# CONTROLLABILITY STUDY ON MULTI-VESSEL BATCH DISTILLATION COLUMN BASED ON LEVEL CONTROL SCHEME

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# ABSTRAK

Penyulingan sekumpul telah menunjukkan kelebihan yang unggul berbanding peroses penyulingan yang lain bagi penghasilan produk kimia yang khusus dan halus, produk farmaseutikal dan produk bermusim. Penyulingan sekumpul dengan tangka berbilang (MVBDC) adalah inovasi daripada penyulingan sekumpul yang memerlukan jumlah tenaga yang tinggi. MVBDC adalah kombinasi antara penerus sekumpul dan kolum pelurut. Oleh kerana tingkah laku dinamik dan keadaan proses tak mantap, strategi sistem kawalan bagi penyulingan sekumpul amat menarik untuk di kaji. Kajian ini membincangkan secara terperinci strategi kawalan penyulingan sekumpul dengan tangka berbilang (MVBDC) yang digunakan untuk memisahkan campuran tidak azeotropik yang terdiri daripada ethanol, 1-propanol dan n-butanol dan MVBDC in beroperasi pada refluks penuh. Pemisahan maksimum dicapai di bawah operasi refluks penuh. Tinggalan di dalam tangki dikekalkan kepada nilai optimum dengan kawalan tahap untuk mengekalkan operasi refluks penuh. Pengawalan dan analisis sensitiviti penting dalam menentukan paras tinggalan pada tangki mana yang perlu di kawal dan untuk mendapatkan prestasi kawalan yang terbaik yang boleh memastikan kualiti produk yang di perlukan tercapai. Jadi, analisis sensitiviti yang merangkumi analisis dinamik yang digunakan untuk menentukan pemboleh ubah bagi pemilihan struktur kawalan berdasarkan sistem terbuka di analisa. Tambahan pula, kajian pengawalan yang merangkumi penalaan pengawal dan interaksi analisis menggunakan RGA dan SVD pada MVBDC berdasarkan system tertutup di kaji. Satu set persamaan model matematik dibangunkan berdasarkan prinsip pertama termasuk keseimbangan berat dan keseimbangan komponen. Model ini di simulasi menggunakan MATLAB Simulink. Penalaan pengawal dilakukan berdasarkan Ziegler Nichols (ZN) kaedah penalaan dan Kawalan Model Dalaman (IMC). Dua kawalan paras dipilih sebagai struktur kawalan berdasarkan analisis sensitiviti. Berdasarkan analisis dinamik, berkadar (P) dan berkadar integral (PI) pengawal digunakan untuk kawalan tahap dan kemudian prestasi dibandingkan. Kemudian, analisis interaksi mengesahkan pasangan yang terbaik bagi pembolehubah dikawal dan dimanipulasi adalah aliran refluks - paras di tangki atas dan aliran bawah - paras di tangka tengah dengan nombor keadaan bersamaan dengan 1.185. Kesimpulannya, dua struktur kawalan paras dengan PI pengawal dengan penalaan IMC dipilih untuk menjamin prestasi pengawal yang terbaik dan memastikan kualiti produk dicapai. Selain daripada memastikan keunggulan prestasi sistem pengawalan MVBDC, sistem pengawalan MVBDC yang kurang interaksi dapat dihasilkan.

# ABSTRACT

Batch distillation has shown superior advantages over other separation processes for the production of fine and specialty chemicals, pharmaceutical products and seasonal campaign products. Multi-vessel batch distillation column (MVBDC) is an innovation of less-energy efficient batch distillation. MVBDC is a combination of batch rectifier and stripper column. Due to its dynamics behavior and unsteady state process, the control system strategy of MVBDC is necessarily interesting to study. This research discusses in detail the control strategy of MVBDC that is used to separate a non-azeotropic multiple component mixture under total reflux operation. A ternary mixture consisting ethanol, 1-propanol and n-butanol was taken as the case for this study. Maximum fractionating was achieved under total reflux operation. The vessel holdup was kept constant to its optimal value by the level control, in order to maintain the total reflux operation. Sensitivity analysis and controllability study are significant to determine the level of vessel holdup needed to be controlled and to obtain the best controller performance that can ensure the product quality achievable. Hence, the sensitivity analysis that covers the dynamic analysis and is used to determine important variables for the selection of control structure based on open loop behavior is analyzed. Furthermore, the controllability study that covers controller tuning and interaction analysis using Relative Gain Array (RGA) and Singular Value Decomposition (SVD) on MVBDC based on closed loop behavior were also studied. A set of mathematical model equations were developed based on first principles including mass balance and component balance. The model was simulated using MATLAB Simulink. Controller tuning was performed based on Ziegler Nichols (ZN) tuning method and Internal Model Control (IMC). A two-level control was selected as the control structure based on sensitivity analysis. Based on dynamic analysis, proportional (P) and proportional integral (PI) controller were applied to level control and then the performance was compared. Interaction analysis confirmed the best pair of controlled and manipulated variables *i.e.* reflux flow-level of top vessel holdup and bottom flow-level of middle vessel with condition number equivalent to 1.185. As a conclusion, two level control structure with PI controller and IMC tuning was chosen in order to guarantee the best performance of controller and to ensure the product quality was achieved Apart from ensuring the best performance of the MVBDC control system, less interactive MVBDC control system is also produced.

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| | |
|---|---|
| A | Antoine coefficient |
| B | Antoine coefficient |
| B | Holdup in reboiler |
| C | Antoine coefficient |
| H | Holdup in top vessel |
| $H_s$ | Stage holdup |
| $K_n$ | Equilibrium constant at n stages |
| $K_{11}$ | Rate of change of $CV_1$ per $MV_1$ |
| $K_{12}$ | Rate of change of $CV_1$ per $MV_2$ |
| $K_{13}$ | Rate of change of $CV_1$ per $MV_3$ |
| $K_{21}$ | Rate of change of $CV_2$ per $MV_1$ |
| $K_{22}$ | Rate of change of $CV_2$ per $MV_2$ |
| $K_{23}$ | Rate of change of $CV_2$ per $MV_3$ |
| $K_{31}$ | Rate of change of $CV_3$ per $MV_1$ |
| $K_{32}$ | Rate of change of $CV_3$ per $MV_2$ |
| $K_{33}$ | Rate of change of $CV_3$ per $MV_3$ |
| $K_1$ | Equilibrium constant stage 1 |
| $K_2$ | Equilibrium constant stage 2 |
| $K_3$ | Equilibrium constant stage 3 |
| $K_4$ | Equilibrium constant stage 4 |
| $K_5$ | Equilibrium constant stage 5 |
| $K_6$ | Equilibrium constant stage 6 |
| $K_7$ | Equilibrium constant stage 7 |
| $K_8$ | Equilibrium constant stage 8 |
| $K_9$ | Equilibrium constant stage 9 |
| $K_{10}$ | Equilibrium constant stage 10 |
| $K_{11}$ | Equilibrium constant stage 11 |
| $K_{12}$ | Equilibrium constant stage 12 |
| $K_{13}$ | Equilibrium constant stage 13 |
| $K_{14}$ | Equilibrium constant stage 14 |
| $L_H$ | Reflux flow (liquid) |

| | |
|---|---|
| $L_M$ | Middle flow (liquid) |
| $L_n$ | Liquid flow at stage n |
| $L_1$ | Liquid flow at stage 1 |
| $L_2$ | Liquid flow at stage 2 |
| $L_3$ | Liquid flow at stage 3 |
| $L_4$ | Liquid flow at stage 4 |
| $L_5$ | Liquid flow at stage 5 |
| $L_6$ | Liquid flow at stage 6 |
| $L_7$ | Liquid flow at stage 7 |
| $L_8$ | Liquid flow at stage 8 |
| $L_9$ | Liquid flow at stage 9 |
| $L_{10}$ | Liquid flow at stage 10 |
| $L_{11}$ | Liquid flow at stage 11 |
| $L_{12}$ | Liquid flow at stage 12 |
| $L_{13}$ | Liquid flow at stage 13 |
| $L_{14}$ | Liquid flow at stage 14 |
| $L_{ns}$ | Bottom flow |
| $L_{n+1}$ | Liquid flow at stage n+1 |
| $p^*$ | Saturated pressure |
| M | Holdup in middle vessel |
| $\tau_c$ | Closed loop time constant |
| $\tau$ | Process time constant |
| $V_B$ | Vapor flow |
| $V_1$ | Vapor flow into condenser |
| $V_2$ | Vapor flow at stage 2 |
| $V_3$ | Vapor flow at stage 3 |
| $V_4$ | Vapor flow at stage 4 |
| $V_5$ | Vapor flow at stage 5 |
| $V_6$ | Vapor flow at stage 6 |
| $V_7$ | Vapor flow at stage 7 |
| $V_8$ | Vapor flow at stage 8 |
| $V_9$ | Vapor flow at stage 9 |
| $V_{10}$ | Vapor flow at stage 10 |

| | |
|---|---|
| $V_{11}$ | Vapor flow at stage 11 |
| $V_{12}$ | Vapor flow at stage 12 |
| $V_{13}$ | Vapor flow at stage 13 |
| $V_{14}$ | Vapor flow at stage 14 |
| $V_n$ | Vapor flow at stage n |
| $V_{n+1}$ | Vapor flow at stage n+1 |
| $x_1$ | Liquid mole fraction at stage 1 |
| $x_2$ | Liquid mole fraction at stage 2 |
| $x_3$ | Liquid mole fraction at stage 3 |
| $x_4$ | Liquid mole fraction at stage 4 |
| $x_5$ | Liquid mole fraction at stage 5 |
| $x_6$ | Liquid mole fraction at stage 6 |
| $x_7$ | Liquid mole fraction at stage 7 |
| $x_8$ | Liquid mole fraction at stage 8 |
| $x_9$ | Liquid mole fraction at stage 9 |
| $x_{10}$ | Liquid mole fraction at stage 10 |
| $x_{11}$ | Liquid mole fraction at stage 11 |
| $x_{12}$ | Liquid mole fraction at stage 12 |
| $x_{13}$ | Liquid mole fraction at stage 13 |
| $x_{14}$ | Liquid mole fraction at stage 14 |
| $x_n$ | Liquid mole fraction at stage n |
| $x_{n+1}$ | Liquid mole fraction at stage n-1 |
| $x_H$ | Liquid mole fraction in top vessel |
| $x_M$ | Liquid mole fraction in middle vessel |
| $x_B$ | Liquid mole fraction in reboiler |
| $x_c$ | Liquid mole fraction in condenser |
| $y_1$ | Vapor mole fraction at stage 1 |
| $y_8$ | Vapor mole fraction at stage 8 |
| $y_n$ | Vapor mole fraction at stage n |
| $y_{n-1}$ | Vapor mole fraction at stage n+1 |

# LIST OF ABBREVIATIONS

| | |
|---|---|
| CMVBD | Cyclic Middle-vessel Batch Distillation |
| IMC-PI | PI controller with Internal Model Control Tuning Method |
| MIMO | Multi input multi output |
| MVBDC | Multi-vessel Batch Distillation Column |
| P | Proportional only controller |
| PI | Proportional integral controller |
| PID | Proportional integral derivative controller |
| RGA | Relative gain array |
| SVD | Singular Value Decomposition |
| ZN | Ziegler Nichols |
| ZN-P | P controller with Ziegler Nichols Tuning Method |
| ZN-PI | PI controller with Ziegler Nichols Tuning Method |

# CHAPTER 1

# INTRODUCTION

The advancement of chemical and biotechnology, pharmaceutical and fine and specialty chemical industries have extensively grown recently and as a result academic interest in batch distillation process is growing rapidly. Production of all those chemicals are examples of such small and multi-product operations where the product are typically required in small volumes and subject to short product cycles and fluctuating market demand (Cheong, 1998). Since the process runs batch wise, the process is operated significantly depending on a batch to batch variability and result to changes due to possible formulation. Hence, without proper control system, process operation faces some risk such as safety issues, economic issues and product specifications issues (Ogunnaike and Ray, 1994). Clearly, process control is very crucial in batch distillation in order to stabilize the process, as well as to maintain production quality and to impart adequate disturbance rejection (Lee et al., 2008).

## 1.1 Multi-vessel Batch Distillation Column (MVBDC)

Simple batch distillation has been known as the oldest separation technique, while the continuous distillation is a result of batch distillation's improvement (Hisyam, 2011). Batch production is very flexible, it can easily be adapted and reconfigured to produce many other products. Batch distillation is particularly suitable for low volume, high value products such as pharmaceuticals, polymers, biotechnological or other fine chemicals (Habobi and Yaseen, 2016).

Recent investigations focused on complex batch distillation column namely middle vessel column and multi-vessel column. Middle vessel column as discussed by Barolo et al. (1996a and 1996b) and Farschman and Diwekar (1998), Gruetzmann et al.

(2006 and 2008) is shown Figure 1.1. Meanwhile, multi-vessel batch distillation column as shown in Figure 1.2 as discussed by Hasebe et al. (1995), Skogestad et al, (1996) and Tang et al. (2014). The main difference between conventional and complex batch distillation is the installation of additional product vessel along the column. In addition, the complex batch distillation consists of reboiler, n-product vessel, n-1 column section and a condenser (Gruetzmann et al., 2006). In the middle vessel column, feed is charged to the middle of column (Barolo et al., 1996a). Meanwhile, in the multi-vessel column feed is charged to all vessels in the column (Bai et al., 2014).

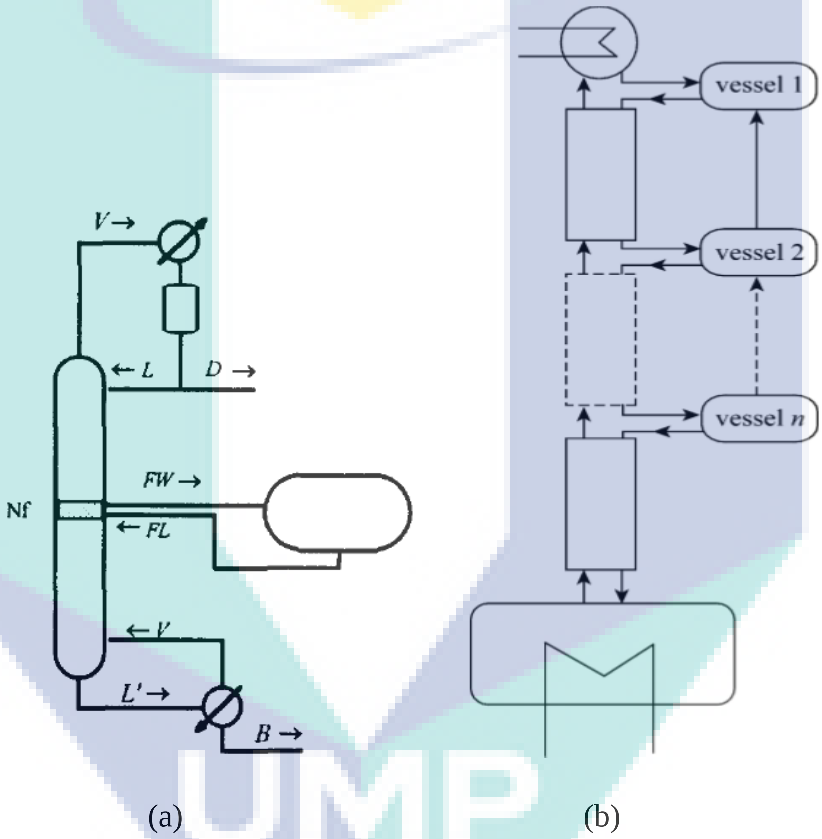



Figure 1.1 Configuration of (a) Middle Vessel Batch Distillation with n-Vessel (b) Multi-Vessel Batch Distillation with n-Vessel

Source: Barolo et al. (1996a); Tang et al. (2014)

Multi-vessel batch distillation column (MVBDC) as shown in Figure 1.2 is said to be more energy efficient due to the multi effect nature of the operation (Hasebe et al., 1995), where the heat required for the separation is supplied only by the reboiler and cooling is done only at the top. In addition MVBDC requires shorter batch time for separation of multiple component mixture (Bai et al., 2014). Hasebe et al. (1995)

mentioned in his paper the energy requirement for MVBDC is lesser and it is comparable to continuous distillation for some separation with multiple components.

Basically, multi-vessel batch distillation is operated under total reflux operation and maximum fractionating capacity and maximum separation efficiency can be achieved under this operation mode (Gruetzmann et al., 2008) because the operation will continue operated in a closed operation. As shown in Figure 1.1 (b), MVBDC consist of 3 identical column, n-vessel mounting along the columns, a reboiler and a condenser. Roughly, at the beginning all vessels are charged with initial feed mixtures (Tang et al., 2014). The operation of MVBDC is continuously operated under total reflux mode (closed operation) with constant heat input. The mixture is continuously circulated in the column and separated until all the product compositions in the vessel reaches its specification and the composition remains unchanged and almost pure product is produced. At this point, the product is collected from the vessels without worrying that the composition decreases by time.

## 1.2 Control of Multi-vessel Batch Distillation

MVBDC is mainly used in the production of high purity products. As mentioned in previous section, maximum fractionating can be achieved if it is operate under total reflux operation. Hence, it is very important to develop MVBDC control structure that can maintain the total reflux operation. Hasebe et al. (1995) suggested that to control the total reflux operation, the level holdup must be kept constant during operation. The holdup is specified initially based on final product composition. According to Skogestad et al., (1996) level control is too sensitive to uncertain initial feed composition with the same set point. Skogestad et al. (1996) mentioned that the adjustment of vessel holdup based on composition is too complicated to implement. Then, in order to overcome this problem, temperature control was introduced by Skogestad et al. (1996) and experimental work was performed by Wittgens et al. (2000).

A comparison of level control structure and temperature control structure was performed by Fanaei et al. (2012). Based on the findings, temperature control is said to be not controllable during the startup operation due to complexity of startup operation. MVBDC with temperature control requires more energy because longer batch time is needed for separation process. Fanaei et al. (2012) mentioned that level control structure gives acceptable performance for MVBDC. The process is more controllable with level control structure compared to temperature control structure. Barolo et al. (1996a) in his study suggested to perform detail analysis such as interaction analysis to select the best control structure for this complex column, since, they are many possible control structures can be chosen.

## 1.3 Problem Statement

In MVBDC system, there are many possible control structures. If the number of vessels used in the column increases the number of variables also increases (Barolo et al., 1996). The selection of the control structure in MVBDC will affect the controllability, performance and production of MVBDC system. Normally, the control structure can be directly obtained from degree of freedom analysis. However, the MVBDC system is dynamic. The dynamic behaviour should be analysed in order to see the effect of each variable toward another. Fruehauf and Mahoney (1993), Barton (1997) and Roat et al. (1988) had performed the analysis to see the behaviour of process variables in order to determine controlled variable, manipulated variables, disturbance variable and set point. As a result, more exact set point and variables can be determined graphically. In MVBDC system, most of past researchers only mentioned the control structure used, they did not highlight the analysis or method used to determine their control structure. This is the reason why MVBDC system with excellent control structure could not be produced.

In order to develop a good control system for MVBDC system, the attention should not only focus on finding the best control structure. The type of controller pairing with a good tuning method is necessary. This can be determined by controllability study to keep the outputs of the process within specified bounds from their set point, in spite of unknown variations such as disturbance and plant changes (Skogestad and Postlethwaite, 2005). For instance, in batch reactive distillation and regular batch distllation distillation, there are controllability study in detail performed by Skogestad and Sorensen (1992) and Nunes et al. (2014). The study focused on the sensitivity and controllability of control strategy in batch system. Different alternative controlled and manipulated variables were discussed. As a result control system that produce stable product quality is considered (Skogestad and Sorensen, 1992). But there is still lack of literature for MVBDC had highlighted the tuning and performance of controller of MVBDC control system which then can be affected to the overall performance and production of MVBDC system.

Hence, by taking into account the issue arisen, which is to develop a good control system for MVBDC system including determining the excellent control structure and good controller performance based on proper method and analysis, this research will try to cover the issues highlight.

## 1.4 Research Objectives

1. To study the sensitivity of dynamic process variables based on open loop behaviour.
2. To study the controllability of MVBDC for separation of non-azeotropic mixture which include determination of closed loop behaviour.

## 1.5 Research Scope

Basically, this research is on the controllability study of MVBDC with level control. This MVBDC will be used to separate ternary non-azeotropic mixtures and operated under total reflux operation. Implementation of process control design will be based on objectives to maintain total reflux operation (maximum fractionating). The best pair of level control loops with the good tuning method will be chosen to control this operation.

First, a set of mathematical model will be develop based on first principle of material balance. The model is simulated using MATLAB Simulink. The open loop model is developed and validated.

Second, a three step sensitivity analysis will be performed in order to determine the suitable controlled variable, manipulated variable and disturbance variable.

Third, interaction analysis using relative gain array (RGA) and singular value analysis (SVD) and dynamic analysis will be performed. This interaction analysis will be used to obtain the exact value condition number in order to choose the least interacting variables.

Fourth, the close loop model will be developed using Simulink with implementation of proportional (P) and proportional integral (PI) controller.

Fifth, the controller tuning based on close loop response will be performed using Ziegler Nichols (ZN) method and Internal Model Control (IMC) method.

Lastly, in order to analyse the controller performance, set point changes analysis will be performed by setting the new set point. Meanwhile, disturbance analysis will be performed by introducing disturbance to the system.

# CHAPTER 2

# LITERATURE REVIEW

In this Chapter 2, a review on the batch distillation researches that have been carried out within the last few years is presented including control, simulation, operation and modelling of middle vessel, multi vessel, regular batch distillation column and continuous distillation column. These review will emphasize the control of batch distillation with intermediate vessels, such as middle vessel and multi-vessel batch distillation.

## 2.1 Multi-vessel Batch Distillation Column (MVBDC)

MVBDC is a combination of a batch rectifier and stripper column that can be used to separate a ternary mixture. MVBDC is used for processing high value products such as specialty and fine chemicals as well as in the production of flavours, fragrances, pharmaceuticals, dyes, and some other products, which are produced in small volume (Salomone et al., 1997) (Roa and Barik., 2012). The idea was first based on the middle vessel column that was mentioned by Robinson and Gilliland (1950) and was first analyzed for purifying binary mixtures by Bortolini and Guaruse (1971). Then, Hasebe et al. (1996) came with the idea to charge a ternary mixture to the middle vessel, so light components are accumulated at the top and heavy components are accumulated at the bottom of the column respectively and the operation stops when intermediate component reaches its desired purity in the middle vessel (Skogestad et al., 1997).

Lastly, Hasebe et al. (1996) came out with the new configuration of batch distillation which is MVBDC under total reflux operation. MVBDC, as a generalization of previously proposed batch distillation schemes, includes the inverted column and the middle-vessel column. It is possible to obtain n pure products by using VBDC in a single batch with n vessels along the column including a reboiler, a condenser and n-2 intermediate vessels (Hasebe et al, 1995) because the system is running in a closed operation (total reflux). The main significance for using the arrangement is that it usually requires less energy or shorter batch time for a given heat input than a regular batch distillation column (Wittgens et al., 2000) (Mahmud et al., 2008).

The operation of MVBDC starts with filling all vessels with a specified amount of feed. The system is perated under total reflux mode, while waiting, holdup in the top drum reaches its on-specification composition (Tang et al., 2014). Bai et al. (2014) mentioned that after the product composition reaches its specification for the first time, all the filled vessels are isolated from the column and the top product in vessel 1 is drained away. The other two vessels (Vessel 2 and Vessel 3) are moved upwards. Consequently, the present holdup in Vessel 1 is the holdup in Vessel 2 and Vessel 2 is the holdup in Vessel 3. At this moment the Vessel 3 is empty and not connected to the column. The remaining filled vessels (Vessel 1 and Vessel 2) are connected to the column and the system repeats under total reflux operation. This cycle is repeated for three time (three time total reflux operation) until product composition in each vessel is completely separated (Bai et al., 2014).

### 2.1.1 Total Reflux Operation of Multi-vessel Batch Distillation Column (MVBDC)

Hasebe et al. (1995) suggested that the simplest strategy for multi-vessel batch distillation is total reflux operation (closed operation). Skogestad et al. (1996) clarified that there are at least two advantages of multi-vessel batch distillation under total reflux operation. First, the operation of multi-vessel batch distillation is simple because no product change-over is required during the operation. Second, the energy requirement may be lesser due to the multi-effect nature of the operation, where the heat required for the separation is supplied only to the reboiler and cooling is done only at top (Skogestad, 1995). Hasebe et al. (1999) compared MVBDC under total reflux operation with rectifier column and found out that energy consumption in multi-vessel column is about half of the cyclic two vessel column. Hilmen (2000) emphasized that MVBDC needs less time than two vessel column. Cyclic method is mostly appropriate for small laboratories (Skouras and Skogestad, 2004).

The latest studies by Tang et al., (2014) focused on the new operation mode of MVBDC which is constant reflux. For this new operation, the whole operation is carried out within infinite reflux (total reflux) ratio until all vessels are filled with desired product. Molar vapor flow is constant during operation (Bai et al., 2014) (Hisyam, 2011). The new mode is apparently offering significant less operation time and maximum fractionating compared with regular constant reflux ratio (CR) in batch distillation. In their study, MVBDC does not adjust the reflux ratio as in a regular CR operation but collects the product by shifting the vessels or switching the valves in the pipelines under total reflux. Thus the new mode is easier to operate without expensive instrumentation. However, more works have to be done in research of mass transfer and control policy in order to make the process a practical technology (Tang et al, 2014).

Warter et al. (2004) performed an experimental investigation of the separation of zeotropic ternary mixture via total reflux operation. Batch distillation with middle vessel as shown in Figure 2.1 was used to carry out separations into three fractions. This configuration is an example of multi-vessel column for laboratory scale. Based on experiment result for separation of zeotropic mixture it shows that a batch distillation with a middle vessel can be easily operated in a total reflux operation with constant holdup mode for removing light and heavy boiling impurities from an intermediate boiling impurity. The high purity is successfully obtained. The result proves that batch distillation with a middle vessel that is operated under total reflux operation offers many practical advantages such as reduction in the temperature in the feed vessel, in the contact time with the hot surface of the reboiler and reduction of time in the duration of the process start-up. It also offers the possibility to reduce the size of reboiler.

Based on the short review, it shows that the MVBDC under total reflux operation offers many practical advantages in specialty chemical, fine chemical, pharmaceutical and biochemical industries. Maximum fractionating and shorter separation time can be achieved under this operation mode. Hence, high purity product can easily be produced.

## 2.2 Control of Multi-vessel Batch Distillation Column (MVBDC)

Basically in MVBDC, the most important controlled objective is to minimise the batch time and maximize the production quality (Skogestad and Sorensen, 1992). It is important to design a control system that satisfies all the controlled objectives. Control involves the manipulation of the material and energy balances in the distillation equipment to affect the product composition and purity. Difficulties arise because of the multitude of potential variable interactions and disturbances that can exist in single-column and in the process of the column (Liptak et al, 2005).

During initial start-up period, the operation must be in total reflux mode (Treybal, 1970). Hence, the accumulator holdup level should be kept constant during the operation using reflux to control level (Hasebe et al, 1995). However, this operation is sensitive to errors in the feed composition and the errors in the level control (Skogestad et al, 1997). A correction on the level set point based on composition measurements is introduced in order to overcome this problem as per stated by Bortolini and Guarise, (1970) and Hasebe et al.(1995) but this makes the control system complicated and requires on-line composition measurements. To avoid these problems, Skogestad et al. (1997) suggested that the accumulator holdup level is indirectly adjusted by using the reflux to control the temperature at some location in the column section shown in Figure 2.1. Skogestad et al. (1997) showed through simulations that this simple way of operation works very well, but the concerns is on the practically, especially for the multi-vessel column (Skogestad et al., 1997).

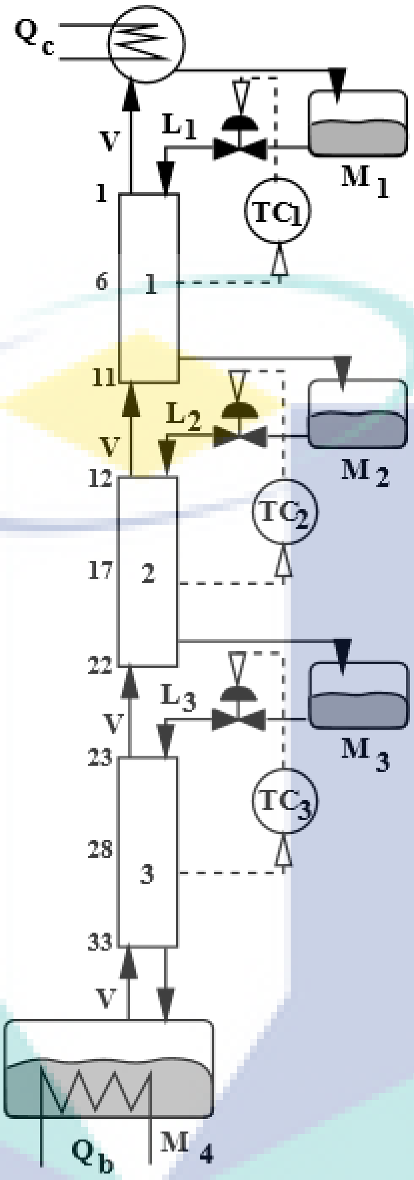



Figure 2.1 Control scheme for closed operation of multi-vessel batch distillation with two intermediate vessel

Source: Skogestad et al. (1997)

Skogestad et al. (1997) studied the feedback control strategy of multi-vessel batch distillation using three proportional only temperature control. The set point was set as the average boiling point of two components being separated in the column section. Two different initial feed composition was considered. As a result, the steady state composition obtained was the same for both feed mixture. This shows that temperature control is not sensitive to the changing feed composition.

Skogestad et al., (1997) also performed the simulation which demonstrated the feasibility of the multi-vessel batch distillation under total reflux. Under this situation the vessel holdup is kept constant. This situation is tested with two different initial feed compositions similar to the temperature control. However, the result shows that, for the steady state composition, level control is very sensitive to the changing feed composition. As a conclusion, by considering the change of initial composition it is more preferable to use the temperature control instead of keeping the holdup constant.

However, in the most recent study by Fanaei et al. (2012), the best control structure is level control because it gives the best system performance compared to the temperature control. The temperature control is said to be not controllable at the start-up period. The process is very highly nonlinear at the start-up and a good controller is necessary. Furthermore, the temperature control is too sensitive to the initial refluxes and needs to have some experience to know how much the reflux valves must be opened to get optimum operation. The schematic diagram for both control structure is shown in Figure 2.2 and Figure 2.3.

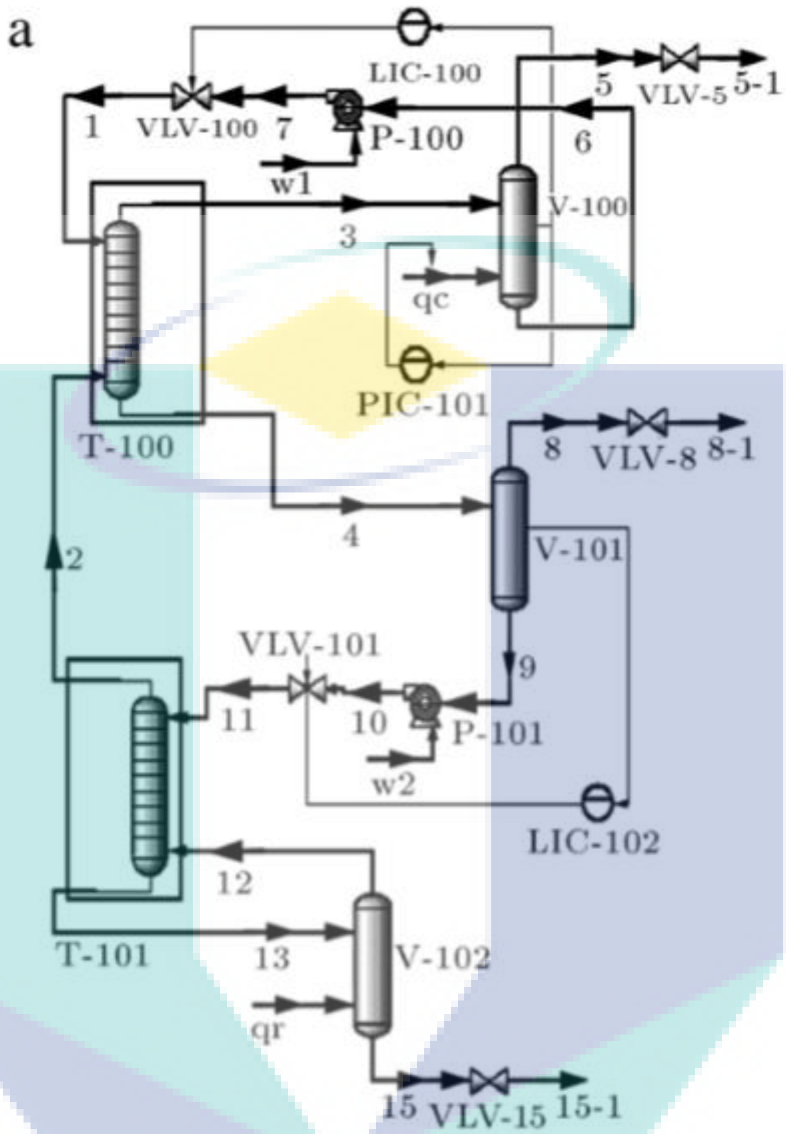



Figure 2.2 Schematic diagram of Modified Middle Vessel Batch Distillation Column with Level Control structure.

Source: Fanaei et al. (2012)

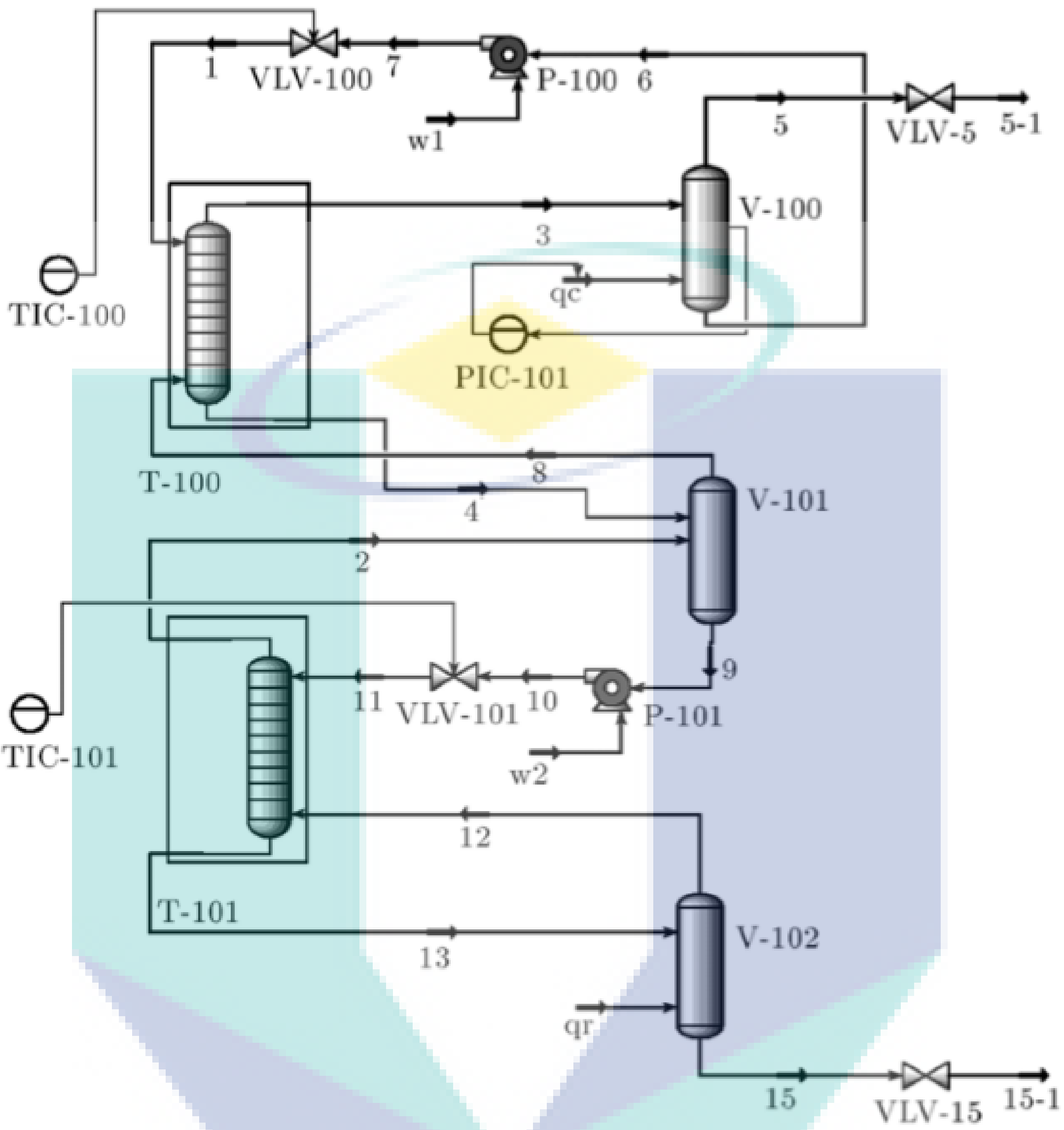



Figure 2.3 Schematic diagram of Modified Middle Vessel Batch Distillation Column with Temperature Control structure.

Source: Fanaei et al. (2012)

MVBDC with the level control structure consists of two level controls and one pressure control as shown in Figure 2.2. MVBDC with temperature control structure consists of two temperature controls as shown in Figure 2.3. In comparison with level control structure, temperature control structure needs more time and energy as shown in Table 2.1.

Table 2.1 Simulation results of energy requirement, time and final compositions

| Column | | Reboiler duty (J x $10^6$) | Condenser duty (J x $10^6$) | Time (min) | Final compositions (molar) |
|---|---|---|---|---|---|
| Conventional | | 2690.7 | 2595.8 | 504.5 | [0.9985,0.99,0.9991] |
| Modified (LC)[a] | | 2152 | 2058.6 | 403.5 | [0.996,0.99,0.9922] |
| Rectifier | Cycle 1 | 1733.3 | 1659.3 | 325 | [0.99,0.535,0.465] |
| | Cycle 2 | 1578.7 | 1550 | 296 | [-,0.99,1] |
| Modified (TC)[b] | | 2453.3 | 2357.4 | 460 | [0.991,0.99,0.9977] |

Source: Fanaei et al. (2012)

Barolo et al. (1996) had studied the middle vessel with level control. Barolo et al. (1996) mentioned that the presence of a middle vessel and stream in (FW) and stream out (FL) from the middle vessel provide an extra degree of freedom for middle vessel batch distillation column as shown in Figure 1.1(a). This situation is similar to multi vessel batch distillation column (MVBDC) but the difference is only the amount of vessel mounted along the column. Hence, in MVBDC if the number of vessel increases, the number of degree of freedom also increases. In normal batch distillation, only one holdup needs to be controlled. In middle vessel column, two holdups need to be set under level controlled. Normally, reflux drum is chosen as the first controlled holdup and remaining controlled variable is middle holdup and bottom holdup. Since this middle vessel column is operated under total reflux operation, the vapor boilup is constant during operation. Thus, the bottom flowrate is set to constant. Hence, middle flowrate is left to controlled level of bottom holdup, which may be impractical. Other alternative is to do a ratio control to control the level in middle holdup and the bottom level controlled by bottom flowrate directly. But all the control structure is depends on the available plant measurement. Specifically, the controllability in term of interaction analysis and sensitivity analysis needs to be studied in detail in order to select the best control structure for each system.

