MODELING & CONTROL pH NEUTRALIZATION PROCESS PILOT PLANT

By

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FINAL REPORT

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CERTIFICATION OF APPROVAL

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Approved:

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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MOHAMAD AFIF BIN ISMAIL

ABSTRACT

pH neutralization process plant are widely used in process industries including wastewater treatment, chemical process, biotechnology and pharmaceuticals. The control of pH process is recognized as a difficult problem due to highly nonlinear characteristics of the process. The purpose need to control the pH value within a specific range is arise from environmental legislative and quality standard. pH control process gives numerous challenges in control strategies. This fact makes the research and development of this process control become more interesting. The first stage of this project is to develop a mathematical model of a specific chemical process, a pH neutralization process. The model would then provide an opportunity for development, testing and evaluation of an advanced form of controller. The second stage of this project concerns the development of advanced forms of controller on the pH model. Fuzzy Logic Controller (FLC) was implemented to control the pH value of this system and the result will compare against conventional PID controller based on performance on simulation test. The simulation is performed in the MATLAB environment using Simulink and Fuzzy Logic Toolbox. The research has been based entirely around a specific pH neutralization process pilot plant installed at the University Teknologi Petronas, Malaysia. The main feature of interest in this pilot plant is that it was built using instrumentation and actuators that are currently used in the process industries. Result shows that Fuzzy Logic Controller perform better than PI Controller.

ACKNOWLEDGEMENT

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1.0 INTRODUCTION

This chapter introduces background information relevant to the project. It also highlights the main issues that drive this research study. To begin the project, there are some objective and scope of study that I am going to refer to.

1.1 Background Study

The control of pH is important in wastewater treatment, chemical process, biotechnology, pharmaceuticals and etc. The purpose of a pH neutralization plant is to neutralize the waste product solution before discharging it to environment. The requirement in terms of pH value to be discharged is in the range of 6 to 8. The reason is to protect rivers and life and to avoid damage due to corrosion.

However, the pH neutralization process is highly non-linear due to the logarithmic relationship between the hydrogen ions concentration [H+] and the pH level, thus posing a challenge for its control. Linear controllers such as the Proportional-Integral-Derivative (PID) controllers are tuned based on the highest gain at the neutralization point of pH 7 in order to keep the loop stable. However, the performance result of PID controller is in poor in the non-linear process [1].

Fuzzy Logic Controllers (FLC) is one of the advance controller that been used to overcome weakness of PID controller. Fuzzy Logic Controller does not need the mathematical model as in the case of conventional control design methods. For non-linear systems, controlling with conventional controllers such as PI, is difficult. Fuzzy Logic Controller provides an effective alternative to classical controllers [2]. By using a linguistic approach, fuzzy set theory can be integrated into control theory using rules of the form, If {condition} Then {action}. Using enough of these rules one can create a functional controller. In the same way, the input variables can be separated into overlapping sets that have a linguistic correlation to form a membership function. The membership values control the degree to which each rule to be fire, illustrating the interdependent relationship between rule sets and membership functions [3].

1.2 Problem Statement

Studies on dynamics and control of pH neutralization have been carried out since 70's until now due to its highly non-linear characteristic [4]. The problem that been faced is how to provide a good dynamic model of a pH neutralization process. Thus, one of the main problems in this research was that the currently available model for pH neutralization process did not represent the pH neutralization plant in industry without modification. The second problem that been faced in this research is the poor control performance established by current control strategies. The major problem contributed to the control performance can be summarized as follows: [9]

i. Increases in plant complexity and strict constraints in terms of environmental and other performance requirements.
ii. The non-linearity of a pH neutralization process is a main cause of difficulty in terms of robust and stable control system design.

1.3 Objectives and Scope of Study

This project will be divided into two main objectives. The first objective is to provide a model for pH neutralization process, based on physical and chemical principles that represent the actual pH neutralization pilot plant in UTP. The second objective of this project is to design, develop and testing an advanced controller on pH model. The proposed type of advanced controller in this project is Fuzzy Logic Controller. Result from Fuzzy Logic Controller will be analyzed and will be compared with conventional feedback approach which is PID controller based on simulation test.

1.4 Relevancy

The main reason of pH neutralization is to protect rivers and life and to avoid damage due to corrosion. It is commonly used in biological treatment, since bacteria are sensitive to rapid outside pH range of 6 to 9. Similarly, aquatic ecosystems are pH sensitive; therefore neutralization of wastewater is required before discharge it to a receiving body [18]. Generally, the purpose of controlling pH value is to neutralize the waste product solution before discharging it to environment. The range value that safe for environment is in the range of 6 to 8. The need to control the pH value within specific level arises from environmental legislative and quality standards which are constantly being revised.

1.5 Feasibility

The feasibility of this project is to complete the research within the scope and time frame, while maintaining substance to this research.

During the first semester (FYP 1), the scope and task that will be covered are:

1. Research on pH neutralization
2. Modeling and simulation of pH neutralization process pilot plant

During the second semester (FYP 2), the scope and task that will be covered are:

1. Further study on controller design
2. Design and developed the proposed controller.
3. Analyze and compare the controller performances.

2.0 LITERATURE REVIEW

This section summaries the literature survey that was conducted before start this research. It covers a short of summary of the characteristic of the pH neutralization process. It also covers about the control method that been used in this research.

2.1 pH Characteristic

There are many reference books in the field shows the equilibrium of chemical processes between acid and alkaline. This section will describe the general properties of acids and bases from a chemical view with some explanations of the acid-base neutralization reaction process. The purpose of this section is to provide important information about the chemical process. The books that been used in this overview mainly well-established textbook.

Concepts Relating to Acids and Bases

As describes in the Arrhenius theory [5], the *universal aqueous acid–base definition* is described as the formation of water from hydrogen ions (H^+) and hydroxide ions (OH^-) is from the dissociation of an acid and base in aqueous solution:

$$H^+ + OH^- \Leftrightarrow H_2O$$

This leads to the definition that in Arrhenius acid–base reactions, the reaction between an acid and a base will produce a formation of salt and water. In other words, this is a neutralization reaction process.

$$acid + base \rightarrow salt + water$$

The positive ion from a base forms a salt with the negative ion from an acid. As an example, two moles of sodium ion (Na^+) from the base sodium hydroxide (NaOH) combine with one mole of sulfate ion (SO_4^{2-}) from sulfuric acid (H_2SO_4) will form one mole of sodium sulfate (Na_2SO_4) . Two moles of water are also formed.

$$2\ NaOH + H_2SO_4 \rightarrow Na_2SO_4 + 2\ H_2O$$

Based on the Bronsted-Lowry theory, an acid is described as a substance that can donate hydrogen ions (H^+) or proton and a base is a substance that can accept a proton. Unlike the previous definitions, the Brønsted–Lowry definition does not refer to the formation of salt and solvent, but it refer to the formation of *conjugate acids* and *conjugate bases* that been produced by the transferring of a proton from the acid to the base [5][6]. In this theory, an acid and a base react not to produce a salt and a solvent, but it will form a new acid and a new base. Thus, the concept of neutralization is absent [7].

For example, the removal of hydrogen ions (H^+) from hydrochloric acid (HCl) produces the chloride ion (Cl^-), this is called as conjugate base of the acid:

$$HCl \rightarrow H^+ + Cl^-$$

The addition of hydrogen ions (H^+) to the hydroxide ion (OH^-) will produces the water (H_2O), it is called as conjugate acid of the base:

$$H^+ + OH^- \rightarrow H_2O$$

There are two types of acid and base which is monoprotic and polyprotic. Monoprotic acids are acids that can release only one proton per molecule and have only one equivalence point. Monoprotic bases are bases that can only react with one proton per molecule and similar to monoprotic acids which are only have one equivalence point. Otherwise, Polyprotic acids are acids that can release more than one protons per molecule. It can be further categorized into diprotic acids and triprotic acids which are can donate two and three protons respectively. Polyprotic bases are bases that can release several protons per molecule. Similar to polyprotic acids, polyprotic bases also can be categorized into diprotic bases and triprotic bases [8].

Sulfuric acid (H_2SO_4) is an example of diprotic acid. This substance ionizes in two different stages that been shown in equation below. Each stage has a different value of dissociation constant which describes the characteristic of the substance itself.

$$H_2SO_4 \Leftrightarrow H^+ + HSO_4^-$$

$$HSO_4^- \Leftrightarrow H^+ + SO_4^{2-}$$

The dissociation constant also can describe the strength of the acids and bases. That means, larger the value of dissociation constant stronger the acid able to donate or ionize all protons in water. Below are the dissociation constant values of sulfuric acid (H_2SO_4):

$$K_1 = \frac{[H^+][HSO_4^-]}{H_2SO_4}$$

$$K_2 = \frac{[H^+][SO_4^{-2}]}{HSO_4^-}$$

A titration curve usually used to describe the characteristic of the acid-base neutralization reaction [9]. Titration curves also shows the important information about the reaction like equilibrium point, types of the acid and base involves (strong or weak and monoprotic or polyprotic) and the total amount of the substance that involved at the end of the process [10].

Figure 2.1 shows example of titration curve for monoprotic acid (hydrocloric acid) and polyprotic acid (phosphoric acid). The figure below describes the behavior of neutralization process is highly nonlinear [9].

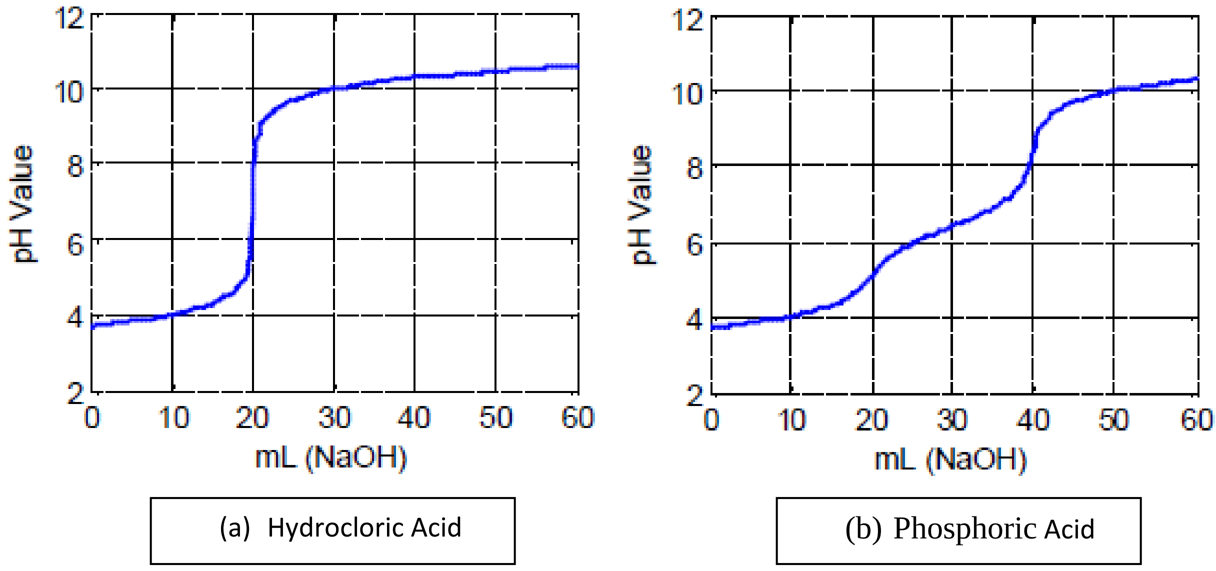


Figure 2.1: Titration curves for monoprotic acid (a) and polyprotic acid (b)

Acidic or alkaline the solution can be determined by using the concentration of hydrogen and hydroxide ions. The solution becomes acidic when the concentration of hydrogen ions greater than the concentration of hydroxide ions. And the solution becomes alkaline if concentration of hydroxide ions greater than concentration on hydrogen. Neutral solution occur when the ions of hydrogen and alkaline become the same.

