure

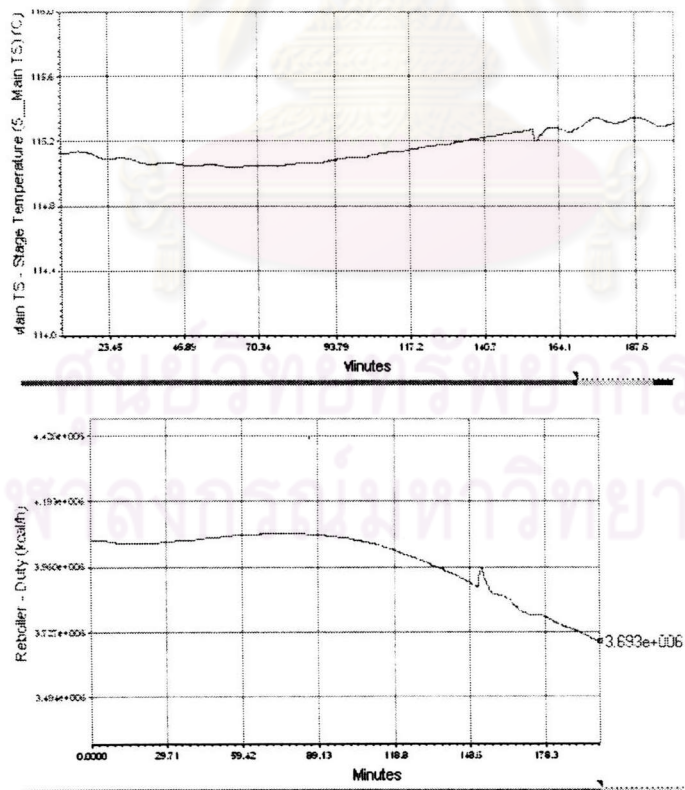


Figure 4.18 (c) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS4 structure

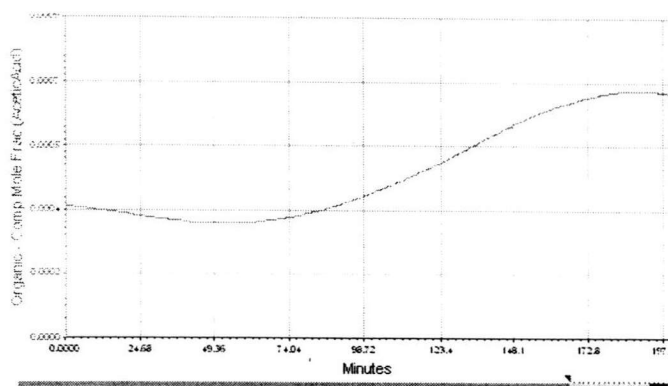


Figure 4.18 (d) Dynamic response of base case to 20 percent increase in acetic acid recycle flow of CS4 structure

The response of changing the recycle flow of acetic acid is shown in Figure 4.18. The total acetic acid increases and reaches the setpoint at 25 minute that is faster than CS1 structure. The reactor temperature and production rate of vinyl acetate do not change. When the decrease in acetic acid flowrate return the setpoint at base case. The production rate increase as the temperature is constant because of unusual reaction rate of vinyl acetate reaction. The feed in column rise results the increase of acetic acid recycle flow. The tray5 temperature and acetic acid composition in overhead column drop. For the reason, heat input manipulates the tray temperature in column increase to the setpoint and also acetic acid composition increases. The change of acetic acid composition in overhead is larger than CS2 and CS3 structures.

4.9 Plantwide Dynamic Simulation

A little guidance for dynamic simulations and tuning method of plantwide systems is as follows:

1. Start with one isolated unit operation. Get its control system installed, tuned, and tested for closed-loop stability and robustness. Then move on to the next unit and repeat. Build up the entire plant one unit at a time until you get all recycles connected.
2. Initially use proportional-only controllers in all loops except flow controllers, where the normal tight tuning can be used ($K_C = 0.5$ and $\tau_1 = 0.3$ minutes). Set the gains in all level controllers (except reactors) equal to 2. Adjust the temperature, pressure, and composition controller gains by trial and error to see if you can line

out the system with the proposed control structure. If P-only control cannot be made to work, PI will not work either. When stable operation is achieved, add a little reset action to each PI controller (one at a time) to pull the process into the setpoint values.