In addition, Hasebe et al. (1999) mentioned that by optimizing the reboiler holdup as a function of time, the separation performance of MVBDC is better compared to rectifying column if the holdup is optimized and kept constant (control). The separation performance of MVBDC can be increased from 18% to 38%. Based on the separation performance, MVBDC with constant holdup policy requires less energy for separation compared to rectifying column. It can be concluded that MVBDC with constant holdup is widely applicable as an effective energy separation system.

Meanwhile, in middle vessel column, Gruetzman et al. (2006) studied control of the cyclic operation of middle vessel batch distillation. This configuration is a special case of a multi-vessel column operating with total reflux and the process control used is very similar with multi-vessel column. As stated by Skogestad et al., (1997), the temperature set point is the average of the boiling point of two components. The knowledge of vapor pressure behaviour is necessary to apply this process control strategy. The detailed information of vapor liquid equilibrium (VLE) need to be provided but in many cases the nature of some cases is not known. The specifications of two temperature set point is problematic and inaccurate (Gruetzman et al, 2006).

Furthermore, control structure based on temperature measurement is optimal controller setting but to optimise the start-up operation, the controller setting must be adjusted. Due to this disadvantage, two step control strategy that considers industrial constraint was proposed by Gruetzmann et al. (2006). First step is mass control strategy and second step is temperature control to ensure the product is achievable. During this first step the holdup is kept constant based on the product specification. At the end of first stage the temperature along the column reached a steady state and the product quality was closed to the desired value. However, there must be slight deviation of product quality. At this point temperature control is implemented. By making small changes of temperature set point, the product quality can be set exactly. At the end of stage two, the product quality can be achieved successfully. The main advantage of this process strategy is the efficient use of all available information during entire process (Gruetzman et al, 2006). Normally, during steady state, temperature remained to its set point. Rarely seen the extreme temperature deviation during the steady state. Hence, temperature control is not very significance during this state.

The study of close loop control of MVBDC is mainly cover the choice of control structure available for this system. However, there are lacks of analysis and methods used to determine the best control structure in order to improve the performance of MVBDC. There is still a room for improvement. Future work in close loop control problems can involve identifying the proper control structure, easy parameter tuning, and focusing on tracing the optimal profiles, as well as on-specification products.

### 2.2.1 Sensitivity analysis

In order to meet the market specifications, plant or process must be regulated at new optimal operating points. Hence, dynamic model should be used for identification and controllability studies. Sensitivity analysis is a part of the controllability study. The purpose of sensitivity analysis includes (Barton et al., 1997):

a) Identify necessary process measurement
b) Determination of appropriate control structure (matching of sensors to control elements) and tuning of control parameters.
c) Validation of control system by predicting plant response to a variety of loads and set point changes.
d) Study sensitivity of process output to range of common disturbance.

There are a few studies in distillation system that consider the sensitivity analysis in their works, e.g. study on the controllability of Bayer distillation process (Barton, 1997). A system of eight coupled and heat integrated distillation columns was used to separate eight crude silanes from a mixture of 40 components. The objective of their study was to study the performance of control system. Sensitivity analysis for dynamic heat integrated column is also presented in this study. The purpose for this analysis is mainly for measurement selection and also to define appropriate control structure. By conducting sensitivity analysis, the optimum temperature for the separation can be determined based on the product composition. Hence, the set point of the temperature control can be determined.

Fruehauf and Mahoney (1993) study was focusing on a control design procedure and an example of application of steady state model technique to an actual column. In his study, the design of single point composition control was presented. The vast majority of columns have one sided composition specifications which can keep both top and bottom product compositions at or below limits for a wide range of disturbances.

The control design procedure includes developing design basis, selecting candidate control scheme, open loop testing, closed loop testing and objectives. The sensitivity analysis is considered in open loop testing procedure. The purpose is to find a candidate temperature sensor location, for temperature control system and to select a candidate control scheme. Based on findings, temperature below the feed appears to be reasonably sensitive to steam rate changes. Thus, temperature does not longer uniquely define compositions under such conditions. The temperature sensor location is set below the feed. In batch distillation column, the study performed by Zamprogna et al (2005) and Zampogna et al. (2005) was using sensitivity analysis to select the number and locations of temperature measurement to be used as a soft sensor inputs for estimating composition profile in a batch distillation column. This sensitivity analysis gives details figure of temperature behaviour in the columns. Hence, accurate decision can be made.

The sensitivity analysis also covers the study on the effect of disturbance on the process system (Raot et al., 1988). This analysis can be used to evaluate the candidate disturbance variables for control system. Emtir et al. (2003) performed the open loop performance for feed rate disturbance for his controllability study on heat-integrated distillation scheme. Figure 2.10 shows the response of disturbance (Feed rate) on the controlled variable (Product mole fraction).

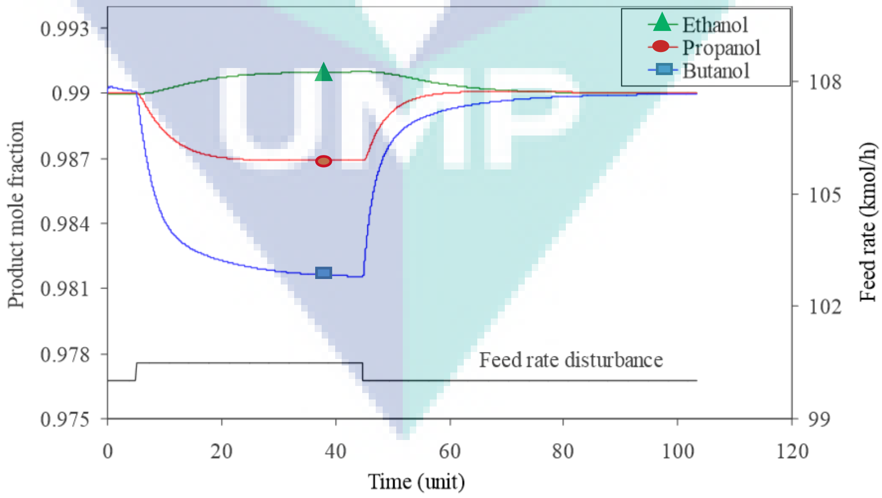



Figure 2.4 Sensitivity Analysis on Product Mole Fraction
Source: Emtir et al., 2003

Based on the response in Figure 2.4, the product mole fraction returns to its set point, although there is disturbance presence at the beginning of operation. Hence, the system is controllable.

Other researches covering sensitivity analysis in batch distillation were highlighted by Nunes (2014). Nunes (2014) performed the sensitivity analysis in order to analyse the impact of the reflux ratio and heat duty supplied to the reboiler. The heat duty and reflux ratio are the main process condition in the batch distillation column. The sensitivity analysis is performed by increasing the reflux ratio +5% and -5% and increasing the heat duty +10% and -10%. Based on the findings, lower reflux ratio and higher heat duty require shorter separation time while higher reflux ratio and lower heat duty require longer separation time. Sensitivity analysis not only use to determine the controlled variable, manipulated variable and disturbance variable, the important process condition can also be determined.

Montes et al. (2015) studied the sensitivity analysis on batch distillation system. The sensitivity analysis has revealed that changes in the heat duty and in the reflux value have a huge impact in the distillate purity. Additionally, step sensitivity analysis that is performed on 2 of 3 analysed variables can possibly detect and correct the mathematical problems within distillation model.

In overall, based on the previous research it is important to consider the sensitivity analysis of the process rather than depending on the calculation of degree of freedom only. The sensitivity analysis shows a clear figure of relation of controlled variable, manipulated variable and disturbance variable and process condition.

### 2.2.2 Loop Interaction: Distillation System

MIMO control system is inherently more complex than SISO control system because process interactions occur between controlled and manipulated variables. A change in manipulated variable will affect all of the controlled variables (Rajaraman, 2016). Due to process interaction, the selection of the best pairing of controlled and manipulated variables for a multi loop control scheme can be a difficult task. There are *n!* possible multi loop configurations for a control problem with *n* controlled variable and *n* manipulated variables, (Seborg, 2010).

The selection of controlled variables is one of the most important tasks in control structure design because this choice can limit the operational (economic) performance of the whole control system (Razzhagi et al., 2009).

In the distillation system there are many interactions that occur; one of the examples is interaction between temperature controllers. Supposing that a top section of temperature controller manipulates the reflux, instead of the shown flow controller and bottom section temperature controller manipulates boil up. The composition of light component will rise in the column feed. The temperature controllers will increase boil up and decrease reflux, if a drop in both control temperatures is sensed. Both temperatures control will return to their set point without interaction if the actions of the two controllers are perfectly matched and instantaneous. However, the two actions are rarely perfectly matched. Usually boil up response is faster. The additional boil up will return the bottom section temperature to its set point but in the same time it will also raise the top section temperature. The top section now calls for more reflux and bottom temperature controller, now will call for less boil up, if it felt the previous reduction in reflux. Each subsequent change will affect both reflux and reboil and the two will cycle (Henry, 1990).

The main works for the selection of manipulated or controlled variable pairings have focused upon using controllability measures, such as relative gain array and structured singular value μ.

#### 2.2.2.1 Relative Gain Array (RGA) Analysis

A MIMO control scheme is important in systems that have multiple dependencies and multiple interactions between different variables. In a distillation column, where a manipulated variable such as the reflux ratio could directly or indirectly affect the feed flow rate, the product composition, and the reboiler energy (Sankaranarayanan and Deepakkumar, 2015).

Thus, understanding the dependence of different manipulated and controlled variables in a MIMO control scheme could be extremely helpful in designing and implementing a control scheme for a process. One method for designing and analysing a MIMO control scheme for a process in steady state is by a Relative Gain Array (RGA).

The RGA describes the impact of each control variable on the output, relative to each control variable's impact on other variables and it is a normalized form of the gain matrix. For all possible input-output variable pairings, the process interactions of open-loop and closed-loop control systems are measured. A ratio of this open-loop gain to this closed-loop gain is determined and the results are displayed in a matrix (Haggblom, 1995).

The array as shown in equation 2.1 is a matrix with one row for each output variable in the MIMO system and one column for each input variable and. This format allows a process engineer to match the input and output variables that have the biggest effect on each other while also minimizing undesired side effects and to easily compare the relative gains associated with each input and output variable pair.

$$RGA = \Lambda = \begin{bmatrix} \lambda_{11} & \lambda_{12} & \cdots & \lambda_{1n} \\ \lambda_{21} & \lambda_{22} & \cdots & \lambda_{2n} \\ \lambda_{31} & \lambda_{32} & \cdots & \lambda_{3n} \\ \lambda_{n1} & \lambda_{n2} & \cdots & \lambda_{nn} \end{bmatrix} \tag{2.1}$$

A study on the interaction analysis of control loop in distillation column is widely covered. As per stated in Sankaranarayanan and Deepakkumar (2015) study, by performing RGA analysis on the control scheme of continuous distillation, the best pair of manipulated and controlled variable is determined. Controller is designed for each best pair of the input and output converting a MIMO system to multivariable SISO system. Based on the findings, reflux flow – distillate composition and reboiler flow – bottom composition are the best pairing. Thus, the desired composition of the bottom and distillate (top) in the distillation column is maintained.

Based on open literature, Skogestad et al. (1994) studied the control strategies of reactive batch distillation. In order to choose the control loop pairing, Skogestad et al. (1994) performed the RGA and sensitivity analysis. From the RGA calculation, loop pairing reboiler duty that is used to control the reactor temperature produced less interaction and either reflux flow or distillate flow to control the distillate composition or loss of reactant. Then, sensitivity analysis based on the effect of disturbance shows that controlling temperature in a tray in a column would give better separation performance.

Other study highlighted by Farschman and Diwekar (1998) showed the interaction of composition control in middle vessel column and mentioned that the degree of interaction between the two composition control loops can be assessed using the relative gain array technique. The analysis shows that the interaction between the two loops for this new column is mostly negligible due to the large time constant of the middle vessel. Furthermore, with the middle vessel column, there is a greater likelihood of reducing the interaction between control loops by varying the parameter q′ (the ratio of the vapor rate in the rectification section of the column to the vapor rate in the stripping section of the column.

Some limitation of RGA stated by Haggblom (1995), the RGA does not contain explicit information on how other inputs $u_k$ effect output $y_i$ when it is paired with input $u_j$ or how $u_j$ affects other outputs $y_k$ when the loop $y_i$ and $u_j$ is closed. Moreover, for 3 by 3 and larger systems, variable pairing based on the RGA may fail because the RGA may be unable to discriminate between several feasible sets, all feasible sets and any feasible set of variable pairings (Haggblom, 1995). Other additional methods can be used to confirm interaction of control loops.

#### 2.2.2.2 Singular Value Decomposition (SVD) Analysis

In particular, the field of control structures, often use singular values and the condition number as measures when comparing or designing different control structures. The basis for directionality analysis usually uses a stem from the singular value decomposition (SVD) of a matrix (Razzhagi et al., 2009).

SVD, that is also called singular value analysis (SVA), is a useful linear, steady-state tool in the control analysis of multivariable chemical processes. By calculating the SVD of the steady state gain matrix the controller pairing and system evaluation can be ascertained to a certain degree (Canter, 1987). Singular matrix can be seen in the form of equation 2.2. SVD considers directional changes in the disturbances. SVD is applied to steady state gain matrix that is decomposed into product of three matrices, A = USVT where U and V are matrix of normalized eigenvectors, S is diagonal matrix of eigenvalues. The condition number (CN) is defined as the ratio between maximum and minimum eigenvalues. Generally if CN < 50 then the system is not prone to sensitivity problems (a small error in process gain will not cause a large error in the controller's reactions) (Sujatha, 2013).

$$S = \begin{bmatrix} \sigma_i & 0 & 0 \\ 0 & \sigma_i & 0 \\ 0 & 0 & \sigma_i \end{bmatrix} \qquad 2.2$$

One main motivation for studying directionality stems (SVD) is that from the common belief that plants with a large CN (plants with high directionality) are potentially hard to control (Razzaghi et al., 2009).

### 2.2.3 Controller Tuning

After the selection of control structure, the most important part is the controller tuning. The good controller tuning would guarantee good controller performance. For many decades, the conventional controller has continued to be the most extensively used as process control technique. The proportional integral derivative (PID) controller is widely used in industrial application due to its ability to compensate many practical industrial (O'Dwyer, 2000). A correctly planned and tuned PID controller has proved to be suitable for the vast majority of industrial control loops, although advanced manage techniques such as model predictive control can provide major improvement (Patil et al, 2017).

The controller performance depends on the controller tuning. Best controller performance is produced from the excellent controller tuning. There are four major characteristics (Figure 2.10) of controller performance (Ishak and Abdullah., 2013):

a) Rise time: The time it takes for the plant output to rise beyond 90% of the desired level.
b) Overshoot: How much the peak level is higher than the steady state, normalized against steady state.
c) Settling time: The times it takes for the system to converge to its steady state.
d) Steady state error: The difference between the steady state output and desired output.

In MVBDC, a number of studies highlight the type of controller used with their tuning method, for example Fanaei et al. (2012) using PI controller for the level control loops. During startup the controller is tuned by trial and error. Then, at the end of the process when the process becomes steady state, the relay feedback auto tuning is used. This PI controller with relay feedback tuning works very well and ensures the controller performance.

Meanwhile, Gruetzmann et al. (2006) used level control with PI controller. The controller parameter has been adjusted by trial and error in a way that a smooth operation has been assured. Whereas, Wittgens et al. (2000) used PI controller to control the temperature control. PI controller is tuning by trial and error method. Each controller gain is set to obtain smooth and good response.

There are vast of controller PI and PID tuning method studies conducted by researchers such as Shamsuzzoha et al. (2007), Priyadarshani and Lather (2013), Shahrokhi and Zamorrodi (2013) and O'Dwyer (2000). As mentioned by O'Dwyer et al. (2000), PI controller with appropriate controller tuning can assure the controllability of the process. The findings of simulation results by O'Dwyer study shows that the stability tends to be assured when a PI controller tuning is used. Based on the review above, in MVBDC, majority used the PI controller is used. The smooth and good response can be successfully produced. However, the tuning method used should be improved. Hence, the review will focus on PI tuning methods. The PI tuning method is classified into two main categories, open loop and closed loop method. Open loop tuning method refers to methods that tune the controller when it is in manual state and the plan operates in open loop (Ishak and Abdullah, 2013). Open loop ZN method is a step response method also known as tangent method. This method provides two most crucial informations used to calculate the optimum PID values namely process dead time and process response rate (Ishak and Hussain, 1999).

The examples of open loop tuning methods include:

a) Open loop Ziegler Nichols (ZN)
b) Chien, Hrones and Reswich (CHR)
c) Cohen Coon
d) Minimum error criteria (IAE, ISE, ITAE)

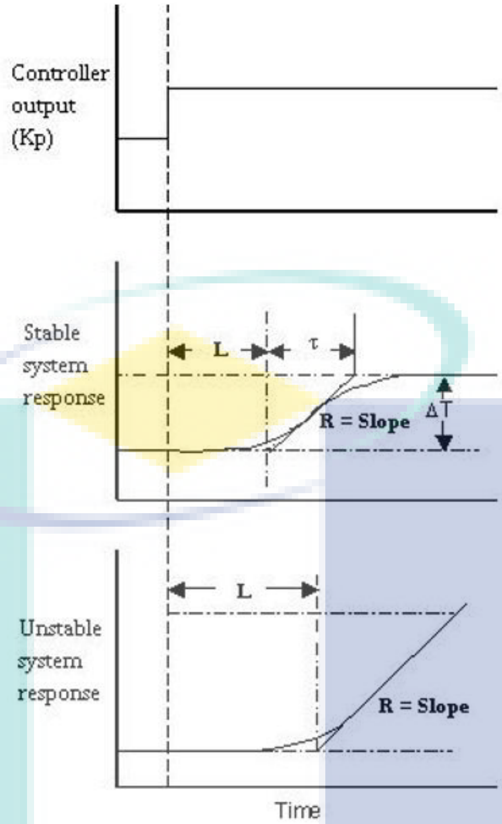



Figure 2.5	Open loop system response

Source: Neil Kuyvenhoven (2002)

Figure 2.5 shows the possible response of an open loop system. The first diagram in the Figure 2.5 shows the controller output response. The second diagram shows the response for self-regulating system. For self-regulating system, the output reaches its steady state at the end of the process. The third diagram shows the response for non-self-regulating system due to the output approaches infinity as time goes by. This system is also known as integrating system (Kuyvenhoven, 2002). The examples of closed loop tuning method include:

a) Closed loop Ziegler Nichols (ZN)
b) Modified Ziegler Nichols
c) Tyrues - Luyben
d) Damped oscillation
e) Internal model control (IMC)

ZN tuning method is a widely used method by researcher and also industry. The capability of the ZN tuning method cannot be denied.  However, there are many advance tuning methods introduced in order to improve the performance of conventional controller since conventional controller such as P, PI and PID are still considered to be the best and simplest controller to be applied to the industry and also in academic study. Nowadays, IMC tuning method is extensively used by researcher to be applied to their application due to its simplicity and performance ability. Hence, two controller tuning methods *i.e* ZN tuning and IMC tuning are chosen and reviewed in details.

#### 2.2.3.1 Ziegler Nichols (ZN) Tuning

Closed loop ZN method was first proposed by Ziegler and Nichols. It is a trial and error tuning method based on sustained oscillation as shown in Figure 2.13. This method is probably the most known and the most widely used method for tuning of PID controllers also known as online or continuous or ultimate gain method (Shahrokhi and Zamorrodi, 2002).

According to the rule, a PID controller is tuned by setting it to P controller only and increasing the value of proportional gain until the system is in a continuous oscillation. The corresponding value of proportional gain is referred to as ultimate gain ($k_u$) and the oscillation period as critical time period ($T_u$) (Priyadarshini and Lather, 2013).

Priyadarshini and Lather (2013) studied different tuning methods to be used with PI and PID controllers. IMC tuning produces better response with no oscillation and less settling time as compared to ZN tuning method. Hence, in the next section the IMC tuning method is reviewed in details.

#### 2.2.3.2 Internal Model Control (IMC) Tuning

The model-based controller design algorithm named IMC was presented by Morari and Zafiriouphriy (1989), which was based upon the internal model principle to combine the process model and external signal dynamics (Priyadarshini and Lather, 2013).

The important research issue for process control engineers is to find a simple design method of a PID type controller with significant performance improvement. Due to the simplicity and improved performance of the internal model control (IMC) based tuning rule, the analytically derived IMC–PID tuning methods have attracted the attention of industrial users over the last decade. The IMC–PID is directly related to the closed loop time constant because it has only one user defined tuning parameter. The IMC–PID tuning and the direct synthesis (DS) approaches are two examples of typical tuning methods based on achieving a desired closed loop response. These methods obtained the PID controller parameters by computing the controller which gives the desired closed loop response (Lee et al, 2008).

IMC is a commonly used technique, which provides a transparent mode for various types of control design and tuning. For the PI controller, IMC technique is used to meet the target of most of the control capacity. Internal Model Control (IMC) design is based on the control system contains the control, a perfect control process can be achieved. Therefore, mathematically perfect control is possible if the control architecture based on an accurate model of the process has been developed (Singh et al. 2014).

#### 2.2.4 Performance Analysis

Normally, after deciding the suitable controller tuning for the process. The performance of the controller should be confirmed by performance analysis. Kesavan et al. (2016) had performed the controller performance analysis for IMC PI controller and schedule PI controller. The performance analysis covers the set point changes analysis and disturbance rejection analysis. A few new set points are selected. For the disturbance rejection analysis, the positive disturbance and negative disturbance are added to the process after the response reaches its settling time. Based on the findings, IMC-PI controller successfully brings the response to its new set point and retains the set point as soon as disturbance is introduced to the system by reducing the settling time of the response.

### 2.3 Modelling and Simulation of Multi-vessel Batch Distillation Column (MVBDC)

In order to analyze the behavior of a chemical process and to study about its control, a mathematical representation of the physical and chemical phenomenon of the process has to be developed. Such a mathematical representation constitutes the model of the system, while the activities leading to the construction of the model will be referred to as modeling.

Simulation is defined as the study of a process or is the imitation of some real thing. Simulation is used in many contexts, including the modelling of natural systems or human systems in order to gain insight into their functioning. Other contexts include simulation of technology for performance optimization, safety engineering, testing, training and education. A great deal of mathematical skill and effort is required to solve even some of the simplest of non-linear equations and such level is usually beyond the reach of the average process engineer (Mahmud, 2008).

An engineering tool for the design and optimisation of steady state and dynamic chemical process is called process simulation. Process simulation offers many benefits such as easier to incorporate actual process data into a simulation model instead of building a pilot plant and its economics (Mahmud, 2008).

### 2.3.1 MATLAB Simulink

MATLAB is a software package which can be used to perform analysis and solve mathematical and engineering problems. It has excellent programming features and graphics capability, very flexible and easy to learn. Simulink is one of the MATLAB tools (Peasly, 2013). MATLAB Simulink provides a nice environment for modelling and simulation of control and embedded systems. Function Blocks are good for designing control application for complex physically distributed systems. An integrated software environment with transformation ability between these two tools will lead to a solution for the validation need for function blocks and also the adoption problems (Yang and Vyatkin, 2009).

Modelling in Simulink is done by creating a network of blocks which are stored in the Simulink library. These blocks in the Simulink library represent common operations for describing and modelling control systems. The blocks include:

1. Mathematical operation (addition, subtraction, multiplication and division)
2. Input sources (Constants, Pulse Generator)
3. Switch, MUX/DEMUX (data manipulation)
4. “Transfer Function” blocks

5. Gain, Saturation, Abs
6. Memory, Integration, Zero-delay
7. S-function (supports external source written in JAVA or C codes)
8. Visualisation blocks (data display etc)
9. State flow (Individual package, but Simulink-based)

Simulink is also capable of storing the output results in a data form, which can be used then for analysis or visualisation inside MATLAB. Sample Simulink models can be seen in the following diagrams (Yang and Vyatkin, 2009). Figure 2.6 is a demo Simulink model provided by Mathworks Inc., representing a distillation column system (Skogestad, 2010).

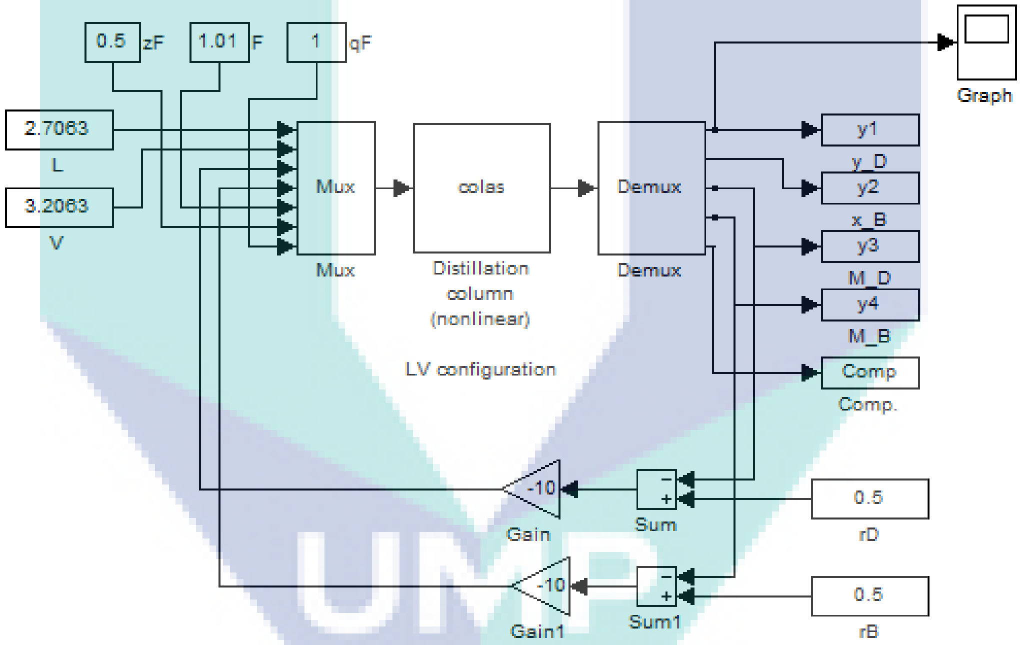



Figure 2.6 Distillation Column Model from Simulink

Source: Skogestad (2017)

MATLAB Simulink is used as a simulator in this research. Simulink Control helps design and analyse plants and control systems model in Simulink and automatically tune controller gains to meet performance requirements. (Peasely, 2013).

MATLAB Simulink has a few unique features including its ability to integrate m-file into its system. Instead of using blocks to represent each differential equation, the equation can be coded in the m-file. MATLAB Simulink can call the m-file function, run the function and lastly obtain the output for the function. It is suitable to solve more than two simultaneous differential equations. Hence, for this study, the model equation is programmed in m-file and the input and output is from Simulink.

## 2.4 Summary of Literature Review

A review of latest research on batch distillation including multi-vessel batch distillation especially, middle-vessel batch distillation and some of continuous distillation is presented in this chapter. Most of researcher are focusing on finding the best control structure for MVBDC in order to give better performance for control system for MVBDC without clearly stated the sensitivity of process variables, the controller tuning procedure and method used and interaction analysis of control loop chosen. All this method and analysis is very important in selection of controlled variable, manipulated variable and disturbance variable and for determining the best control system performance. The list of past research on controllability study various distillation system is stated in Table 2.4. Moreover, it is ensured the performance of the MVBDC system by developing a reliable and good control strategy.

Table 2.2 List of Past Researches on Controllability Study

| No. | Distillation System | Selected Control Structure | Controllability Study | References |
|---|---|---|---|---|
| 1 | Batch Distillation combined with Batch Reactor | Temperature control | Yes (only cover interaction analysis by RGA method) | Skogestad and Sorensen (1992) |
| 2 | Multi Vessel Batch column | Level control | No | Hasebe et al. (1995) |
| 3 | Multi Vessel Batch column | Temperature control | No | Skogestad et al. (1996) |
| 4 | Middle Vessel Batch column | Level control | No | Barolo et al. (1996a) |
| 5 | Middle Vessel Batch Column | Composition control | Yes (only cover interaction analysis by RGA) | Farschman and Diwekar (1998) |
| 6 | Multi-vessel Batch Distillation Column | Temperature control | No sensitivity analysis and interaction analysis. Controller tuning method by try and error | Wittgens (2000) |
| 7 | Cyclic Middle Vessel Batch Column | Level and temperature control | No sensitivity analysis and interaction analysis. Controller tuning method by try and error | Gruetzmann et al (2006) |
| 8 | Multi-vessel Batch Distillation Column | Level and temperature control | No sensitivity analysis and interaction analysis. Controller tuning method by try and error | Alex et al. (2010) |
| 9 | Multi Vessel Batch Distillation column | Level control | No | Fanaei et al. (2012) |
| 10 | Regular Batch Distillation Column | Level and Pressure control | Yes. Sensitivity analysis only. | Nunes (2014)dl |
| 11 | Regular Batch Distillation Column | Temperature control | Yes. Sensitivity analysis only. | Montes et al. (2015) |

# CHAPTER 3

# METHODOLOGY

Modelling, simulation, sensitivity analysis and controllability analysis are presented in this study. The modelling of multi-vessel batch distillation column (MVBDC) is carried out to develop the mathematical model equations including mass balance, component balance, Antoine equation, equity equation, equilibrium equation and bubble point equation for MVBDC system. The simulation of MVBDC is performed using MATLAB Simulink. Closed loop model is simulated by adapting the Proportional (P) only and Proportional Integral (PI) controller. A three-step sensitivity analysis and dynamic analysis are performed. Interaction analysis of control loop is performed in order to select the best control loop pairing. Meanwhile, controllability analysis covers controller tuning and controller performance analysis that is performed in order to obtain the best controller performance. The overall flow of the methodology is shown in Figure 3.1.

This overall methodology is divided into three stages. Stage 1 covers the modelling and degree of freedom analysis. Stage 2 covers the open loop testing including the sensitivity analysis, dynamic analysis and interaction analysis, while stage 3 covers closed loop testing including controller tuning and performance analysis.

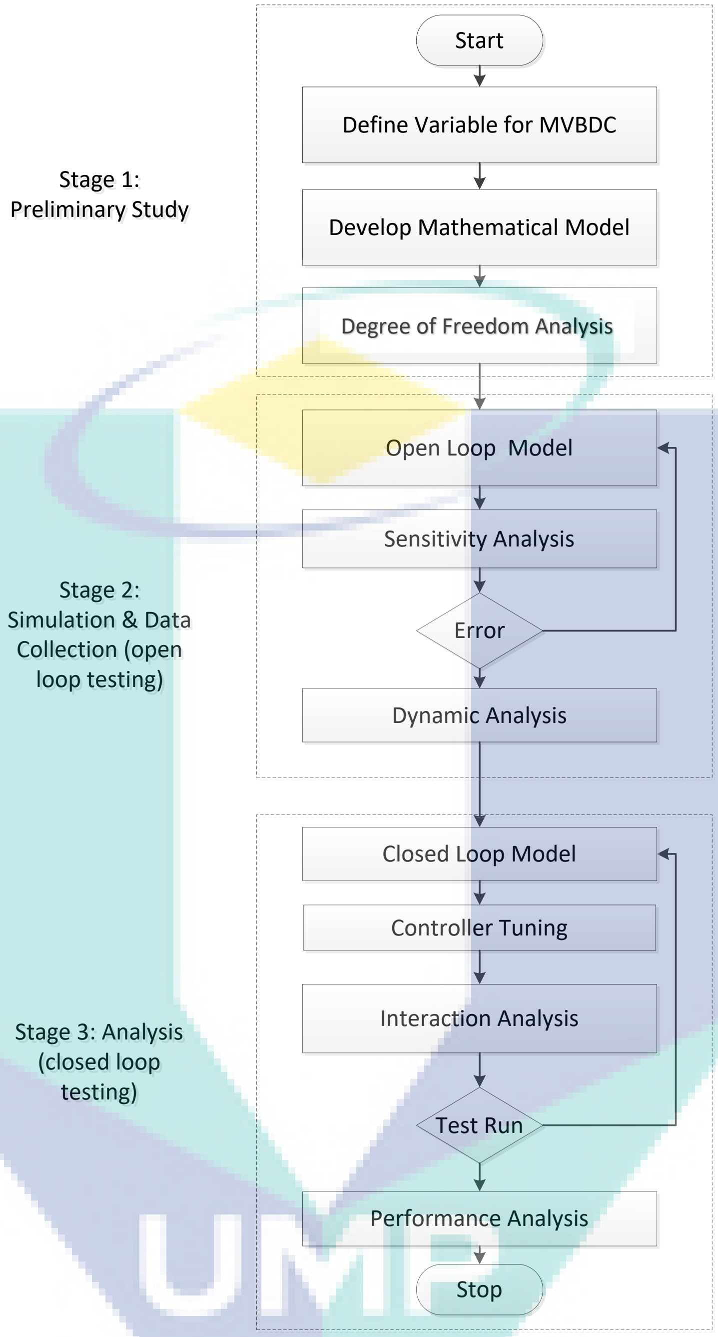



Figure 3.1 Flowchart of Overall Methodology of MVBDC

## 3.1 Design Parameter of Multi-vessel Batch Distillation Column (MVBDC)

The design and operation of multi-vessel batch distillation column is based on the secondary data as calculation basis by Hisyam (2011). The design parameter of this MVBDC is listed in Table 3.1 including column design, holdup volume, column pressure, feed composition and flow rates. Figure 3.2 is a schematic diagram of multi-vessel batch distillation for this study. The MVBDC configuration is based on Hisyam

(2011). This MVBDC consists of condenser, reboiler, top vessel, middle vessel and two columns identical same number of stages.

The top vessel, middle vessel and reboiler are charged with feed mixture consisting of ethanol, 1-propanol and n-butanol as stated in Table 3.3 and Table 3.4. As shown in Figure 3.2, the heat is supplied by reboiler ($V_3$) and the system starts to operate under total reflux operation. Vapor from the reboiler goes up through column 2 and then column 1. At the condenser, vapour ($V_1$) is condensed to the liquid ($L_H$). The liquid flows down through column 1 and then column 2 and lastly to the reboiler. The liquid accumulates in the vessel according to its boiling point. The operation continues under total reflux until the system reaches steady state and product compositions remains constant in all vessels and the highest product purity is obtained. During steady state condition, the top vessel is enriched with lightest components (ethanol) while middle vessel is enriched with second lightest components (1-propanol) and the heaviest components (n-butanol) remains in reboiler. Product withdrawal for all vessel is started after this point. The holdup in each vessel is calculated in advance based on product composition measurement. There are two case studies considered in this study.

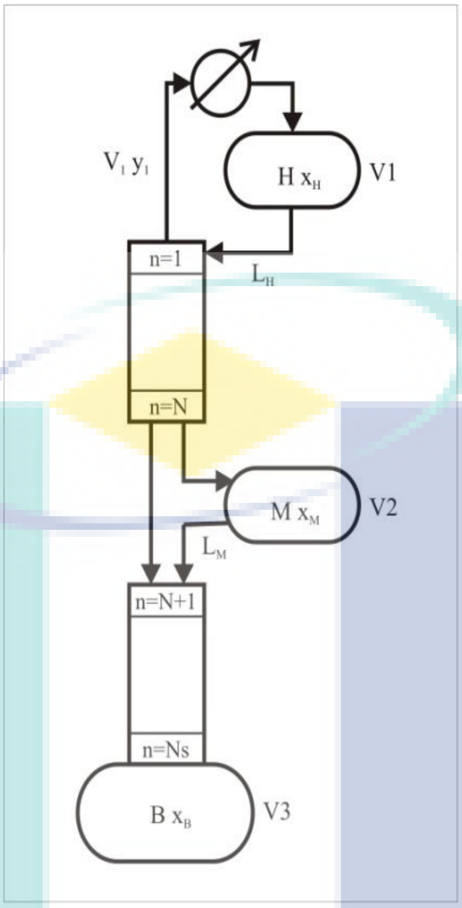



Figure 3.2 Schematic diagram of Multi-vessel Batch Distillation Column.
Source: Hisyam, (2011)

The simulation model of MVBDC is validated by secondary data by Hisyam (2011) and used as the basis of calculation. The number of stages as per stated in Table 3.1 is estimated based on the concept of height equivalent to a theoretical plate (HETP). Each column is calculated separately. The first column is calculated based on the mixture of ethanol and 1-propanol while in the second column based on the mixture of 1-propanol and n-butanol. Vessel holdup is specified first based on the feed composition. This is to ensure the product composition within the specification. Heat input to the system is assumed to be constant during the operation. Thus, the vapour flow is also constant.

Table 3.1 The design parameter of MVBDC

| Parameter | Value | |
|---|---|---|
| Number of stages | 7 (column 1) | |
| | 7 (column 2) | |
| Components | Ethanol, 1-propanol and n-butanol | |
| Feed composition | Case 1 : (0.17,0.415,0.415) | |
| | Case 2 : (0.40,0.20,0.40) | |
| Type of column | Packed column | |
| Length of column, cm | 50 | |
| Column Diameter, cm | 2.5 | |
| Vessel Volume, ml | (400,400,400) | |
| Holdup Volume, ml | Case 1 | Case 2 |
| a) Top vessel | 100 | 400 |
| b) Middle vessel | 400 | 50 |
| c) Bottom (still pot) | 400 | 400 |
| Column Pressure, atm | 1 | |
| Pressure Drop, atm | 0.01 | |
| Vapour Flowrate, mol/s | 0.1 | |
| Liquid Flowrate, mol/s | 0.1 | |
| Heat Input, kJ/s | 4.775 | |

A non-azeotropic ternary mixture is used to represent the multicomponent in this study. The ternary mixture consists of ethanol, 1-propanol and n-butanol. The physical properties of the mixture is shown in Table 3.2. Non-azeotropic behaviour of a mixture means that each component is possible to be separated to obtain pure products. In this case, ethanol, 1-propanol, and n-butanol mixture has a possibility to be processed to obtain pure products. Hence, two cases are considered in this study. The objective of case (1) is to obtain the purest top product (ethanol), while for case (2) the most pure middle product (1-propanol) is considered as the main product.

Table 3.2 Physical properties of ternary mixtures

| Properties | Ethanol | 1-Propanol | N-Butanol |
|---|---|---|---|
| Appearance | Clear | Clear | Clear |
| Formula | $C_2H_5OH$ | $C_3H_8O$ | $C_4H_{10}O$ |
| Molecular weight, g/mole | 46.07 | 60.0950 | 74.1216 |
| Boiling point, K | 351.65 | 370.3 | 390.6 |
| Density, g/m$^3$ | 789 | 804.13 | 809.70 |

Source: Felder et al., (2000)

### 3.1.1 Case (1): Top Product Recovery

For case (1), the vessel holdup and initial composition are stated in Table 3.3. For case (1), the objective is to remove the intermediate and bottom components from the top product in the same time, purer top product is targeted. In this case, the vessel holdup in top vessel is set lower compared to middle vessel and reboiler (stillpot) in order to suite with initial composition feed into the system.