Based on Sørenso theory, the pH value can be measured through mathematical equation below:

$$pH = -log_{10}[H^+]$$

The result of the equation has been scale between 1 and 14. The mixed solutions become neutral when the pH value is 7. If the pH value is more than 7, the mixed solution been classified as alkaline and below than 7 the mixed solution is acidic.

2.2 pH Control Techniques

In industry, there are many control strategies of pH neutralization process that been used. The general aim in this form of control is to maintain the pH value within a liquid at a specific level (usually 6 to 8). This can be important in order to comply with and satisfy certain environmental requirements or quality standards. This section contains a short review about the controller that been used by the author for this project. Firstly, the conventional PID control strategy been applied to the model to control pH value. For further improvement, a new approach of advance control strategy had been also applied to the system. The type of advance controller that been used by the author is Fuzzy Logic Controller.

2.2.1 Overview of pH Control

pH control need to maintain the pH value during continuous operation at certain value by manipulating the acid or the alkaline flow stream. Usually in most industrial application, the desired value that been choose is around 7 (usually 6 to 8). This is the safest value for portable water, utility water used in industry, or waste disposed water [11].

pH neutralization is well known as a difficult problem to control. The difficulty comes from the high non-linearity of the process around the neutralization point. The non-linearity seems in the S-shape of the titration curve associated with pH processes. The process gain grows drastically at the intermediate region of the S-shape curve. This behavior is the main cause why the pH neutralization is hard to control. Moreover, the shape of the titration curve is distorted when the feed condition changes. This situation gives more complexity to the control system. For this reason, pH control is still in the research by many researchers in the world [12].

pH control methods can be divided into three main categories which is open loop control method, feedback control method and feedforward control method .

2.2.2 Conventional Feedback Controller

The most widely used simple feedback control strategy that been applied in many process control is the PID algorithm [9]. PID controller is short term from proportional, integral and derivative controller. The PID controller calculates an error value as the difference between a measured process variable and a desired setpoint. The controller tries to minimize the error by adjusting the process control inputs. The PID controller calculation is based on three separate constant parameters which is proportional (P), integral (I) and derivative (D) values. These values can be interpreted in terms of time: P depends on the present error, I on the accumulation of past errors, and D is a prediction of future errors, based on current rate of change [13]. The weighted sum of these three actions then been used to adjust the process by a control element. Below is the figure of PID controller block diagram.

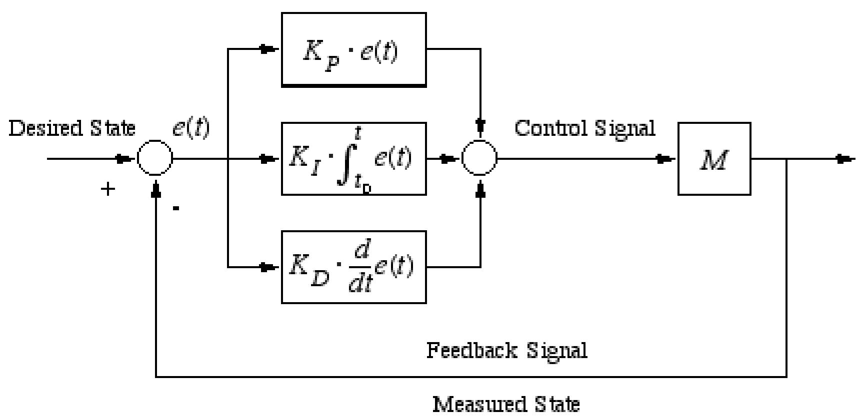


Figure 2.2: A block diagram of a PID controller

In the lack of knowledge of the basic process, PID controller is the best controller [14]. By tuning the three PID parameters, the controller can provide control action designed for specific process requirements. The response of the controller can be described in terms of the responsiveness of the controller to an error based on the degree of the controller overshoots the setpoint and the degree of system oscillation. However, using the PID algorithm for control purpose does not guarantee optimal control performance of the system and system stability.

Some applications may require using only one or two actions to provide the appropriate system control. This can be done by setting the other parameters value to zero. A PID controller will be called a PI, PD, P or I controller in the absence of the respective control actions. PI controllers are commonly been used, since derivative action is sensitive to measurement noise. However the absence of the integral action may prevent the system from reaching the setpoint value.

2.2.3 Fuzzy Logic Control

The idea of Fuzzy Logic was conceived by Lotfi Zadeh, a professor at the University of California at Berkley. Firstly, Fuzzy Logic was presented not as a control methodology but as a way of processing data by allowing partial set membership rather than crisp set membership or non-membership. This approach starts to apply in control system in the 70's due to lack of computer capability prior to that time. Professor Lotfi Zadeh reasoned that people do not require precise, numerical information input, and yet they are capable of highly adaptive control. It would be much more effective if the feedback controllers could be programmed to accept noisy and imprecise input [15].

Fuzzy logic is a problem solving control system methodology that provides itself to implementation in systems ranging from simple, small, embedded micro-controllers to large, networked, multi-channel PC or workstation-based data acquisition and control systems. It also can be implemented in hardware and software. Fuzzy Logic provides a simple way to arrive at a definite conclusion based upon vague, ambiguous, imprecise, noisy, or missing input information. Fuzzy Logic approach to control problems mimics how a person would make decisions [16].

In compare with the conventional controllers, Fuzzy Logic Controllers have a high ability to control nonlinear, time–invariant, time-delayed and complex processes. Unlike the conventional controllers, the procedures of fuzzy control algorithm involve the powerful software and big volume of memory to implement [17].

The block diagram of a fuzzy controller is shown in Figure 2.3. The fuzzy controller consists of the following four elements [16]:

1. A Rule-Base (a set of If-Then rules): contains a fuzzy logic quantification of the expert's language description of how to achieve good control.
2. An Inference Mechanism (also called an "inference engine" or "fuzzy inference" module): emulates the expert's decision making in interpreting and applying knowledge about how best to control the plant.
3. A Fuzzification interface: converts controller inputs into information that the inference mechanism can easily use to activate and apply the rules.
4. A Defuzzification interface: converts the conclusions of the inference mechanism into actual inputs for the process.

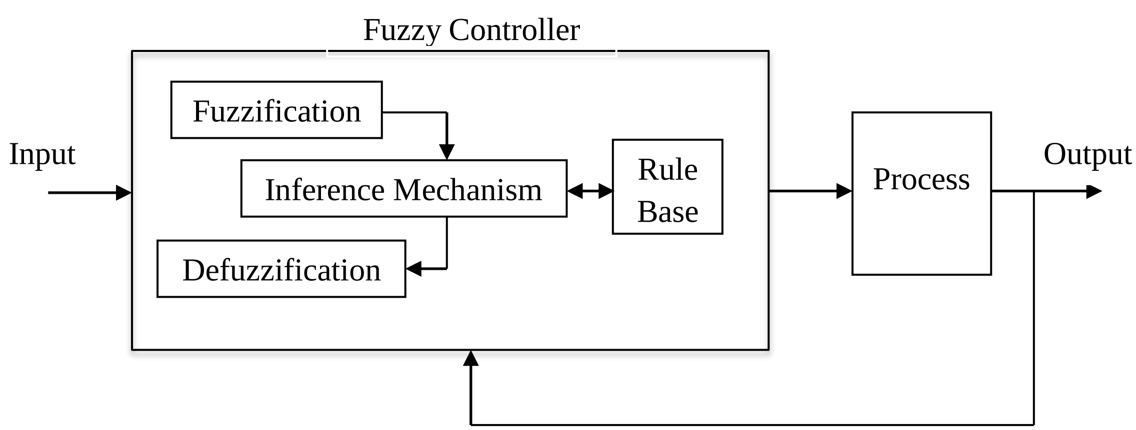


Figure 2.3: Fuzzy Controller Architecture

3.0 METHODOLOGY

Methodology is a chapter which will cover the process and flow through this project. Although we have project activities and Gantt chart, we will also brief the milestone and equipment used.

3.1 Research Methodology

This project divided into two stages. The first one is to provide a nonlinear pH neutralization model that can represent the actual pH neutralization plant in UTP. The second goal is to develop and testing an advanced form of controller to the pH model. The controller proposed for this project is Fuzzy Logic Controller. Result from this advanced controller will be compared with conventional PID controller. Figure 3.1 shows the detailed flow of this project.

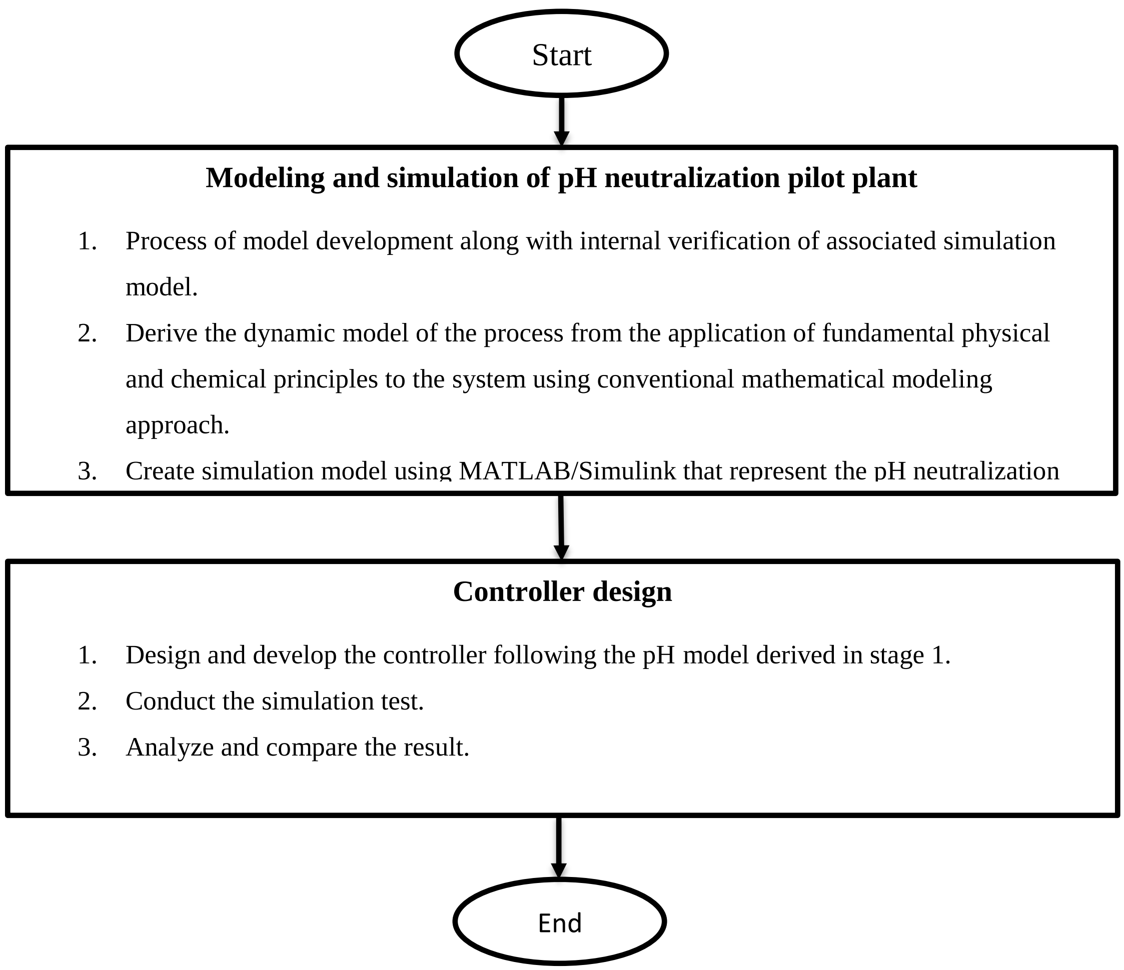


Figure 3.1: Flow of research activities

Figure 3.2 shows the detailed flowchart of FYP 1 and figure 3.3 shows the detailed flowchart for FYP 2. The work for this project will always follow and refer this flowchat.