3. Perform a relay-feedback test on each temperature, pressure, and composition loop one at a time to determine the ultimate gains and frequencies. Remember to place reasonable lags and deadtimes in these loops in your simulation program. Temperature measurement lags are 0.3 to 0.5 minutes in liquid streams and 0.6 to 1.5 minute in gas streams. The relay-feedback test does not work unless the loop has a phase angle less than -180° , so the process transfer function must be at least third-order or contain deadtime.
4. Use PI controllers in most temperature, pressure, and composition loops. Derivative action can help improve dynamics in some noise free situations, but if you cannot get good control using only PI in a simulation environment, it is doubtful that the control of a real plant will be good.
5. Ziegler-Nichols settings should be used only if tight control is desired and large swings in the manipulated variable can be tolerated. Reactor temperature control is a typical example:

$$K_C = K_u / 2.2$$

$$\tau_I = P_u / 1.2$$

6. Use Tyreus-Luyben settings for most loops, particularly distillation columns where large swings in vapor boilup or reflux are undesirable. These settings are much more conservative (robust) than Ziegler-Nichols:

$$K_C = K_u / 3.2$$

$$\tau_I = 2.2P_u$$

7. Once the entire plant is running, use different disturbances with different magnitudes and in different directions to test the control structure. If large changes in manipulated variables are required to get to the new steady state, the control structure will perform poorly.

Table 4.2 shown the tuning parameters all of control structure.

Table 4.2 Tuning parameters

The same control loops of all control structures had the same tuning parameters. The following loops are:

LOOP	CS1		CS2		CS3		CS4	
	K_c	τ_i	K_c	τ_i	K_c	τ_i	K_c	τ_i
Oxygen composition	1.43	0.71	1.43	0.71	1.43	0.71	1.43	0.71
Gas recycle stream pressure	2	-	2	-	2	-	2	-
Vaporizer level	2	-	2	-	2	-	2	-
Reactor inlet temperature	2	0.176	2	0.176	2	0.176	2	0.176
Reactor outlet temperature	0.5	2	0.5	2	0.5	2	0.5	2
Separator temperature	0.09	0.147	0.09	0.147	0.09	0.147	0.09	0.147
Separator level	2	-	2	-	2	-	2	-
Absorber level	2	-	2	-	2	-	2	-
Absorber scrub flowrate	0.5	0.1	0.5	0.1	0.5	0.1	0.5	0.1
Circulation stream flowrate	0.5	0.3	0.5	0.3	0.5	0.3	0.5	0.3
Absorber scrub temperature	0.5	5	0.5	5	0.5	5	0.5	5
Circulation stream temperature	2	10	2	10	2	10	2	10
% CO ₂ in the gas recycle	0.05	10	0.05	10	0.05	10	0.05	10
% C ₂ H ₆ in the gas recycle	0.05	10	0.05	10	0.05	10	0.05	10
Decanter temperature	0.08	0.119	0.08	0.119	0.08	0.119	0.08	0.119
Decanter organic level	2	-	2	-	2	-	2	-
Decanter aqueous level	2	-	2	-	2	-	2	-
Column pressure	6	0.23	6	0.23	6	0.23	6	0.23

The different control loops of each structure had some different tuning parameters.

LOOP	CS1		CS2		CS3		CS4	
	K_c	τ_i	K_c	τ_i	K_c	τ_i	K_c	τ_i
% H ₂ O in column	1	25.9	58	0.327	58	0.327	1	25.9
Fifth tray temperature	19.7	1.88	19.7	1.88	1	2	19.7	1.88
Column base level	2	-	2	-	2	-	2	-
Total acetic acid flowrate	0.5	0.1	0.5	0.1	0.5	0.1	0.5	0.1
Fresh ethylene feed	-	-	-	-	-	-	0.5	0.3

4.10 Control structure Comparison

4.10.1 Change in reactor temperature

- Decrease temperature

The starting conditions are the base-case design where reactor exit temperature is 159.5 °C. The reactor temperature controller is tuned at this operating point. Step changes of 8 °C decreasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

Figure 4.19-4.21 give results for step increase in the reactor temperature controller setpoint from 159.5 to 151.5 °C. When the reactor temperature decreases, the production rate of vinyl acetate decreases because of the reaction rate depends on the temperature. A large amount of reactants remain in the reactor so the fresh feed of reactants decrease. The heat input of separator decrease because low temperature inlet in separator.