Table 3.3 Vessel Holdup and Initial Composition for Case (1)

| Components | Vessel Holdup, ml | Initial Composition, mole fraction | Holdup Height, cm |
|---|---|---|---|
| Ethanol | 100.0 | 0.1700 | 1.30 |
| 1-propanol | 400.0 | 0.4150 | 5.30 |
| n-butanol | 400.0 | 0.4150 | 5.30 |

### 3.1.2 Case (2): Middle Product Recovery

For case (2), the objective is to remove the top and bottom components from intermediate product. The purer intermediate product is targeted in this case. The vessel holdup and initial composition for case (2) are stated in Table 3.4. The vessel holdup in middle vessel is set lower compared to top vessel and reboiler (stillpot) in order to suite the initial feed composition for 1-propanol which is set lower compared to ethanol and n-butanol. The lower the vessel holdup the higher product purity can be achieved (Hisyam, 2011).

Table 3.4 Vessel Holdup and Initial Composition for Case (2)

| Components | Vessel Holdup, ml | Initial Composition, mole fraction | Holdup Height, cm |
|---|---|---|---|
| Ethanol | 400.0 | 0.4000 | 5.30 |
| 1-propanol | 50.00 | 0.2000 | 0.66 |
| n-butanol | 400.0 | 0.4000 | 5.30 |

## 3.2 Mathematical Model of Multi-vessel Batch Distillation Column (MVBDC)

The mathematical equations are developed based on the following assumptions. (1) Total condenser (condenser is not considered as stage). The entire vapour from stage 1 is totally condensed in the condenser and then recycled back to the column. (2) Constant vapour rate. (3) Molar vapour rate is always equal to the liquid rate. (4)

Constant molar over flow. (5) Total reflux operation. Hence, the entire liquid from the condenser is recycled back to the column. (6) Each stage is in equilibrium condition. There is no accumulation in the stage (Hisyam, 2011). The mathematical model of the separation process in a multi-vessel batch distillation can be presented in Eq. (1)-(16) and the modelling is adapted from Tang et al. (2014) and some modification are introduced to suit with this MVBDC configuration. The equations include mass balance and component balance equations at each point, equilibrium equation, equation of unity, and Antoine equation for predicting the components boiling point.

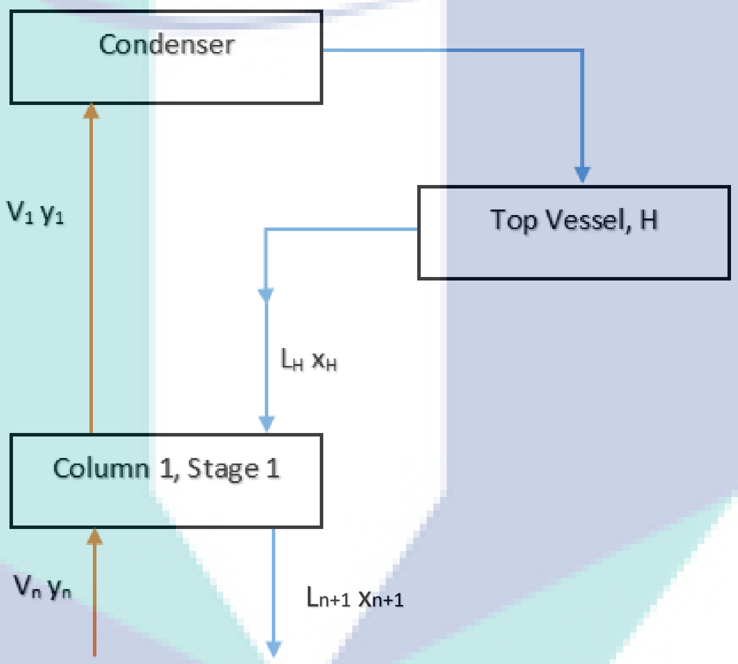



Figure 3.3 Schematic Diagram for Liquid and Vapor Flow For Top Vessel

1. Top Vessel
   a) Overall Mass Balance

$$\frac{dH}{dt} = V_1 - L_H \tag{3.1}$$

   b) Component Balance

$$\frac{d(H.x_H)}{dt} = V_1 y_1 - L_H x_H \tag{3.2}$$

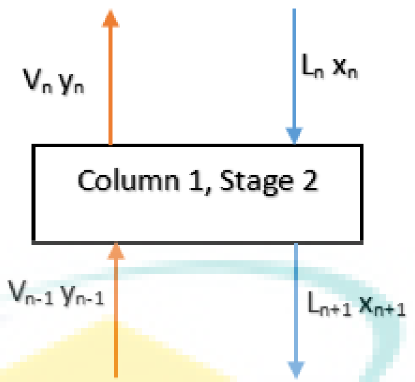



Figure 3.4 Schematic Diagram for Liquid and Vapor Flow For Stage 2

2. Stage Column 1

a) Overall Mass Balance

Stage n=1

$$\frac{dH_1}{dt} = V_n + L_H - V_1 - L_{n+1} \tag{3.3}$$

Stage n=n+1 to 7

$$\frac{dH_n}{dt} = V_{n-1} + L_n - V_n - L_{n+1} \tag{3.4}$$

b) Component Balance

Stage n=1

$$\frac{d(H_1.x_1)}{dt} = V_{n-1}y_{n-1} + L_H x_H - V_1 y_1 - L_{n+1}x_{n+1} \tag{3.5}$$

Stage n=n+1 to 7

$$\frac{d(H_n.x_n)}{dt} = V_{n-1}y_{n-1} + L_n x_n - V_n y_n - L_{n+1}x_{n+1} \tag{3.6}$$

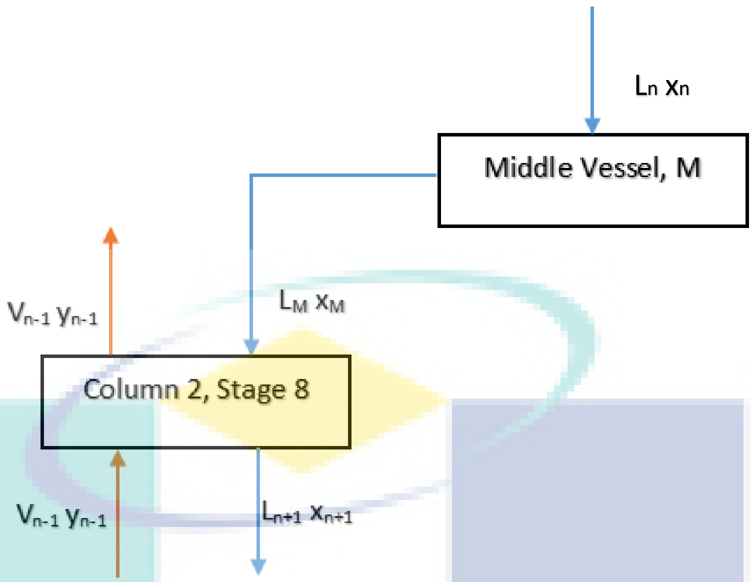



Figure 3.5 Schematic Diagram for Liquid and Vapor Flow For Middle Vessel

3. Middle vessel

a) Overall Mass Balance

$$\frac{dM}{dt} = L_{n1} - L_M \tag{3.7}$$

b) Component Balance

$$\frac{d(M.x_M)}{dt} = L_n x_n - L_M x_M \tag{3.8}$$

4. Stage Column 2

a) Overall Mass Balance

Stage n=8

$$\frac{dH_8}{dt} = V_{n-1} + L_M - V_8 - L_{n+1} \tag{3.9}$$

Stage n=n+1 to 14

$$\frac{dH_n}{dt} = V_{n-1} + L_n - V_n - L_{n+1} \tag{3.10}$$

b) Component Balance

Stage n=8

$$\frac{d(H_8.x_8)}{dt} = V_{n-1}y_{n-1} + L_M x_M - V_8 y_8 - L_{n+1}x_{n+1} \tag{3.11}$$

Stage n=n+1 to 14

$$\frac{d(H_n.x_n)}{dt} = V_{n-1}y_{n-1} + L_n x_n - V_n y_n - L_{n+1}x_{n+1} \qquad 3.12$$

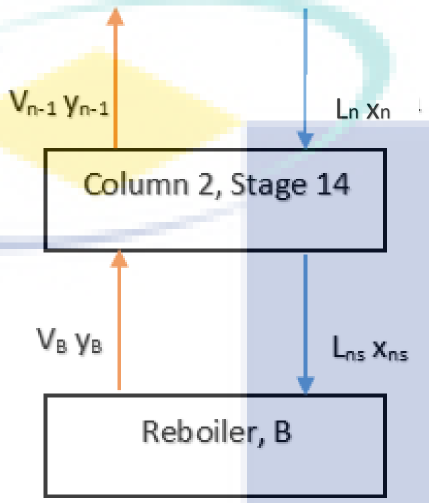



Figure 3.6 Schematic Diagram for Liquid and Vapor Flow For Reboiler

5. Reboiler
   a) Overall Mass Balance

$$\frac{dB}{dt} = L_{ns} - V_B \qquad 3.13$$

   b) Component Balance

$$\frac{d(B.x_B)}{dt} = L_{ns}x_{ns} - V_B y_B \qquad 3.14$$

6. Equilibrium constant

$$y_i = K_i x_i \text{ (Raoult's Law)} \qquad 3.15$$

$$K = \frac{p*}{P} \qquad 3.16$$

7. Equation of unity

$$\sum y_i = 1 \qquad 3.17$$

8. Antoine equation

$$\log p*(bar) = A - \frac{B}{C + T(K)} \qquad 3.18$$

Table 3.5-3.7 show the Antoine coefficient used for the VLE calculation. The Antoine coefficient is selected based on the boiling point of the components. The calculation of the temperature of each stages in this MVBDC is based on the VLE calculation then followed by calculation of composition based on component and mass balance.

Table 3.5 Antoine Coefficient for Ethanol for certain temperature

| **Temperature (K)** | **A** | **B** | **C** |
|---|---|---|---|
| 364.8-513.91 | 4.92531 | 1432.526 | -61.819 |
| 292.77-366.63 | 5.24677 | 1598.673 | -46.424 |
| 273.15-351.70 | 5.37229 | 1670.409 | -40.191 |

Source: National Institute of Standard and Technology (2016)

Table 3.6 Antoine Coefficient for 1-propanol for certain temperature

| **Temperature (K)** | **A** | **B** | **C** |
|---|---|---|---|
| 333.32-377.72 | 4.87602 | 1441.629 | -74.299 |
| 292.40-370.50 | 5.31384 | 1690.864 | -51.804 |
| 405.46-536.71 | 4.59871 | 1300.491 | -86.364 |

Source: National Institute of Standard and Technology (2016)

Table 3.7 Antoine Coefficient for N-butanol for certain temperature

| **Temperature (K)** | **A** | **B** | **C** |
|---|---|---|---|
| 295.8-391.0 | 4.54607 | 1351.555 | -93.34 |
| 391.0-479.0 | 4.39031 | 1254.502 | -105.246 |
| 419.34-562.98 | 4.42921 | 1305.001 | -94.676 |
| 362.36-398.83 | 4.50393 | 1313.878 | -98.789 |

Source: National Institute of Standard and Technology (2016)

### 3.2.1 Degree of Freedom Analysis

In order to proceed with design of control system, the degree of freedom analysis is necessary. All the variables listed are based on the mathematical model from the previous section. The manipulated variables are selected based on the available controlled variable, while disturbance variable is selected based on the most severe situation if the variable starts to fluctuate.

Number of variables =237

($V_1$, $H_1$, $H_2$, $H_3$, $H_4$, $H_5$, $H_6$, $H_7$, $H_8$, $H_9$, $H_{10}$, $H_{11}$, $H_{12}$, $H_{13}$, $H_{14}$, $L_H$, $y_{1a}$, $y_{1b}$, $y_{1c}$, $x_{Ha}$, $x_{Hb}$,

$x_{Hc}$, $H$, $M$, $L_M$, $L_1$, $B$, $x_{1a}$, $x_{1b}$, $x_{1c}$, $y_{Ba}$, $y_{Bb}$, $y_{Bc}$, $x_{Ma}$, $x_{Mb}$, $x_{Mc}$, $L_{ns}$, $V_B$, $x_{nsa}$, $x_{nsb}$, $x_{nsc}$, $V_2$, $V_3$, $V_4$, $V_5$, $V_6$, $V_7$, $V_8$, $V_9$, $V_{10}$, $V_{11}$, $V_{12}$, $V_{13}$, $V_{14}$, $L_2$, $L_3$, $L_4$, $L_5$, $L_6$, $L_7$, $L_8$, $L_9$, $L_{10}$, $L_{11}$, $L_{12}$, $L_{13}$, $L_{14}$, $x_{2a}$, $x_{3a}$, $x_{4a}$, $x_{5a}$, $x_{6a}$, $x_{7a}$, $x_{8a}$, $x_{9a}$, $x_{10a}$, $x_{11a}$, $x_{12a}$, $x_{13a}$, $x_{14a}$, $x_{2b}$, $x_{3b}$, $x_{4b}$, $x_{5b}$, $x_{6b}$, $x_{7b}$, $x_{8b}$, $x_{9b}$, $x_{10b}$, $x_{11b}$, $x_{12b}$, $x_{13b}$, $x_{14b}$, $x_{2c}$, $x_{3c}$, $x_{4c}$, $x_{5c}$, $x_{6c}$, $x_{7c}$, $x_{8c}$, $x_{9c}$, $x_{10c}$, $x_{11c}$, $x_{12c}$, $x_{13c}$, $x_{14c}$, $y_{2a}$, $y_{3a}$, $y_{4a}$, $y_{5a}$, $y_{6a}$, $y_{7a}$, $y_{8a}$, $y_{9a}$, $y_{10a}$, $y_{11a}$, $y_{12a}$, $y_{13a}$, $y_{14a}$, $y_{2b}$, $y_{3b}$, $y_{4b}$, $y_{5b}$, $y_{6b}$, $y_{7b}$, $y_{8b}$, $y_{9b}$, $y_{10b}$, $y_{11b}$, $y_{12b}$, $y_{13b}$, $y_{14b}$, $y_{2c}$, $y_{3c}$, $y_{4c}$, $y_{5c}$, $y_{6c}$, $y_{7c}$, $y_{8c}$, $y_{9c}$, $y_{10c}$, $y_{11c}$, $y_{12c}$, $y_{13c}$, $y_{14c}$, $K_{1a}$, $K_{1b}$, $K_{1c}$, $K_{2a}$, $K_{3a}$, $K_{4a}$, $K_{5a}$, $K_{6a}$, $K_{7a}$, $K_{8a}$, $K_{9a}$, $K_{10a}$, $K_{11a}$, $K_{12a}$, $K_{13a}$, $K_{14a}$, $K_{2b}$, $K_{3b}$, $K_{4b}$, $K_{5b}$, $K_{6b}$, $K_{7b}$, $K_{8b}$, $K_{9b}$, $K_{10b}$, $K_{11b}$, $K_{12b}$, $K_{13b}$, $K_{14b}$, $K_{2c}$, $K_{3c}$, $K_{4c}$, $K_{5c}$, $K_{6c}$, $K_{7c}$, $K_{8c}$, $K_{9c}$, $K_{10c}$, $K_{11c}$, $K_{12c}$, $K_{13c}$, $K_{14c}$, $K_{Ba}$, $K_{Bb}$, $K_{Bc}$, $P_{1a}$, $P_{1b}$, $P_{1c}$, $P_{2a}$, $P_{3a}$, $P_{4a}$, $P_{5a}$, $P_{6a}$, $P_{7a}$, $P_{8a}$, $P_{9a}$, $P_{10a}$, $P_{11a}$, $P_{12a}$, $P_{13a}$, $P_{14xa}$, $P_{2b}$, $P_{3b}$, $P_{4b}$, $P_{5b}$, $P_{6b}$, $P_{7b}$, $P_{8b}$, $P_{9b}$, $P_{10b}$, $P_{11b}$, $P_{12b}$, $P_{13b}$, $P_{14b}$, $P_{2c}$, $P_{3c}$, $P_{4c}$, $P_{5c}$, $P_{6c}$, $P_{7c}$, $P_{8c}$, $P_{9c}$, $P_{10c}$, $P_{11c}$, $P_{12c}$, $P_{13c}$, $P_{14c}$, $P_{Ba}$, $P_{Bb}$, $P_{1c}$, $T_{guess}$)

Number of equations =203

4 equation at top vessel

4 equation at middle vessel

4 equation at reboiler

21 equation at column 1

21 equation at column 2

14 equation for both column

45 equilibrium constant equation

| | |
|---|---|
| | 45 Raoult's law equation |
| | 45 Antoine equation |
| Number of controlled variable | =3 (from equation mass balance in top vessel, middle vessel and reboiler) |
| Number of specified variable | =28 |
| | ($V_{n-1}$, $V_n$, $L_{n+1}$, $V_1$ , $T_{guess}$) |
| Number of available disturbance and manipulated variables | =3 ($L_M$ ,$L_H$ and $L_{ns}$) |
| Degree of Freedom | =0 |

In the analysis, notation 'a' is for ethanol, 'b' is for 1-propanol and 'c' is for n-butanol. Based on the calculation, three variables are available, *i.e.* middle flow ($L_m$) bottom flow ($L_{ns}$) and reflux flow ($L_H$) and three controlled variable level holdup in top vessel, middle vessel and reboiler. However, these variables will undergo sensitivity analysis and interaction analysis in order to select the manipulated ad disturbance variable

The main goal of this research is to study the controllability of the MVBDC using level control. This controllability study is also used to confirm the decision of control design chosen for this system. As mentioned in DOF analysis, two or three level control can be chosen based on the degree of freedom analysis. The objective of this level control is to ensure that the holdup in each vessel is kept constant. This is because the MVBDC system is operated under total reflux mode. Hasebe et al. (1995) mentioned that in order to maintain the total reflux operation, the vessel holdup must be kept constant. Total reflux operation provides many advantages such as the maximum fractionating capacity, easier operation control and fewer disturbances to product quality and yield (Bai et al., 2014; Tang et al., 2014). Purer overhead product can be obtained under this operation mode. Hence, this level control not only maintains the vessel holdup, it can also indirectly control and maintain the desired product quality and specification.

## 3.3 Open Loop Testing

As mentioned at the beginning of this chapter, the open loop testing covers sensitivity analysis, dynamic analysis and interaction analysis. At this stage the best pairing of control structure will be determined. It starts with evaluating the process variable until analysis of interaction of each available controlled variable and manipulated variable. The open loop model of MVBDC is shown in Figure 3.7.

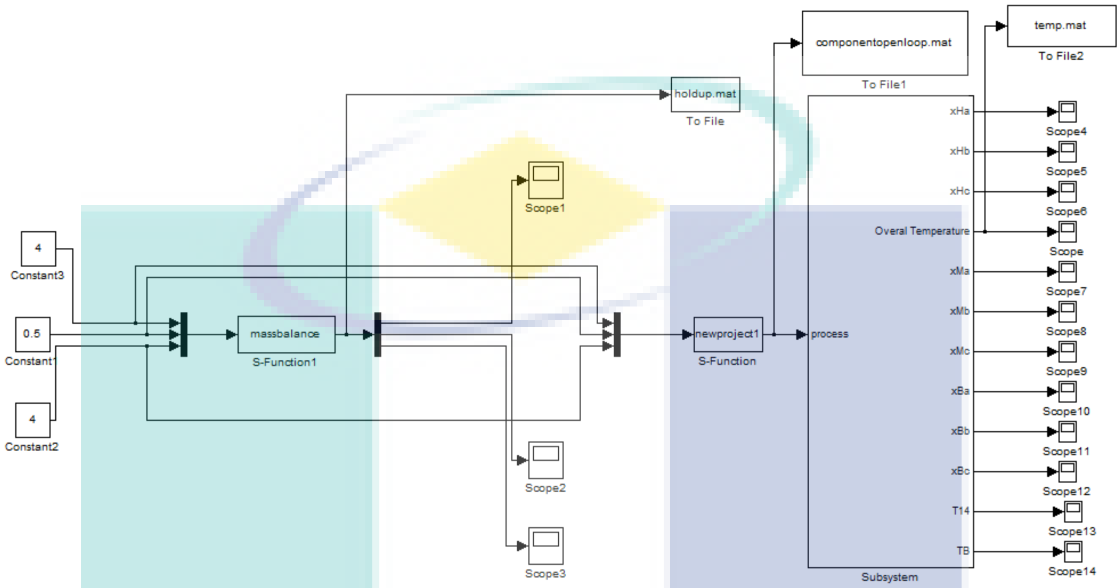



Figure 3.7 Open loop model of MVBDC

MVBDC is simulated using MATLAB Simulink. This model is divided into two main s-function blocks. First block is mass balance block. Second block is component balance block named newproject1. This simulink model is integrated with m-file from MATLAB. The mathematical equation is coded in m-file as attached in Appendix A and then run in Simulink. The input of m-file is from Simulink block while output of m-file is displayed in Simulink plot. The input of mass balance block is flow of liquid in each stream and the output is the level holdup for each vessel. Meanwhile, the input of the component block is holdup for each vessel. VLE calculation is also included in the component balance s-function block. The composition of each stage is determined based on the guessed temperature for each stage. The guessed temperature is based on boiling point of product. The block diagram in Simulink is the graphical approaches for visualizing the system s. The m-file is used to code the mathematical equation because it is more user friendly to solve the large number of simultaneous equation (Nehra, 2014). This model will be validated using secondary data (Hisyam, 2011).

### 3.3.1 Sensitivity Analysis

The purpose of sensitivity analysis is to select candidate control scheme as well as to determine the disturbance variable and indirect control variable (Fruehauf et al., 1993). A three steps of sensitivity analysis are performed. First step is plotting the level of vessel holdup (input) against composition profile (output). The objective is to observe the effect of level of vessel holdup toward product composition. At the same time, the set point value can be estimated and the indirect control variable can be confirmed. Second step is plotting the available controlled variables (output) against manipulated variables (input) in order to observe the effect of flow of liquid stream entering the vessel toward the level of vessel holdup and to select the suitable control structure.

Lastly, plotting available disturbance variables against controlled variables. The objective is to see effect of candidate disturbance variables toward the level of each vessel holdup and choose the suitable disturbance variable (Roat et al., 1988). Due to assumption made in mathematical model, the energy balance is not considered. Hence, the heat input to the system is constant. Based on the DOF analysis, available manipulated and disturbance variables are reflux flow, middle and bottom flow. The disturbances and manipulated variables is then be selected in this sensitivity analysis. The illustration of sensitivity analysis block diagram is shown in Figure 3.8. For each step the input is varied from +20% to -20%.

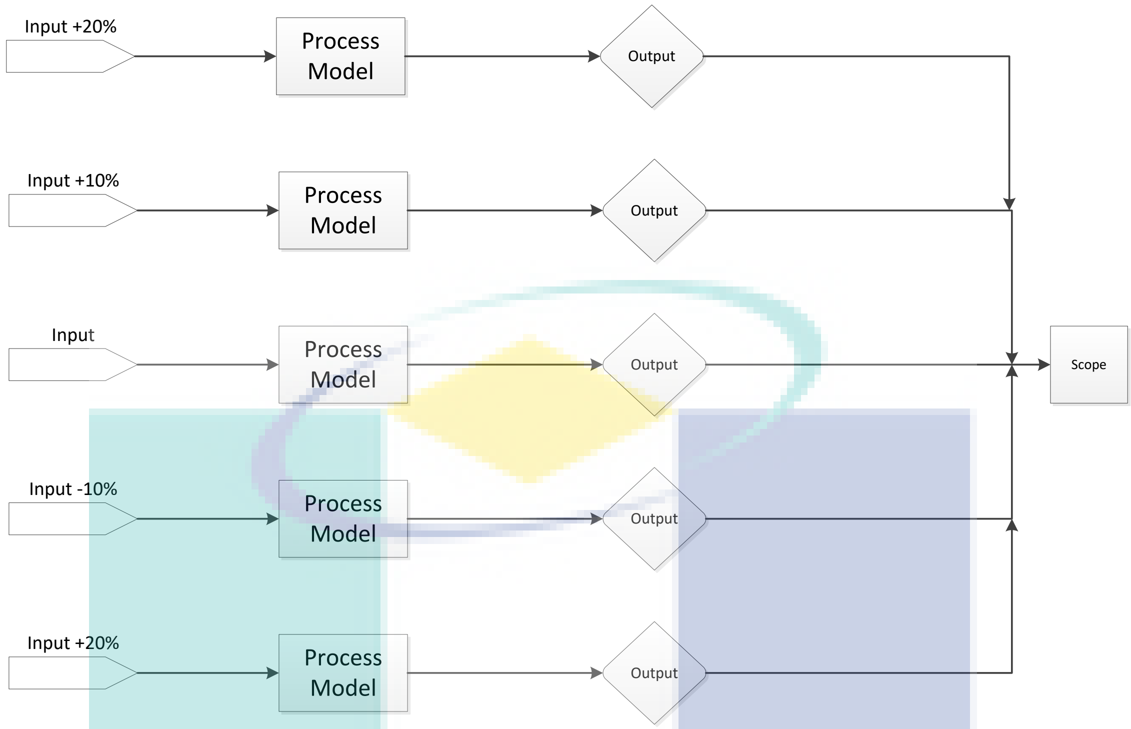



Figure 3.8 Illustration Block diagram for Sensitivity Analysis

### 3.3.2 Interaction Analysis

The suitable control structure obtained from sensitivity analysis is then analyse by interaction analysis in order to choose the best pairing of control loops. For the interaction analysis, two method are applied *i.e* relative gain array (RGA) and singular value decomposition (SVD). RGA is used to determine the available pairing of control loops. Meanwhile, SVD is used to determine and choose the best pair of manipulated and controlled variable with the least condition number (CN). The RGA can be calculated based on the following equation (Sankaranarayanan and Deepakkumar, 2015):

$$\lambda_{11} = \frac{\left.{}^{\delta_{yi}}\!/_{\delta_{uj}}\right|_{u}}{\left.{}^{\delta_{yi}}\!/_{\delta_{uj}}\right|_{y}} \tag{3.19}$$

$$RGA = \Lambda = \begin{bmatrix} \lambda_{11} & \lambda_{12} & \lambda_{13} \\ \lambda_{21} & \lambda_{22} & \lambda_{23} \\ \lambda_{31} & \lambda_{32} & \lambda_{33} \end{bmatrix} \tag{3.20}$$

The SVD procedure is listed as follow:

a. Determine the singular matrix,

$$A = USV^T \tag{3.21}$$

$$S = \begin{bmatrix} \sigma_i & 0 & 0 \\ 0 & \sigma_i & 0 \\ 0 & 0 & \sigma_i \end{bmatrix} \tag{3.22}$$

Where the matrix U and matrix V are unitary and matrix S contains a diagonal singular values, $\sigma_i$ arranged in a descending order. If one eigenvalue is very small compared to others then very large change in one or more manipulated variable is required to control the process.

b. Determine the condition number (CN),

$$CN = \frac{\sigma_{\text{large}}}{\sigma_{\text{small}}} \tag{3.23}$$

The condition number is used as an input-output controllability measure and in particular it has been postulated that a large condition number indicates sensitivity to uncertainty. $\sigma_{large}$ refers to the highest $\sigma_i$ in the diagonal matrix, S. Meanwhile, $\sigma_{small}$ refers to the smallest $\sigma_i$ in diagonal matrix, S.

### 3.3.3 Dynamic Analysis

Dynamic analysis is performed on the manipulated variable (input) and controlled variable (output). The input (manipulated variable) is increased and decreased by +10 % to -10 %. The illustration of dynamic analysis is shown in Figure 3.9. The response is then referred to initial set value and the response out from the last block is analysed. The aim of this analysis is to determine whether process is linear or nonlinear. The linearity and nonlinearity of process is determined by analysing the trends of the response produced by level in vessel holdup for varied step input (Melnik et al. 2005). If the system is linear, conventional controller such as Proportional (P), Proportional Integral (PI) and Proportional Integral Derivative (PID) can be directly used. However in this study PID controller is not considered since only level control is used. P and PI controller are sufficient enough to control the level in vessel holdup.

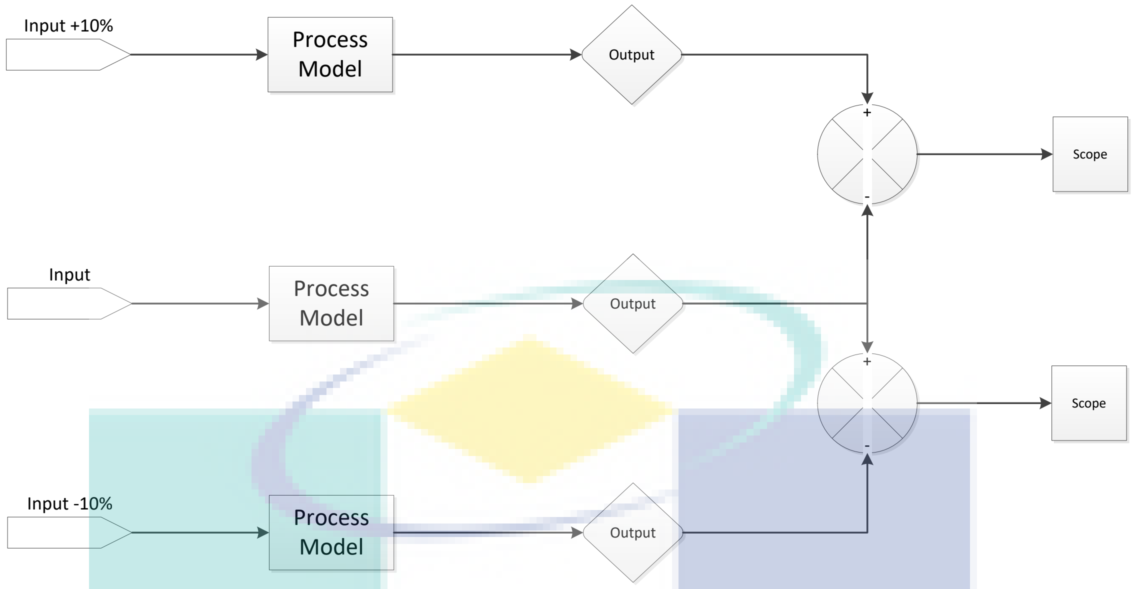



Figure 3.9 Illustration Block Diagram for Dynamic Analysis

## 3.4 Closed Loop Testing

Closed loop testing covers the controller tuning based on ZN closed loop tuning method and IMC tuning method for case (1) and case (2) and controller performance analysis based on set point changes analysis and disturbance analysis. Figure 3.10 shows the closedd loop model for MVBDC.

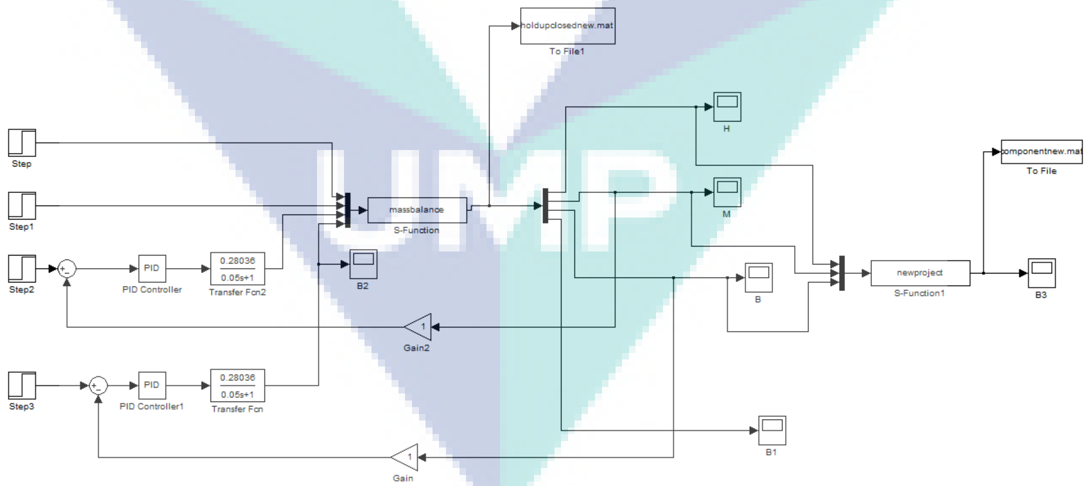



Figure 3.10 Closed loop model of MVBDC

Two controllers will be implemented into the closed loop system using general PID block. The mode of controllers can be changed by changing the parameter in PID block. The valve equation block is placed in series with PID block. The valve equation is stated in Appendix C. The input of mass balance is vessel holdup. The output from the mass balance block become the input of the component balance block (newproject). This shows the correlation between two blocks. The output of the component block are compositions of each stage and also temperatures of each stage.

### 3.4.1 Controller Tuning

Tuning method based on Ziegler Nichols (ZN) and Internal model control (IMC) tuning is performed and compared in this study. Tuning setting that gives the best controller performance and ensures the stability of system will be chosen. The response of the system must have no overshoot, lower settling time and rise time.

For the ZN closed loop method, the proportional value at which the oscillations become constant is defined as ultimate gain, Ku. The ultimate period, Tu, is the period of oscillations for ultimate gain. The ultimate gain and ultimate period is implemented to the ZN closedd loop formulae shown in Table 3.6.

Table 3.8 Tuning parameter of Ziegler Nichols closedd loop method

| | $K_P$ | $T_I$ | $T_D$ |
|---|---|---|---|
| P | 0.5Ku | - | - |
| PI | 0.45Ku | Tu/1.2 | - |
| PID | 0.6Ku | Tu/2 | Tu/8 |

Source: Shahrokhi et al. (2013)

For IMC tuning, the closedd loop time constant, $\tau_c$ is set based on Rivera et al. (1986) approximation ($\tau_c > \tau$). This closedd loop time constant is applied to the controller setting for IMC-based PID for integrating process as shown in Table 3.7.

Table 3.9 IMC-Based PID Controller Setting

| Model | $K_cK$ | $T_I$ | $T_D$ |
|---|---|---|---|
| K/s | $2/\tau_c$ | $2\tau_c$ | - |

Source: Chien and Fruehauf (1990)

### 3.4.2 Performance Analysis

The performance analysis covers the set point changes test and disturbance rejection test in order to study the performance and stability of controllers. A good controller can produce stable and smooth response in whatever situation even though the process condition is changing, the presence of unwanted disturbance and the set point is changing.

For the set point change analysis, the set point for both level holdup is changed from initial value stated in Table 3.3 and 3.4 to (1.56:6.36) for case (1) and (6.36:0.79) for case (2). The level in vessel holdup is expected to achieve the new set point successfully. Meanwhile, the disturbance for this system chosen in sensitivity and interaction analysis is introduced to the system after the operation reaches steady state. The disturbance value is been manipulated during the process to observe the response of the controller. The level in vessel holdup is expected to produce stable response and the set point should be achieved.

### 3.5 Simulation of Multi-vessel Batch Distillation Column (MVBDC)

MATLAB Simulink is used as a simulator in this study. MATLAB Simulink has a few unique features including its ability to integrate m-file into its system. Instead of using blocks to represent each differential equation, the equation can be coded in the m-file. MATLAB Simulink can call the m-file function, run the function and lastly obtain the output for the function. It is suitable to solve more than two simultaneous differential equations. Hence, for this study, the model equation is programmed in m-file and the input and output is from Simulink.

### 3.6 Summary of Methodology

Basically, in the Chapter 3, the overall methodology is divided into three stages. In the first stage the modelling of MVBDC is determined and degree of freedom analysis is performed. As a result the available variables for manipulated variable and controlled variable are determined. The available variables will be further analysed in the next chapter in order to choose the best pairing of control loops. The second stage of methodology is open loop testing. The open loop testing procedure and method including sensitivity, interaction and dynamic analysis are explained briefly in Chapter 3 and will be performed in the next chapter. Lastly, the third stage covers closed loop testing including the controller tuning and controller performance analysis. The method and procedure for this closed loop testing is explained extensively.

# CHAPTER 4

# RESULTS AND DISCUSSIONS

In this chapter, the simulation and control of multi-vessel batch distillation is presented. This chapter consists of two parts. First part in sub chapter 4.1 covers the simulation of MVBDC. The tool used for the simulation is MATLAB. Second part in sub chapter 4.2 covers the controllability study of MVBDC.

## 4.1 Introduction

The simulation for this MVBDC is based on the mathematical modelling including mass balance, component balance, Antoine equation, equilibrium equation and equation of unity. In industry, modelling is used to reduce the number of experiments, which require expensive manpower and utilization costs (Peasley, 2013). The mathematical model is shown in section 3.2.

The simulation model with four inputs *i.e.* reflux flow, middle flow, bottom flow and vapour flow is shown in Figure 3.11. From the control point of view, based on the degree of freedom (DOF) analysis, there are three controlled objectives available *i.e.* to control the level of holdup in top vessel, level of holdup in middle vessel and level of holdup in reboiler. The level control loops will be selected in the sensitivity and interaction analysis in this chapter. The controller tuning based on ZN tuning and IMC tuning method is also covered in this chapter. Tuning method that produced smooth and stable response will be chosen.

Two cases are presented. The detail explanation of both cases are mentioned in section 3.11 and 3.1.2.

### 4.1.1 Model Validation

Model validation is important to prove the reliability and credibility of the simulation model. As mentioned previously, the model validation is performed using secondary data by Hisyam (2011) as shown in Table 3.1 and then the data is used in this study. The configuration of MVBDC applied in this study is shown in Figure 3.1 and the simulation is performed in MATLAB Simulink. The separation behaviour of non-azeotropic mixture consisting of ethanol, 1-propanol and n-butanol was highlighted in his study.

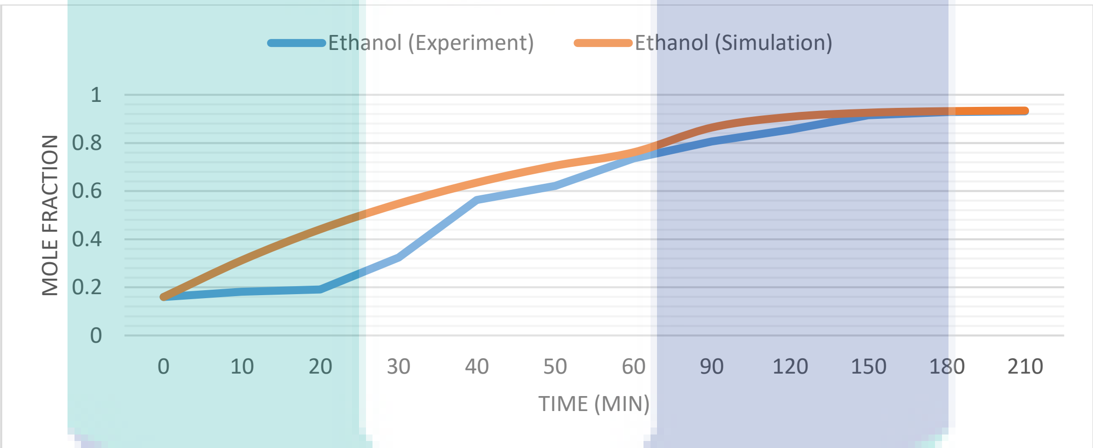



Figure 4.1 Composition Profile of Ethanol (Validation)
Source: Hisyam (2011)

Figure 4.1 shows a composition profile of ethanol from experimental work secondary data and simulation data. When comparing simulation results with experimental results, there is disagreement at the beginning. This might be caused by start-up step that possibly has different model (Warter et al., 2004). There are some assumptions or parameters that were not included in the modelling to represent the startup model since the startup model was not studied. The modelling is simulated based on model during steady state operation. Hisyam (2011) only shows the experimental data for ethanol composition in his work. He mentioned that the other data is calculated by mass and component balance and simulated in Matlab and Aspen. The composition of ethanol obtained by Hisyam and the simulation are stated in the Table 4.1. The large error (40%) at the beginning occured because the startup modelling is not considered. This is because the modelling of startup operation is considerably complex and is not investigated in this study. After the operation reaches steady state, the error value gets smaller approximately to 0.4% at 180 minutes. The error is low enough. Hence, the model developed in Simulink is validated and can be used further in this study.