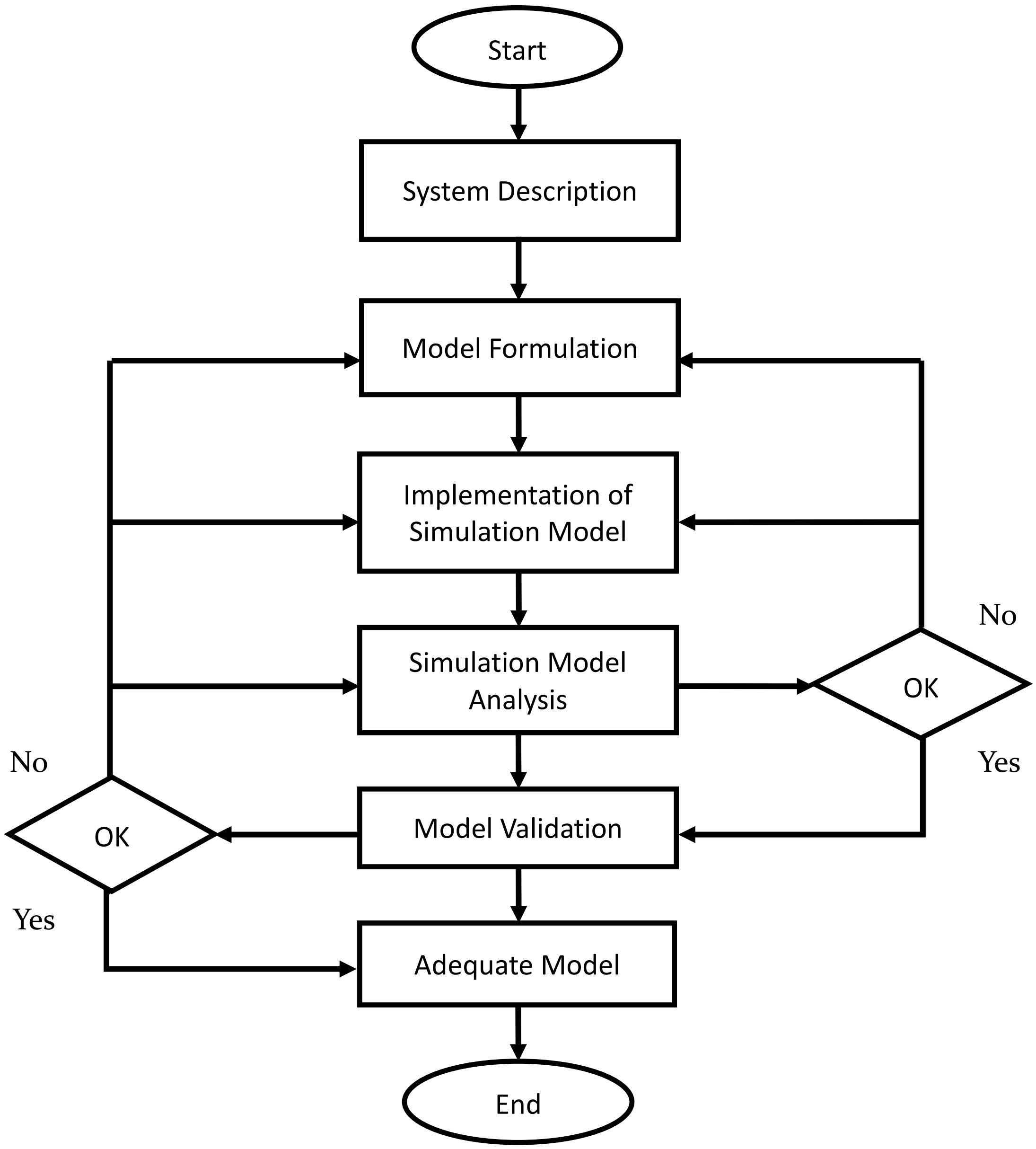


Figure 3.2: The flowchart of the modeling process

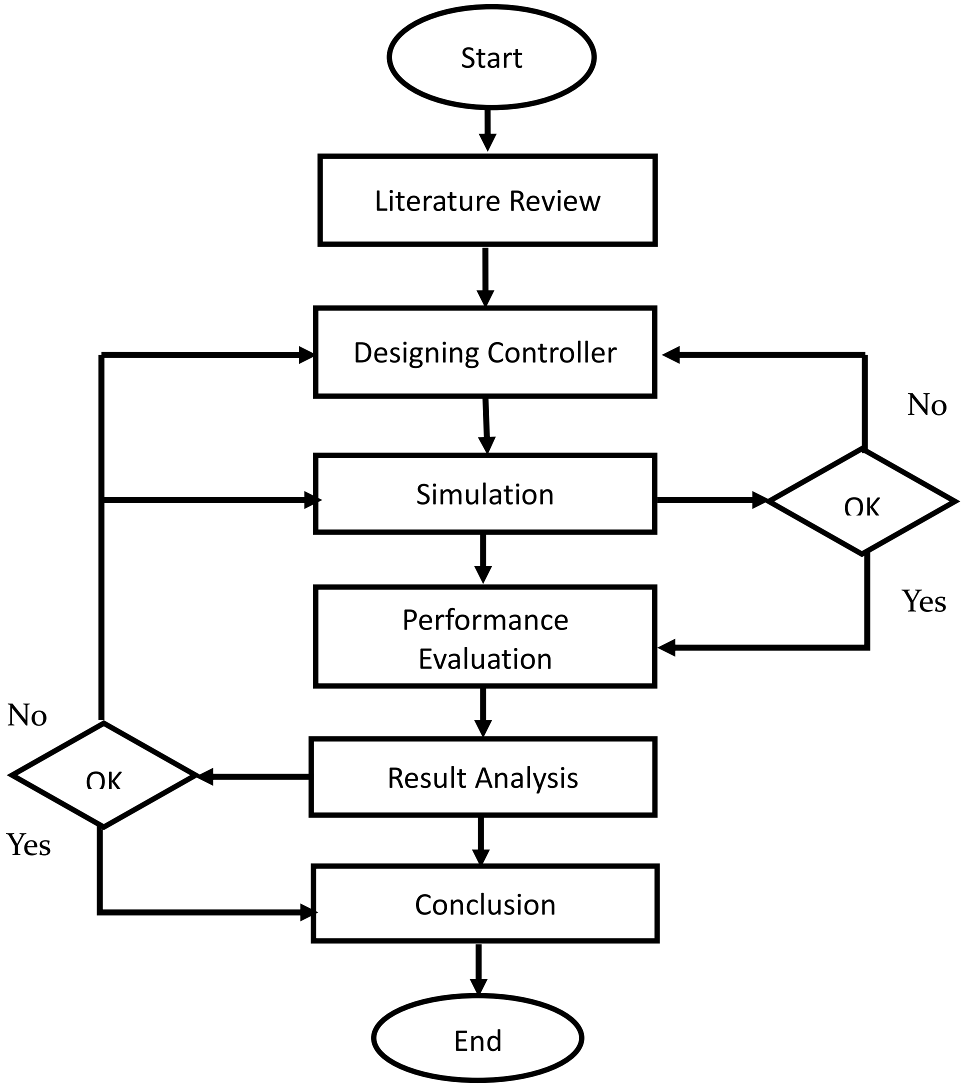


Figure 3.3: The flowchart of the designing controller

3.2 Project Activities

There are quite number of activities in this project such as;

1. Reading and research material that could expand knowledge about:
    a. Chemical reaction
    b. Modeling techniques
    c. pH control strategy
    d. MATLAB/ Simulink
2. Developed a suitable model for pH neutralization system based on requirement parameters and control strategy.
3. Design and develop pH controller for this system.
4. Conduct the simulation test.
5. Analyze and compare the result.

3.3 Key Milestone

Table below shows the research milestone of this project:

		FYP 1														FYP 2													
	Activities	1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Completion on the study of pH neutralization process control strategies, modeling and simulation of pH plant.						*																						
2	Completion on the developing the model. Develop a suitable model for pH neutralization system.													*															
3	Completion on the design, devolop pH controller and test the controller																				*								
4	Completion on the data analysis																										*		
5	Research completion																												*

Table 3.1: Research Milestone

3.4 Gantt Chart

Table below shows the gantt chart for FYP 1 & FYP 2:

No.	Activity	FYP 1														FYP 2														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Literature study and review	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■			
	1. Study of fundamental of pH:	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■			
	a. Concept of pH	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■			
	b. Titration Curve	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■			
	c. acid-base equilibra and calculation	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■			
	2. Plant process control	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■			
	3. Study about modeling techniques	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■			
	4. Study about the suitable controller	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■			
2	Submit extended proposal						■																							
3	Develop a model for pH neutralization system based on requirement parameters and control strategy							■	■	■	■	■	■	■																
4	Viva: proposal defence									■																				
5	Submit draft report FYP 1													■																
6	Submit final report FYP 1														■															
7	Design, develop pH controller															■	■	■	■	■	■									
8	Carry out simulation and test the developed simulator																		■	■	■									
9	Collect data result and analysis the data																			■	■	■								
10	Carry out experiment																						■	■	■	■				
11	Collect and analyse the experiment result																						■	■	■	■				
12	Data analysis																							■	■	■	■			
13	Submit draft report FYP 2																											■		
14	Submit final report FYP 2																												■	
15	Viva																													■

Table 3.2: Gantt Chart

3.5 Tool and Equipment:

The tool and equipment that been used in this project is:

1. pH neutralization plant located at block 23, UTP
2. MATLAB/Simulink

4.0 MODELING AND SIMULATION OF THE pH NEUTRALIZATION PILOT PLANT

In this section, the research involves process model development along with internal verification of simulation model and external validation of model from test data obtained from open loop and simple closed loop tests carried out on the actual plant.

The model of the process has been derived from the application of fundamental physical and chemical principles using a conventional mathematical modeling approach. This chapter will describe about the detail of the development of pH neutralization process. The model of the pH neutralization is based on the actual pilot plant located at block 23, UTP known as Chemical Pilot Plant. The flowchart that been describe in Chapter 3 provides a useful guideline which was followed throughout the process of developing the model of the plant.

4.1 Overview of the pH Neutralization Process Modeling

As been stated before, the design of pH neutralization model is based on actual pilot plant that been used in this project. Piping and Instrument Diagram of this pilot plant been shown in Figure 4.1.

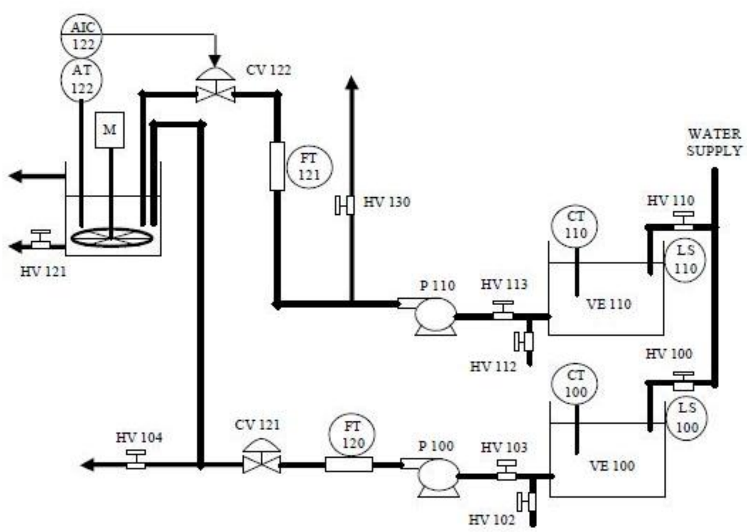


Figure 4.1: Piping and Instrument Diagram (P&ID) of the Pilot Plant

This diagram provides useful information about the overall process, the piping layout and the instrument equipment in this pilot plant. As shown in the figure, this pilot plant has three main tanks which is an acid tank (VE100), an alkaline tank (VE110) and a process tank (VE120). There are two pumps that been used to pumped the acid stream and alkaline stream which is P100 and P110. Flow transmitter FT120 and FT 121 used to indicate the flowrate of acid and alkaline streams respectively.