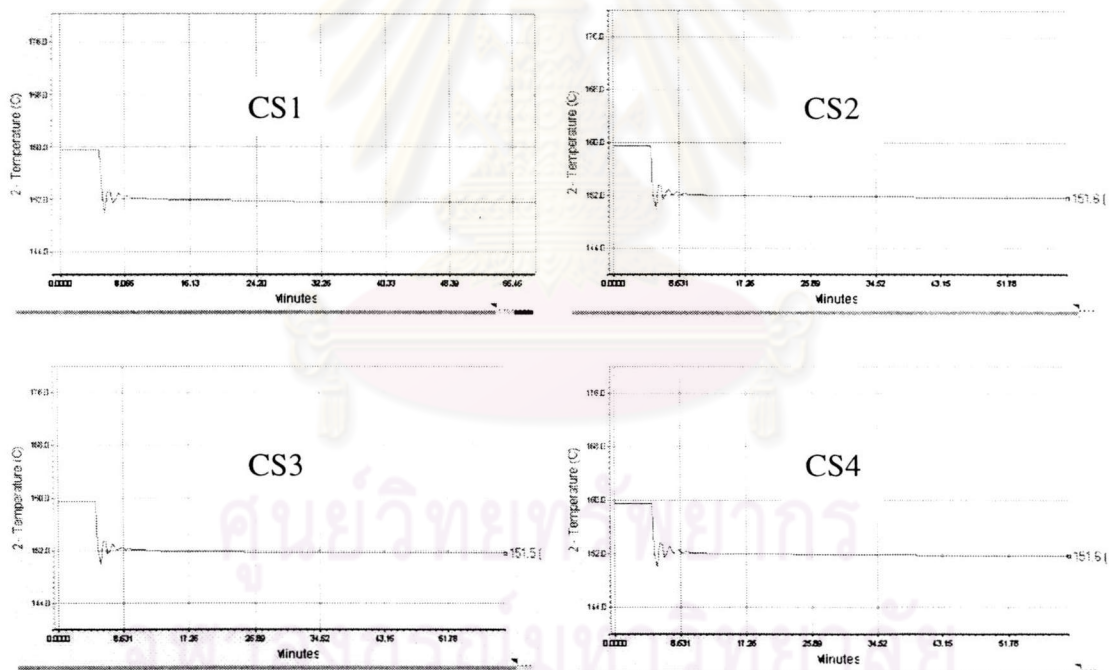


Figure 4.19 Comparison reactor temperature responses when the temperature decreases.

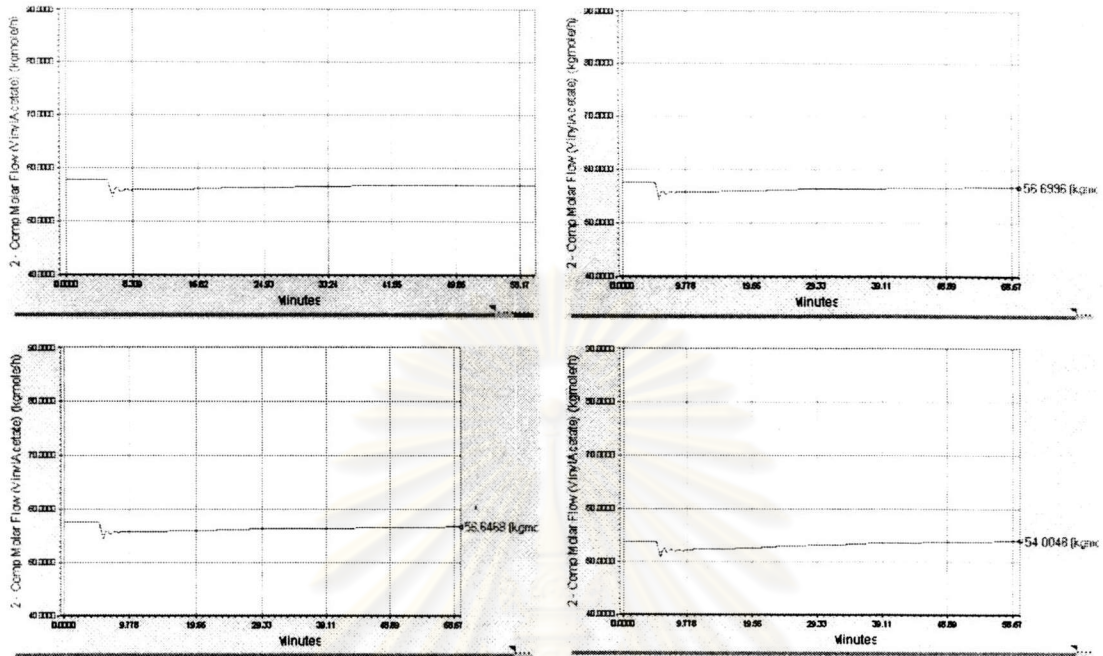


Figure 4.20 Comparison reactor production of VAc responses when the temperature decreases.