Table 4.1 Composition of Components in Top Vessel for Validation

| Time (min) | Secondary Data | Simulation Data |
|---|---|---|
| 0 | 0.160 | 0.160 |
| 10 | 0.182 | 0.312 |
| 20 | 0.191 | 0.441 |
| 30 | 0.324 | 0.548 |
| 40 | 0.563 | 0.635 |
| 50 | 0.621 | 0.705 |
| 60 | 0.735 | 0.761 |
| 90 | 0.806 | 0.864 |
| 120 | 0.855 | 0.908 |
| 150 | 0.915 | 0.926 |
| 180 | 0.928 | 0.932 |
| 210 | 0.931 | 0.934 |

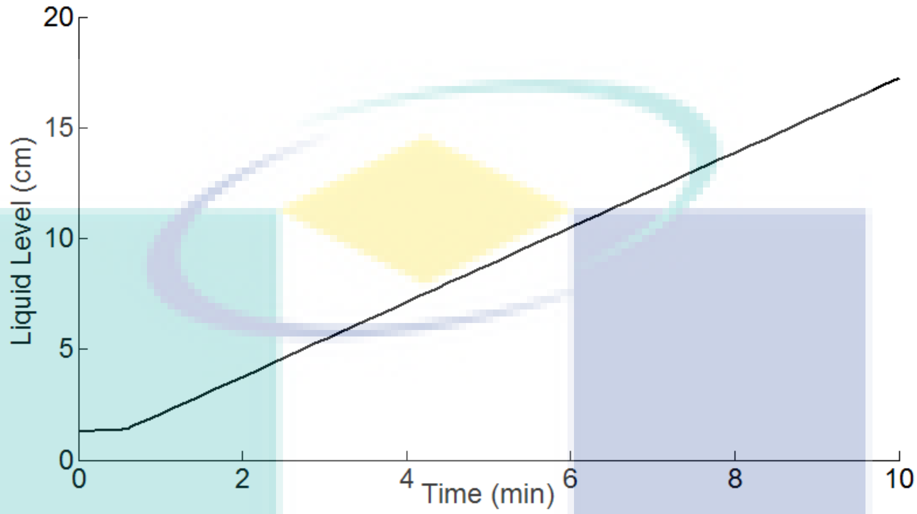
## 4.2 Controllability of Multi-vessel Batch Distillation Column (MVBDC)

In this research, controllability study is divided into two parts, *i.e.* open loop testing and closed loop testing. Open loop testing covers sensitivity analysis and dynamic analysis. Meanwhile, closed loop testing covers closed loop controller tuning, interaction analysis and performance analysis. Basically, the first part covers the selection of control structure and selection of controllers. The second part covers the performance of the control system.

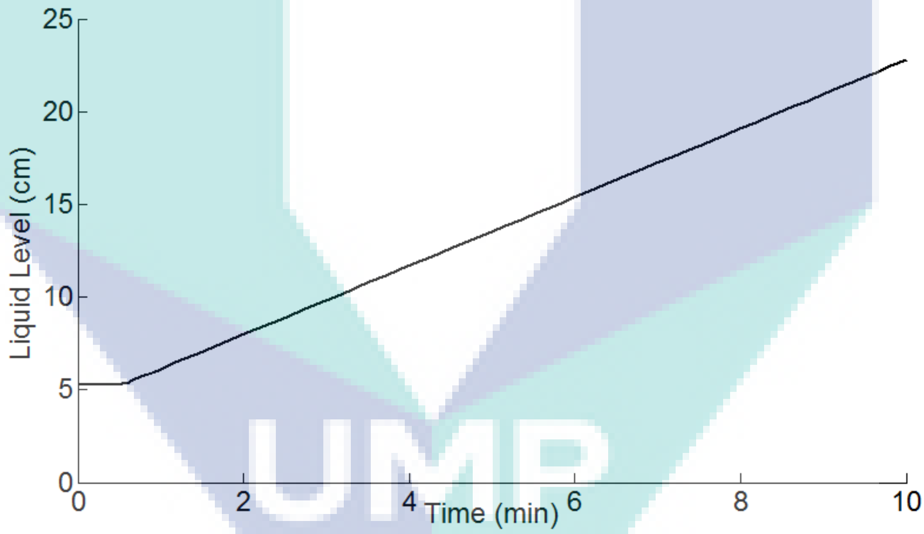
### 4.2.1 Open Loop Testing

The purpose of first part in open loop testing is to perform the input step to the control loop in order to determine the process categories.

Figure 4.2 shows the response of liquid level of open loop model for case (1). The step time is 0.5 minutes for both liquid level in case (1). Based on Figure 4.2, the vessels holdup responses keep increasing until the process reaches its limit. There are no steady state achieved for both top and middle liquid levels because the process is non-self-regulating process that also called as integrating process.



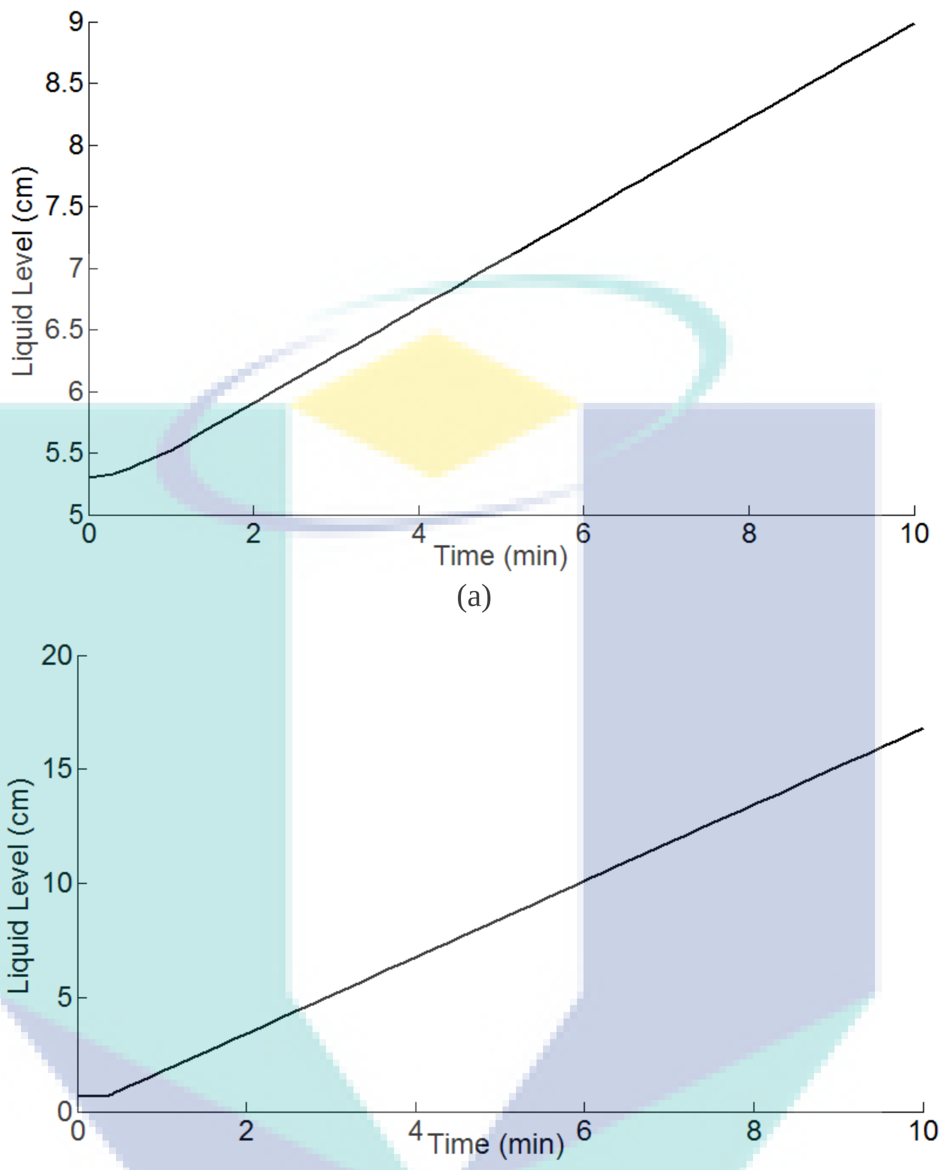

(a)




(b)

Figure 4.2 Open Loop Liquid Level Profile for Case (1) (a) Top Vessel (b) Middle Vessel

Meanwhile, Figure 4.3 shows the response of the liquid level of open loop model for case (2).For case (2) the step size is 0.3 minutes. The response for both liquid level increases over time. Hence, for both cases the open loop process can be categorized as non-self-regulating process (Kuyvenhoven, 2002). The difference between case (1) and case (2) is that the liquid level in middle vessel for case (2) is lower compared to case (1) as explained in chapter 4.1.



(a)


(b)

Figure 4.3 Open Loop Liquid level Profile for Case (2) (a) Top Vessel (b) Middle Vessel

#### 4.2.1.1 Sensitivity Analysis

As mentioned in Chapter 3, a three-step of sensitivity analysis is performed. First step is to analyse the effect of liquid level on the product composition (Fruehauf et al., 1993) (Barton, 1997) (Roat et al, 1988). The liquid level is manipulated in order to monitor the changes of product composition. The set point of the liquid level can be determined based on this step (Roat et al, 1988). The optimal liquid level is specified in advanced based on composition of product (Hasebe et al, 1997) (Hasebe et al, 1999). The liquid level is increased by +20% and -20% from initial value. Three sets of liquid level which are +20%, -20% and initial value (0%) are plotted in one graph in order to see the sensitivity of product composition toward liquid level. Case (1) and case (2) are considered for all three steps of sensitivity analysis. Figure 4.4 shows the composition profile of ethanol in top vessel for case (1).

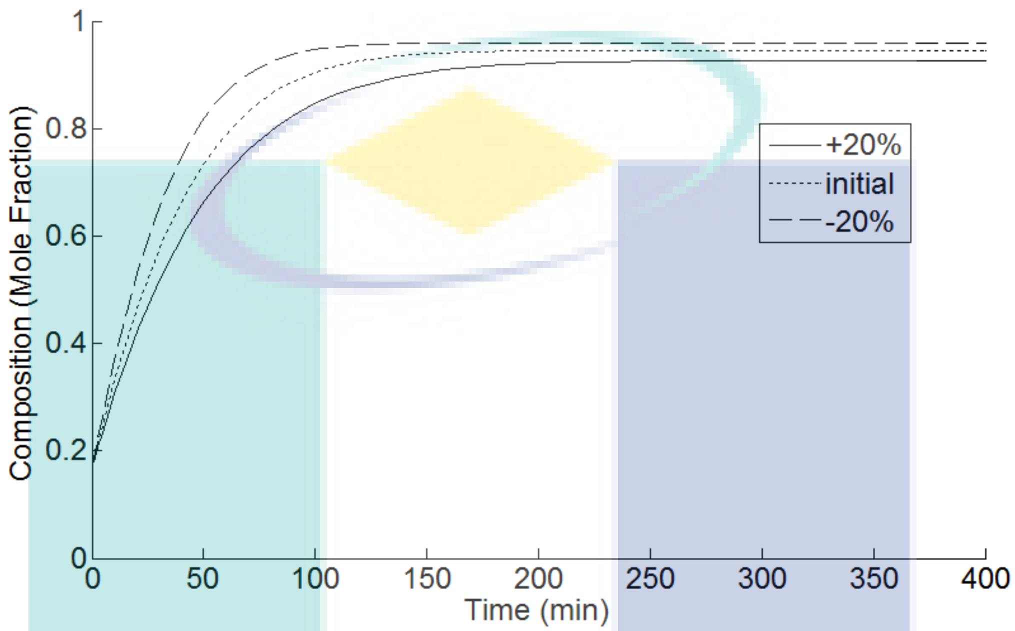



Figure 4.4 Sensitivity Analysis of Ethanol Composition toward Variable Liquid Level for Case (1) (Top Vessel)

For case (1), the liquid level in top vessel is increased +20% and -20%, while holdup in middle vessel and reboiler remain constant in order to analyse the effect of top product composition toward the changes of liquid level in top vessel. The liquid level in top vessel is set to 1.56 cm and 1.04 cm for +20% and -20% respectively. Based on Table 4.2, the product composition of ethanol with liquid level (+20%) is 0.926 and the product composition of ethanol with liquid level (-20%) is 0.958. While the product composition of ethanol with liquid level (initial or 0%) is 0.945. If the holdup is decreased, the purity of product is increased. This is because the volume of mixture is low, more product can be separated. The product composition is the indirect controlled variable in this study since the product composition is sensitive to the holdup changes.

Table 4.2 Composition of Components in Top Vessel for Case (1) (Sensitivity Analysis)

| | **+20%** | **Initial Value** | **-20%** |
|---|---|---|---|
| Ethanol | 0.926000 | 0.945000 | 0.958000 |
| 1-propanol | 0.073400 | 0.054900 | 0.042300 |
| n-butanol | 0.000009 | 0.000006 | 0.000004 |

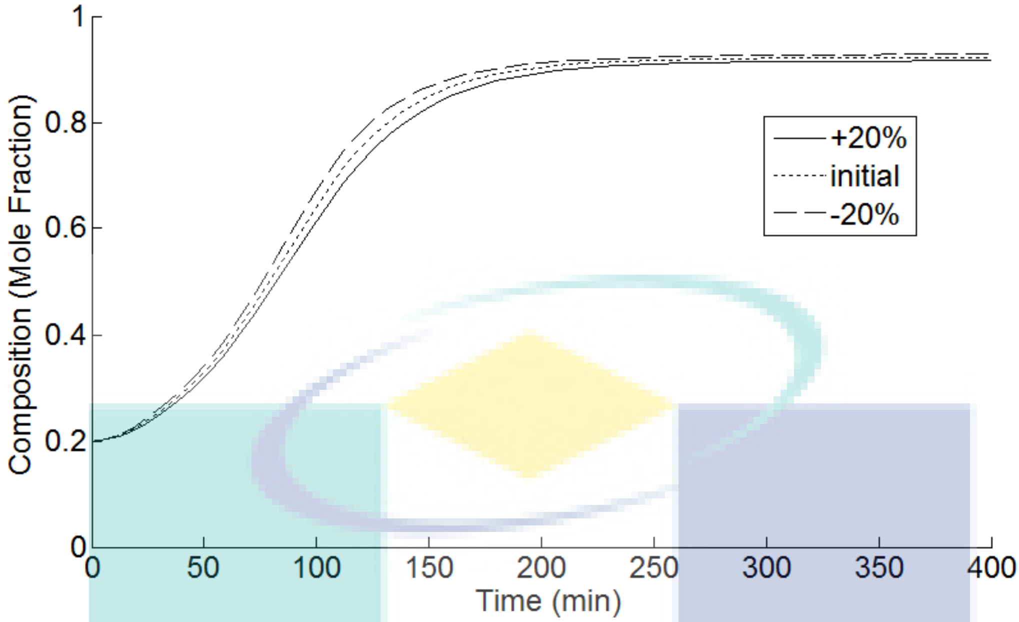



Figure 4.5 Sensitivity Analysis of 1-propanol Composition toward Variable Liquid Level for Case (2) (Middle Vessel)

In order to analyse the effect of middle product composition toward the changes of liquid level in middle vessel, for case (2), the liquid level in middle vessel is increased by 20% and -20%, while liquid level in top vessel and reboiler remain constant. Based on Figure 4.6, 0.9254, 0.929 and 0.9333 of 1-propanol is obtained for liquid level +20%, 0% and -20% respectively. The lower the liquid level the higher the product purity (Hisyam, 2011). However, in industry, it is hard to set liquid level to the lowest value due to equipment constraint and measurement constraint. Hence, the optimum liquid level must be chosen. In this study, for case (1) in order to produce 0.94 ethanol the level in liquid level in top vessel is set to 1.3 cm and level in reboiler holdup is set to 5.3 cm and liquid level in reboiler is set to 5.3 cm. For case (2) in order to produce 0.92 1-propanol the liquid level in top vessel is set to 5.3 cm, the liquid level in middle vessel is set 0.66 cm and liquid level in reboiler is set to 5.3 cm.

Table 4.3 Composition of Components in Middle Vessel for Case (2) (Sensitivity Analysis)

| | **+20%** | **Initial Value** | **-20%** |
|---|---|---|---|
| Ethanol | 0.0598 | 0.0568 | 0.0538 |
| 1-propanol | 0.9254 | 0.9294 | 0.9333 |
| n-butanol | 0.0148 | 0.0138 | 0.0128 |

The second step involves the sensitivity analysis of liquid level towards reflux flow and bottom flow. Reflux and bottom flow is manipulated +20%, +10%, 0%, -10% and -20% in order to analyse the effect of liquid level toward changes of reflux flow (top vessel) and bottom flow (middle vessel).

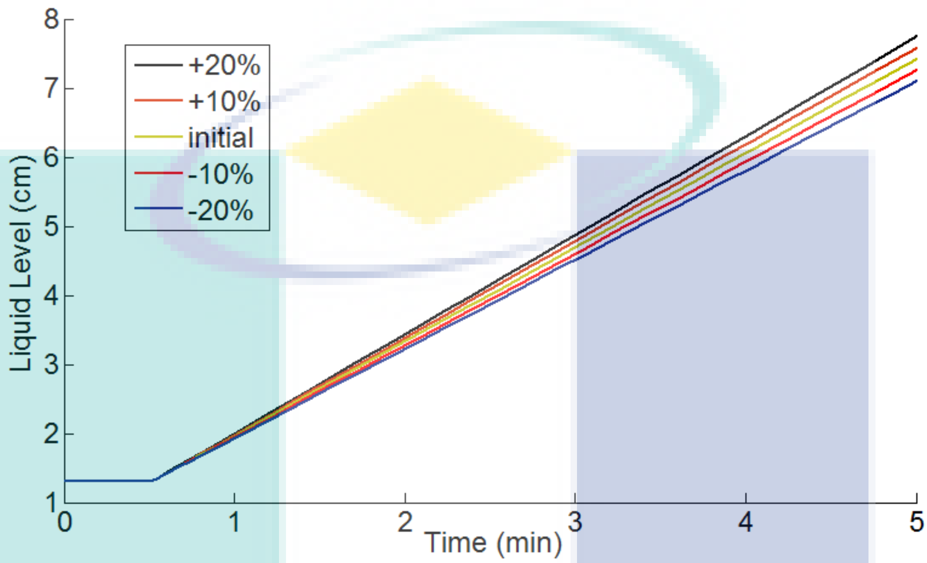



Figure 4.6 Sensitivity Analysis of Liquid Level (Open Loop) Profile for Case (1) (Top Vessel)

Based on the Figure 4.6, the liquid level (+20%) is steeper compared to liquid level (-20%). The liquid level (-10%) is less steeper compared to liquid level (+10%). All the liquid level with variable reflux flow is increased. When the reflux flow increased the liquid level of top vessel also increased. This is because the process is circulated in close operation, when reflux flow increases more liquid goes down and causes more vapor to form. Thus, more liquid is condensed and cause the level to increase. In order to control the level in top vessel, the reflux flow should be manipulated. The sensitivity analysis of middle vessel is shown in Figure 4.7.

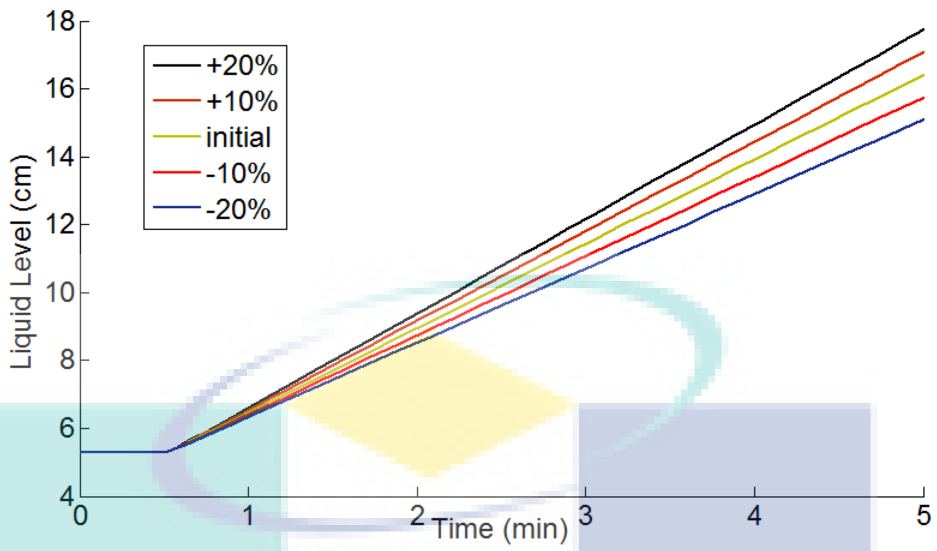



Figure 4.7 Sensitivity Analysis of Liquid Level (Open Loop) Profile for Case (1) (Middle Vessel)

The liquid level profile increased consistently with increasing bottom flow from -20% to +20% as shown in Figure 4.7. The liquid level with bottom flow at (+20% and +10%) is steeper compared to liquid level with bottom flow at (-20% and -10%). When the bottom flow decreased, the level of holdup also decreased. The liquid level for middle vessel is sensitive to bottom flow because when the bottom flow is decreased, less liquid goes down and less vapor is formed. Since the process operation is circulated, the lesser liquid condensed at the top causes the level of middle vessel to decrease. In order to control the level in middle vessel the bottom flow should be manipulated. The sensitivity analysis is proceeded with case (2). Figure 4.8 shows the sensitivity analysis of liquid level in middle vessel for case (2).

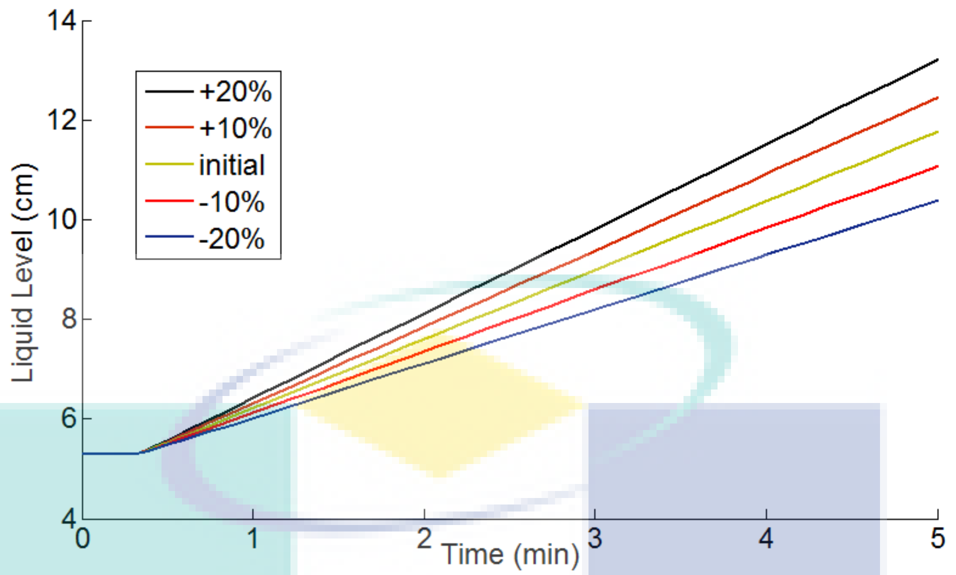



Figure 4.8 Sensitivity Analysis of Liquid Level (Open Loop) Profile for Case (2) (Top Vessel)

According to Figure 4.8, the liquid level (+20%) is steeper compared to liquid level (-20%). The liquid level (-10%) is less steeper compared to liquid level (+10%). When the reflux flow increased the liquid level also increased exhibiting similar behaviour as case (1). The liquid level in top vessel is sensitive to reflux flow. Similar to case (1), reflux flow should be manipulated in order to control the liquid level of top vessel. The sensitivity analysis of middle vessel for case (2) is shown in Figure 4.19.

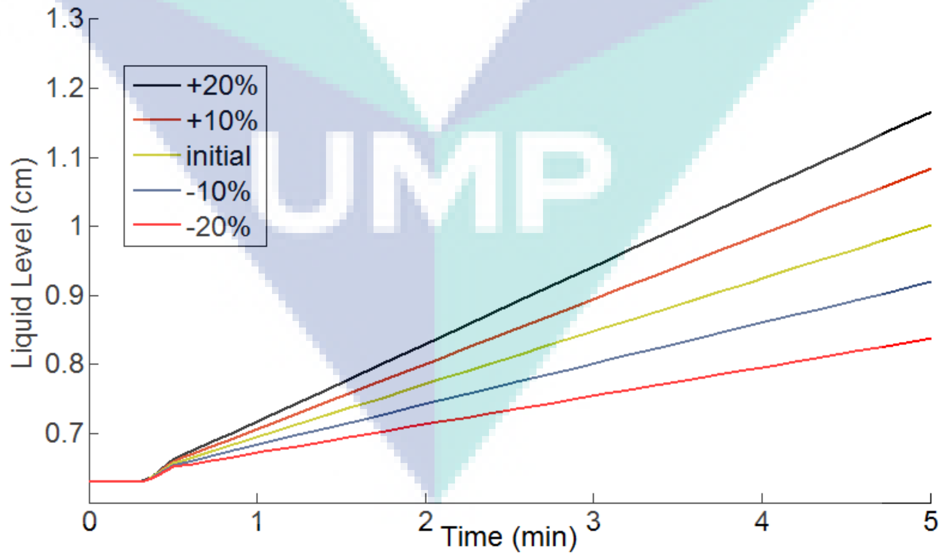



Figure 4.9 Sensitivity Analysis of Liquid Level (Open Loop) Profile for Case (2) (Middle Vessel)

The liquid level profile increased consistently with increasing bottom flow from -20% to +20% as shown Figure 4.9. The liquid level with bottom flow (+20% and +10%) is steeper compared to liquid level with bottom flow (-20% and -10%). Hence, the liquid level for middle vessel is sensitive to bottom flow similar behaviour occurred in case (2). In this step two sensitivity analysis, for case (1) and case (2), the liquid level in top vessel is sensitive to reflux flow and the liquid level in middle vessel is sensitive to bottom flow because every change of input is results in change of output. This behaviour shows that control system is necessary. The output becomes the controlled variable (level) and the input becomes manipulated variable (flow). For both cases, the controlled variable is liquid level in top vessel and middle vessel is paired with the manipulated variable that is reflux flow and bottom flow. The sensitivity analysis is proceeded to step three for case (1).

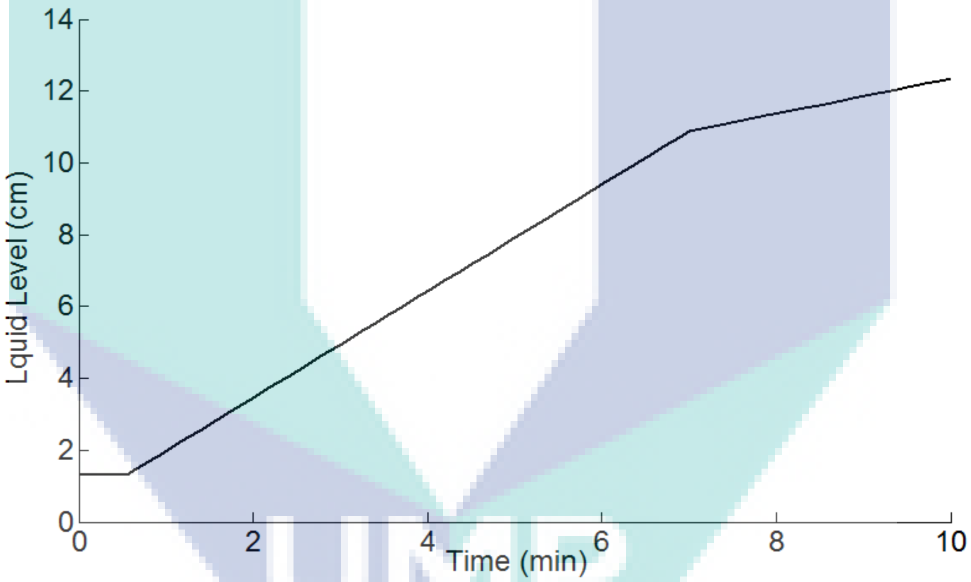



Figure 4.10 Sensitivity Analysis of Liquid Level in Top Vessel toward Middle Flow for Case (1) (Top Vessel)

In this step three, the effect of liquid level is analysed depending on the most effected variable that is coming into the system. This most effected variable is then considered as the disturbance variable to the system (Raot et al., 1988). In this MVBDC system the available disturbance for MVBDC system is middle flow. At the beginning the system is operated at steady state. After 7 minutes of operation the process behave dynamically by decreasing the middle flow to 1 $cm^3$/min for both case (1) and case (2). Figure 4.10 shows the effect of liquid level in top vessel toward the changes of middle flow for case (1). Based on Figure 4.10, when the sudden decreased of middle flow happens after 7 minutes, the level in middle vessel also decreased. As a conclusion, it is confirmed that the middle flow is the disturbance in middle vessel. Due to decrease of middle flow, less liquid flows down to the column and result to decrease of reboiler holdup. Thus, less vapor is formed and condensed to liquid. Hence the level in top vessel decreases. In order to keep the holdup constant, it is important to control the liquid level in the top vessel to maintain the product purity. This level control can ensure that the disturbance is rejected to the system. The disturbance rejection for close loop system is discussed in detail in sub chapter 4.2.2.3. Then, the liquid level profile for middle vessel is shown Figure 4.11.

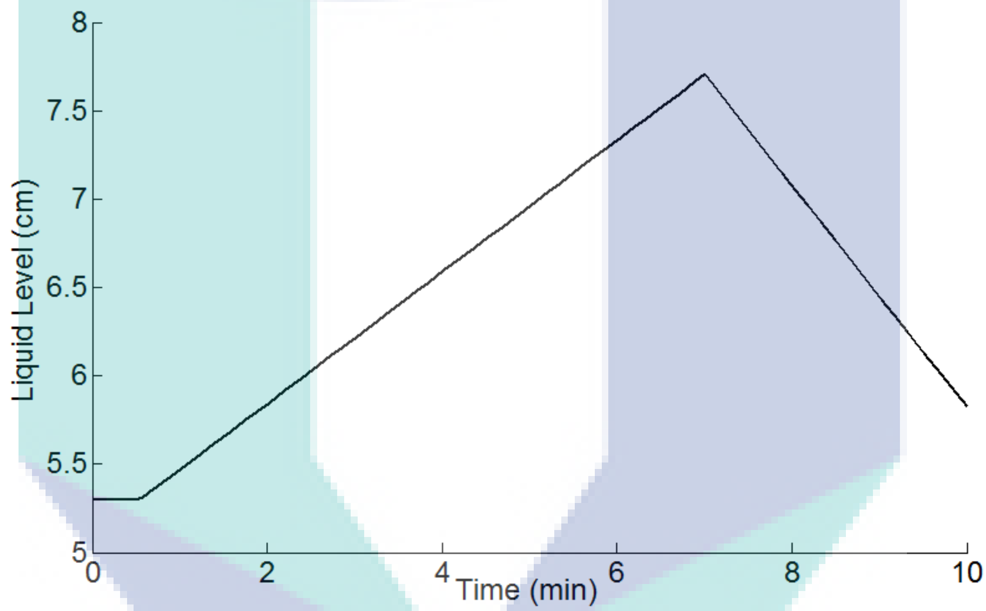



Figure 4.11 Sensitivity Analysis of Liquid Level in Middle Vessel toward Middle Flow for Case (1) (Middle Vessel)

In the middle vessel, the middle flow is decreased about 1cm$^3$/min. Based on the degree of freedom calculation as mentioned in chapter 3, the middle flow is one of the disturbance variables. The changes of middle flow affects the reboiler level as shown in Figure 4.11. If the middle flow decreased the level in top vessel liquid level also decreased, while if the middle flow decreased the level in middle vessel in liquid level also decreased.

The sensitivity analysis is also performed for case (2). The sensitivity analysis of liquid level profile toward disturbance for case (2) is shown in Figure 4.12 and 4.13.

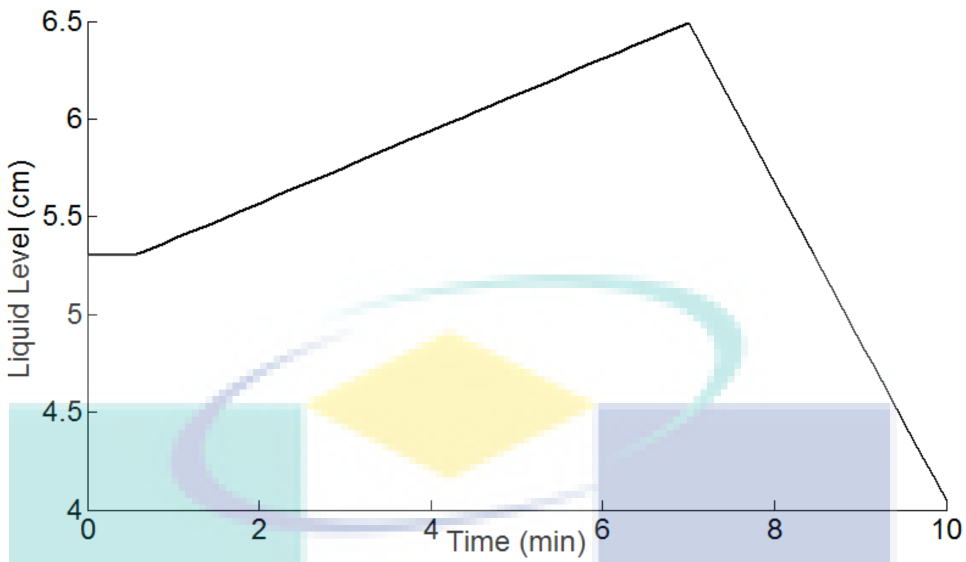



Figure 4.12 Sensitivity Analysis of Liquid Level in Top Vessel toward Middle Flow for Case (2) (Top Vessel)

For case 2, the disturbance variables for top vessel and middle vessel is similar to case (1). The middle flow is also decreased about 1 $cm^3$/min at 7 min during operation. The effect of liquid level in top vessel toward change of middle flow is shown in Figure 4.12. According to Figure 4.12, liquid level in top vessel is sensitive to change of middle flow. When the middle flow decreased the liquid level in top vessel also decreased. This is because less liquid flows down to columns and reboiler. Thus, less vapor is formed and less liquid is condensed and result to increase of the level of holdup in the vessel. This result confirmed that middle flow is the most affected variable to top vessel. A level control is necessary in this top vessel in order to reject disturbance coming to the system. This sensitivity analysis is continued in middle vessel as shown in Figure 4.13.

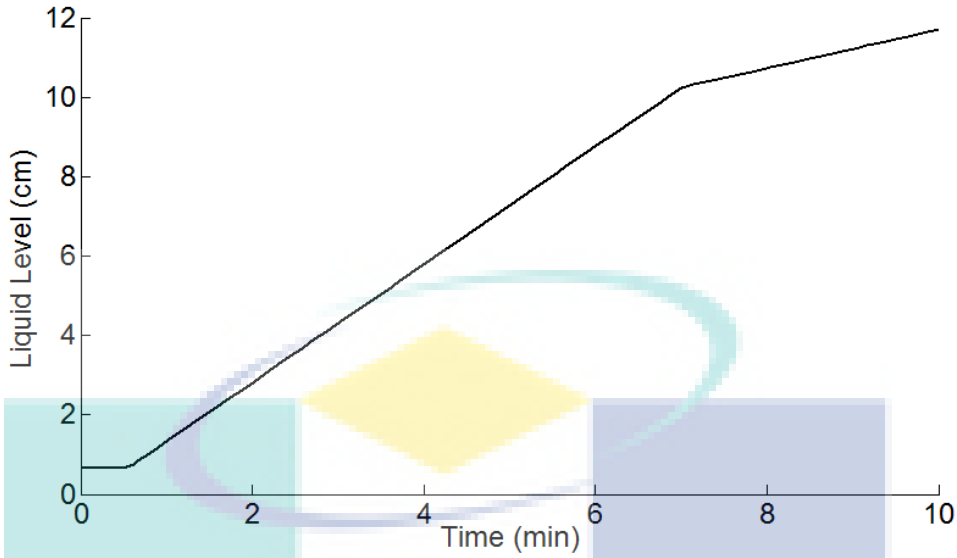



Figure 4.13 Sensitivity Analysis of Liquid Level in Middle Vessel toward Middle Flow for Case (2) (Middle Vessel)

Based on Figure 4.13, when the middle flow is decreased about 1 $cm^3$ /min at 7 minutes during operation the liquid level in middle vessel is also decreased similar to behaviour as case (1). This shows that the liquid level in middle vessel is sensitive to middle flow. A level control is necessary to reject the disturbance coming to the system.

### 4.2.1.2 Interaction Analysis

In this section, two methods are considered *i.e* relative gain array (RGA) and singular value decomposition (SVD). RGA is one of the method for designing and analysing a MIMO control scheme for a process in steady state. This RGA calculation is performed for case (1) only since both cases are implemented on the same system, the only difference is the objective to achieve.

The relative gain array can be evaluated from steady state gain matrix. Six tests run are performed. Table 4.4 shows the test run of the close loop system with variable manipulated variable. Manipulated variable 1 ($MV_1$) is reflux flow, manipulated variable 2 ($MV_2$) is middle flow, manipulated variable 3 ($MV_3$) is bottom flow, controlled variable 1 ($CV_1$) is level of holdup in top vessel, controlled variable 2 ($CV_2$) is level of holdup in middle vessel and controlled variable 3 ($CV_3$) is level of holdup in middle vessel.