The flowrate of acid and alkaline can be controlled with control valve which is CV121 is for the acid stream and CV122 is for the alkaline stream. AG120 is a motorized agitator that used to mis up the solution in the process tank. The pH value been measured by pH sensor (AT122) located in process tank.

The solution that been used in this project is strong acid and strong base. The type of acid and base that been used is Sulphuric Acid (H_2SO_4) and Sodium Hydroxide (NaOH) respectively. The mathematical modeling of this process that been design needs to follow the equation and the constant value of this type of solution itself.

The making process of modeling this plant also needs some of assumptions to reduce model complexity. Without these assumptions, the model presents computational difficulties and can consist of major problems in terms of validation and tuning. The assumptions involves in this modeling are as follows [9]:

i. The acid and alkaline solutions in the reactor tank are perfectly mixed at all times and a lumped parameter compartmental form of model can be used.
ii. The acid-base reaction process in the reactor tank is instantaneous and isothermal.
iii. The dissociation of acid and base reaction is complete and the attainment of equilibrium is fast.
iv. No other reactions occur in the reactor tank.
v. The volume of the solution in the tank is constant.
vi. The time constants for the control valves and measuring instruments are negligible compared to those of the process.

4.2 Development of the Mathematical Model

The formulation of the model is based on fundamental principles. The first principle is known as conservation balance principle. The general equation for conservation of material for the pH process is:

$$\left(\begin{array}{c}\text{Rate of}\\ \text{accumulation of}\\ \text{non-reactant}\\ \text{species witin}\\ \text{element volume}\end{array}\right) = \left(\begin{array}{c}\text{Rate of flow non-}\\ \text{reactant species}\\ \text{into element}\\ \text{volume}\end{array}\right) - \left(\begin{array}{c}\text{Rate of flow non-}\\ \text{reactant species}\\ \text{out of element}\\ \text{volume}\end{array}\right)$$

Two equations have been derived based on this general equation to get the mathematical equation of pH process in the CSTR system. These equations follow the general approach that been used by many previous researchers in this field. The equation as below:

$$V\frac{da}{dt} = F_1C_1 - (F_1 + F_2)a$$

$$V\frac{db}{dt} = F_2C_2 - (F_1 + F_2)\beta$$

V = *Volume in the reactor tank*

F_1 =*Flowrates of acid*

F_2 =*Flowrates of base*

C_1 =*Concentration of acid*

C_2 =*Concentration of base*

α is the non-reactant component of the acid and β is the non-reactant component of alkaline. These variables are defined in the equation below:

$$\alpha = [H_2SO_4] + [HSO_4^-] + [SO_4^{-2}]$$

$$\beta = [Na^+]$$

Next is to identify the electroneutrality condition of non-reactant components. Electroneutrality means the solutions are electrically neutral.

The condition is as below:

$$[Na^{+}] + [H^{+}] = [OH^{-}] + [HSO_4^{-}] + 2[SO_4^{-2}]$$

Then, the equilibrium constant that been applied to the acid-base system is:

i. Sulphuric Acid (H_2SO_4)

$$K_1 = \frac{[H^{+}][HSO_4^{-}]}{[H_2SO_4]}$$
$$= \mathbf{1.0 \text{ x } 10^{3}}$$

$$K_2 = \frac{[H^{+}][SO_4^{-2}]}{[HSO_4^{-}]}$$
$$= \mathbf{1.2 \text{ x } 10^{-2}}$$

ii. Water *(H_2O)*

$$K_w = [H^{+}][OH^{-}]$$
$$= \mathbf{1.0 \text{ x } 10^{14}}$$

As shown in the equation above, there are two value of acid dissociation constant for sulphuric acid which is K_1 and K_2. It is because of the sulphuric acid is a diprotic acid. Which means this type of acid have two equilibrium point.

Thus, the pH value can be calculated by using the decimal logarithm that measures the ion concentration. The equation is as below:

$$pH = -log_{10}[H^{+}]$$

In order to find the value of the hydrogen ion $[H^+]$, the electroneutrality equation needs to solve. Then, subtitute the non-reactant component equation and the value of equilibrium constant into the electroneutrality equation to get the final equation in polynomial form.

The final equation also can be known as pH equation as below:

$$[H^+]^4 + a_1 [H^+] + a_2 [H^+] + a_3 [H^+] + a_4$$

Where:

$$a_1 = K_1 + \beta$$

$$\alpha_2 = \beta K_1 + K_1K_2 - K_w - K_1\alpha$$

$$\alpha_3 = \beta K_1K_2 - K_1K_w - 2K_1K_2\alpha$$

$$a_4 = -K_1K_2K_w$$

From the equation above (the final equation), the pH neutralization process model can be developed represent pH neutralization process pilot plant that been used in this project. Figure 4.2 shows the model that creates using MATLAB/Simulink interface.

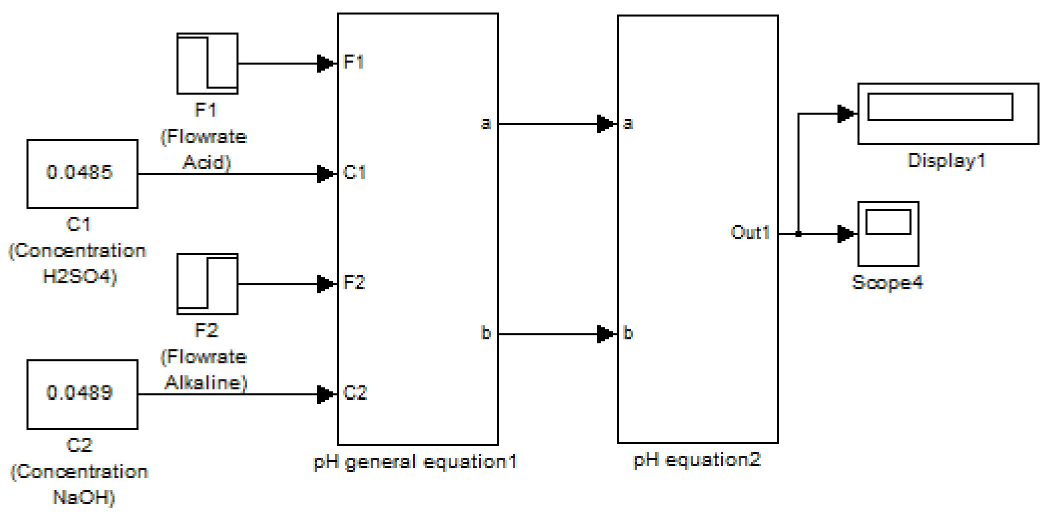


Figure 4.2: MATLAB/Simulink Blocks of the pH Neutralization on Process Model

From the figure above, the first block is the dynamic part. This block involves the equation of the pH process in the CSTR system. The remains equation is located at the second block. All equation in the second block is run by S-Function which is a computer language description of a Simulink block written in MATLAB. Using S-Function function in this part can reduce block diagram in Simulink and make the system easier to understand.

The next stage is model analysis. The objective of this stage is to analyses and evaluates the dynamic response of the developed model whether the simulation result is acceptable or not. The analysis of this model is based on comparison between the results from simulation and experimental. The next part will shows the detail of simulation result and experimental result of this process.

4.3 Open Loop Experiment Results

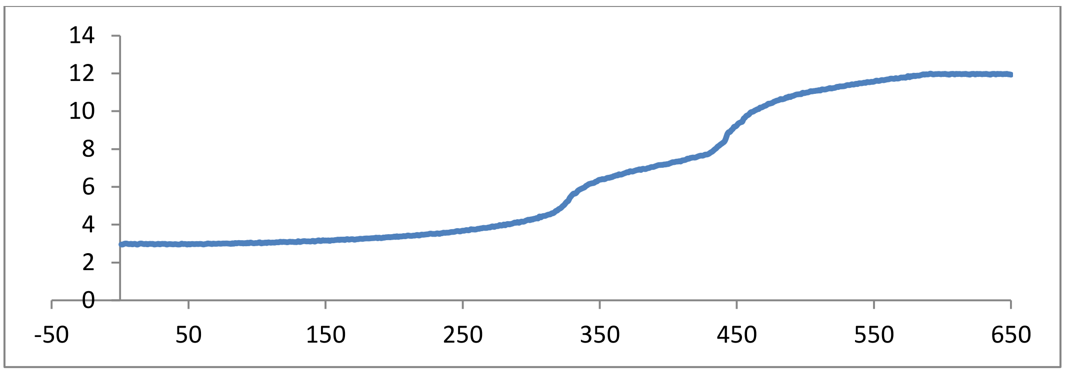
At this stage, two experiments were carried out to provide information about the dynamic behavior of the pH neutralization process. The result from this experiment are used to compare with simulation result that been design before.

4.3.1 Experimental Result

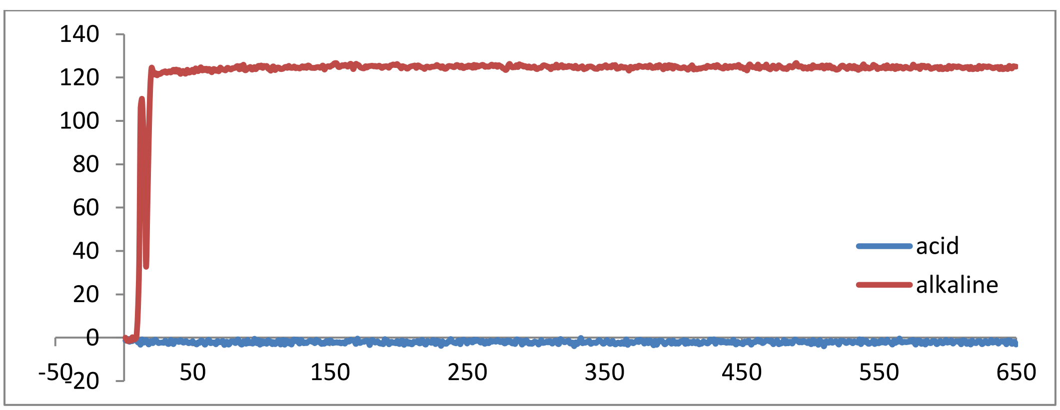
These experiments involves a continuous process, the reactor tank is filled until reach the maximum level (80 L) and the level will be constant since the flow going out from the tank at this point. The initial pH value will be set to a desired value by controlling acid and alkaline stream manually.

Two experiments were carried out to provide information about the dynamic behavior of the pH neutralization process. The result used to validate the developed model described in previous section. The first of this experiment involves a step change of alkaline flow where the acid stream was set to the fully closed position.

Figure 4.3 shows the result of the first experiment. To get the dynamic response of pH neutralization, the initial pH in this experiment was set to the lowest possible value. The preparation of this experiment requires a lot of acid solution to bring down the pH value to the lowest possible value. After several trial, the value of initial pH that been set for this experiment is 3. Another parameter that been looking before run this experiment been shows in Table 4.1.