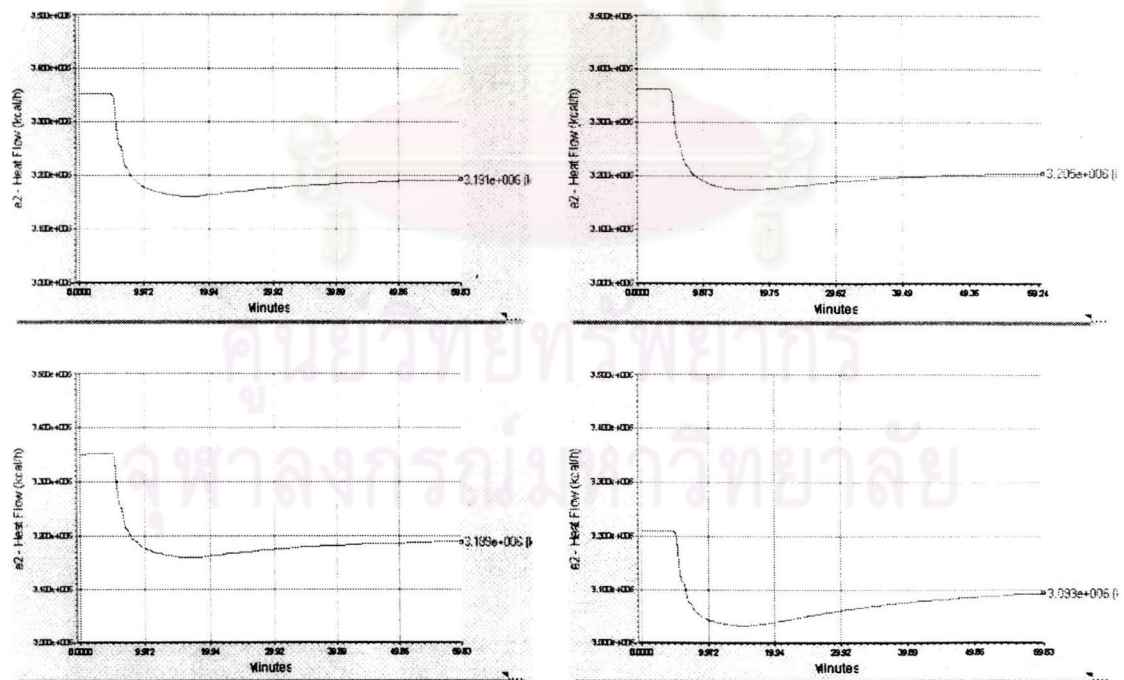


Figure 4.21 Comparison heat duty in separator cooler responses when the temperature decreases.

- increase temperature

Step changes of 6 °C decreasing at time 5 minutes and 60 minutes are made in the setpoint of the reactor temperature controller.