Table 4.4 Test Run of Variable Manipulated Variable against Controlled Variable

| | $MV_1$ | $MV_2$ | $MV_3$ | $CV_1$ | $CV_2$ | $CV_3$ |
|---|---|---|---|---|---|---|
| Run 1 | 0.1696 | 0.1249 | 0.09 | 6.63 | 2.65 | 2.65 |
| Run 2 | 0.3097 | 0.1249 | 0.09 | 3.97 | 3.97 | 3.97 |
| Run 3 | 0.49987 | 0.07749 | 0.09 | 1.3 | 6.63 | 3.97 |
| Run 4 | 0.49987 | 0.25395 | 0.09 | 2.65 | 2.65 | 6.63 |
| Run 5 | 0.49987 | 0.1249 | 0.1414 | 1.3 | 3.97 | 6.63 |
| Run 6 | 0.49987 | 0.1249 | 0.0226 | 2.65 | 6.63 | 2.65 |

Equation 4.1-4.9 shows the calculation of rate of change of controlled variable per change of manipulated variable. The matrix in equation 4.10 shows the result from calculation:

$$K_{11} = \frac{\Delta CV_1}{\Delta MV_1} = -18.986 \tag{4. 1}$$

$$K_{12} = \frac{\Delta CV_1}{\Delta MV_2} = 7.650 \tag{4. 2}$$

$$K_{13} = \frac{\Delta CV_1}{\Delta MV_3} = -11.366 \tag{4. 3}$$

$$K_{21} = \frac{\Delta CV_2}{\Delta MV_1} = 9.421 \tag{4. 4}$$

$$K_{22} = \frac{\Delta CV_2}{\Delta MV_2} = -22.554 \tag{4. 5}$$

$$K_{23} = \frac{\Delta CV_2}{\Delta MV_3} = -22.396 \tag{4. 6}$$

$$K_{31} = \frac{\Delta CV_3}{\Delta MV_1} = 9.421 \tag{4. 7}$$

$$K_{32} = \frac{\Delta CV_3}{\Delta MV_2} = 15.074 \tag{4. 8}$$

$$K_{33} = \frac{\Delta CV_3}{\Delta MV_3} = 33.510 \tag{4. 9}$$

$$RGA = \Lambda = \begin{bmatrix} -66.491 & 33.746 & 33.746 \\ 33.746 & -99.941 & 67.195 \\ 33.746 & 67.1954 & -99.941 \end{bmatrix} \quad 4.10$$

Based on the RGA eigenvalue, the positive eigenvalue should be chosen as the best pairing of controlled and manipulated variables. Based on the results, there are a few possible pairings that can be chosen, such as ($CV_1$ – $MV_2$), ($CV_1$ – MV3), ($CV_2$ – $MV_1$), ($CV_2$ – $MV_3$) and ($CV_1$ – $MV_3$) and ($CV_3$ – $MV_2$).The RGA method cannot confirm and justify the best pairing of controlled and manipulated variable. Hence, SVD analysis is used to determine the best pairing of controlled and manipulated variable.

SVD is to decompose a rectangular matrix into three simple matrices *i.e* two orthogonal matrices and one diagonal matrices (Abdi, 2007). This singular matrix is then analysed based on the $\sigma_i$ obtained. The condition number is calculated based on $\sigma_i$. In this study, the SVD calculation is computed in Matlab as shown in Appendix B. The calculation of SVD is stated in equation 4.11 – 4.14.

$$A = \begin{bmatrix} -18.986 & 7.650 & -11.366 \\ 9.421 & -22.554 & -22.396 \\ 9.421 & 15.074 & 33.510 \end{bmatrix} \quad 4.11$$

$$A = U \sum V^T$$

Where,

$$U = \begin{bmatrix} -0.1591 & 0.8042 & 0.5727 \\ -0.6157 & -0.5343 & 0.5792 \\ 0.7718 & -0.2605 & 0.5801 \end{bmatrix} \quad 4.12$$

$$S = \begin{bmatrix} 48.2666 & 0 & 0 \\ 0 & 27.5049 & 0 \\ 0 & 0 & 0.0899 \end{bmatrix} \quad 4.13$$

$$V = \begin{bmatrix} 0.0930 & -0.8274 & 0.5539 \\ 0.5035 & 0.5190 & 0.6907 \\ 0.8590 & -0.2147 & -0.4649 \end{bmatrix} \quad 4.14$$

Based on the singular matrix, the largest gain is 48.2666 and the smallest gain is 0.0899. At this stage, a conclusion of best pairing of controlled and manipulated variable is still not obtained. This 3 x 3 matrix should be reduced to 9 set of 2 x 2 matrix in order to calculate the condition number of each loop. The paired with the highest condition number is called ill-conditioned. It should be eliminated due to high interaction between loops (Skogestad et al, 1992). Table 4.5 shows the condition number of each 2 x 2 pairing. The calculation in Matlab software for each 2 x 2 pairing is shown in appendix C.

Table 4.5 Condition Number for Different 2 x 2 Pairings.

| Pairing Number | Controlled Variables | Manipulated Variables | Condition Number |
|---|---|---|---|
| 1 | $y_1,y_2$ | $u_1,u_2$ | 2.445 |
| 2 | $y_1,y_2$ | $u_1,u_3$ | 1.185 |
| 3 | $y_1,y_2$ | $u_2,u_3$ | 2.381 |
| 4 | $y_1,y_3$ | $u_2,u_1$ | 1.254 |
| 5 | $y_1,y_3$ | $u_1,u_3$ | 2.866 |
| 6 | $y_1,y_3$ | $u_2,u_3$ | 3.292 |
| 7 | $y_2,y_3$ | $u_2,u_1$ | 2.101 |
| 8 | $y_2,y_3$ | $u_1,u_3$ | 3.098 |
| 9 | $y_2,y_3$ | $u_2,u_3$ | 5.461 |

Based on the Table 4.13, the pairing number 2 has obtained the lowest condition number due to low interaction between control loops (Skogestad et al, 2005). Hence, level in top liquid level and reflux flow and level in middle liquid level and bottom flow is the best pairing for this MVBDC control system. Performing RGA and SVD together with sensitivity analysis results in a more complete column control design procedure. This analysis could be used to screen perspective control strategies which could then be simulated to more completely examine each candidate's disturbance rejection ability (Roat et al, 1988).

#### 4.2.1.3 Dynamic Analysis

Dynamic analysis is important in determining the behaviour of the process. Besides, it is also used to determine the type of controller used for the system. Advanced controller is not necessary for linear system, linear controller such as proportional (P) only, proportional integral (PI) and proportional integral derivative (PID) is sufficient enough (Raptis and Valvanis, 2011) (Maheswari et al., 2014). Two cases are considered in this analysis similar to previous analysis. This dynamic analysis is a continuation from the previous sensitivity analysis. Basically, this dynamic analysis is based on graphical method (Melnik et al, 2005).

The liquid level response profile for case (1) shown in Figure 4.14 - 4.15 and case (2) shown in Figure 4.16 - 4.17 is analysed based on the behaviour of responses and the differences of each liquid level responses. Each holdup response in case (1) and case (2) increased linearly. The difference between each responses is identical. No uneven or unstable responses are presented on the Figure 4.14 - 4.17. Hence, the system is linear for middle vessel and reboiler in case (1) (Melnik et al., 2005). Thus, the conventional controller is sufficient enough to control the level control in top vessel.

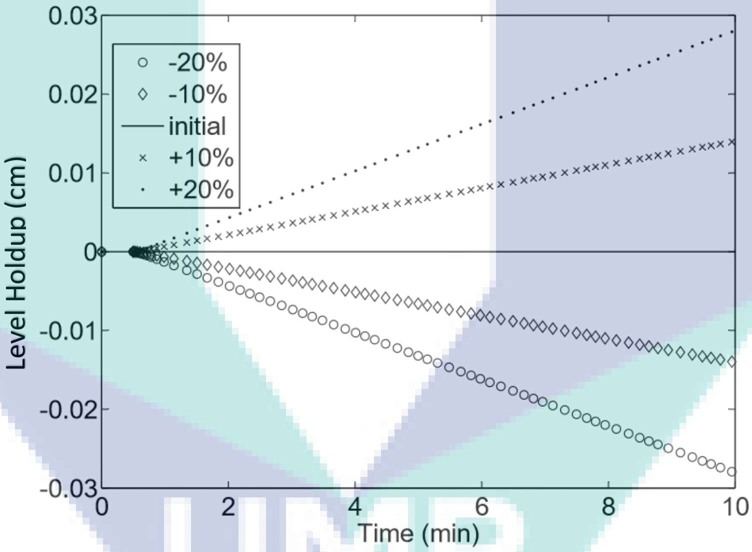



Figure 4.14 Dynamic Analysis of Liquid Level Profile for Case (1) (Top Vessel)

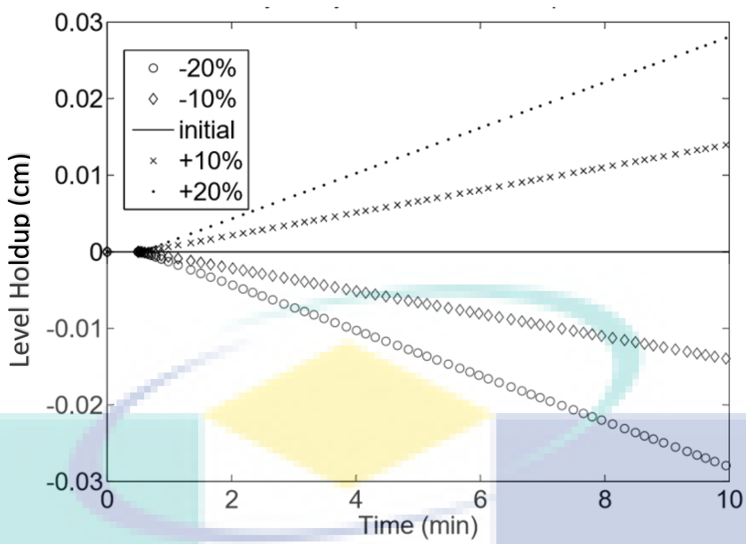



Figure 4.15 Dynamic Analysis of Liquid Level Profile for Case (1) (Middle Vessel)

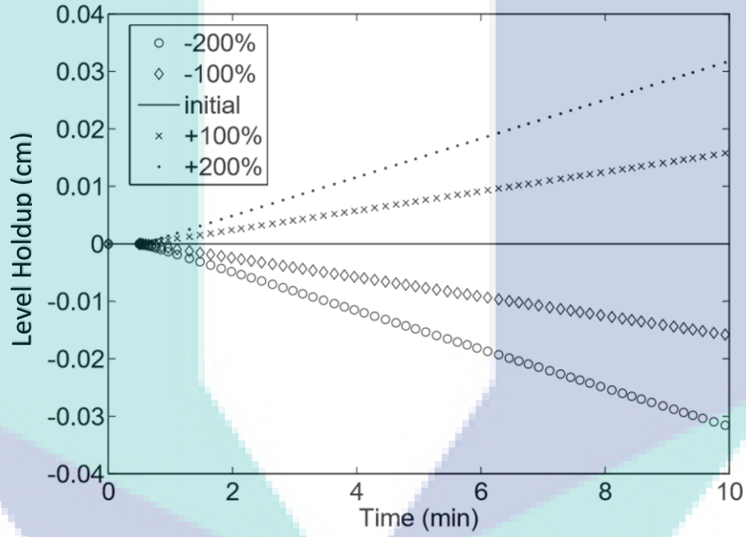



Figure 4.16 Dynamic Analysis of Liquid Level Profile for Case (2) (Top Vessel)

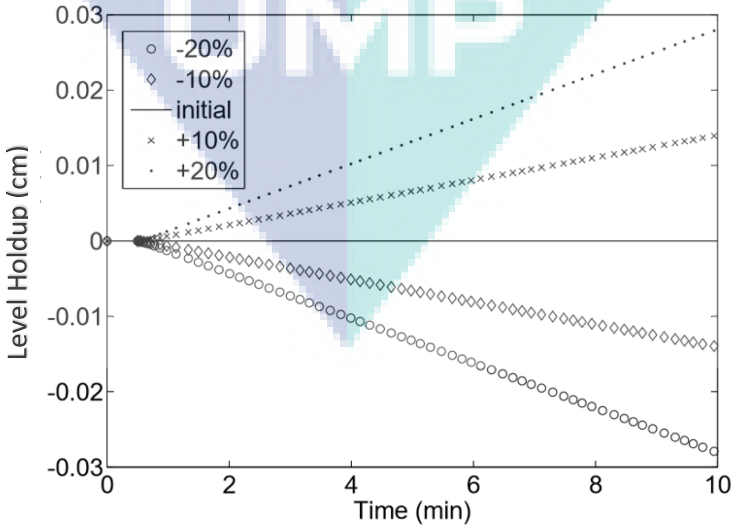



Figure 4.17 Dynamic Analysis of Liquid Level Profile for Case (2) (Middle Vessel)

Based on the dynamic analysis, the conventional (linear) controller is chosen for both liquid level in both cases. Hence, proportional controller and proportional integral controller are chosen for study in detail.

### 4.2.2 Closed loop Testing

This closed loop testing covers the controller tuning based on Ziegler Nichols (ZN) closed loop tuning method and IMC tuning method for case (1) and case (2).

#### 4.2.2.1 Controller Tuning

As mentioned earlier, the closed loop controller tuning is performed based on ZN closed loop tuning and IMC tuning. The performance of controller for both methods are compared. Two cases are considered for this closed loop controller tuning which are case (1) and case (2). For ZN closed loop tuning, the formula for controller gain is based on Table 3.7. The ultimate gain and ultimate period are obtained by trial and error method. The controller is set to auto mode. The integral value is set to infinity and the derivative value is set to zero. The proportional value is increased from one until the response start to oscillate (Haugen, 2010).

For IMC tuning, the formula of controller gain is based on Table 3.8. The closed loop time constant, $\tau_{c,}$ is set higher than $\tau$ (Rivera et al., 1986). The controller gain setting is based on integrating process (Fruehauf et al, 1990).

**a. Case (1): Top Product Recovery**

For top vessel, the ultimate gain, Ku and ultimate period, Tu are 15 and 0.7 minutes as shown Figure 4.18. The value are applied to the formula of ZN closed loop tuning in Table 3.7. The calculation of P only controller gain for reboiler is written in equation 4.1 and the calculation of PI controller gain for top vessel is written in equation 4.2 and 4.3:

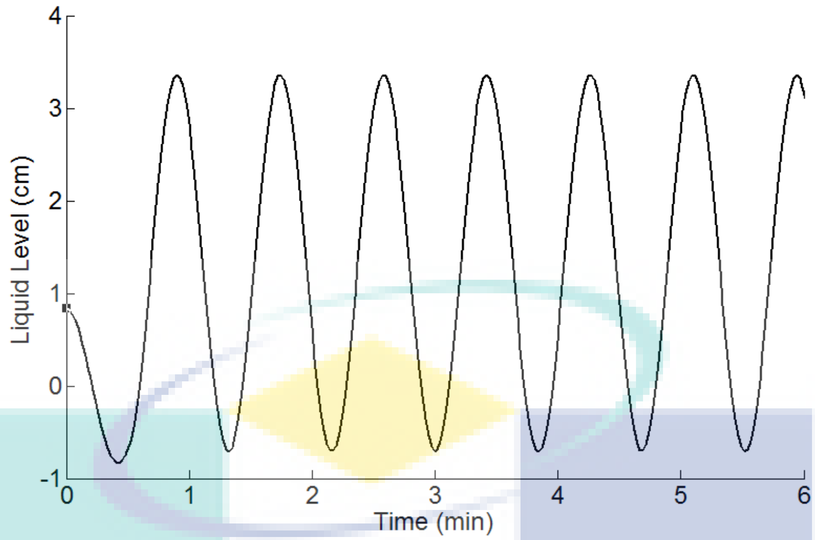



Figure 4.18 Liquid Level Oscillatory Response Case (1) (Top Vessel)

$$P = 0.5 \times 15 = 7.500 \quad 4.15$$

$$P = 0.45 \times 15 = 6.75 \quad 4.16$$

$$I = 0.7 \div 1.2 = 0.583 \quad 4.17$$

For middle vessel, the ultimate gain, Ku and ultimate period, Tu are 10 and 0.9 minutes respectively as shown in Figure 4.19. The value are applied to the formula of ZN closed loop tuning in Table 3.7. The calculation of P only controller gain for top vessel is written in equation 4.4 and the calculation of PI controller gain for top vessel is written in equation 4.5 and 4.6:

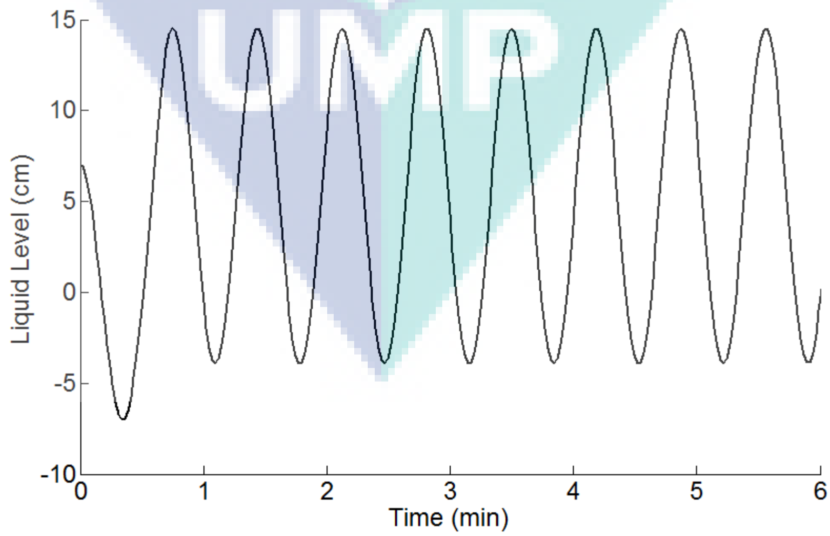


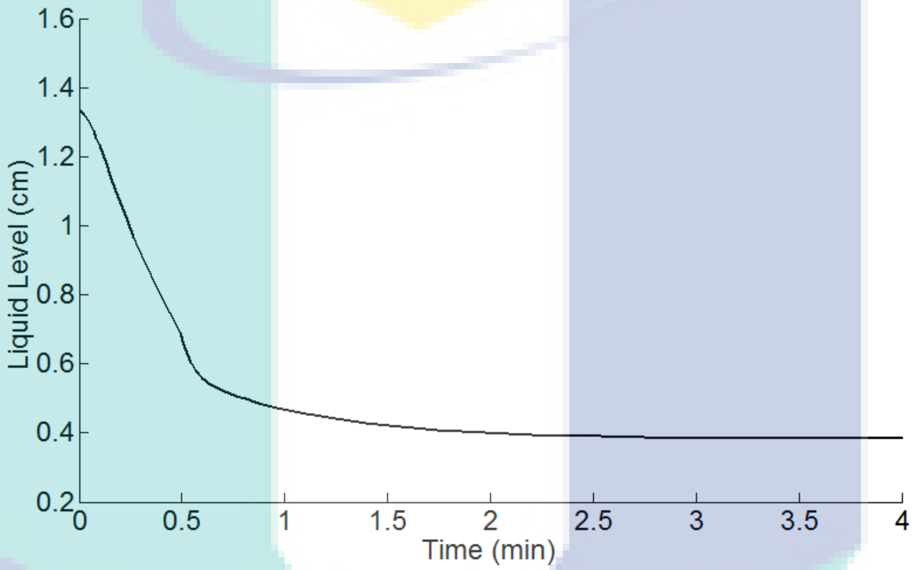

Figure 4.19 Liquid Level Oscillatory Response Case (1) (Middle Vessel)

$$P = 0.5 \times 10 = 5.0 \tag{4.18}$$

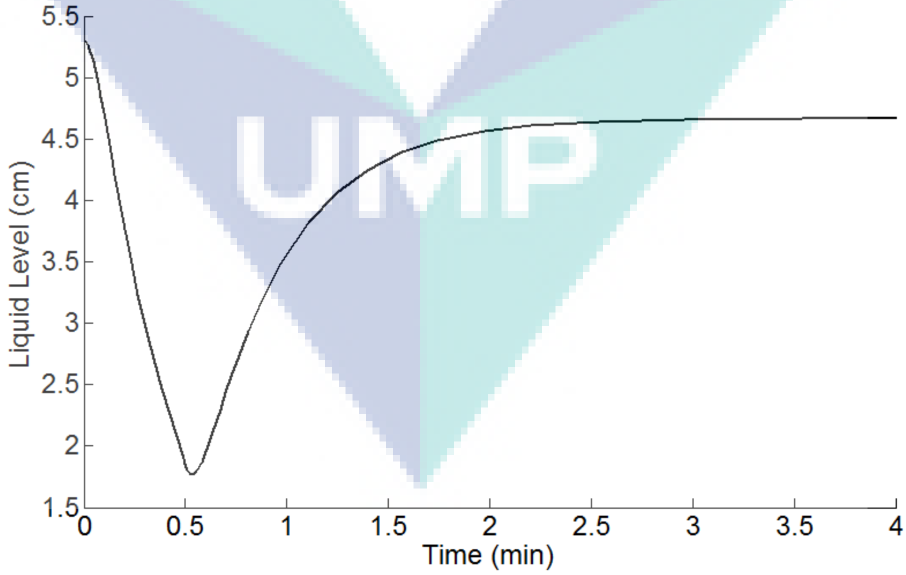
$$P = 0.45 \times 10 = 4.5 \tag{4.19}$$

$$I = 0.9 \div 1.2 = 0.75 \tag{4.20}$$

The controller gain from equation 4.4 - 4.6 is applied to PID block in a closed loop simulation model. Figure 4.20 shows the response of liquid level for both vessel.



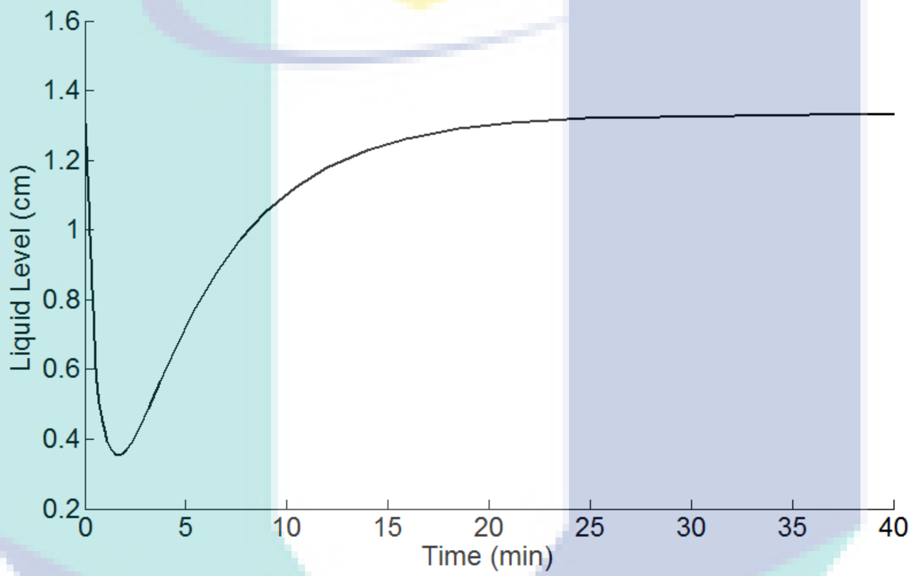

(a)



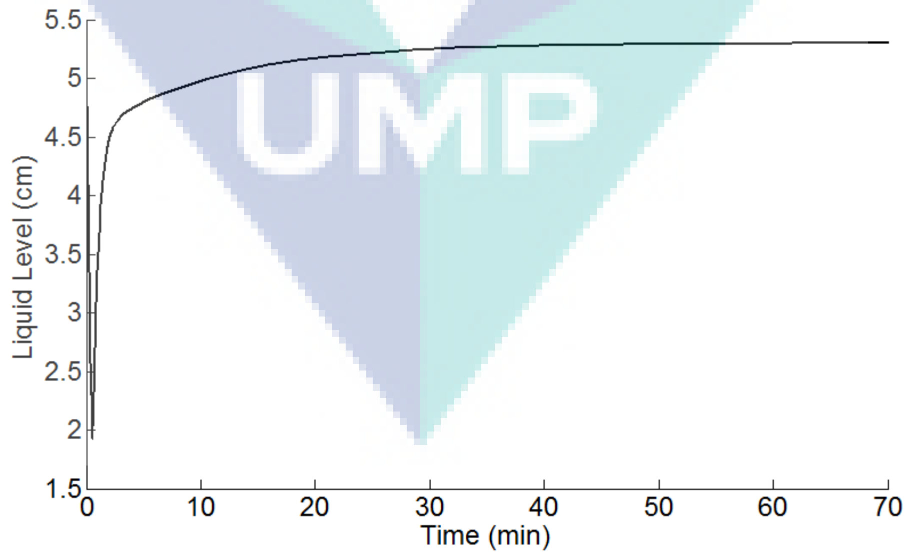

(b)

Figure 4.20 P Controller Liquid Level Profile with Closed Loop ZN Tuning Case (1) (a) Top Vessel (b) Middle Vessel

The response for both vessels is stable and no overshoot is produced. The holdup in top vessel and middle vessel do not achieve its set point 5.3 cm for both holdup. Based on Figure 4.18, the offset from set point for top liquid level is about 71.30 % (0.38 cm) and middle vessel 11.89 % (4.67 cm). The offset is due to higher sustained error being produced (Anthony et al., 2014). Hence, PI controller is necessary to bring the liquid level to its set point (Ishak et al., 2013). The integral action is used to reduce sustained error produced by proportional action (Rao et al., 2014).




(a)




(b)

Figure 4.21 PI Controller Liquid Level Profile with Closed Loop ZN Tuning Case (1) (a) Top Vessel (b) Middle Vessel

Based on Figure 4.21, there are no overshoot produced in the level response for top vessel and middle vessel, respectively. The liquid level response for top vessel and middle vessel reaches its set point which is 1.3 cm and 5.3 cm for top vessel and middle vessel respectively. Based on the Figure 4.21, at the beginning of process the response experience suddenly decreases due to the decreasing liquid level. After the control action starts the liquid level response increases and reaches set point. The settling time for top vessel response is less than 35 minutes and middle vessel is 50 minutes. The rise time for top vessel is 15 minutes and for reboiler is 5 minutes. The settling time is quite longer because of the slow response produced by PI controller by ZN tuning method (Priyadarshani and Lather, 2013). Hence, the PI controller is then tuned by IMC method. The controller gain for IMC tuning method stated in Table 4.5 is based on the formula mentioned in Table 3.8. The results are shown in Figure 4.20.

Table 4.6 Controller Gain for IMC Tuning Method for Case (1)

| | **Top Liquid level** ($\tau_c = 0.53$) | **Middle Liquid level** ($\tau_c = 0.52$) |
|---|---|---|
| P | 3.774 | 3.846 |
| I | 1.060 | 1.040 |

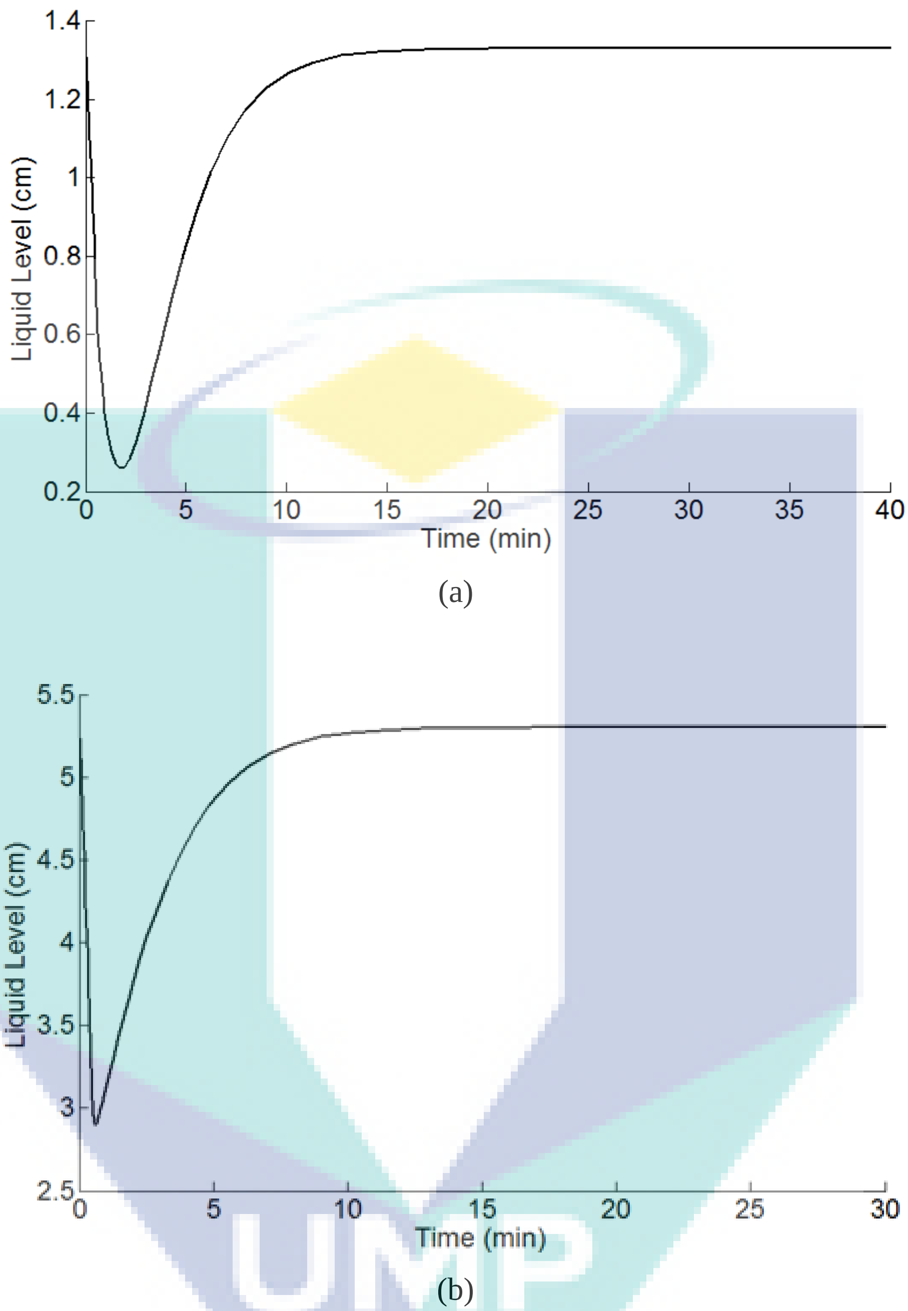
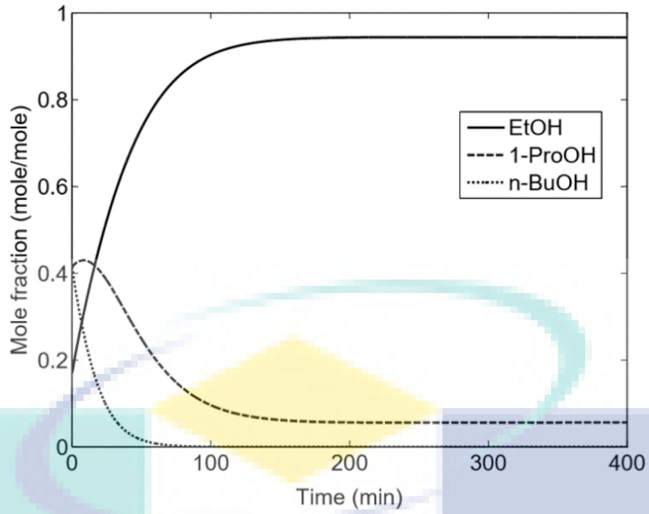
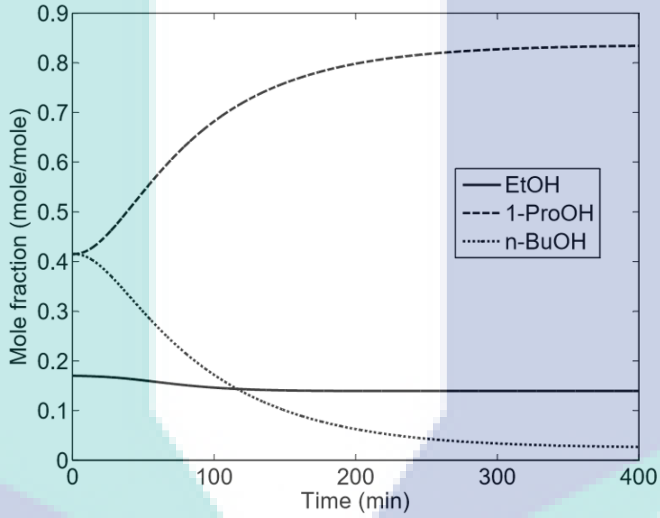



Figure 4.22 PI Controller Liquid Level Profile with Closed Loop IMC Tuning Case (1) (a) Top Vessel (b) Middle Vessel

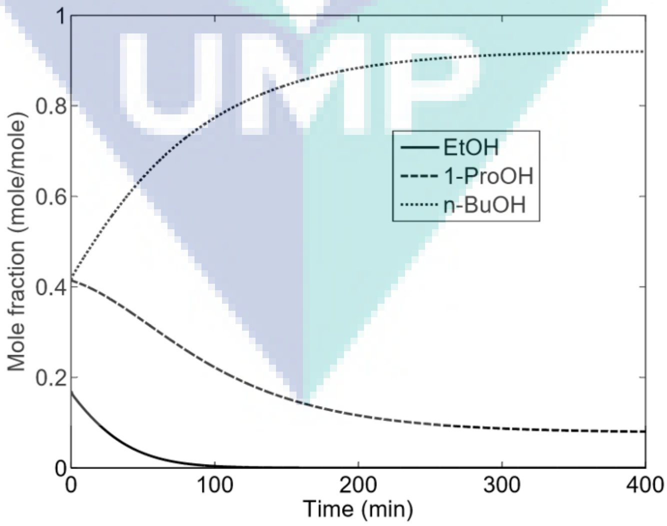
Based on Figure 4.22, the settling time for middle vessel and reboiler is reduced to 20 minute and 15 minute. The rise time is also reduced to 8 minutes and 5 minutes respectively. No overshoot is produced. From the time domain response IMC based system produces less settling time and overshoot because its has advantage of internal model control that includes the characterization of conventional PID controller (Priyadarshini and Lather, 2013). The composition profiles for each controller tuning ~~is~~ are presented in Figure 4.21 - 4.23.




(a)




(b)




(c)

Figure 4.23 Composition Profile for (a) Top Vessel (b) Middle Vessel (c) Reboiler for Closed Loop ZN Tuning of P Controller for Liquid Level (Case (1))

Figure 4.23 shows the composition profile for liquid level with ZN-P controller. Based on Figure 4.23, the top product is enriched with ethanol, the middle product enriched with 1-propanol and bottom product is enriched with n-butanol. The composition of top product (ethanol) is the highest. The intermediate and bottom product are successfully removed from top product. Hence, the objective of case (1) to produce the purest top product is achieved. The settling time of top product, middle product and bottom product are 200 minutes, 300 minutes and 300 minutes respectively. The composition of the components in each vessel is presented in Table 4.7.

Table 4.7 Composition of Components for Vessels with Closed Loop Tuning P Controller for Level Control (Case (1))

| **Components** | **Composition in Top Vessel** | **Composition in Middle Vessel** | **Composition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.943700 | 0.139400 | 0.000111 |
| 1-Propanol | 0.056320 | 0.833800 | 0.080260 |
| n-Butanol | 0.000006 | 0.026790 | 0.919700 |

From Table 4.7, the top product composition is 0.9437, the middle product composition is 0.8338 and the bottom product composition is 0.9197. In top vessel, there are small amount of impurity contributed by 1-propanol (0.056320) and almost no n-butanol remained. In middle vessel, the impurities are contributed by ethanol (0.1394) and n-butanol (0.02679). While, in reboiler, the impurities are contributed by 1-propanol (0.08026) and almost no ethanol remained. As mentioned in 4.2.1.1, the objective of case (1) to produce 0.94 ethanol is achieved. From the sensitivity analysis, composition of final product is said to be indirectly related to level control. In order to obtain desired product composition, the liquid level must be kept constant at its optimal value calculated in advance (Hasebe et al, 1997) (Hasebe et al, 1999). Although the objective of case (1) is achieved with P controller, the liquid level response is unsatisfactory. The set point is not achievable. Thus, the PI controller must be implemented.

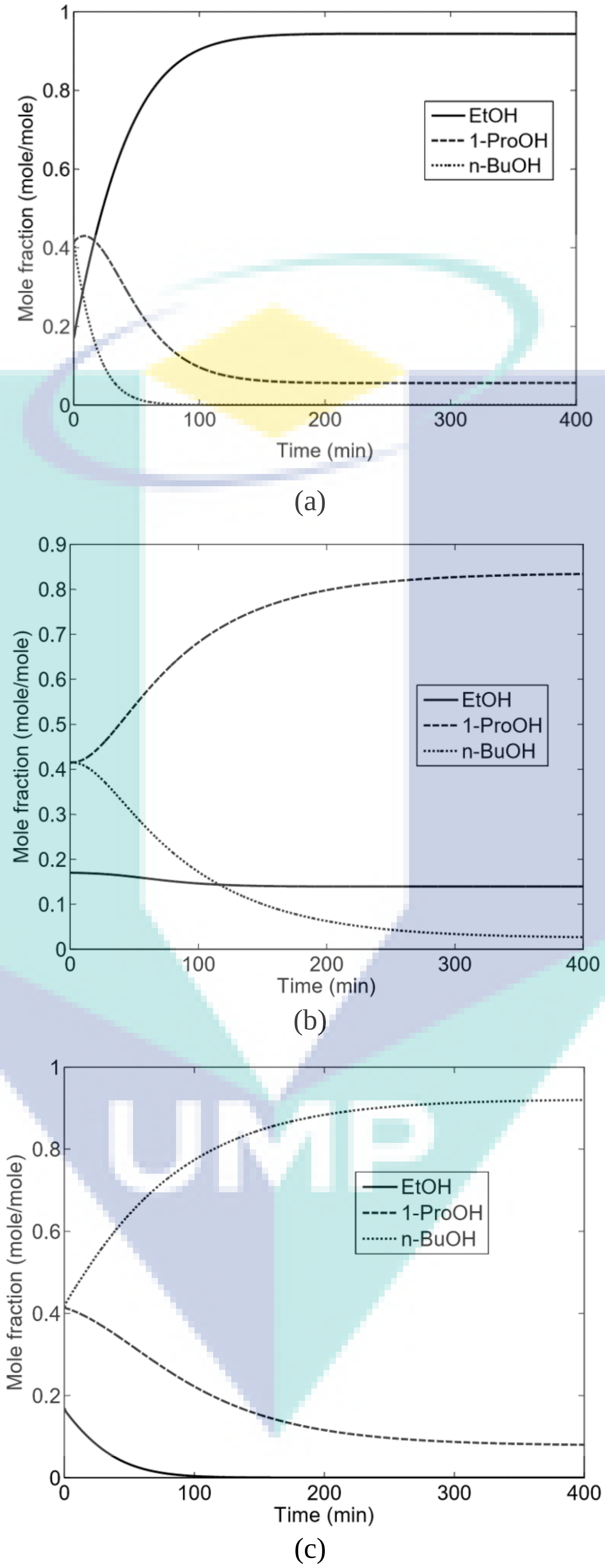


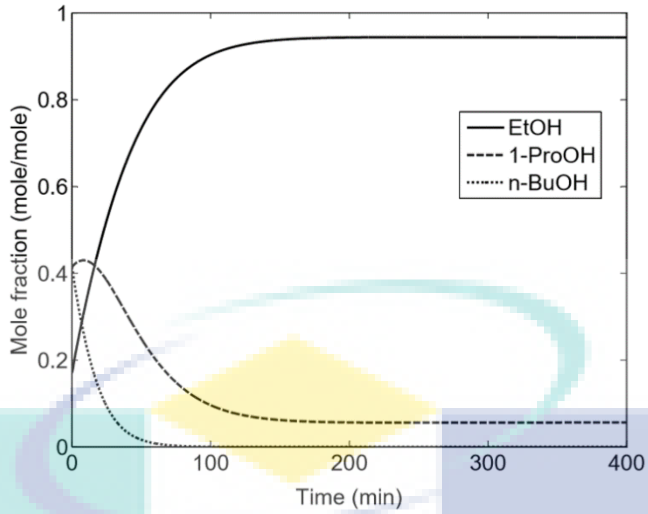

Figure 4.24 Composition Profile for (a) Top Vessel (b) Middle Vessel (c) Reboiler for Liquid Level with Closed Loop ZN Tuning PI Controller (Case (1))

From Figure 4.24(a) - (c), the composition profile for liquid level with ZN-PI controller produces similar response as before for liquid level with ZN-P controller. The top product is enriched with ethanol, middle product is enriched with 1-propanol and bottom product enriched of n-butanol. However, the settling time and composition of product slightly changes. The settling time of top product is reduced to 190 minutes. Meanwhile the settling time for middle and bottom product are 300 minutes. The product composition for each component in each vessel stated in Table 4.7.