(a) Process reaction curve of the experiment



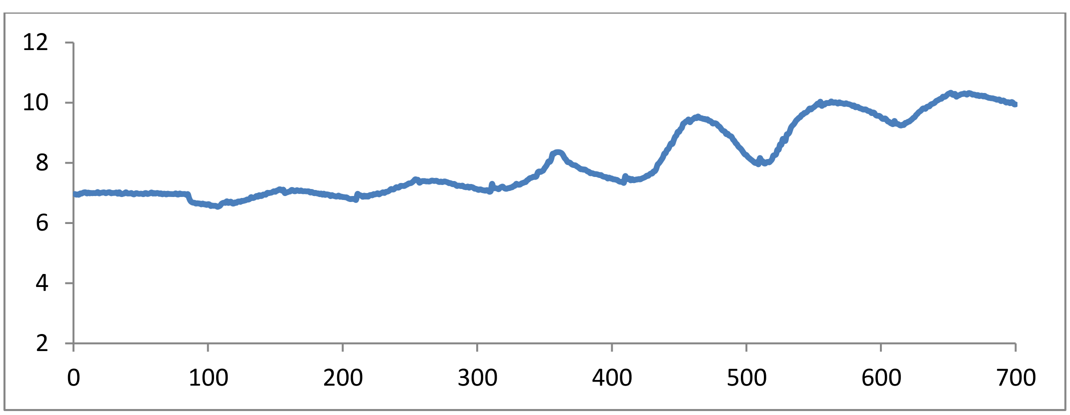
(b) Flowrate of the acid and alkaline streams

Figure 4.3: Experimental results during a test involving a step change of the flow rate for the alkaline stream

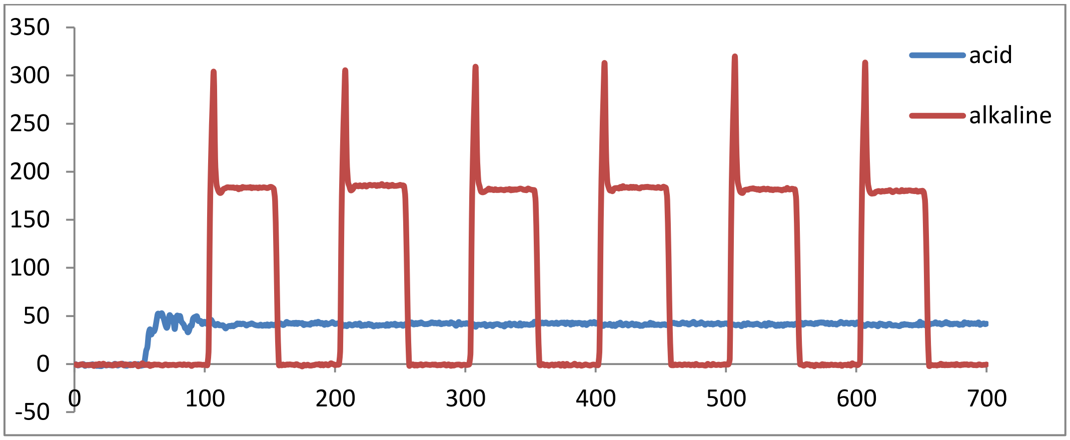
The experimental result as shown in Figure 4.3 shows clearly the nonlinearity of the process. The dynamic behavior shows two different equilibrium points. The equilibrium point depends on the value of dissociation constants. As explained before, sulphuric acid which is type of diprotic acid theoretically have two equilibrium points. However, due to the first dissociation constant that large compare to the second one, the equilibrium cannot be seen clearly on the titration curve.

As shown in the result figure before, it is believed that the dissociation constant for the acid has been decreased due to some other reaction in process tank. Process of dilute acid in water may have an additional and unknown source of hydrogen and hydroxyl ions. This situation will make the ionic strength been decrease and as a result the dissociation constant will decrease too.

The result from the second experiment is shown in Figure 4.4. It shows another dynamic response of pH neutralization process. The objective of this experiment is to obtain further insight about how to control the alkaline stream and the pH value in the reactor tank with a constant flow of acid stream. In this experiment, the initial value that been set is 7. The acid valve was set to an opening which provided a constant flow value of acid stream. The flowrate of alkaline had been set to square wave variation with same value of alkaline stream at the peak. Another parameter of this experiment has been shows in Table 4.2.



(a) Dynamic response from the experiment



(b) Flowrate of the acid and alkaline

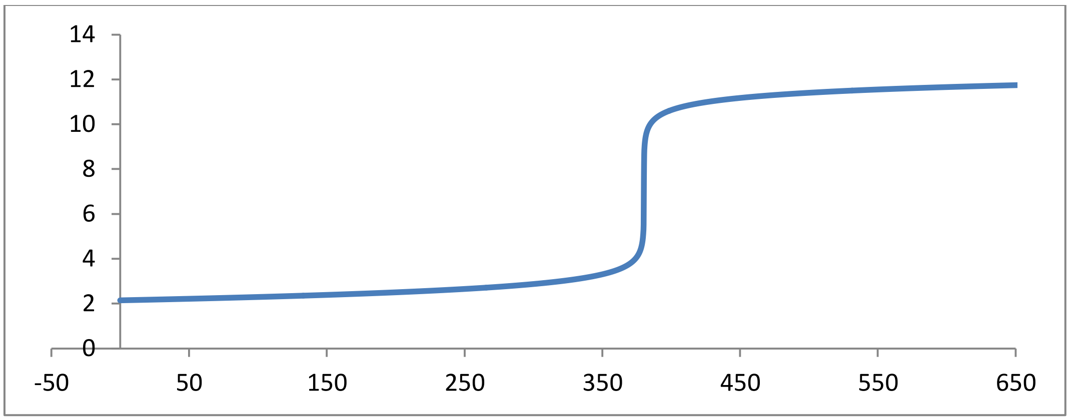
Figure 4.4: The dynamic response for square-wave variation of alkaline experiment

Experiment	Concentration		Flowrate	
	Acid	**Alkaline**	**Acid**	**Alkaline**
Experiment 1 (Process reaction curve)	0.0523	0.05361	The valve is open until the pH value reach the required initial value in reactor tank.	Increased from zero to 125.14L/h at 20 sec.
Experiment 2 (Square wave signal)	0.0523	0.0536	Increased from zero to 40.5L/h at 50 sec	Square wave signal with period of 50s and flowrate of 185.15 L/h

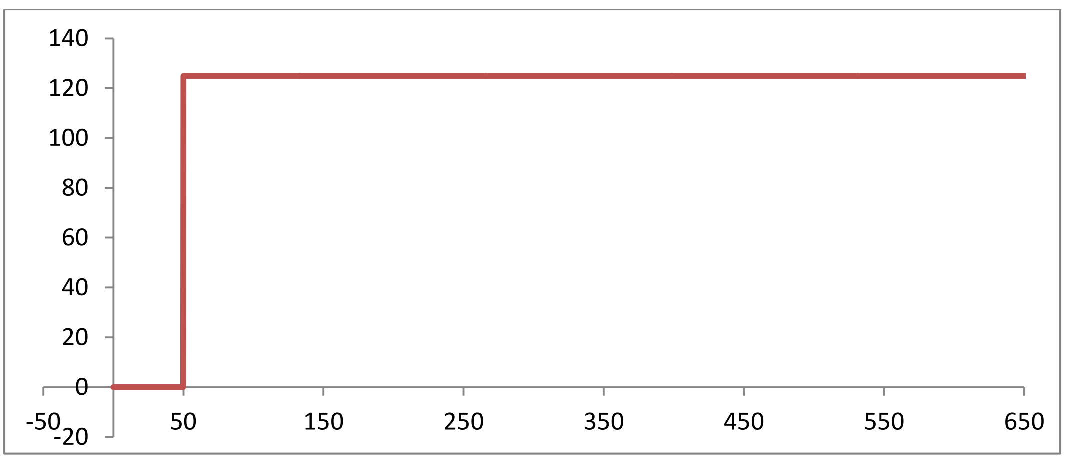
Table 4.1: Parameter setting for experimental work

4.3.2 Simulation Result

For simulation part, computational method was carried out to simulate the experiment before. The simulated experiments were based on actual setting and parameter that been used in experimental part. The results then been compared to get validation for the model. Figure 4.5 and Figure 4.6 shows the graphical simulation result and Table 4.2 shows the parameter setting for the simulation method.

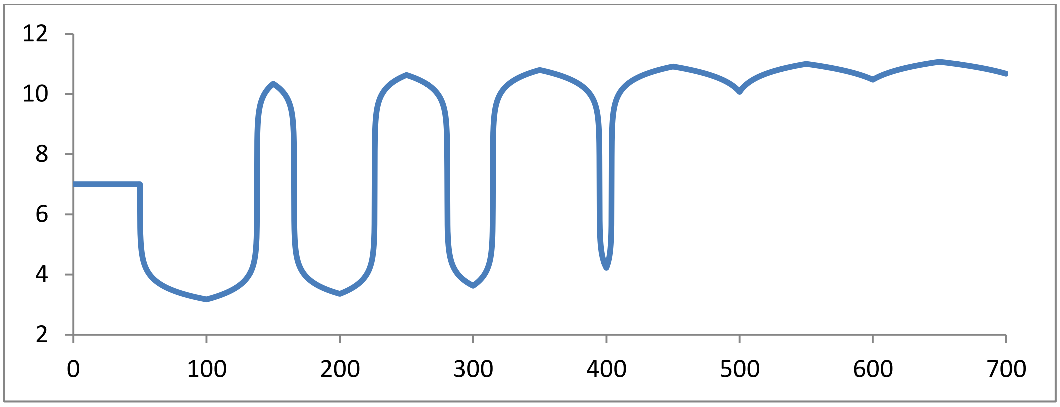


(a) Process reaction curve for simulation 1

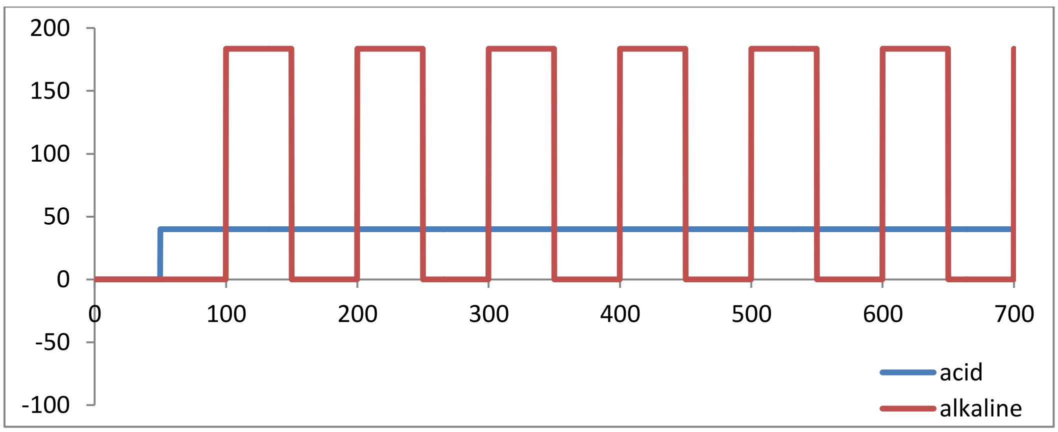


(b) Step change of alkaline stream

Figure 4.5: Process Reaction Curve from the pH Model



(a) Dynamic response of simulation for experiment 2



(b) Flowrate of acid and alkaline

Figure 4.6: Dynamic response of pH model for experiment 2

Experiment	Concentration		Flowrate	
	Acid	**Alkaline**	**Acid**	**Alkaline**
Experiment 1 (Process reaction curve)	0.0523	0.0536	Step change until the pH value reaches the required initial value in reactor tank.	Increased from zero to 124.92L/h at 50 sec.
Experiment 2 (Square wave signal)	0.0523	0.0536	Increased from zero to 40L/h at 50 sec	Square wave signal with period of 50s and flowrate of 183.6 L/h

Table 4.2: Parameter setting for simulation work

Simulation result shown in Figure 4.5 and Figure 4.6 should be similar to the actual response obtained in the experimental. But the result shows clearly that the dynamic responses from both experiment of simulation model do not properly represent the response as the actual pilot plant done. So, the model needs further investigation and modification of the pH neutralization process model. The next section will describe some modification to the pH neutralization process model in order to make the pH dynamic model more reasonable.