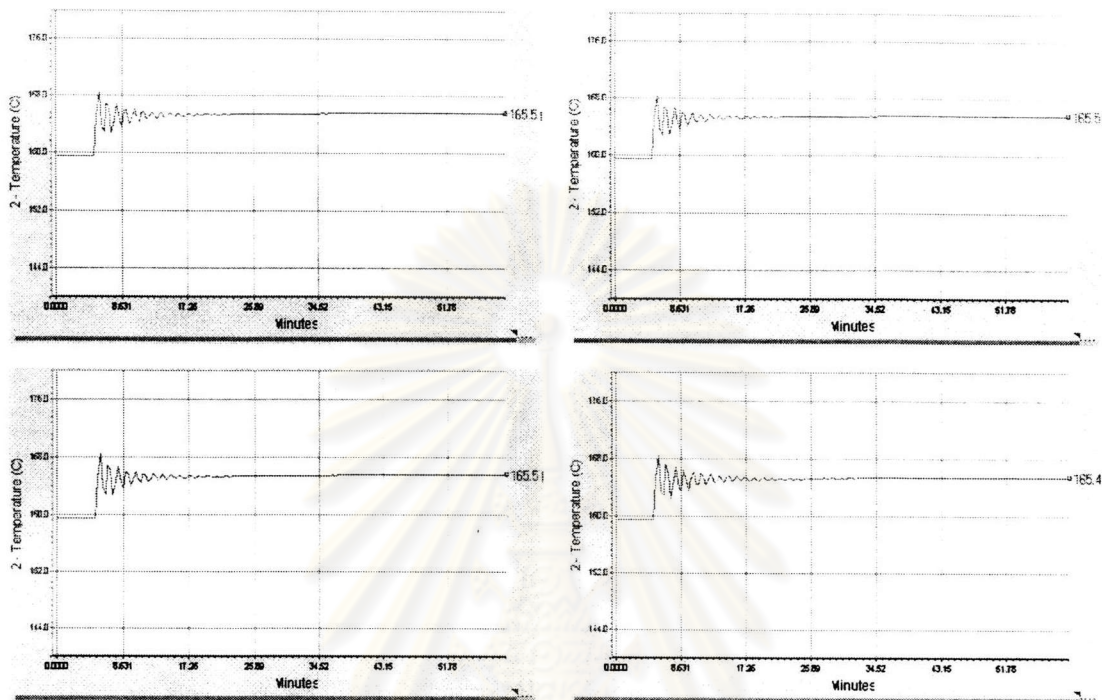


Figure 4.22 Comparison reactor temperature responses when the temperature increases.

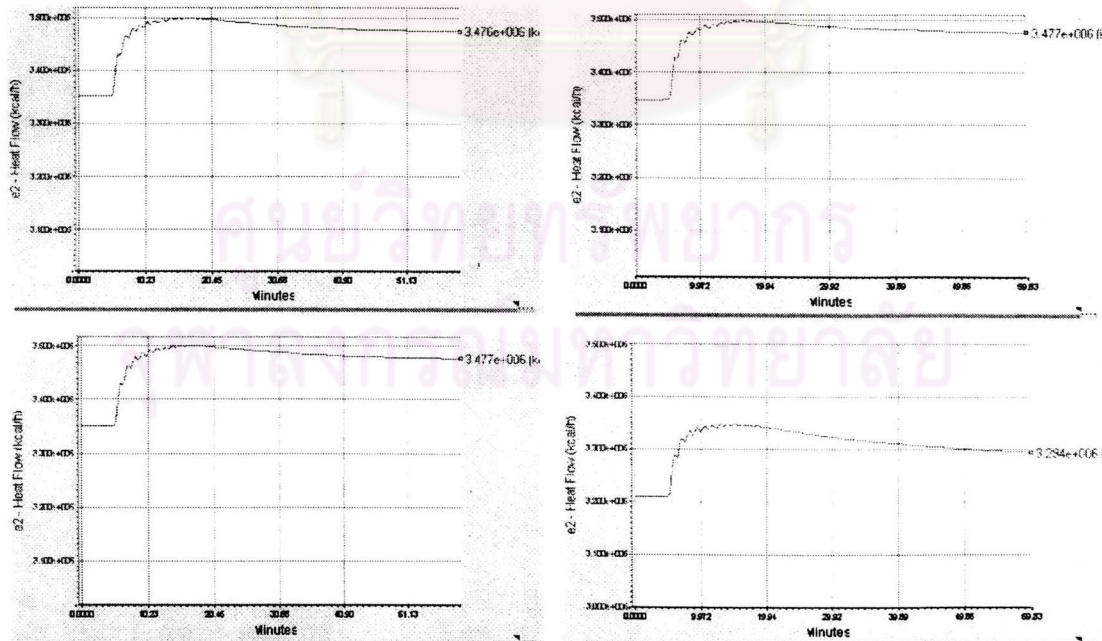


Figure 4.23 Comparison heat duty in separator cooler responses when the temperature increases.

less change in CS3 but the column temperature large change in this scheme. The CS3 structure is used the bottom product to manipulate the VAc composition or the tray5 temperature that gives the large effect to control composition more than heat input. However, all of control structure can achieve the product quality of Vinyl acetate process.