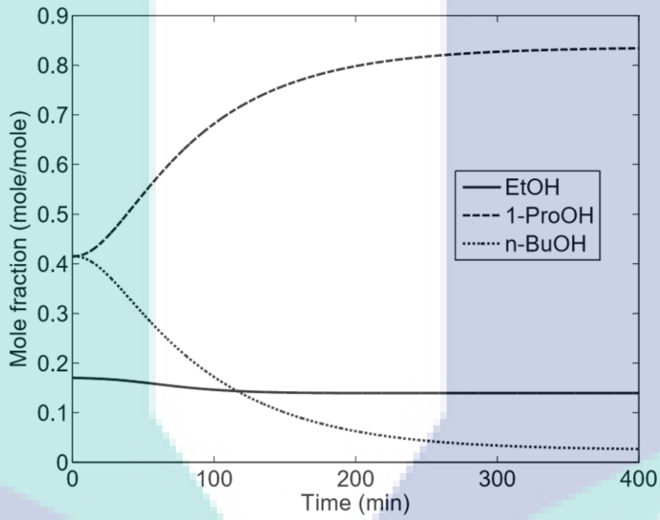
Table 4.8 Composition of Components for Vessels with Closed Loop ZN Tuning PI Controller for Level Control (Case (1))

| **Components** | **Composition in Top Vessel** | **Compositionin Middle Vessel** | **Composition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.943600 | 0.139300 | 0.000111 |
| 1-Propanol | 0.056380 | 0.834200 | 0.080960 |
| n-Butanol | 0.000006 | 0.026530 | 0.918900 |

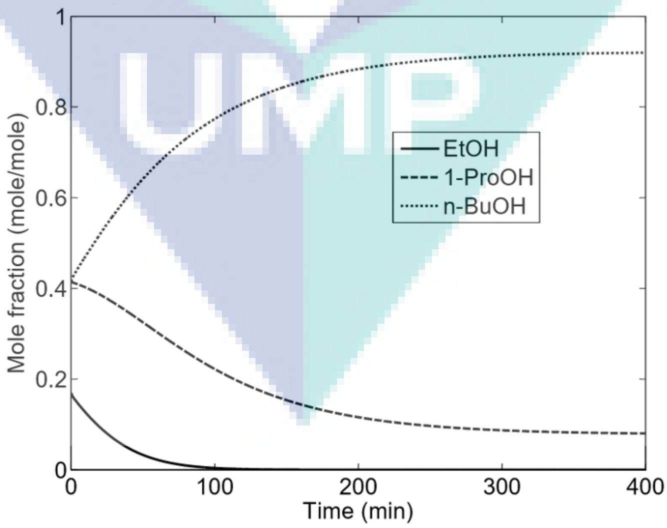
Based on the Table 4.8, the top product (ethanol) composition is 0.9436, the middle product (1-propanol) composition is 0.8342 and bottom product (n-butanol) composition is 0.9189. The composition of top product is slightly decreased and the composition of middle and bottom product is slightly increased from composition with ZN-P controller liquid level. This is due to response of liquid level in middle vessel and bottom vessel reaches its set point and caused the middle and bottom product composition to increase. It can be concluded that the holdup must be kept constant to its specified value calculated in advanced in sensitivity analysis in order to improve the product purity (Hasebe et al, 1997) (Hasebe et al, 1999). However the composition is still maintained above 0.94 mole. The objective of case (1) is achieved. In order to obtain the best controller performance, the tuning method is proceed with IMC tuning method.




(a)




(b)




(c)

Figure 4.25 Composition Profile for Top Vessel, Middle Vessel and Reboiler for Liquid Level with IMC-PI Controller (Case (1))

Figure 4.25 (a) - (c) show the composition profile of liquid level with IMC-PI controller. From the Figure 4.25 (a) - (c), same as previous the top product is enriched with ethanol, middle product is enriched with 1-propanol and bottom product enriched of n-butanol. The settling time for top product is 180 minutes, middle product is 280 minutes and bottom product is 290 minutes. IMC-PI controller successfully reduced the settling time for all product. This is because settling time of liquid level with IMC-PI controller is also decreased compared to ZN-PI controller. The behaviour of product composition is depending on the liquid level. The composition of product for liquid level with IMC-PI controller is stated in Table 4.8.

Table 4.9 Composition of Components for Vessels with IMC Tuning PI Controller for Level Control (Case (1))

| Components | Composition in Top Vessel | Composition in Middle Vessel | Composition in Reboiler |
|---|---|---|---|
| Ethanol | 0.943700 | 0.139500 | 0.000112 |
| 1-Propanol | 0.056310 | 0.834000 | 0.083160 |
| n-Butanol | 0.000006 | 0.026490 | 0.918500 |

The composition of ethanol is 0.9437, the composition of 1-propanol is 0.834 mole and the composition of n-butanol is 0.9185 as stated in Table 4.9. The composition of ethanol is slightly increased, the composition of ethanol is slightly increased compared to previous composition with liquid level with ZN-PI controller shown in Table 4.8. The objective of case (1) is successfully achieved. As a conclusion, the liquid level with IMC-PI controller has not even kept the holdup constant to its set point, the composition of product is also improved from previous liquid level with ZN-PI controller. The controller tuning then proceeds to case (2).

**b. Case (2): Middle Product Recovery**

For top vessel, the ultimate gain, Ku and ultimate period, Tu obtained are 30 and 0.44 minutes as shown in Figure 4.26. The value are applied to the formula of ZN closed loop tuning in Table 3.7. The calculation of P only controller gain for top vessel is written in equation 4.21 and the calculation of PI controller gain for top vessel is written in equation 4.22 and 4.23:

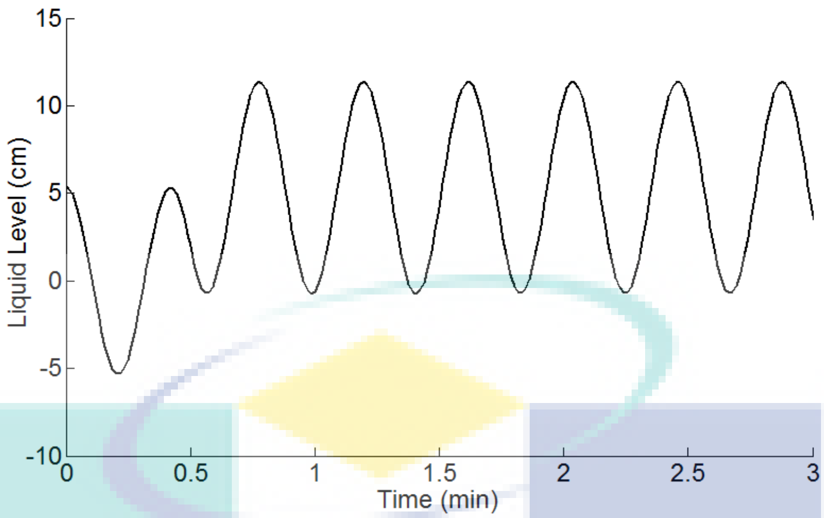



Figure 4.26 Liquid Level Oscillatory Response Case (2) (Top Vessel)

$$P = 0.5 \times 30 = 15 \qquad 4.21$$

$$P = 0.45 \times 30 = 13.5 \qquad 4.22$$

$$I = 0.4 \div 1.2 = 0.333 \qquad 4.23$$

For middle vessel, the ultimate gain, Ku and ultimate period, Tu obtained are 40 and 0.5 minute as shown in Figure 4.27. The values are applied to the formula of ZN closed loop tuning in Table 3.7. The calculation of P only controller gain for middle vessel is written in equation 4.24 and the calculation of PI controller gain for top vessel is written in equation 4.25 and 4.26:

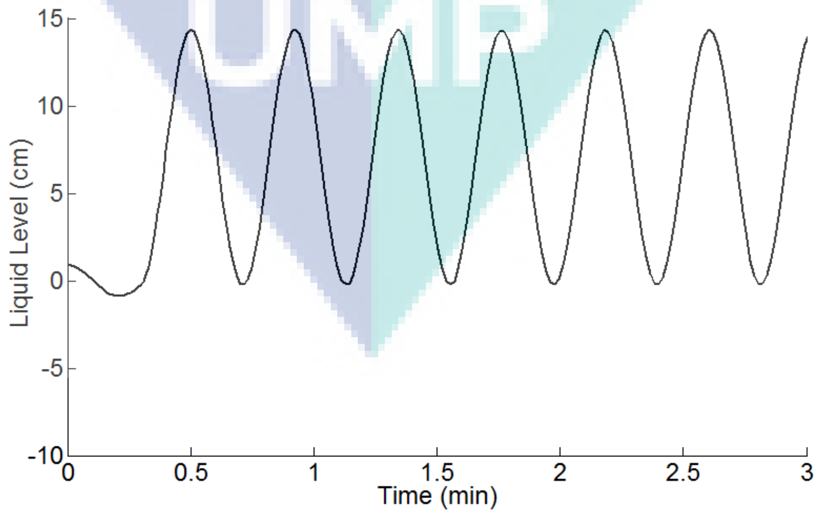


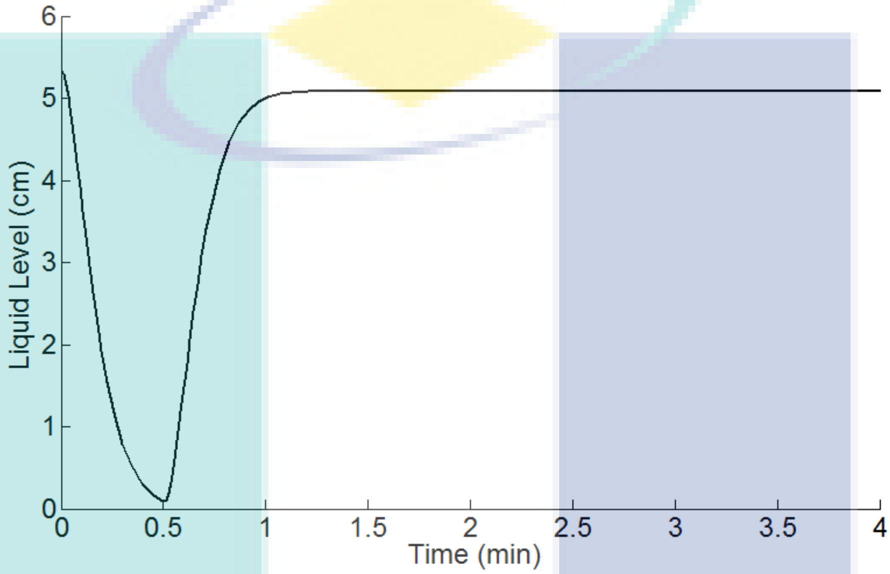

Figure 4.27 Liquid Level Oscillatory Response Case (2) (Middle Vessel)

$$P = 0.5 \times 40 = 20 \qquad 4.24$$

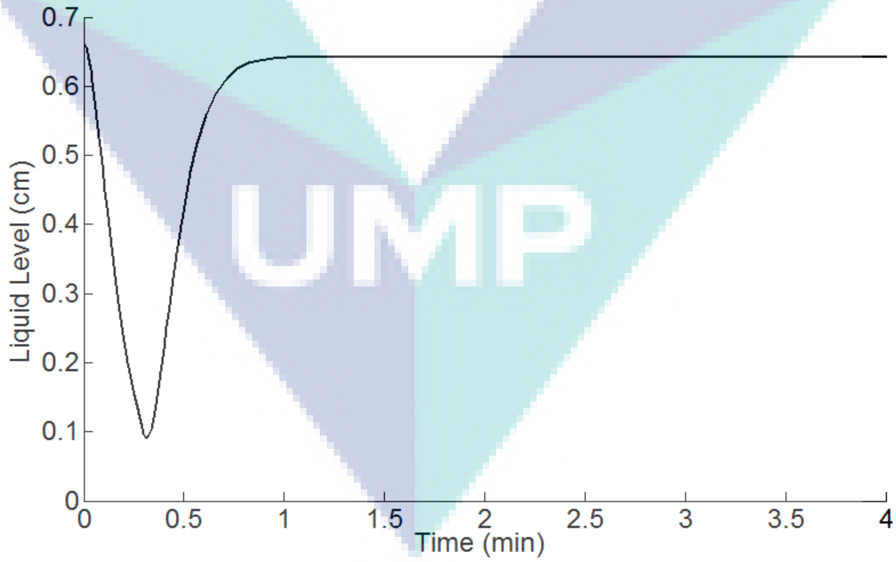
$$P = 0.45 \times 40 = 18 \qquad 4.25$$

$$I = 0.50 \div 1.2 = 0.417 \qquad 4.26$$

The controller gain from the calculation is applied to closed loop simulation model. Figure 4.46 shows the response of liquid level for both vessels




(a)




(b)

Figure 4.28 P Controller Liquid Level Profile with Closed Loop ZN Tuning Case (2) (a) Top Vessel (b) Middle Vessel

According to Figure 4.28, the response of liquid level is stable. There is no overshoot present. The settling time is less than 5 minutes. Liquid level response in top vessel and middle vessel do not achieve its set point 5.3 cm and 0.66 cm respectively. The offset from set point for liquid level in top vessel is about 3.98% (5.089 cm) percent. Meanwhile in middle vessel it is about 3.03% (0.644 cm). The offset is caused by the sustained error. The P controller has not managed to eliminate the offset (Anthony, 2014). The PI controller is applied in order to push the liquid level to its desired set point and to maintain the product composition (Ishak et al., 2013). An integral mode controller responds to the integral of the difference between set point and measured value. Thus, PI controller reduced the sustained error and pushed the output to its set point (Rao et al, 2014).

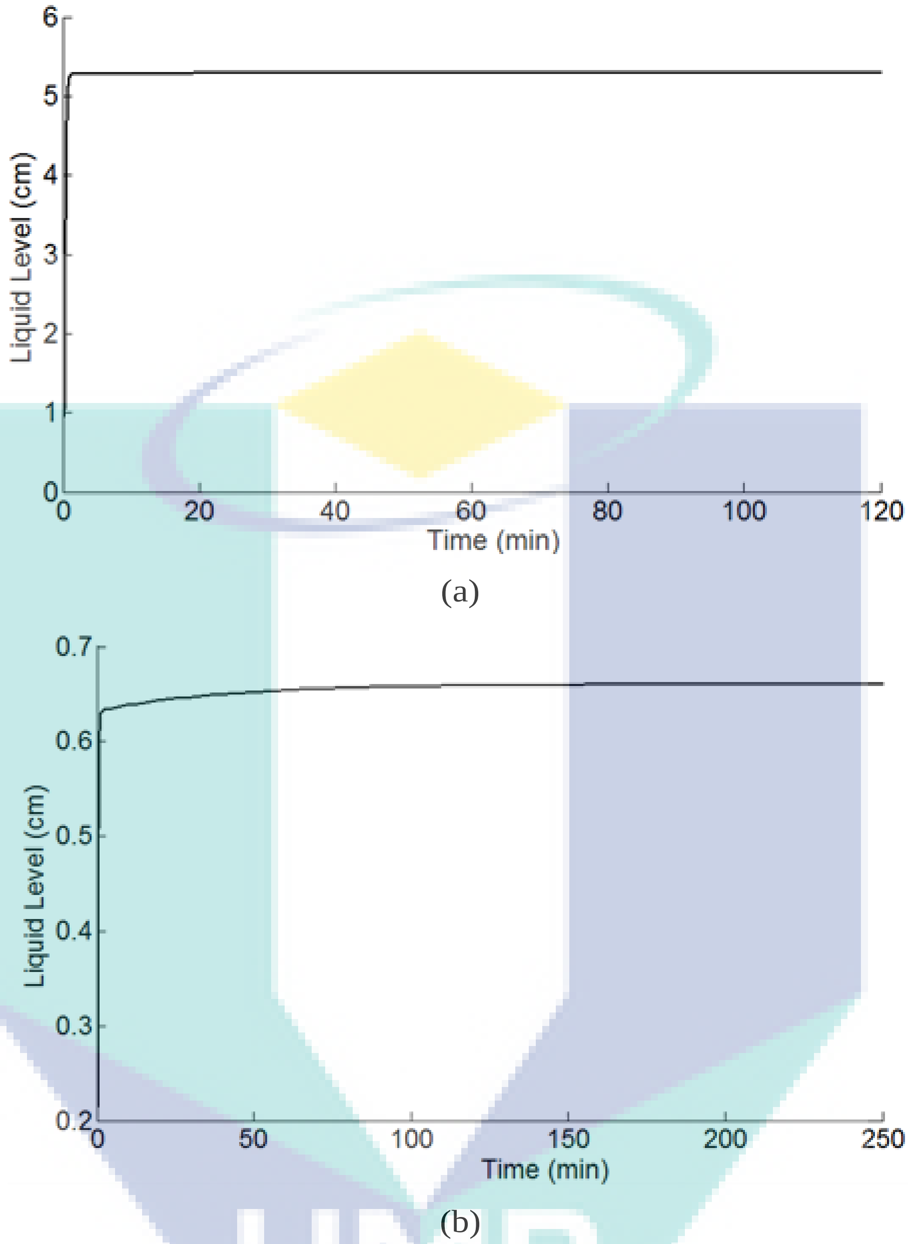


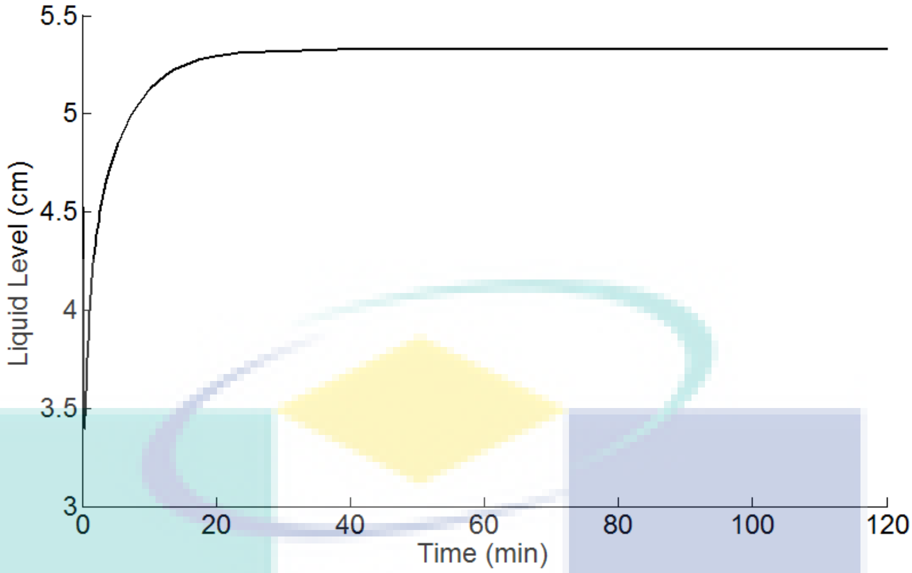

Figure 4.29 PI Controller Liquid Level Profile with Closed Loop ZN Tuning Case (2) (a) Top Vessel (b) Middle Vessel

Liquid levels for both top vessel and middle vessel reaches its set point which is 5.3 cm and 0.66 cm for middle vessel and reboiler respectively as shown in Figure 4.29. The settling time is 60 minutes for middle holdup response and 190 minutes for liquid level response in middle vessel. The rise time for liquid level in top vessel is 5 minutes and for reboiler holdup is 50 minutes. There is no overshoot produced in the liquid level response for both top vessel and middle vessel. Both responses are moving downward at the beginning of operation and increasing towards the set point. This is due to its slight decrease in liquid level during start-up period. The settling time and the rise time for liquid level with ZN-PI is too high. Hence, the IMC tuning is implemented to the PI controller to improve the response of liquid level. The value for proportional and integral gain for IMC-PI controller is stated in Table 4.9 is based on the formula of controller gain mentioned in Table 3.8.

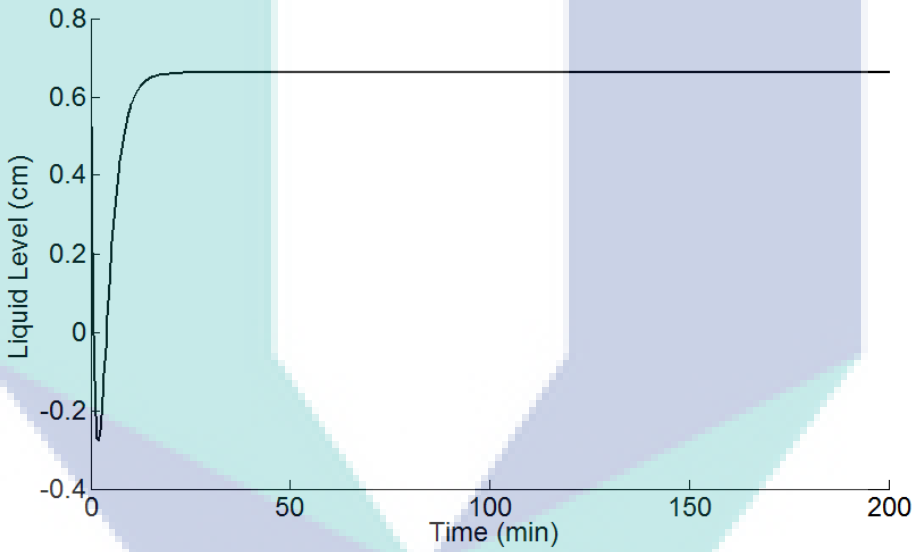
Table 4.10 Controller Gain for IMC Tuning Method for Case (2)

| | **Top Liquid level** ($\tau_c = 0.5$) | **Middle Liquid level** ($\tau_c = 0.4$) |
|---|---|---|
| P | 4.0 | 5.0 |
| I | 1.0 | 0.8 |

The liquid level response for liquid level with IMC-PI controller is shown in Figure 4.27.




(a)




(b)

Figure 4.30 PI Controller Liquid Level Profile with Closed Loop IMC Tuning Case (2) (a) Top Vessel (b) Middle Vessel

According to Figure 4.30, the stable response is obtained with no oscillation. No overshoot is produced for both holdup. The settling time for top vessel liquid level response is less 35 minutes and for middle vessel liquid level response is 25 minutes. The rise time for both liquid level response is less than 8 minutes. As a conclusion, IMC improve the holdup response by reducing the settling time and rise time and eliminating overshoot. This is because IMC possess the advantage of internal model control that has used the characterization of conventional PID controller (Priyadharsini and Lather, 2013).

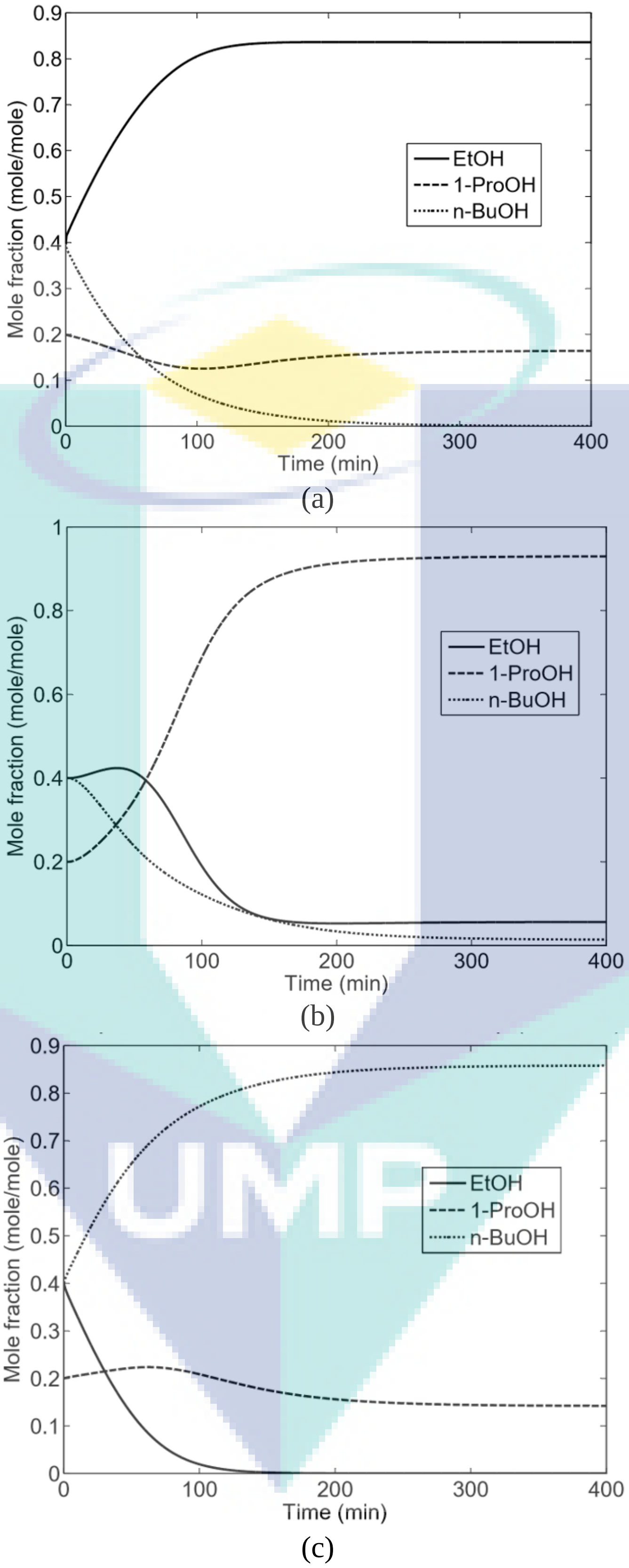



Figure 4.31 Composition Profile for (a) Top Vessel (b) Middle Vessel (c) Reboiler for P Controller Liquid Level with Closed Loop ZN-P Tuning (Case (2))

Based on Figure 4.31, the top product is enriched with ethanol, the middle product is enriched with 1-propanol and bottom product is enriched with n-butanol. The purest product obtained for case (2) is 1-propanol (middle product). The top product and bottom product is successfully removed from middle product. Thus, the objective of case (2) is achieved. The settling time for top product, middle product and bottom product is 180 minutes, 290 minutes and 300 minutes respectively. The composition of the components in each vessel is presented in Table 4.11.

Table 4.11 Composition of Components for Vessels with Closed Loop Tuning P Controller for Level Control (Case (2))

| **Components** | **Composition in Top Vessel** | **Composition in Middle Vessel** | **Composition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.854800 | 0.058440 | 0.000070 |
| 1-Propanol | 0.145000 | 0.928600 | 0.150700 |
| n-Butanol | 0.000292 | 0.013000 | 0.849200 |

Based on Table 4.11, the middle product composition produced is 0.928, the purest among those three components. Meanwhile, the top product composition is 0.855 and bottom product is 0.849. The objective for case (2) which is to produce 0.92 of middle product is achieved. Although the objective is achieved. The liquid level response is unsatisfactory with P controller. The liquid level response is not achieved for both holdup. Hence, the PI controller is implemented. The composition profile for components for liquid level with PI controller is shown in Figure 4.28 (a) - (c).

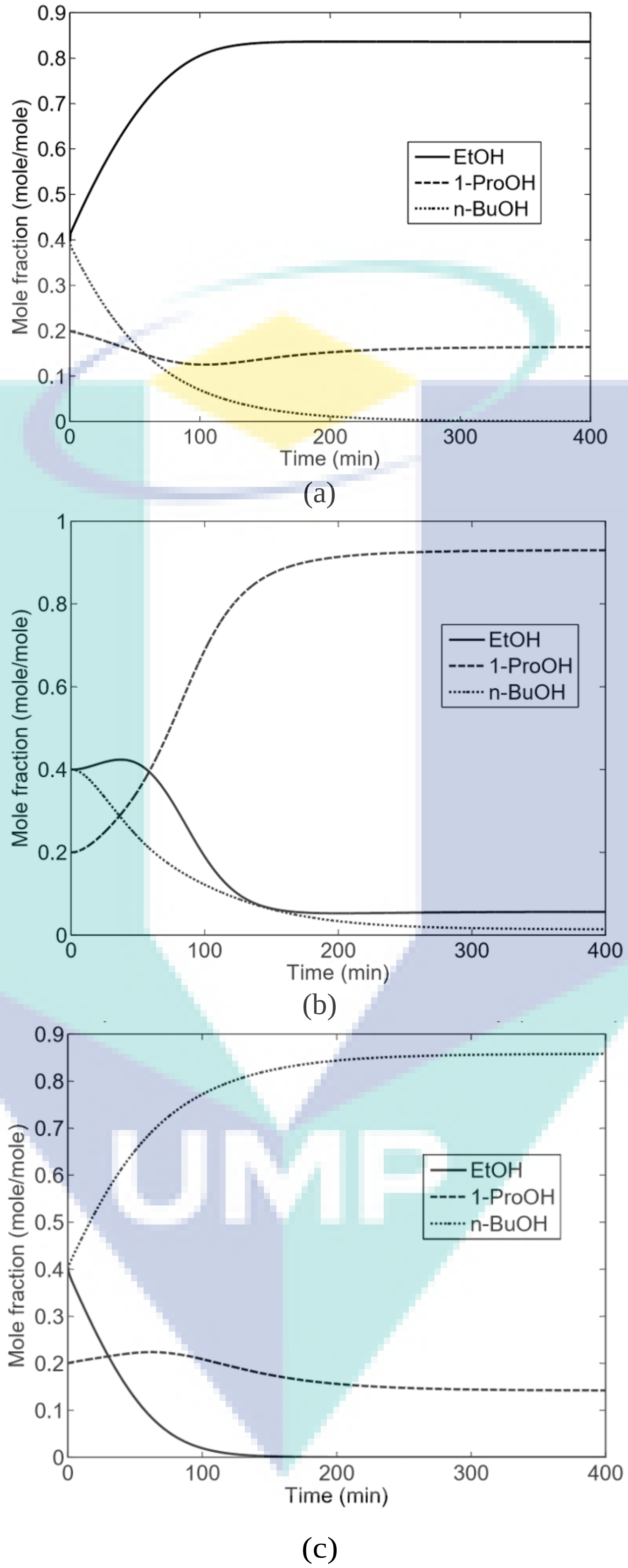



Figure 4.32 Composition Profile for Top Vessel, Middle Vessel and Reboiler for PI Controller Liquid Level with Closed Loop ZN Tuning (Case (2)).

Figure 4.32 (a) - (c) show the composition profile of liquid level with ZN-PI controller. From the Figure 4.32 (a) - (c), the top product is enriched with ethanol, middle product is enriched with 1-propanol and bottom product enriched of n-butanol. The settling time for top product, middle product and bottom product is 180 minutes, 300 minutes and 300 minutes respectively. The composition of product for liquid level with ZN-PI controller is stated in Table 4.12.

Table 4.12 Composition of Components for Vessels with Closed Loop ZN Tuning PI Controller for Level Control (Case (2))

| **Components** | **Composition in Top Vessel** | **Composition in Middle Vessel** | **Composition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.853800 | 0.058020 | 0.000070 |
| 1-Propanol | 0.145900 | 0.928500 | 0.146700 |
| n-Butanol | 0.000296 | 0.013430 | 0.853200 |

The composition of top product is 0.856, the composition of middle product is 0.928 and the composition of bottom product is 0.853 as stated in Table 4.12. The composition of top and middle product remained unchanged similar to liquid level with ZN-P controller shown in Table 4.11. The objective of case (1) to produce 0.92 ethanol is successfully achieved but the holdup response obtained possess high settling time and rise time. Hence, to obtain the smoother response with lower settling time and rise time and higher product purity, IMC-PI controller is applied.

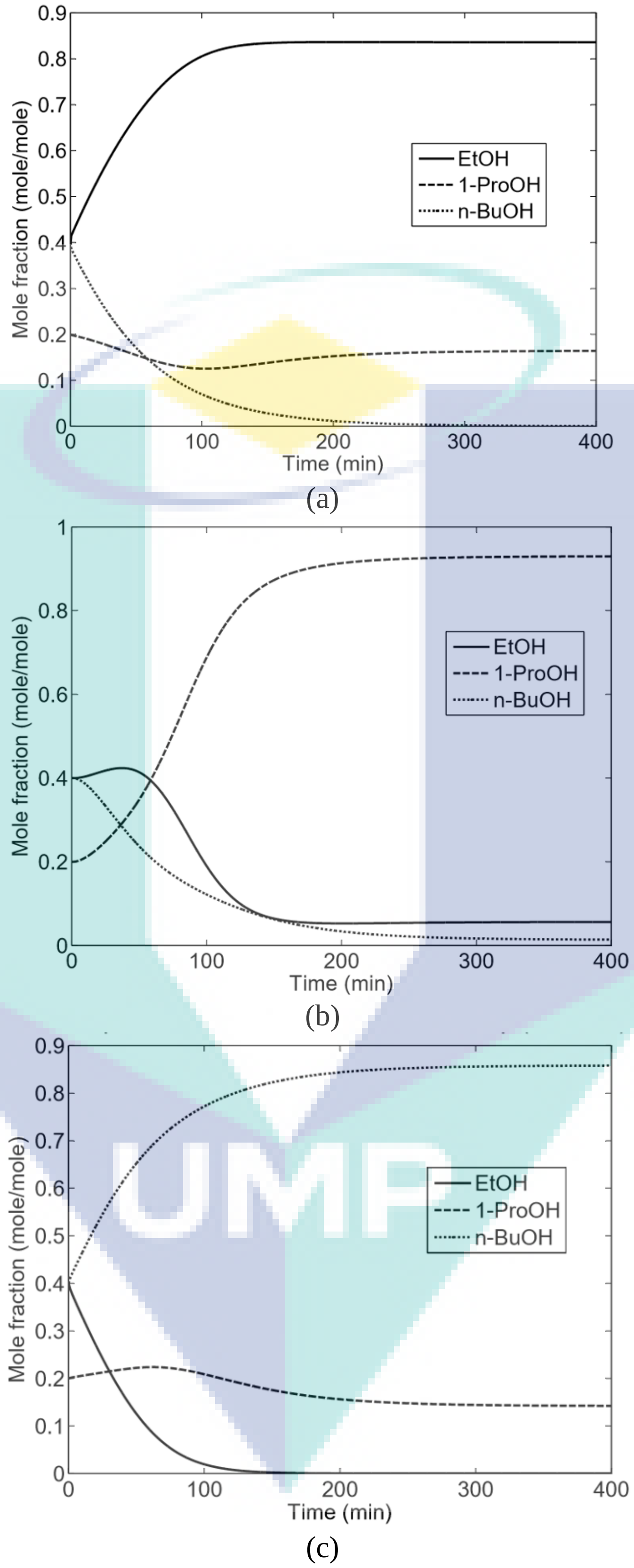



Figure 4.33 Composition Profile for (a) Top Vessel (b) Middle Vessel (c) Reboiler for PI Controller Liquid Level with Closed Loop IMC Tuning (Case (2)).

Based on the composition profile in Figure 4.33 (a) - (c), the settling time of top product is 160 minutes, middle product is 230 minutes and bottom product is 260 minutes. The settling time of the product is decreased compared to the settling time for composition profile in Figure 4.33 (a) - (c). This is because of liquid level with IMC-PI controller produce faster response due to its advantage of internal model characterization (Priyadarshini and Lather, 2013)

Table 4.13 Composition of Components for Vessels with IMC Tuning PI Controller for Level Control (Case (2))

| **Components** | **Composition in Top Vessel** | **Composition in Middle Vessel** | **Composition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.851700 | 0.057160 | 0.000070 |
| 1-Propanol | 0.148000 | 0.929100 | 0.144400 |
| n-Butanol | 0.000299 | 0.013700 | 0.855000 |

Based on the Table 4.13, the middle product (1-propanol) composition for liquid level with IMC-PI controller has increased to almost 0.93. The composition of top product (ethanol) and bottom product (n-butanol) is 0.851 and 0.855 respectively. The result shows a slight decreased of top product composition for liquid level with IMC-PI controller compared to composition of top product with liquid level with ZN-PI controller. IMC-PI controller has improved the response for top vessel and middle vessel liquid level. Additionally, the middle product composition are also improved. From the result, when the composition of middle product is increased the top product composition is also decreased. This situation occurs due to interaction between the loops.

#### 4.2.2.3 Performance Analysis

Under this performance analysis, the controller for both level control is tested. The analysis covers the set point change analysis and disturbance rejection analysis. The intention for this analysis is to analyse the performance of controller, if there is any changes in process condition and unwanted disturbance present (Ishak et al., 2013). The performance analysis is performed for both IMC and ZN tuning method for PI controller. Similar to previous section two cases are considered. Table 4.14 shows the new set point value for each case.