4.4 Development of the Modified pH Model

The investigation will focused mainly on the dissociation constant of the acid solution. A similar approach to determine the dissociation constant of the acid solution, which is based on the titration curves will be used as a guidelines. Based on the process reaction curve before, the graphical approached was used to calculated the dissociation constants as below [19] [20].

i. First dissociation constant, K_1

$$pK_1 = -\log K_1$$
$$K_1 = \text{antilog}(-pK_1)$$
$$= 6.31 \times 10^{-4}$$

ii. First dissociation constant, K_2

$$pK_2 = -\log K_2$$
$$K_1 = \text{antilog}(-pK_2)$$
$$= 1.59 \times 10^{-7}$$

Figure 4.7 and Figure 4.8 shows the simulation result of both experiment for modified pH model. The parameter and structure of model be maintained except the values of dissociation constant. The dynamic response shown in figure 4.7 has similar pattern as compared to the dynamic response from experimental method. There are two equilibrium points represent two dissociation constant for diprotic acid. Besides, the modified pH model has shown very encouraging results compared to the experimental work. However, the result of dynamic response shown in Figure 4.8 not exactly same as in the experimental results shown in Figure 4.4. Next section will evaluate the performance of this modified model of experiment 2 based on results from modified model and experimental work.

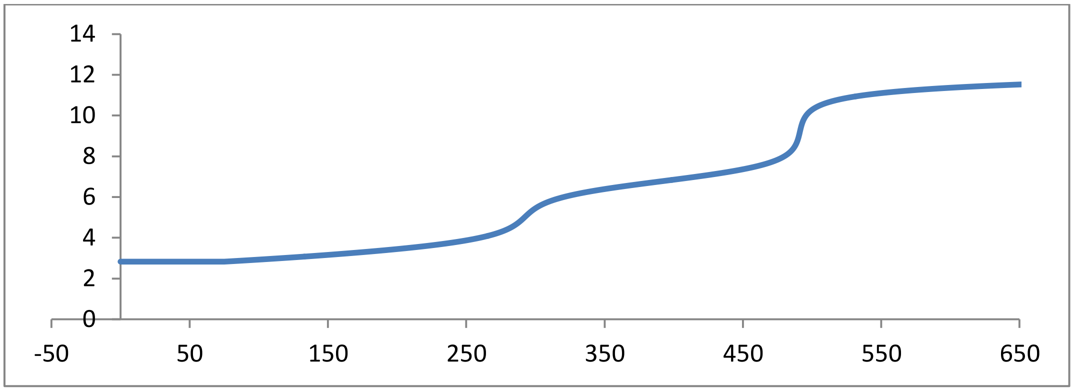


Figure 4.7: Process reaction curve from the modified pH model

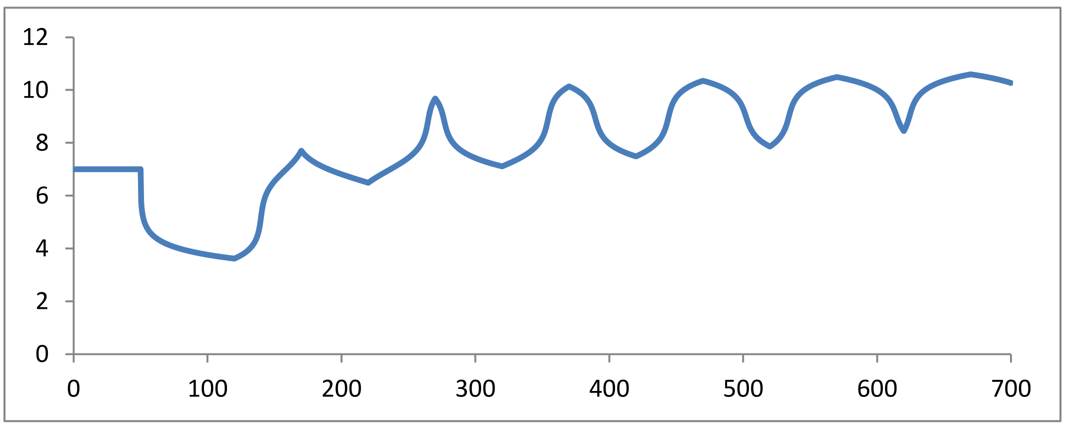
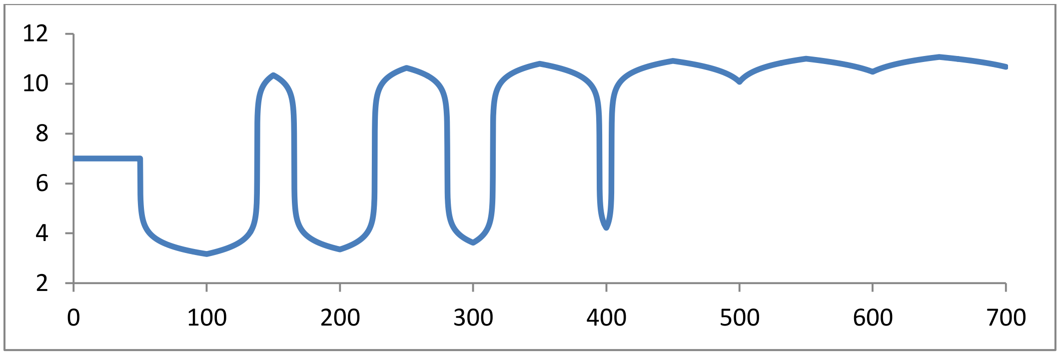


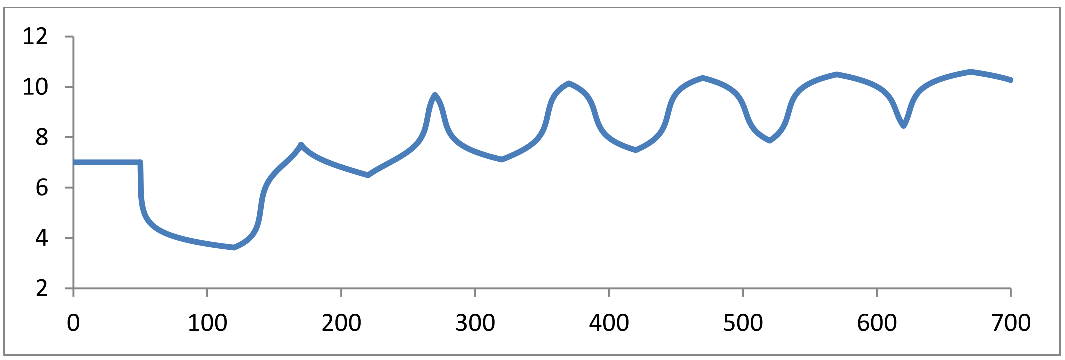
Figure 4.8: Dynamic response of pH modified model for experiment 2

4.5 Evaluation of the Modified model

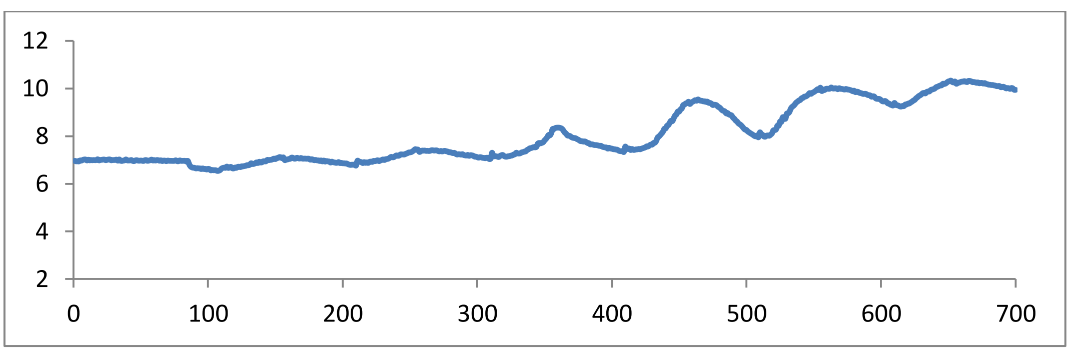
Figure 4.9 shows the comparison between the first model, modified model and experimental work for experiment 2. It may be seen that there are distinctly different dynamic responses for the three cases presented.



(a) Dynamic response from the first model



(b) Dynamic response from the modified model



(c) Dynamic response from the actual experiment

Figure 4.9: Comparison of dynamic response in three different works

As shown in figure 4.9, the first model did not give the reasonable response as the actual experiment. The pH range of this model is between 4 and 10. This response does not represent the actual dynamic response of the pH neutralization pilot plant. The modified model shows the improvement of the dynamic response and looks more likely as the actual experiment. However, in the first 150 seconds, the pH value decreases rapidly unlike the actual experiment. This is due to the mathematical modeling that acts fast than the real one. However, the modified model still can be accepted compared to the first model. Next chapter is to design the controller based on this modified model.

5.0 DEVELOPMENT OF A CONVENTIONAL PI CONTROLLER

This section will provide an overview of PID controller in this process which is specific to the pilot plant that been used in this project. This project will be used Proportional Plus Integral (PI) controller without the derivative component. This controller already been used in process industry since 1940s and remains the most commonly used algorithm today due to fact that this controller is very simple in structure and easy to design.

The PID controller has three adjustable control parameters that which are proportional, integral and derivatives that affect the control performance. If all the parameters have been set properly, this controller is able to provide a reasonably good performance. However, the control performance also depends on the process. The nonlinear processes are predictably more difficult to control compared to linear process by using the PID algorithm. This is because parameter values of the controller that are design for one part of the operating range may be completely unsuitable for some other operating point.

5.1 Overview of the PID Controller

Basically a PID controller has three control modes which are Proportional, Integral and Derivative. The proportional mode is a simple gain factor that makes the control action proportional to the error signal. The error increases, the adjustment to the manipulated variable also increase. This control mode can reduce the offset error but it does not give a zero offset in process application.

The second mode in the PID controller is integral mode. This integral mode achieves zero offset although it adjust the manipulated variable in a slower manner than the proportional mode. This giving poor dynamic performance and can make the system unstable if it tuned improperly.

The last mode is the derivative mode. This mode has no direct influence to the final steady state value of error. However, the derivative mode can provide rapid correction on the rate of change of the controlled variable. In many cases the derivative mode did not use due to increasing the effect of measurement noise that can damage the overall performance of the controller.

In pH neutralization process, many of previous researchers have used PI controller as a benchmark to compare to other controller. This research using the PI controller as a reference against Fuzzy Logic Controller as a type of advanced controller.

Figure 5.1 shows the MATLAB/ Simulink that represent PI controller for the pH neutralization pilot plant. In PID block below, the tuning is made by change the value of proportional and integral only and the derivative gain will be leave as zero value. The saturation block that shows in Figure 5.1 is to set the lower and the upper limit by the user. For this process the value that been set for lower limit is 0 and 100 for the upper limit. These values represent the position of a valve that cannot be open over the limit value which is 100% for fully open condition and cannot be negative value since 0% is the fully closed condition.

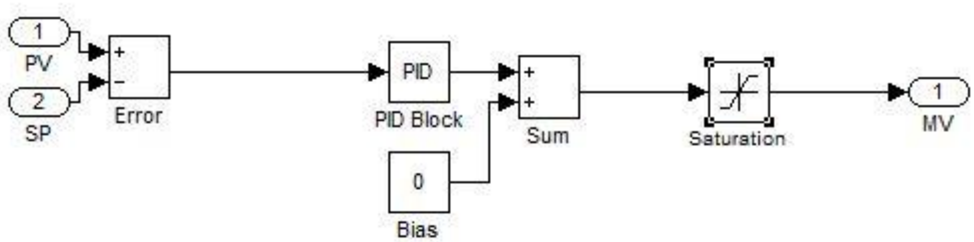


Figure 5.1: MATLAB/ Simulink of PI controller

5.2 Simulation Work of the PI Controller

Figure 5.2 shows the MATLAB/ Simulink block diagram that represent the complete form of pH neutralization pilot plant including PI controller. FCV 121 is the block diagram that represents flow control valve movement for the alkaline stream. The method that been using to represent this flow control valve is by using a first order transfer function to provide linearized model of flow process of control valve itself.