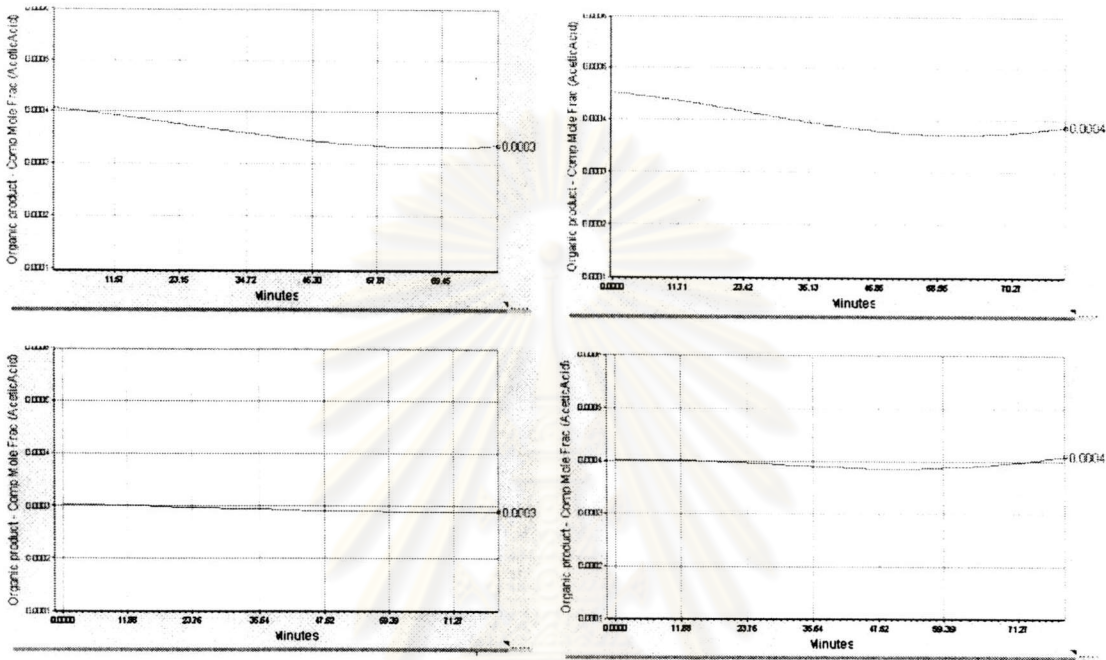


Figure 4.25 Comparison HAc conc. in organic product responses

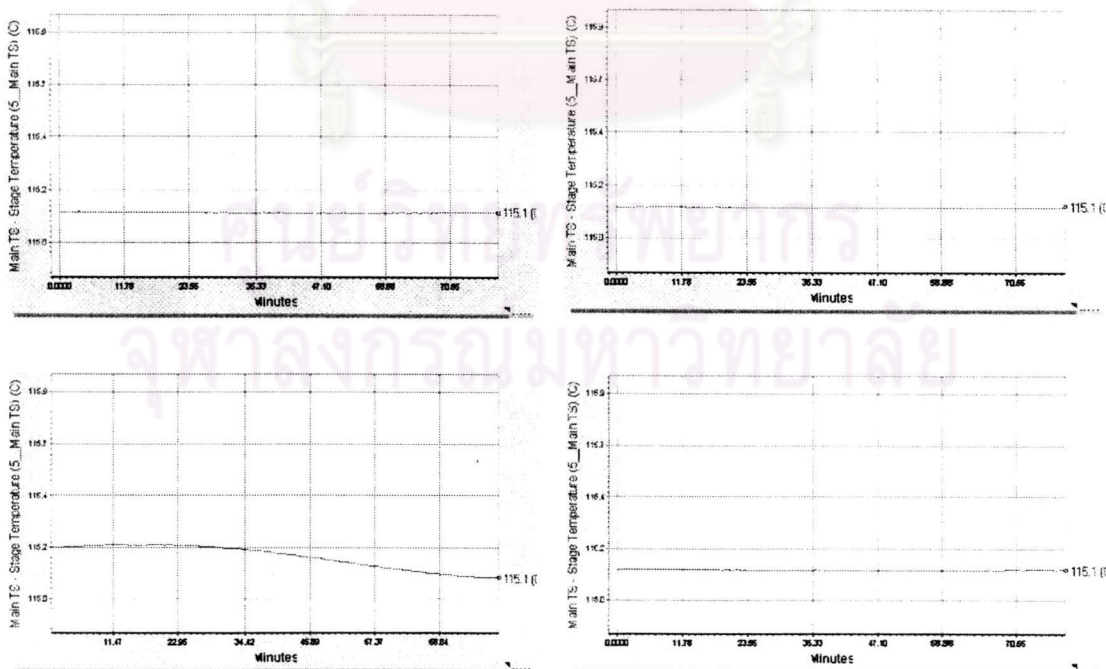


Figure 4.26 Comparison column tray 5 temperature responses