Table 4.14 New Set Point for Liquid level for Each Cases

| | Level in Top Liquid level (cm) | Level in Middle Liquid level (cm) |
|---|---|---|
| Case (1) | 1.56 | 6.36 |
| Case (2) | 6.36 | 0.79 |

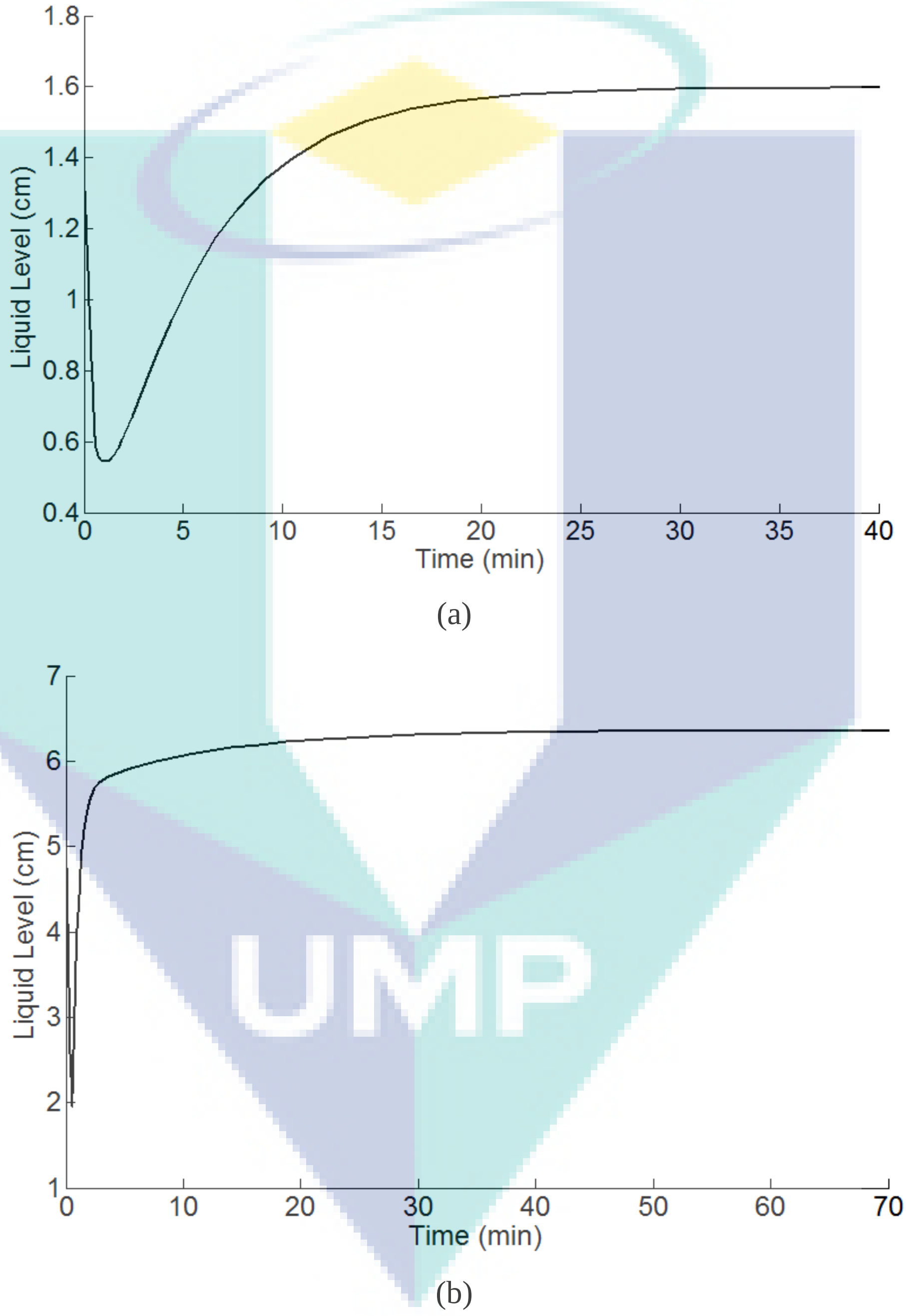
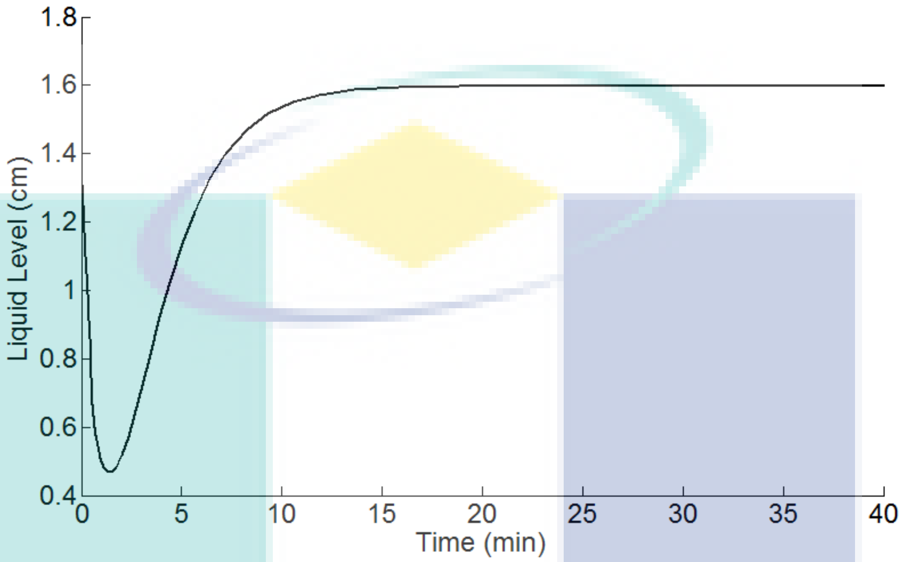
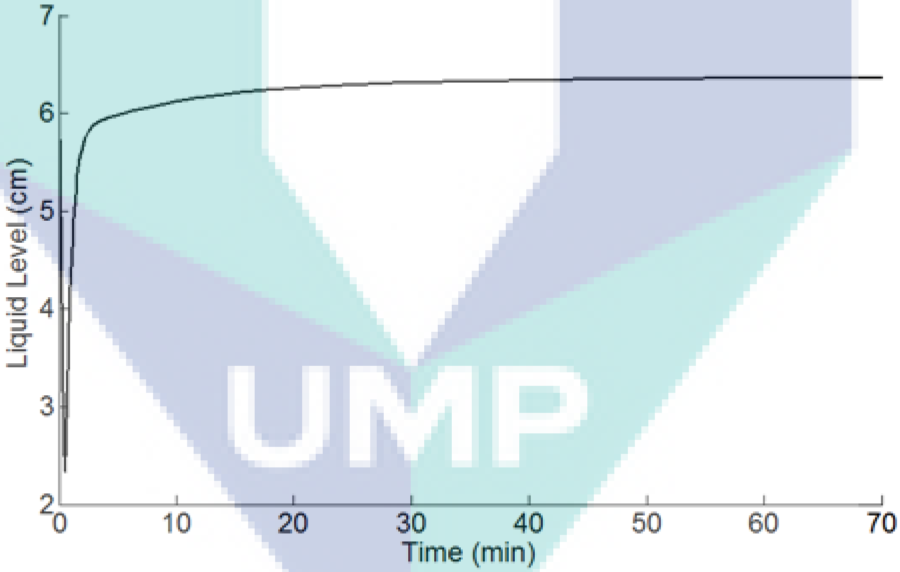



Figure 4.34 PI Controller Liquid Level Profile with Closed Loop ZN Tuning for Set Point Change Case (1) (a) Top Vessel (b) Middle Vessel

Figure 4.34 shows the liquid level with PI controller closed loop ZN tuning for case (1). The liquid level response produced is stable and the new set point for both holdup is achievable. The set points obtained are 1.56 cm and 6.36 cm for top vessel and middle vessel respectively. The set point analysis proceeds with liquid level with IMC-PI controller for case (1).




(a)




(b)

Figure 4.35 PI Controller Liquid Level Profile with Closed Loop IMC Tuning for Set Point Change Case (1) (a) Top Vessel (b) Middle Vessel

Figure 4.35 shows the liquid level with IMC-PI controller. The response obtained is stable and the new set point is achievable for top vessel (1.56 cm) and middle vessel (6.36 cm). The settling time for liquid level with IMC-PI controller is reduced compared to liquid level with ZN-PI controller. The set point change proceeds to case (2)

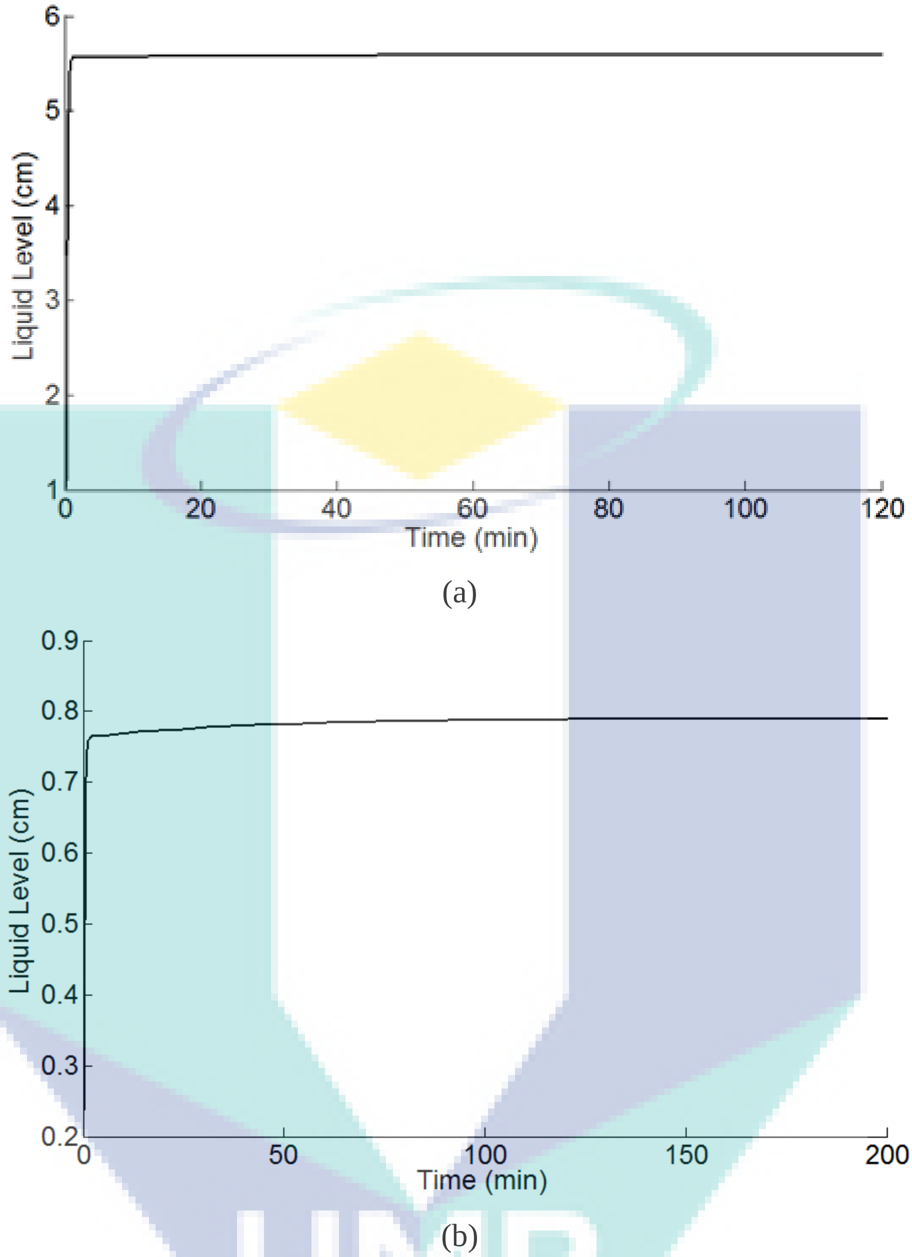
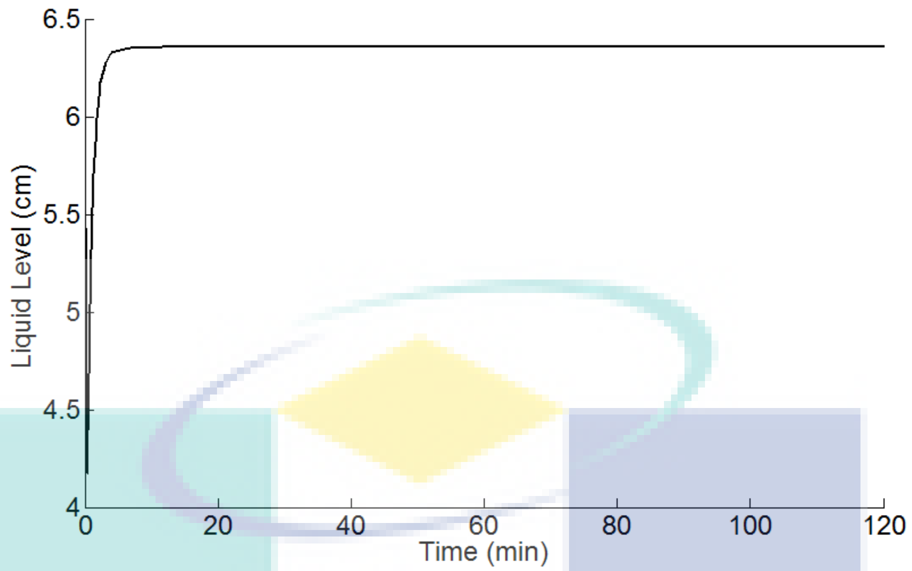
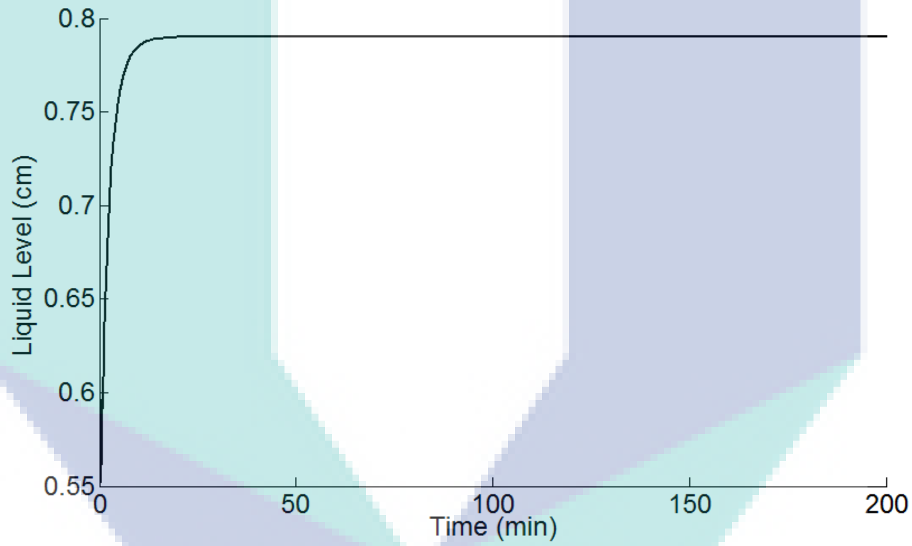



Figure 4.36 PI Controller Liquid Level Profile with Closed Loop ZN Tuning for Set Point Change Case (2) (a) Top Vessel (b) Middle Vessel

The set point for both holdups are achievable for the liquid level with PI controller closed loop ZN tuning. The set points obtained are 0.79 cm and 6.36 cm for top vessel and middle vessel respectively. The response obtained is stable. The set point change for liquid level with IMC-PI controller is shown in Figure 4.36.




(a)

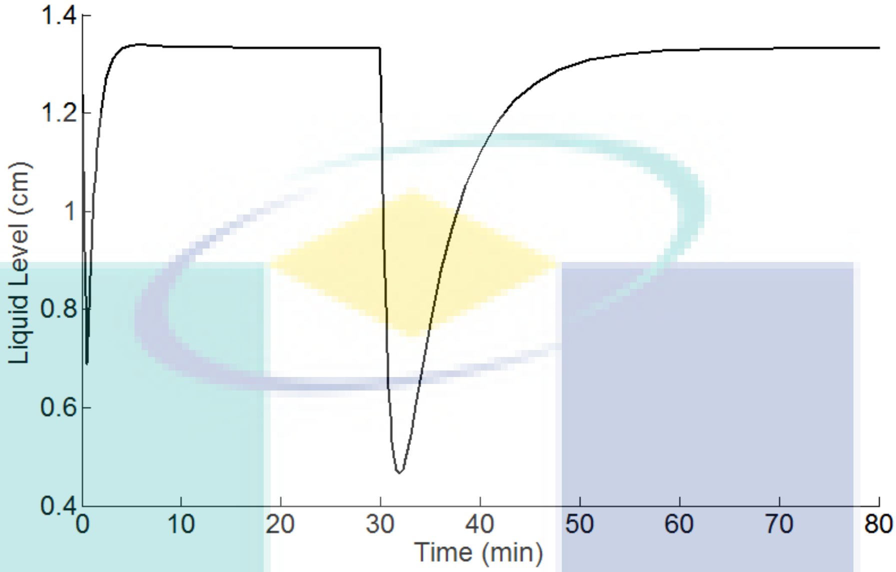



(b)

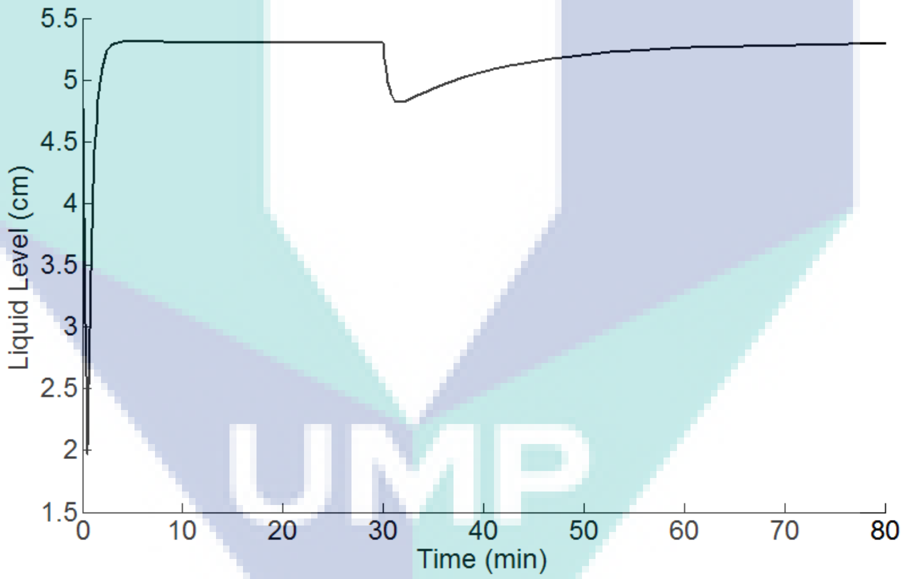
Figure 4.37 PI Controller Liquid Level Profile with Closed Loop IMC Tuning for Set Point Change Case (2) (a) Top Vessel (b) Middle Vessel

Figure 4.37 shows the liquid level with IMC-PI controller. The response obtained is stable and the new set point is achievable for middle vessel 0.79 cm and reboiler 6.36 cm. The settling time for liquid level with IMC-PI controller is shorter compared to liquid level with ZN-PI controller.

From sensitivity analysis, the most affecting variables to the system is middle flow. Hence, the middle flow is chosen as disturbance variables. The middle flow experience sudden decreased about 1 $cm^3$/min after 30 minutes during dynamic operation and the liquid level response in top vessel and middle vessel is analysed. The disturbance rejection analysis is performed on the liquid level with ZN-PI and IMC-PI controller.



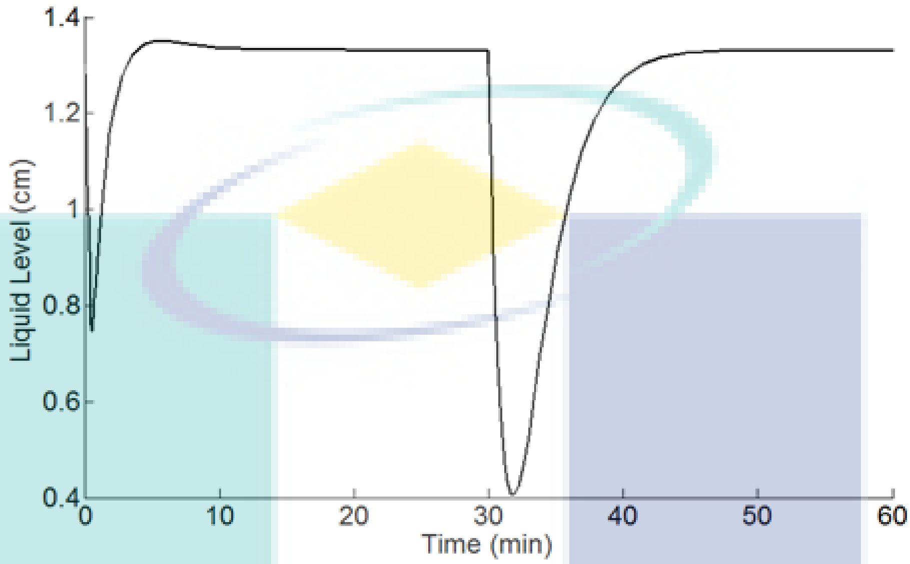

(a)



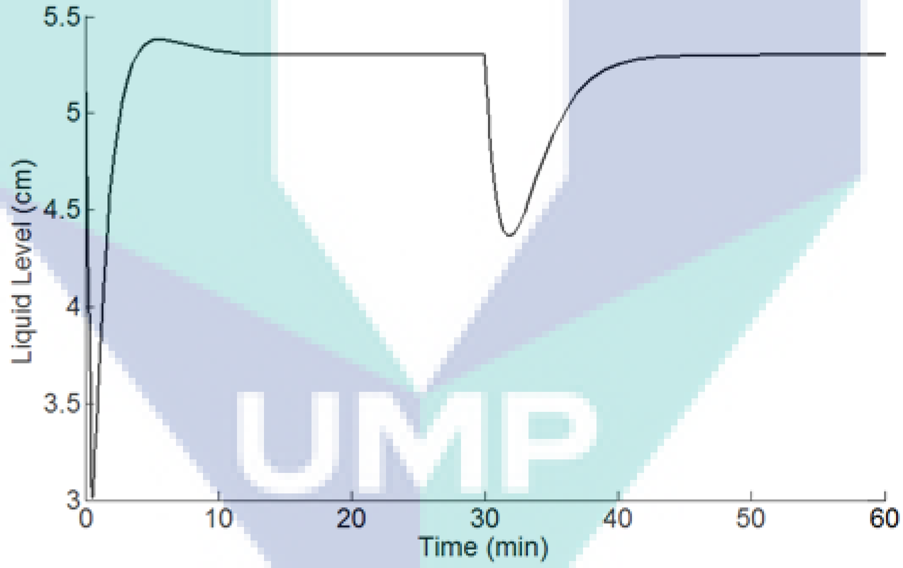

(b)

Figure 4.38 ZN-PI Controller Liquid Level Profile for Disturbance Rejection Case (1) (a) Top Vessel (b) Middle Vessel

For liquid level with PI controller, the controller gain from ZN tuning is applied. The set point is successfully achieved for both vessels. No overshoot is produced in both holdup response. The liquid level response is suddenly decreased after the disturbance is introduced. However, the ZN-PI controller successfully bring the liquid level response in top vessel to its set points immediately after 25 minutes the disturbance is introduced. While, in middle vessel, the liquid level response return to its set point after 30 minutes the disturbance is introduced. The time taken for response to its set point is quite longer. Hence, the disturbance rejection is proceed with IMC-PI controller. The liquid level with IMC- PI controller for case (1) is shown in Figure 4.37.



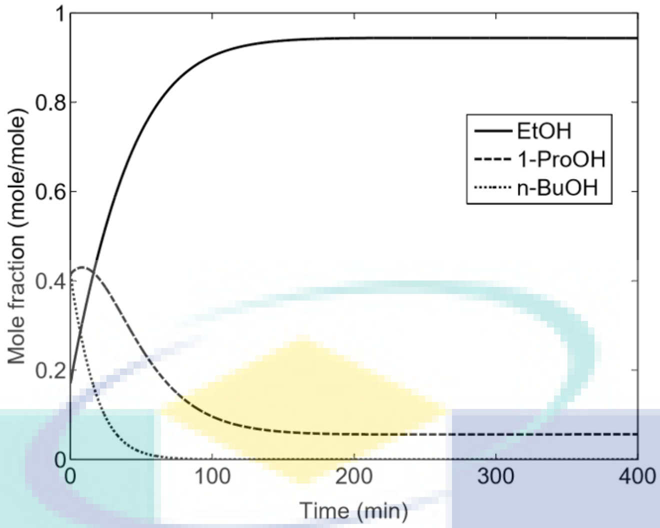

(a)



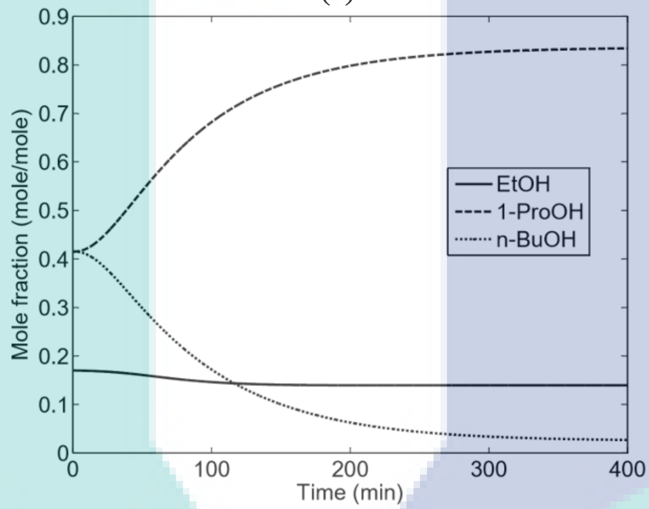

(b)

Figure 4.39 IMC-PI Controller Liquid level Profile for Disturbance Rejection Case (1)

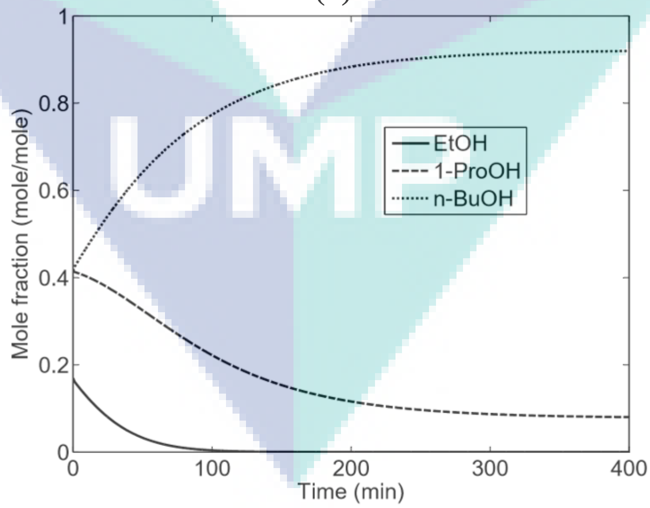
For liquid level with IMC-PI controller, the set point has successfully achieved for top vessel and middle vessel. There is small overshoot in both liquid level response that is 1.50% (1.35 cm) for top vessel and 1.56% (5.38 cm) for middle vessel. The response is stable. The settling time is shorter for liquid level with IMC-PI controller compared to liquid level with ZN-PI controller. The liquid level response immediately return to its set point after the disturbance is introduced. Liquid level response in top vessel take about 12 minutes to return to the set point and liquid level response in middle vessel take about 10 minutes to return its set point after disturbance is introduced. The IMC-PI controller has not even rejected the disturbance coming to the system, the time take for the response return to its set point is shorter compared to ZN-PI controller. The composition profile for disturbance rejection with ZN-PI is shown in Figure 4.36 (a) - (c).




(a)




(b)




(c)

Figure 4.40 Composition Profile for (a) Top Vessel (b) Middle Vessel (c) Reboiler for ZN Tuning PI Controller Liquid Level for Disturbance Rejection (Case (1))

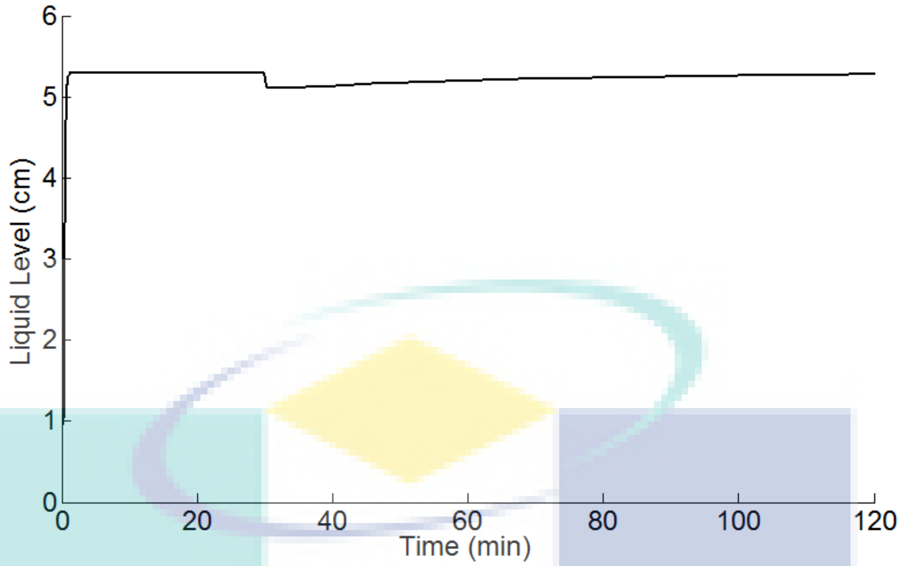
The composition profile for liquid level with IMC-PI and ZN-PI controller after disturbance produced shows similar trending as previous liquid level with IMC-PI and ZN-PI controller with closed loop tuning (without disturbance). Due to above matter, composition profile with ZN-PI controller liquid level is shown. The settling time and the composition of the component for both PI controller is stated in Table 4.15 and 4.16. The composition of top product, middle product and bottom product slightly changes. The settling time is also slightly higher when compared to the system without the presence of disturbance. However, IMC-PI controller produces lower settling time for composition profile and composition of top product is also higher compared to the composition profile for liquid level with ZN-PI controller. Both ZN-PI and IMC-PI controller successfully rejects disturbance to the system (Rao et al, 2014). However, IMC-PI give better controller performance by reducing the settling time and improving the product composition.

Table 4.15 Composition and Settling Time of Components for Vessels with Closed Loop ZN Tuning PI Controller for Level Control (Case (1)) (Disturbance Rejection)

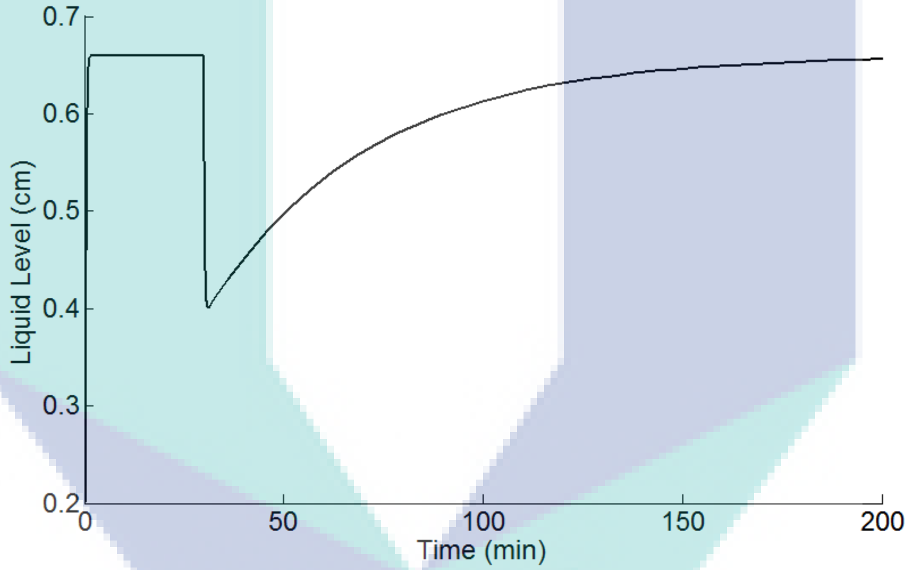
| **Components** | **Composition in Top Vessel** | **Composition in Middle Vessel** | **Compsoition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.942600 | 0.137100 | 0.000107 |
| 1-Propanol | 0.057420 | 0.835500 | 0.083160 |
| n-Butanol | 0.000006 | 0.027400 | 0.918500 |
| Settling Time (minutes) | 200 | 320 | 320 |

Table 4.16 Composition and Settling Time of Components for Vessels with Closed Loop IMC Tuning PI Controller for Level Control (Case (1)) (Disturbance Rejection)

| **Components** | **Composition in Top Vessel** | **Composition in Middle Vessel** | **Composition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.947000 | 0.147300 | 0.000129 |
| 1-Propanol | 0.052990 | 0.830600 | 0.872300 |
| n-Butanol | 0.000005 | 0.022050 | 0.912600 |
| Settling Time (minutes) | 190 | 300 | 300 |



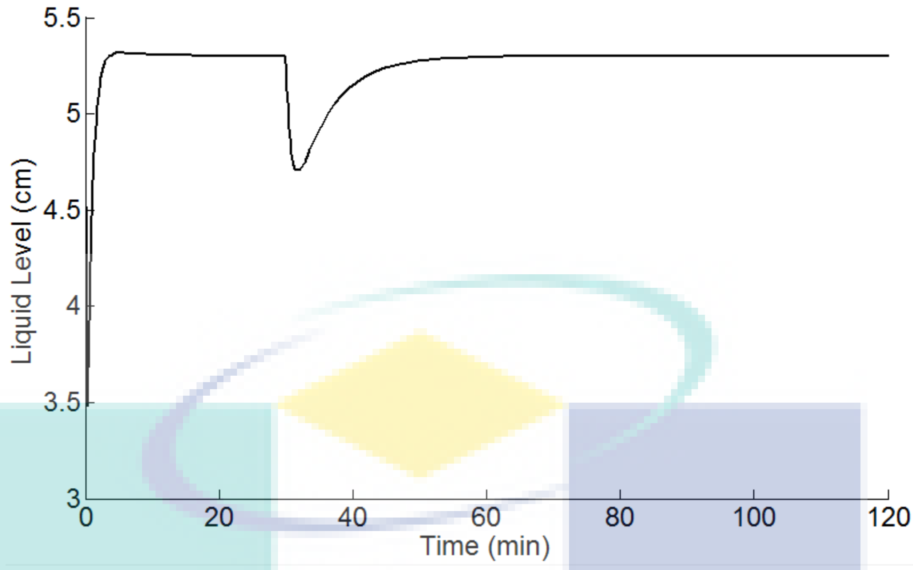

(a)



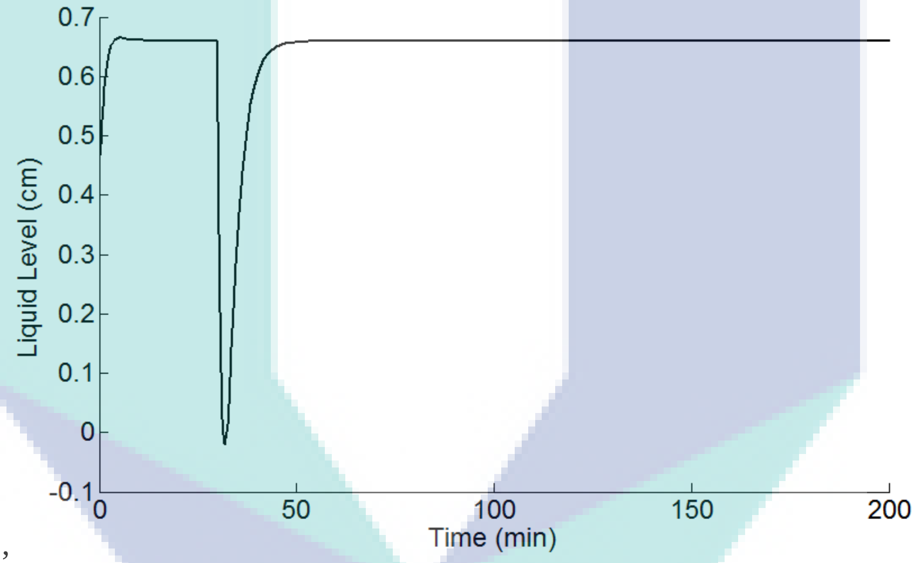

(b)

Figure 4.41 ZN-PI Controller Liquid Level Profile for Disturbance Rejection Case (2)

Based on the liquid level profile in Figure 4.41, the liquid level response obtained for top vessel and middle vessel reaches set point. After disturbance is introduced during 30 minutes, the liquid level response decreased suddenly. However, the ZN-PI controller successfully push the response to its set point. For top vessel, it take 50 minutes for liquid level response to retain to its set point. Meanwhile, for middle vessel, it take 60 minutes for liquid level response to retain to its set point. The composition profile for all vessels for liquid level with PI controller is shown in Figure 4.39 (a) - (c).




(a)

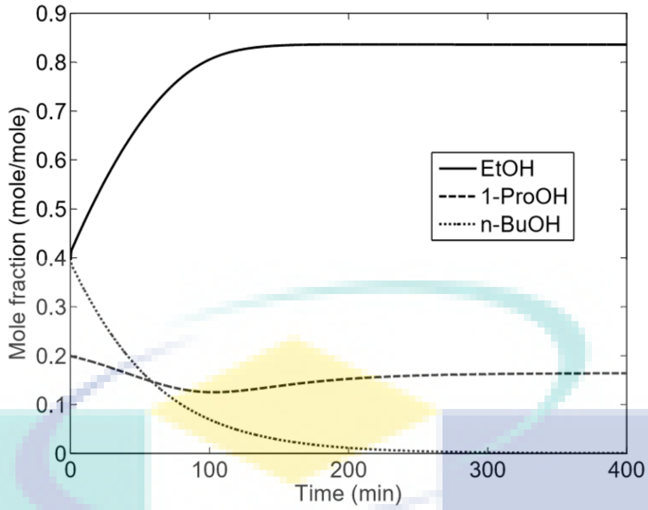



,

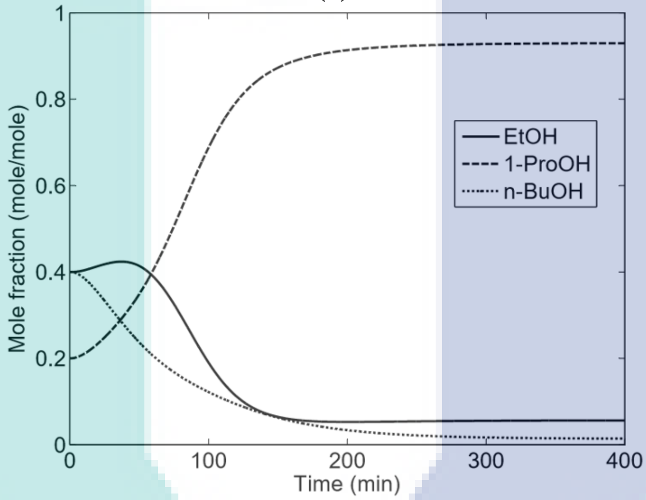
(b)

Figure 4.42 IMC-PI Controller Liquid level Profile for Disturbance Rejection Case (2)

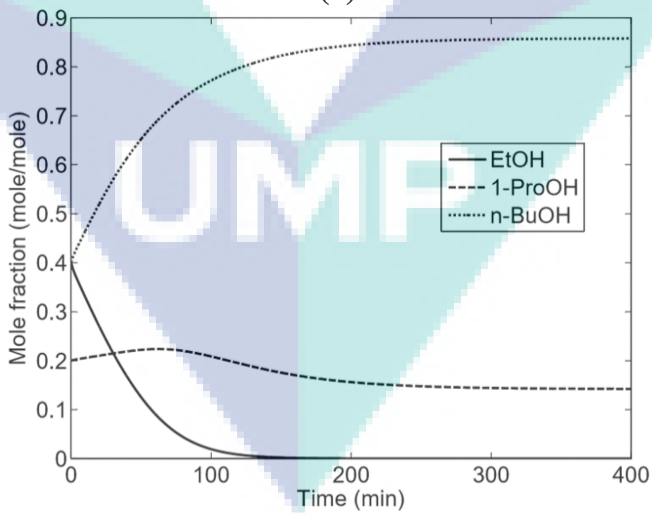
Figure 4.42 shows the disturbance rejection for liquid level with IMC-PI controller. The set point has successfully achieved for both vessel. The disturbance is introduced after 30 minutes, the middle flow is decreased about 0.7 $cm^3$/min. After the disturbance is introduced, the liquid level response in the top vessel decreased and the IMC-PI controller immediately push the response to its set point. For liquid level in top vessel, IMC-PI controller take 25 minutes to bring the response to set point and for liquid level in middle vessel, IMC-PI controller take 20 minutes to push the response to set point. There is no overshoot in both liquid level response. The response is stable. The IMC-PI controller successfully rejects the disturbance coming to the system.