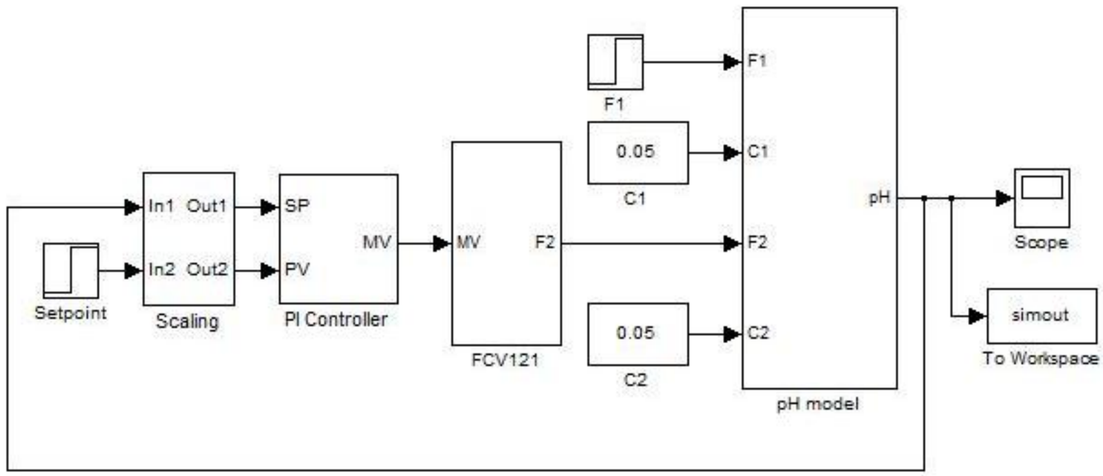


Figure 5.2: MATLAB/ Simulink represent the complete form of pH neutralization pilot plant including PI controller

5.3 Tuning Method for PI Controller

The most widely method that been used for establish appropriate parameter value for PID controller is Ziegler-Nichols tuning method. This method works very well especially in nonlinear systems which using trial and error procedure to determine the parameter values for PID.

There are some procedures that need to follow using this method. Firstly, the proportional gain been set to the minimum value and zero value for the integral and derivative gain. The proportional gain must be adjusted until the amplitude of oscillations become constant. The final value of this proportional gain is called as ultimate gain (Gu) and the period of one oscillation is called (Pu).

Table 5.1 shows the Ziegler-Nichols tuning formula. This formula based on two values that been mentioned earlier which is ultimate gain (Gu) and period for one oscillation (Pu). The value of controller parameter that been determined using this formula will apply to the PID controller directly.

	P	PI	PID
Proportional, K_P	0.5Gu	0.45Gu	0.6Gu
Integral, T_I	-	1.2K_P/Pu	2K_P/Pu
Derivative, T_D	-	-	K_PPu/8

Table 5.1: Ziegler-Nichols Tuning Formula

After several trials, the proportional gain that gives the constant oscillation is 20. Figure 5.3 shows the pH response in the process tank that get from this proportional gain

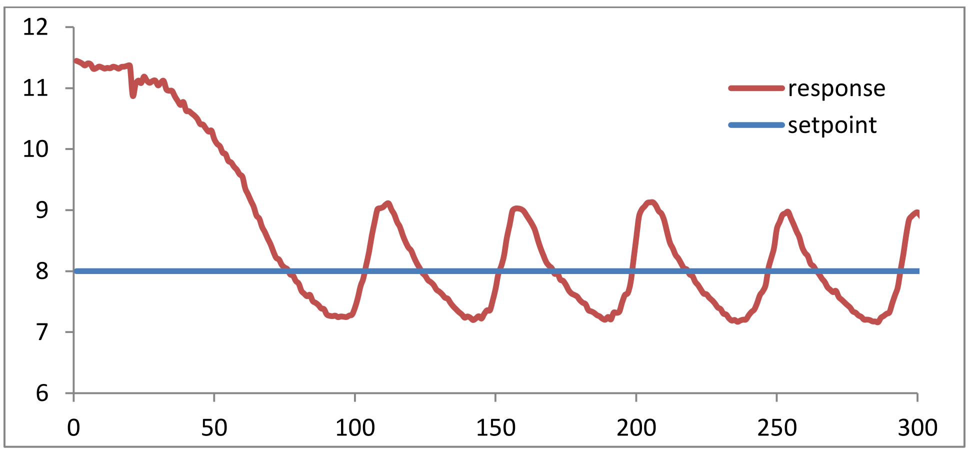


Figure 5.3: PID tuning response

As shown in Figure 5.3, the ultimate proportional gain from this experiment is 20. The period for one oscillation that get from this tuning approximately in 48 seconds. The PI controller parameter been calculated as follows:

Proportional Gain, K_P = 0.45 x 20

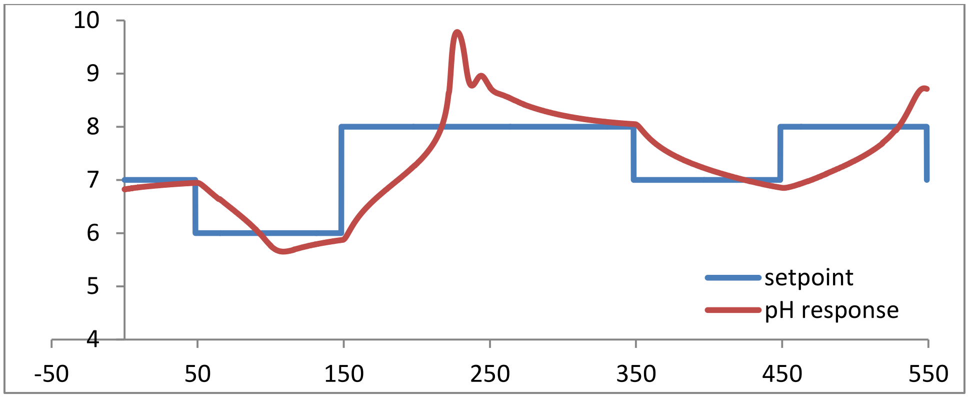
= 9

Integral Gain, T_I = (1.2 x 9) / 48

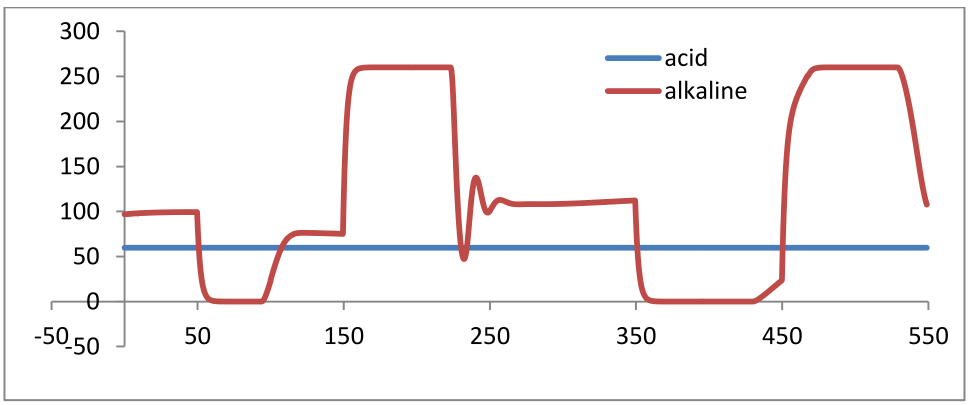
=0.225

5.4 Simulation Results of PI Controller

Figure 5.4 shows the performance of PI controller that response to the setpoint changes. The parameter that been used in proportional and integral gain is 9 and 0.225 that get from tuning method before.



(a) pH response



(b) Flowrate of alkaline and acid stream

Figure 5.4: PI controller performance

As be seen in Figure 5.4, the performance of the PI controller not too encourage since the process value does not follow setpoint value in the required time. The overshoot value also is quite high that occur when the value of setpoint increase or decrease. The performance will compare to the Fuzzy Logic Controller that will design in next chapter.

6.0 DEVELOPMENT OF A FUZZY LOGIC CONTROLLER

This chapter describes the development of an advanced controller which is Fuzzy Logic Controller. The design of Fuzzy Logic Controller based on certain set point only which are 8 since the requirement need from environmental legislative is range of 6 to 8. Simulation of this model is run by MATLAB/ Simulink.

6.1 Fuzzy Inference System of pH Neutralization Process

The set point that need for this process is around 6 to 8. The controller that been design by the author is for set point of 8 only by controlling the alkaline flow. Figure 6.1 shows the MATLAB/ Simulink model including Fuzzy Logic Controller.

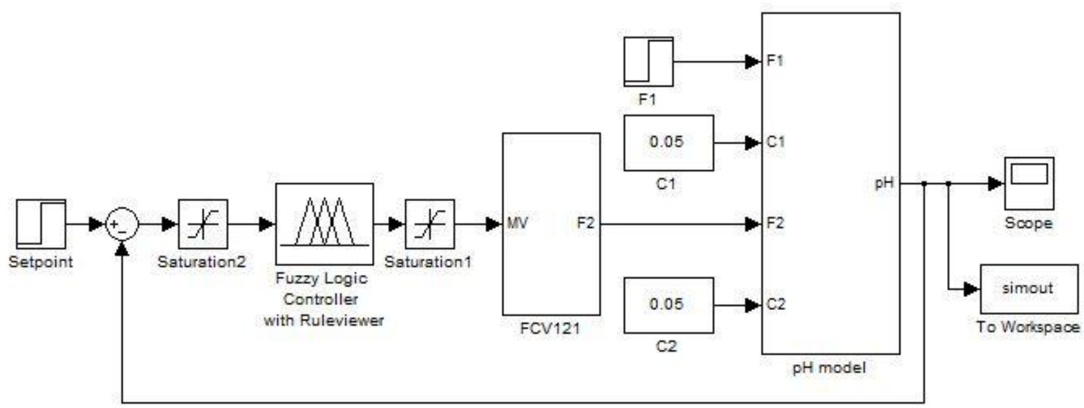


Figure 6.1: MATLAB/ Simulink represent the complete form of pH neutralization pilot plant including Fuzzy Logic Controller

The range of error for this controller is between -3 to 3 and this is matched to the range for pH value for neutralization process which is in range from 6 to 8. The output of the controller will control the flowrate of the alkaline stream. The range that been used for the output is configured in between of -100L/h to 180L/h. The saturation block that comes after Fuzzy Logic Controller is from 0 L/h to 180L/h. This range of this value been selected based on alkaline control valve which is minimum value is 0 L/h (fully closed) and 180 L/h for maximum value (fully open).

The membership functions for the input set of the controller shown in Figure 6.2 and description that been setting for this controller shown in Table 6.1. There are nine groups that been used for membership function in this pH controller. The range in the middle point seems to be detailed to ensure the smoothness of settling condition. However, the performance of the Fuzzy Logic Controller is based on the combination of the input and output sets.

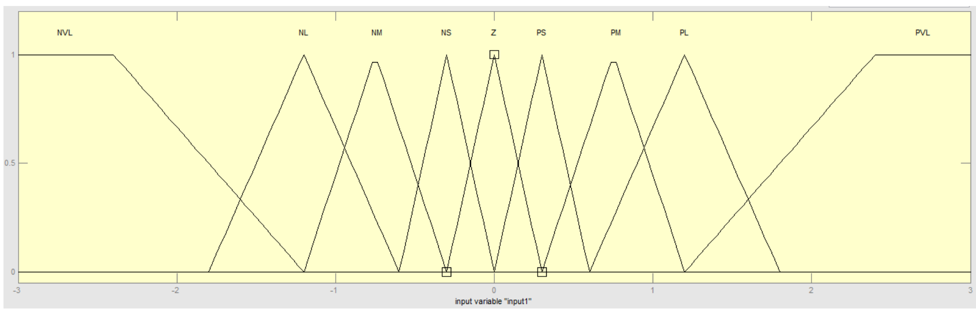


Figure 6.2: Membership function for input set

Symbol	Description	Type
NVL	Negative Very Large	Trapezoid
NL	Negative Large	Triangular
NM	Negative Medium	Triangular
NS	Negative Small	Triangular
Z	Zero	Triangular
PS	Positive Small	Triangular
PM	Positive Medium	Triangular
PL	Positive Large	Triangular
PVL	Positive Very Large	Trapezoid

Table 6.1: Membership function description for input set

Figure 6.3 and Table 6.2 shows the membership functions of the output set and their description. The parameter values of this output determined based on trial and error procedure. The design of the output step need understanding of alkaline flow when it needed to increase or decrease based on error of pH value to make a reasonable time response.