(a)




(b)




(c)

Figure 4.43 Composition Profile for (a) Top Vessel (b) Middle Vessel (c) Reboiler for ZN tuning PI Controller Liquid level for Disturbance Rejection (Case (2))

Based on Figure 4.43, the composition profile of top product, middle product and bottom product remains constant. The composition profile for liquid level with both PI controller for case (2) after disturbance present shows similar product composition trending compared to previous liquid level with PI controller with IMC-PI and ZN-PI closed loop tuning (without disturbance). Due to the above matter, composition profile with ZN-PI controller liquid level is shown. The settling time and the composition of the component for both PI controller is stated in Table 4.17 and 4.18 Although, the composition of middle product is decreased when the disturbance is injected to the system, liquid level with IMC-PI controller successfully produces lower settling time for composition profile and produces higher product composition compared to the liquid level with ZN-PI controller. From the results shown for set point changes analysis and disturbance analysis, it shows that for both cases the IMC-PI controller is good enough to control the MVBDC system based on their performance and stability. IMC-PI controller is able to react and reject the disturbance and ensure controlled variable always is at its desired set point. Moreover, the IMC tuning method is straightforward and easy to implement. The IMC–PID tuning rule is directly related to the closed–loop time constant and has only one user defined tuning parameter (Lee et al, 2008)

Table 4.17 Composition and Settling Time of Components for Vessels with Closed Loop ZN Tuning PI Controller for Level Control (Case (2)) (Disturbance Rejection)

| **Components** | **Composition in Top Vessel** | **Composition in Middle Vessel** | **Composition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.855000 | 0.058510 | 0.000070 |
| 1-Propanol | 0.144700 | 0.928200 | 0.148200 |
| n-Butanol | 0.000305 | 0.013290 | 0.851700 |
| Settling Time (minutes) | 180 | 280 | 300 |

Table 4.18 Composition and Settling Time of Components for Vessels with Closed Loop IMC Tuning PI Controller for Level Control (Case (2)) (Disturbance Rejection)

| **Components** | **Composition in Top Vessel** | **Composition in Middle Vessel** | **Composition in Reboiler** |
|---|---|---|---|
| Ethanol | 0.852400 | 0.057430 | 0.000070 |
| 1-Propanol | 0.147300 | 0.928900 | 0.145100 |
| n-Butanol | 0.000313 | 0.013650 | 0.854800 |
| Settling Time (minutes) | 170 | 250 | 270 |

## 4.3 Summary of Result and Discussion

In this chapter, simulation of MVBDC based on mathematical model has been developed in chapter 3 and the controllability study has been performed. As a result, two level controls are chosen and confirmed by sensitivity analysis. Reflux flow – top vessel liquid level and bottom flow – middle vessel liquid level are chosen as best pairings based on the RGA and SVD analysis with condition number 1.18. The conventional controller is implemented to these two level controls. In order to obtain the best controller performance the IMC-PI controller is used. IMC-PI controller reduce the settling time and rise time of liquid level response. The control system implemented to this MVBDC is stable and gives good performance since it can react very well to the set point changes and unwanted disturbance.

# CHAPTER 5

# CONCLUSION AND RECOMMENDATION

In this chapter, the conclusion and recommendations based on the controllability study of multi-vessel batch distillation column are presented.

## 5.1 Conclusion

1. Mathematical model is developed based on first principles including total mass balance and component balance. All the model equations are solved simultaneously in MATLAB Simulink. The model is validated based on the secondary data by Hisyam (2011). The simulation result from Simulink shows good agreement with simulation and experimental data from secondary data. Instead of degree of freedom analysis, the controlled variables, manipulated variables and disturbance variables can be identified by performing sensitivity analysis. Besides, sensitivity analysis helps to determine the set point of top vessel and middle vessel level holdup for case (1) is 1.33 cm and 5.3 cm respectively and case (2) is 5.3 cm and 0.66 cm respectively. Pure top product (0.940 ethanol) is targeted to produce for case (1) and pure middle product (0.920 1-propanol) is successfully produced for case (2). Based on sensitivity analysis, vessel holdup will influence the final product composition. Hence, the level control chosen for both top vessel and middle vessel level holdup will indirectly control the product composition. In consequence of no heat balance is considered in the model development, the disturbance is decided based on the available variables from mass and component balance, which mostly effects the controlled variable directly and indirectly. The disturbance variable is the middle flow. Interaction analysis based on relative gain array (RGA) calculation shows that a few possible pairings can be chosen such as ($CV_1$ – $MV_2$), ($CV_1$ – MV3), ($CV_2$ – $MV_1$),

$(CV_2 - MV_3)$ and $(CV_1 - MV_3)$ and $(CV_3 - MV_2)$ as the best manipulated and controlled variable pairing. SVD analysis is performed to determine the best pairing. $(CV_1 - MV_1)$, level in top vessel level holdup – reflux flow and $(CV_2 - MV_3)$, level in middle vessel level holdup – bottom flow is chosen as the best pairings with lowest condition number 1.185. Dynamic analysis shows that the process is linear. Hence, linear controllers such as proportional (P) only, proportional integral (PI) and proportional integral derivative (PID) are sufficient enough to control the system.

2. In order to obtain the best controller response, the controller must be tuned to suitable gain value. In this study, open loop and closed loop tuning are performed based on Ziegler Nichols (ZN) tuning method and Internal Model Control (IMC) tuning method. Based on the simulation result, it shows that the P controller based on ZN tuning closed loop method cannot force the level for both holdup to its set point. Due to limitation of the P controller. Hence, the controller tuning is proceeded to PI controller based on ZN closed loop tuning method. Thus, the controller response for top vessel and middle vessel level holdup for both cases (1) and case (2) is improved. However, the settling time and the rise time obtained is too high. The composition of top product (0.943 ethanol) for case (1) and middle product (0.928 1-propanol) for case (2) is improved. In order to reduce the settling time and the rise time produced in both holdup, the IMC tuning is performed. As a result, the faster response obtained with lower settling time and rise time. In addition, the quality of final product composition increases and settling time of composition also decreases. Thus, the PI controller with IMC tuning is the best for middle and reboiler level control for both cases in order to obtain the best controller response. Finally, set point changes and disturbance changes analysis are performed in order to analyse the controller performance. PI controller with IMC tuning gives the best performance for top vessel level holdup and middle vessel level holdup control on both cases due to lowest settling time and higher product purity produced. The response obtained is stable for disturbance rejection analysis even though the disturbance is introduced to the system and for the set point changes analysis the controller successfully brings the level control to its new set point. As a conclusion, the PI level control with IMC tuning is able to ensure the performance and its stability.

## 5.2 Recommendations

Based on the study presented in this thesis, there are a few possible research areas that could be further extended in future, these are listed as follow:

1. Controllability study on the other control structures studied by previous researcher on multi-vessel batch distillation column such as temperature control.
2. Decoupling of two level control structures in multi-vessel batch distillation system.
3. Addition of temperature control to the multi-vessel batch distillation column with two level control system and study their interaction in details.

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# APPENDIX A

# M-FILE CODE FOR SIMULINK MODEL

## A.1 M-file code of main program (newproject.m)

```
function [sys,x0] = multivess3comp(t,x,u,flag)

PH=1;
PB=1.25;
NT=7;
NT1=14;
for nt=1:NT;
  P(nt)=PH+((nt).*(PB-PH))./NT;
end
PM=1;
for nt=8:NT1;
  P(nt)=PH+((nt).*(PB-PH))./NT1  ;
end

H=100;
M=400;
B=400;
Hs=1;

V=10;
L=V;

%Comp Ethanol=1; 1-propanol=2; n_butanol=3;
xawal=[0.17 0.415 0.415];

switch flag,
case 1, %persamaan diff

 %===============Liquid komposisi
```

```
%di H
x1H=x(1);
x2H=x(2);
x3H=x(3);
%di stage N=1:7
for n=1:7
    x1(n)=x((n*3)+1);
    x2(n)=x((n*3)+2);
    x3(n)=x((n*3)+3);
end
%di M
x1M=x(25);
x2M=x(26);
x3M=x(27);
%di N=8:14
for N=1:7
    x1(7+N)=x(((8+N)*3)+1);
    x2(7+N)=x(((8+N)*3)+2);
    x3(7+N)=x(((8+N)*3)+3);
end
%di B
x1B=x(49);
x2B=x(50);
x3B=x(51);

for ntb = 1:14,
        [y1(ntb),y2(ntb),y3(ntb),T(ntb)]=bublTi(x1(ntb),x2(ntb),x3(ntb),P(ntb));
end
[y1B,y2B,y3B,TB]=bublTi(x1B,x2B,x3B,PB);

dxTdt = [(L/H)*(y1(1)-x1H),... %di H  dx1Hdt =x(1)
         (L/H)*(y2(1)-x2H),... %      dx2Hdt =x(2)
         (L/H)*(y3(1)-x3H),... %      dx3Hdt =x(3)
(1/Hs)*(V*(y1(2)-y1(1))+L*(x1H-x1(1))),... %      dx11dt=x(4)  %di tray 1
```

```
(1/Hs)*(V*(y2(2)-y2(1))+L*(x2H-x2(1))),... %     dx21dt=x(5)
(1/Hs)*(V*(y3(2)-y3(1))+L*(x3H-x3(1))),... %     dx31dt=x(6)
(1/Hs)*(V*(y1(3)-y1(2))+L*(x1(1)-x1(2))),... %   dx12dt=x(7)%di tray 2: N
(1/Hs)*(V*(y2(3)-y2(2))+L*(x2(1)-x2(2))),... %   dx22dt=x(8)
(1/Hs)*(V*(y3(3)-y3(2))+L*(x3(1)-x3(2))),... %   dx32dt=x(9)
(1/Hs)*(V*(y1(4)-y1(3))+L*(x1(2)-x1(3))),... %   dx13dt=x(10)
(1/Hs)*(V*(y2(4)-y2(3))+L*(x2(2)-x2(3))),... %   dx23dt=x(11)
(1/Hs)*(V*(y3(4)-y3(3))+L*(x3(2)-x3(3))),... %   dx33dt=x(12)
(1/Hs)*(V*(y1(5)-y1(4))+L*(x1(3)-x1(4))),... %   dx14dt=x(13)
(1/Hs)*(V*(y2(5)-y2(4))+L*(x2(3)-x2(4))),... %   dx24dt=x(14)
(1/Hs)*(V*(y3(5)-y3(4))+L*(x3(3)-x3(4))),... %   dx34dt=x(15)
(1/Hs)*(V*(y1(6)-y1(5))+L*(x1(4)-x1(5))),... %   dx15dt=x(16)
(1/Hs)*(V*(y2(6)-y2(5))+L*(x2(4)-x2(5))),... %   dx25dt=x(17)
(1/Hs)*(V*(y3(6)-y3(5))+L*(x3(4)-x3(5))),... %   dx35dt=x(18)
(1/Hs)*(V*(y1(7)-y1(6))+L*(x1(5)-x1(6))),... %   dx16dt=x(19)
(1/Hs)*(V*(y2(7)-y2(6))+L*(x2(5)-x2(6))),... %   dx26dt=x(20)
(1/Hs)*(V*(y3(7)-y3(6))+L*(x3(5)-x3(6))),... %   dx36dt=x(21)
(1/Hs)*(V*(y1(8)-y1(7))+L*(x1(6)-x1(7))),... %   dx17dt=x(22)
(1/Hs)*(V*(y2(8)-y2(7))+L*(x2(6)-x2(7))),... %   dx27dt=x(23)
(1/Hs)*(V*(y3(8)-y3(7))+L*(x3(6)-x3(7))),... %   dx37dt=x(24)
(L/M)*(x1(7)-x1M),... %   dx1Mdt=x(25)%di M
(L/M)*(x2(7)-x2M),... %   dx2Mdt=x(26)
(L/M)*(x3(7)-x3M),... %   dx3Mdt=x(27)
(1/Hs)*(V*(y1(9)-y1(8))+L*(x1M-x1(8))),... %   dx18dt=x(28)%di N+1 :
(1/Hs)*(V*(y2(9)-y2(8))+L*(x2M-x2(8))),... %   dx28dt=x(29)
(1/Hs)*(V*(y3(9)-y3(8))+L*(x3M-x3(8))),... %   dx38dt=x(30)
(1/Hs)*(V*(y1(10)-y1(9))+L*(x1(8)-x1(9))),... %   dx19dt=x(31)
(1/Hs)*(V*(y2(10)-y2(9))+L*(x2(8)-x2(9))),... %   dx29dt=x(32)
(1/Hs)*(V*(y3(10)-y3(9))+L*(x3(8)-x3(9))),... %   dx39dt=x(33)
(1/Hs)*(V*(y1(11)-y1(10))+L*(x1(9)-x1(10))),... %   dx110dt=x(34)
(1/Hs)*(V*(y2(11)-y2(10))+L*(x2(9)-x2(10))),... %   dx210dt=x(35)
(1/Hs)*(V*(y3(11)-y3(10))+L*(x3(9)-x3(10))),... %   dx310dt=x(36)
(1/Hs)*(V*(y1(12)-y1(11))+L*(x1(10)-x1(11))),... %   dx111dt=x(37)
(1/Hs)*(V*(y2(12)-y2(11))+L*(x2(10)-x2(11))),... %   dx211dt=x(38)
```

```
(1/Hs)*(V*(y3(12)-y3(11))+L*(x3(10)-x3(11))),... %    dx311dt=x(39)
(1/Hs)*(V*(y1(13)-y1(12))+L*(x1(11)-x1(12))),... %    dx112dt=x(40)
(1/Hs)*(V*(y2(13)-y2(12))+L*(x2(11)-x2(12))),... %    dx212dt=x(41)
(1/Hs)*(V*(y3(13)-y3(12))+L*(x3(11)-x3(12))),... %    dx312dt=x(42)
(1/Hs)*(V*(y1(14)-y1(13))+L*(x1(12)-x1(13))),... %    dx113dt=x(43)
(1/Hs)*(V*(y2(14)-y2(13))+L*(x2(12)-x2(13))),... %    dx213dt=x(44)
(1/Hs)*(V*(y3(14)-y3(13))+L*(x3(12)-x3(13))),... %    dx313dt=x(45)
(1/Hs)*(V*(y1B-y1(14))+L*(x1(13)-x1(14))),... %    dx114dt=x(46)
(1/Hs)*(V*(y2B-y2(14))+L*(x2(13)-x2(14))),... %    dx214dt=x(47)
(1/Hs)*(V*(y3B-y3(14))+L*(x3(13)-x3(14))),... %    dx314dt=x(48)
(L/B)*(x1(14)-y1B),... %    dx1Bdt=x(49)%di B
(L/B)*(x2(14)-y2B),... %    dx2Bdt=x(50)
(L/B)*(x3(14)-y3B)]; %    dx3Bdt=x(51)

sys = dxTdt;
case 3, % outputnya
x1H=x(1); x2H=x(2); x3H=x(3);
x11=x(4);x21=x(5);x31=x(6);
x12=x(7);x22=x(8);x32=x(9);
x13=x(10);x23=x(11);x33=x(12);
x14=x(13);x24=x(14);x34=x(15);
x15=x(16); x25=x(17); x35=x(18);
x16=x(19);x26=x(20);x36=x(21);
x17=x(22);x27=x(23);x37=x(24);
x1M=x(25); x2M=x(26); x3M=x(27);
x18=x(28);x28=x(29);x38=x(30);
x19=x(31);x29=x(32);x39=x(33);
x110=x(34);x210=x(35);x310=x(36);
x111=x(37);x211=x(38);x311=x(39);
x112=x(40); x212=x(41); x312=x(42);
x113=x(43);x213=x(44);x313=x(45);
x114=x(46);x214=x(47);x314=x(48);
x1B=x(49); x2B=x(50); x3B=x(51);
sys =[x1H x2H x3H ...
```

```
    x11 x21 x31 ...
    x12 x22 x32 ...
    x13 x23 x33 ...
    x14 x24 x34 ...
    x15 x25 x35 ...
    x16 x26 x36 ...
    x17 x27 x37 ...
    x1M x2M x3M ...
    x18 x28 x38 ...
    x19 x29 x39 ...
    x110 x210 x310 ...
    x111 x211 x311 ...
    x112 x212 x312 ...
    x113 x213 x313 ...
    x114 x214 x314 ...
    x1B x2B x3B ];

case 0, %

NumContStates = 51; NumOutputs = 51; NumInputs = 0;
sys = [NumContStates,0,NumOutputs,NumInputs,0,0];
x0 = [xawal...
      xawal xawal xawal xawal xawal xawal xawal ...
      xawal ...
      xawal xawal xawal xawal xawal xawal xawal ...
      xawal];
case { 2, 4, 9 },
sys = [];
Otherwise
% error(['Unhandled flag = ',num2str(flag)]);
end


function [y1, y2 ,y3 ,t] = bublTi(x1,x2,x3,P)
```

```
xi=[x1 x2 x3];
tisat=tsati(P);
iter = 1;
max=100;
g=ones(1,3);
tet=ones(1,3);
T(1)=sum(xi.*tisat);
pis=psati(T(1));
%misal j species 2
ps=sum(((xi.*g)./tet).*(pis/pis(2)));
psat2(1)=P/ps;
  f=((xi.*g).*pis)./(tet*P);
%mulai loop
for j=2:max
    T(j)=tsat2(psat2(j-1));
    pis=psati(T(j));
    %g=feval('gammai',xi,T(j));
    %tet=feval('tetai',f,T(j),P);
    f=((xi.*g).*pis)./(tet*P);
    ps=sum(((xi.*g)./tet).*(pis/pis(2)));
    psat2(j)=P/ps;
    if abs(T(j) - T(j-1)) < 0.02
        break;
    end
  iter =j;
end
if (iter >= max)
    disp('berhenti');
end

a=sum(f);
b=f/a;
y1=b(1);
y2=b(2);
```

```
y3=b(3);
t=T(j);

function tsat =tsati(P)
%P=2;
%mencari tsat comp 1, ethanol
c  =[4.92531   1432.526      -61.819;
    4.87601    1441.629      -74.299;
    4.54607    1351.555      -93.34];
A=c(:,1);
B=c(:,2);
C=c(:,3);
tsat=B./(A-log10(P))-C;
tsat=tsat';

function tsat =tsat2(P)
 %mencari tsat comp 2, 1-propanol
 c=[4.87601   1441.629      -74.299];
 A=c(1);
 B=c(2);
 C=c(3);
 tsat=B/(A-log10(P))-C;

function pisat= psati(t)
%t=350;
%antoine coef P(bar) T (K)
c  =[4.92531   1432.526      -61.819;
    4.87601    1441.629      -74.299;
    4.54607    1351.555      -93.34];
A=c(:,1);
B=c(:,2);
C=c(:,3);
logPi=A-B./(t+C);
pisat=10.^(logPi);
```

```
pisat=pisat';

function pisat= psat2(t)
%t=350;
%antoine coef P(bar) T (K)
c  =[4.92531    1432.526        -61.819];
A=c(1);
B=c(2);
C=c(3);
logPi=A-B/(t+C);
pisat=10^(logPi);
```

## A.2 M-File code of second main program (massbaalance.m)

```
function [sys,x0,str,ts] = massbalance(t,x,u,flag)
switch flag,
   %%%%%%%%%%%%%%%%%%
   % Initialization %
   %%%%%%%%%%%%%%%%%%
   case 0,
      [sys,x0,str,ts]=mdlInitializeSizes;
      %%%%%%%%%%%%%%%
      % Derivatives %
      %%%%%%%%%%%%%%%
   case 1,
      sys=mdlDerivatives(t,x,u);
      %%%%%%%%%%%
      % Outputs %
      %%%%%%%%%%%
   case 3,
      sys=mdlOutputs(t,x,u);
      %%%%%%%%%%%%%%%%%%%
      % Unhandled flags %
      %%%%%%%%%%%%%%%%%%%
```

```
    case { 2, 4, 9 },
        sys = [];
        %%%%%%%%%%%%%%%%%%%%
        % Unexpected flags %
        %%%%%%%%%%%%%%%%%%%%
    otherwise     error(['Unhandled flag = ',num2str(flag)]);
end
% end wpfun1
%
%
%=============================================================================
% mdlInitializeSizes
% Return the sizes, initial conditions, and sample times for the Sfunction.
%=============================================================================
%
function [sys,x0,str,ts]=mdlInitializeSizes
sizes = simsizes;
sizes.NumContStates  = 4;
sizes.NumDiscStates  = 0;
sizes.NumOutputs     = 4;
sizes.NumInputs      = 4;
sizes.DirFeedthrough = 1;
sizes.NumSampleTimes = 1;
sys = simsizes(sizes);
x0  = [1; 4; 4];
str = [];
ts  = [0 0];
% end mdlInitializeSizes
%
%=============================================================================
% mdlDerivatives
```

```
% Return the derivatives for the continuous states.
%=============================================================
========
%
function sys=mdlDerivatives(t,x,u)
%model parameters
V=10;   %K/W
L=10;   %K/W
% input
x(1)=u(1);
x(2)=u(2);
x(3)=u(3);
x(4)=u(4);
dxdt(1)=x(4)-x(1);
dxdt(2)=x(1)-x(3);
dxdt(3)=x(2)-x(4);
dxdt(4)=x(4);
sys = [dxdt(1); dxdt(2); dxdt(3)];
% end mdlDerivatives
%
%=============================================================
========
% mdlOutputs
% Return the block outputs.
%=============================================================
========
%
function sys=mdlOutputs(t,x,u)
sys = x;
% end mdlOutputs
```

### A.3 M-File code of program to generate composition profile (component12.m)

```
load xmbd.mat;
```

```
tm=Data(1,:);
x1H=Data(2,:);
x2H=Data(3,:);
x3H=Data(4,:);
x1M=Data(26,:);
x2M=Data(27,:);
x3M=Data(28,:);
x1B=Data(50,:);
x2B=Data(51,:);
x3B=Data(52,:);

%Grafik konsentrasi vs time pada Vessel 1 (top)
figure(1)
plot (tm,x1H,'-k+',tm,x2H,'-kd',tm,x3H,'-k*')
legend('EtOH','1-ProOH','n-BuOH')
title('Liquid composition in top vessel')
xlabel('Time (min)')
ylabel('Mole fraction')

%Grafik konsentrasi vs time pada Vessel 2 (middle)
figure(2)
plot (tm,x1M,'-k+',tm,x2M,'-kd',tm,x3M,'-k*')
legend('EtOH','1-ProOH','n-BuOH')
title('Liquid composition in midle vessel')
xlabel('Time (min)')
ylabel('Mole fraction')

%Grafik konsentrasi vs time pada Vessel 3 (bottom)
figure(3)
plot (tm,x1B,'-k+',tm,x2B,'-kd',tm,x3B,'-k*')
legend('EtOH','1-ProOH','n-BuOH')
title('Liquid composition in bottom vessel')
xlabel('Time (min)')
ylabel('Mole fraction')
```

### A.4 M-File code of program to generate level holdup profile (holdupclosed.m)

```
clear all;
load holdupori.mat;
tm=Data(1,:);
M=Data(3,:);
B=Data(4,:);

%tw=[0 10 20 30 40 50 60 90 120 150 180];
%x3Be=[];
%xlswrite('(400-325-400)-(055-010-035)-m.xls',tm,'tm');
%xlswrite('(400-325-400)-(055-010-035)-m.xls',x2M,'x2M');
%xlswrite('(400-325-400)-(055-010-035)-m.xls',x1H,'x1H');

%Grafik konsentrasi vs time pada Vessel 1 (top)
figure(1)
plot (tm,M,'-k',tm,H,'--k')
%tw,x2Me,'^k')
%title('Equimolar feed (400;100;400)')
legend('Middle Vessel', 'Reboiler')
%,'1-ProOH-sim','1-ProOH-exp','n-BuOH-sim','n-BuOH-exp')
xlabel('Time (min)')
ylabel('Level Holdup (m)')
```

# APPENDIX B

# SINGULAR VALUE DECOMPOSITION MATLAB CALCULATION

## B.1 SVD for 3 x 3 matrix

```
A =

  -18.9864    7.6505  -11.3664
    9.4218  -22.5547  -22.3960
    9.4218   15.0742   33.5099

>> [U,S,V]=svd(A)

U =

   -0.1591    0.8042    0.5727
   -0.6157   -0.5343    0.5792
    0.7718   -0.2605    0.5801

S =

   48.2666         0         0
         0   27.5049         0
         0         0    0.0899

V =

    0.0930   -0.8274    0.5539
    0.5035    0.5190    0.6907
    0.8590   -0.2147   -0.4649
```

## B.2 SVD for 9 set of 2 x 2 matrix

```
>> b=[-18.9864 7.6504; 9.4218 -22.5547];
>> u=svd(b)
u =
   29.5100
   12.0689
>> b1=29.510/12.0689
b1 =
```

```
   2.4451

>> c=[-18.9864 9.4218; -11.3664 -22.396];
>> u=svd(c)
u =
  25.1178
  21.1926
>> c1=25.1178/21.1926
c1 =
   1.1852

>> d=[7.6504 -22.5547; -11.3664 -22.396];
>> u=svd(d)
u =
  31.9123
  13.4025
>> d1=31.9123/13.4025
d1 =
   2.3811

>> e=[7.6504 -18.9864; 15.074 9.4218];
>> u=svd(e)
u =
  21.1971
  16.9024
>> e1=21.1971/16.9024
e1 =
   1.2541

>> f=[-18.9864 -11.3664; 9.4218 33.5098];
>> u=svd(f)
u =
  38.9456
  13.5866
```

```
>> f1=38.9456/13.5866
f1 =
    2.8665

>> g=[7.6504 -11.3664; 15.074 33.5098];
>> u=svd(g)
u =
   37.5224
   11.3985
>> g1=37.5224/11.3985
g1 =
    3.2919

>> h=[-22.5547 9.4218; 15.07424 9.4218];
>> u=svd(h)
u =
   27.2893
   12.9916
>> h1=27.2893/12.9916
h1 =
    2.1005

>> i=[9.4218 -22.396; 9.4218 33.50986];
>> u=svd(i)
u =
   40.3984
   13.0385
>> i1=40.3984/13.0385
i1 =
    3.0984

>> j=[-22.5547 -22.396; 15.0742 33.50986];
>> u=svd(j)
u =
```

```
   47.7897
    8.7509
>> j1=47.7897/8.7509
j1 =
    5.4611
```

# APPENDIX C

# CONTROL AND SIMULATION BY MATLAB/SIMULINK

## C.1 Level Control Closed System

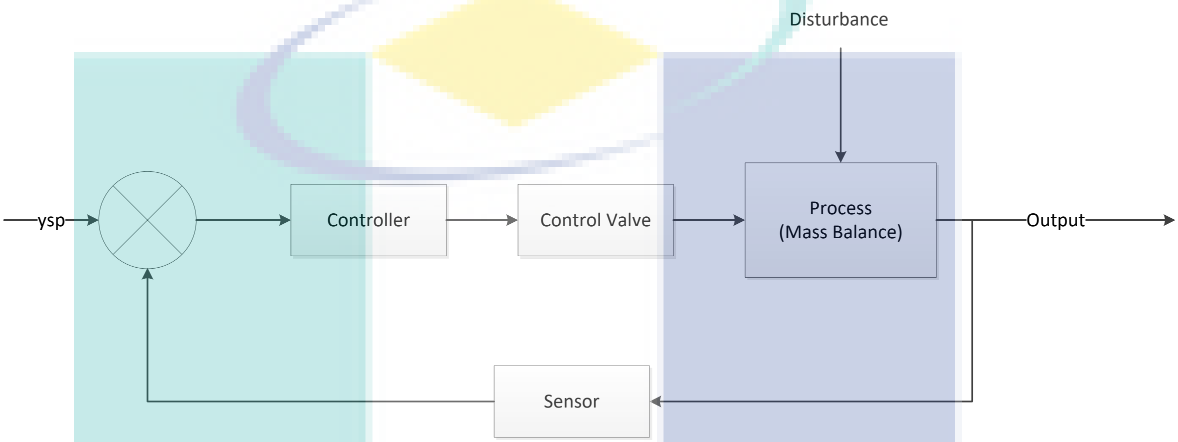



Figure C. 1 Level Control Loop Block Diagram

The control valve equation is stated in equation C.1. $K_V$ is equal to 0.28036 and $\tau_v$ is equal to 0.05. The valve coefficient and time constant is based on manufacturer valve written in Smith and Corripio (2006).

$$\frac{K_v}{\tau_v s+1} \qquad \text{C. 1}$$

## C.2 Multi-vessel Batch Distillation Column Control System

The configuration of MVBDC with two level control is shown in Figure C.2.

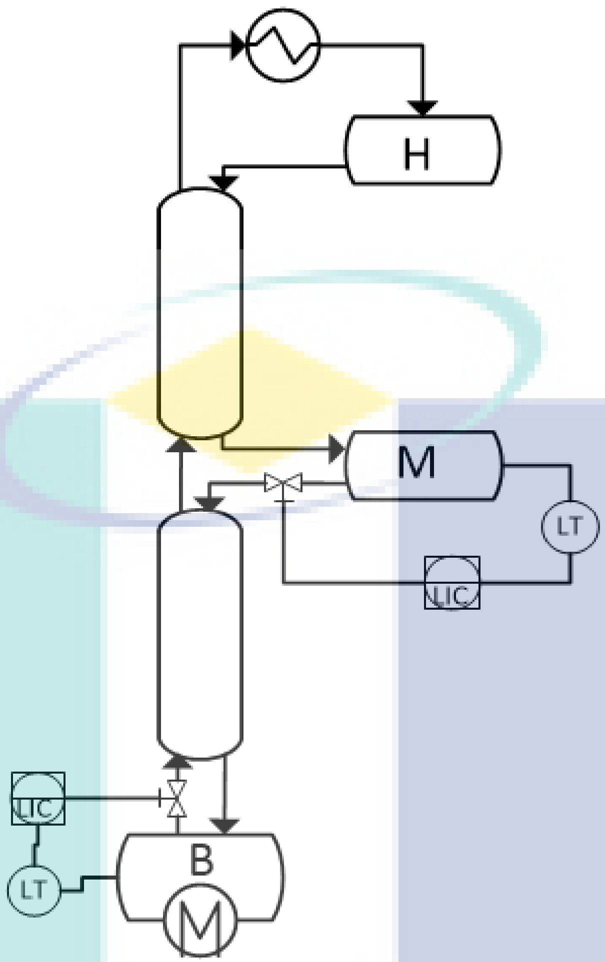
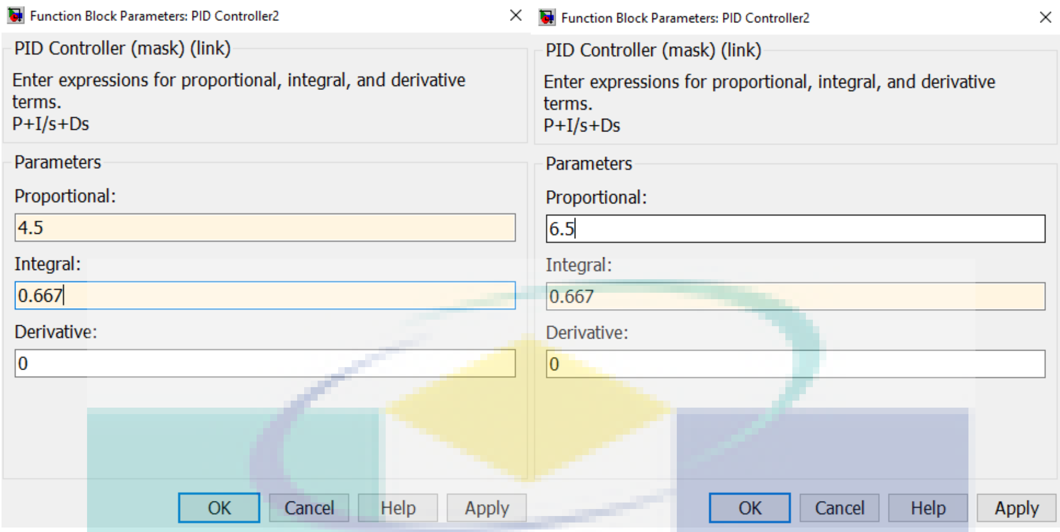



Figure C. 2 MVBDC Configuration with Two Level Control

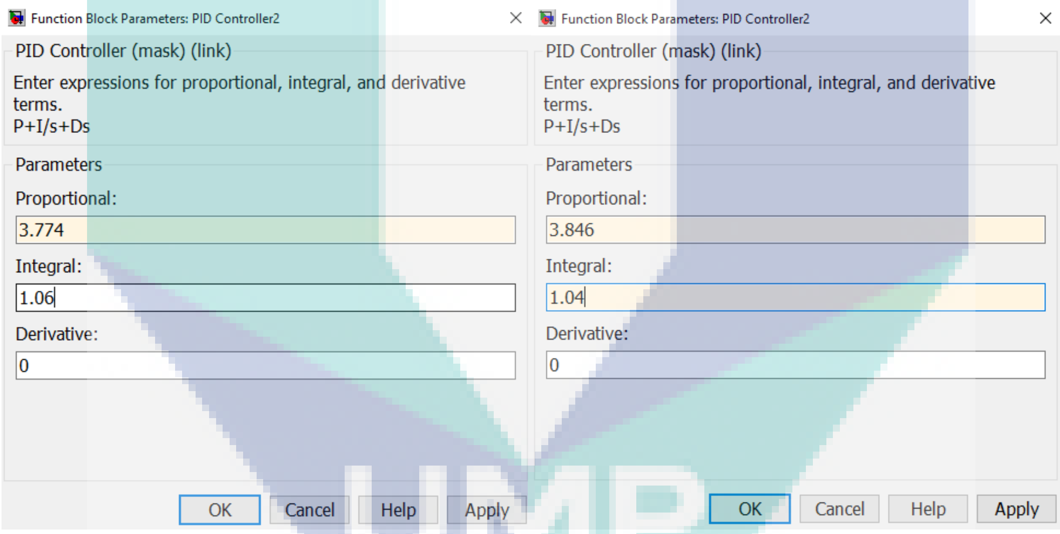
## C.3 Controller Tuning Block Diagram

The block diagram of PID Controller for both level in middle vessel and reboiler holdup for case (1) and case (2) for ZN tuning and IMC tuning is shown in Figure C.2-C.5.



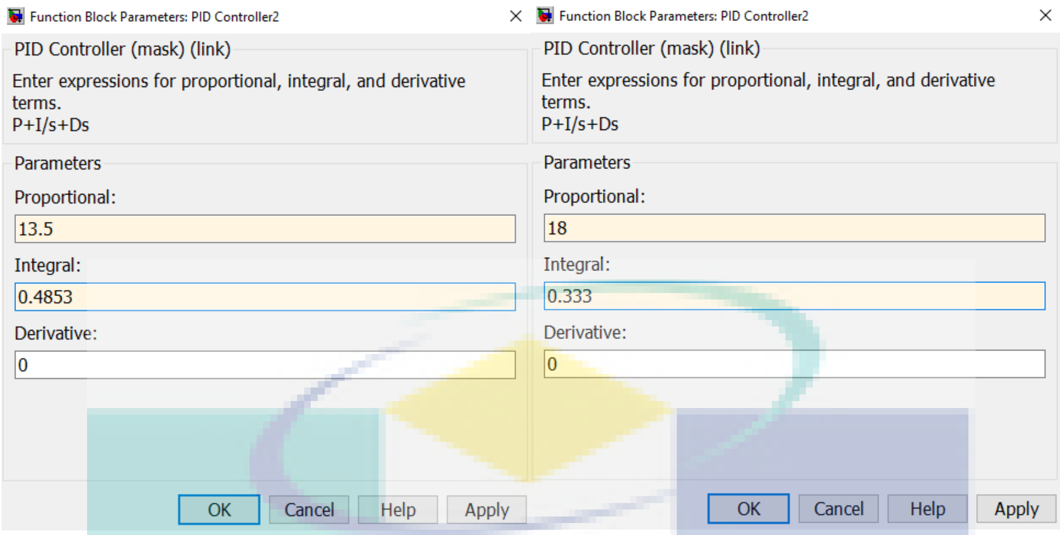

(a) (b)

Figure C. 3 PID Controller Block Diagram for (a) Top Vessel (b) Middle Vessel with ZN-PI Controller Case (1)



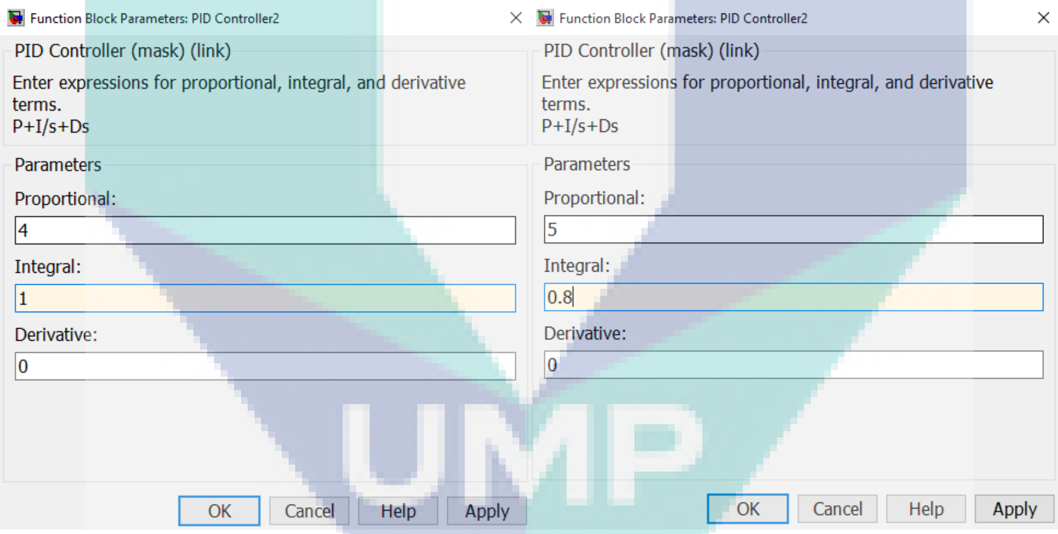

(a) (b)

Figure C. 4 PID Controller Block Diagram for (a) Top Vessel b) Middle Vessel with IMC-PI Controller Case (2)




(a) (b)

Figure C. 5 PID Controller Block Diagram for a) Top Vessel b) Middle Vessel with ZN-PI Controller Case (2)




(a) (b)

Figure C. 6 PID Controller Block Diagram for a) Top Vessel b) Middle Vessel with IMC-PI Controller Case (2)