In the output set, the membership function also has nine groups same as the input set. The triangular shape at the middle seems very narrow. The reason is to make a small step change based on small pH error then make the alkaline valve react accordingly. If the membership function is too large, it will make poor performance of the controller with large overshoot and unwanted oscillation.

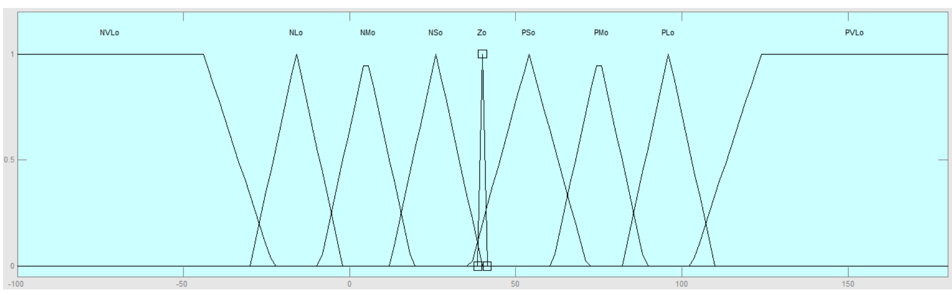


Figure 6.3: Membership function for output set

Symbol	Description	Type
NVLo	Negative Very Large	Trapezoid
NLo	Negative Large	Triangular
NMo	Negative Medium	Triangular
NSo	Negative Small	Triangular
Zo	Zero	Triangular
PSo	Positive Small	Triangular
PMo	Positive Medium	Triangular
PLo	Positive Large	Triangular
PVLo	Positive Very Large	Trapezoid

Table 6.2: Membership function description for output set

Table 6.3 shows the relationship between the input and the output of the pH Fuzzy Logic Controller. Since this system represents straightforward process which is one input and one output case, the if-then rule statement be apply for this controller. The idea of how this controller work is shown in Figure 6.4. When the error is in positive value, the valve should take the action to increase the flowrate by increase the opening percentage of valve. On the other hand, when the error comes to negative value, the opening valve should be decrease to slowdown the flowrate. This form is seen to be linear to error values.

No	Statement	Input	Statement	Output
1	IF	NVL	THEN	NVLo
2	IF	NL	THEN	NLo
3	IF	NM	THEN	NMo
4	IF	NS	THEN	NSo
5	IF	Z	THEN	Zo
6	IF	PS	THEN	PSo
7	IF	PM	THEN	PMo
8	IF	PL	THEN	PLo
9	IF	PVL	THEN	PVLo

Table 6.3: If-then rule statement for Fuzzy Logic Diagram

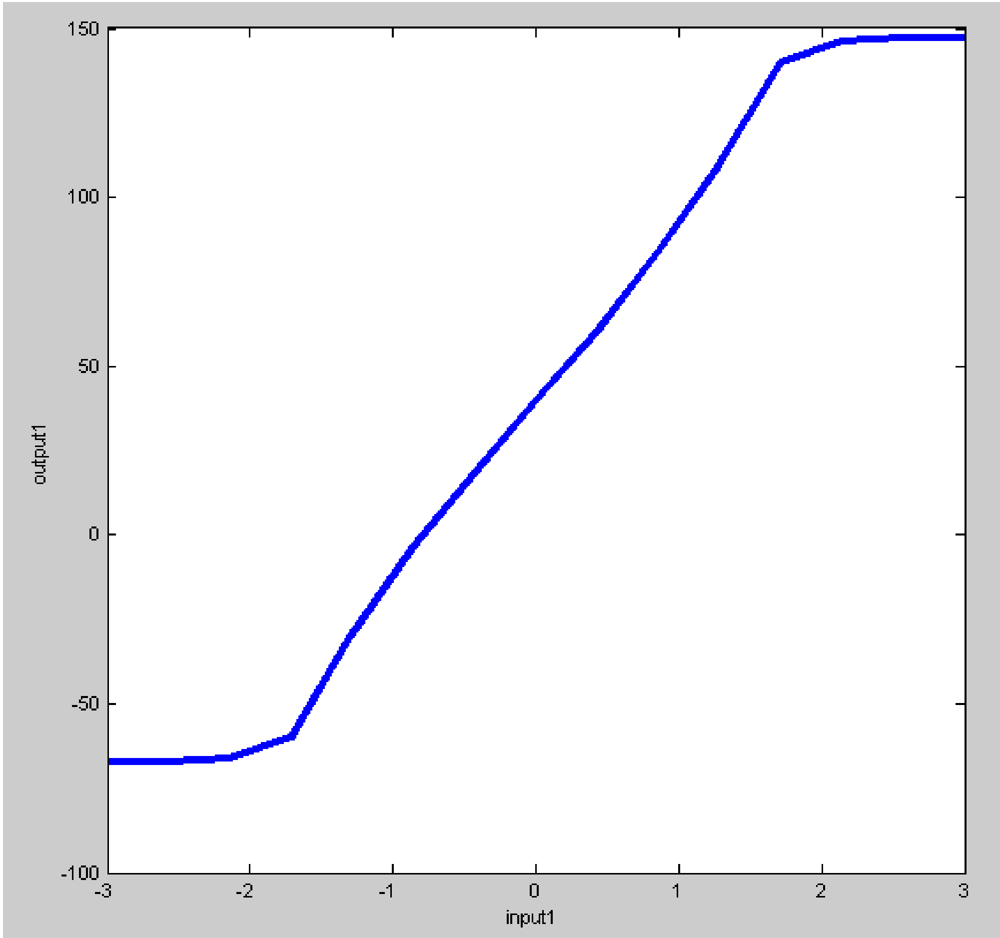
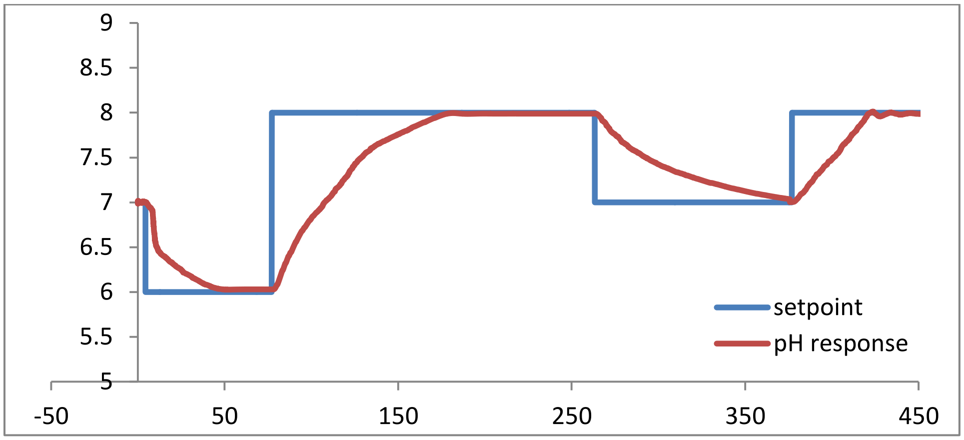


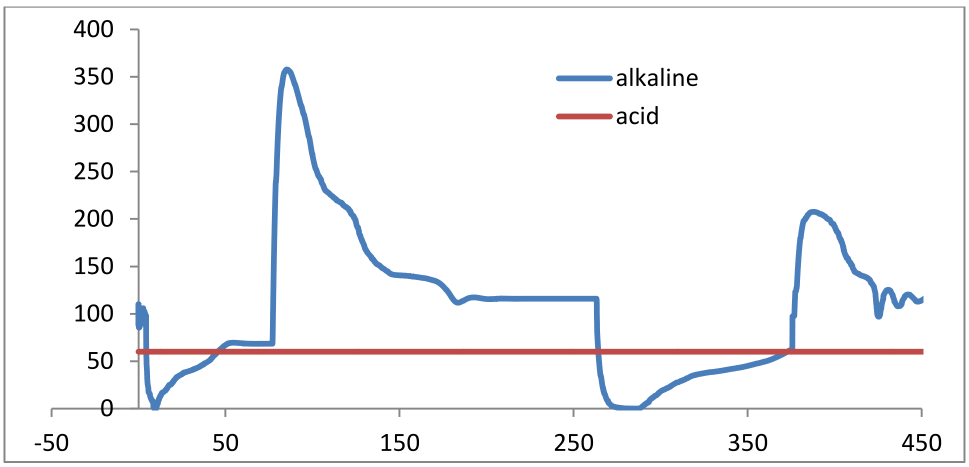
Figure 6.4: Fuzzy Logic Controller Response

6.2 Simulation Results of Fuzzy Logic Controller

Figure 6.5 shows the performance of Fuzzy Logic Controller due to changes of setpoint values. The results shown in this section based on performance of MATLAB/ Simulink modified model with Fuzzy Logic Controller. Setpoint values that been choose in this test in between 6 to 8.



(a) pH response



(b) Flowrate of alkaline and acid stream

Figure 6.5: Fuzzy Logic Controller performance

As Shown in the Figure 6.5, the Fuzzy Logic Controller seems stable since the process value follows the setpoint values. There are no overshoot in this controller performance. The IAE value is smaller compared to the PID controller. The settling time that been seen in Fuzzy Logic controller also smaller compared to PID controller. The comparison between two controllers will be discussed further in next part.

6.3 Comparison between Fuzzy Logic Controller and PI Controller

Next is the comparison between PI controller and Fuzzy Logic Controller. The controller only response to a single step input which is from initial value 6 to 7. The comparison is based on offset, integral absolute error (IAE), settling time, rise time and percentage overshoot. Figure 10 shows the result of two different controller performances.

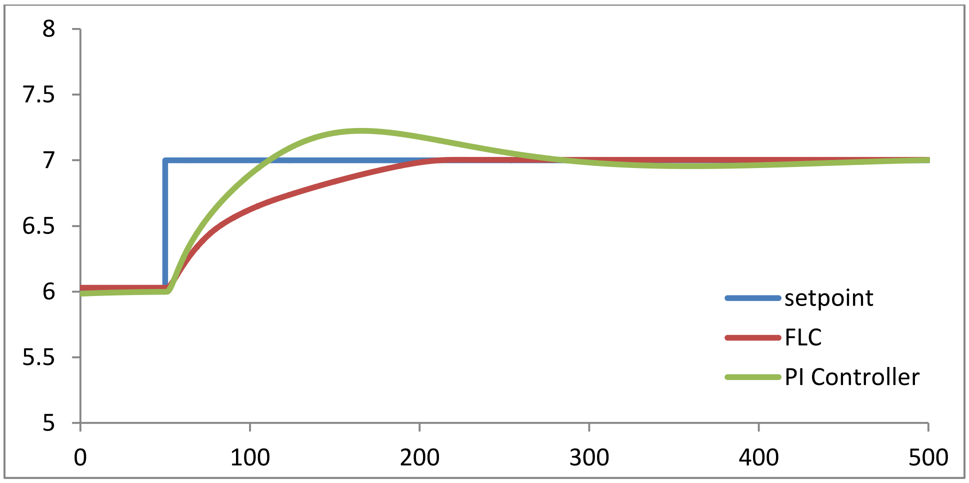


Figure 6.6: Comparison of the response between PI Controller and Fuzzy Logic Controller

As shown clearly in the figure above, the performance of Fuzzy Logic Controller much better compare to PI Controller. Table below summarizes results of performance between two controllers.

Control Performance	PI Controller	Fuzzy Logic Controller
Offset	Zero	Zero
IAE	406.1	65.18
Rise Time, T_r	54	153
Settling Time, T_s	434	153
Percentage Overshoot	21.3%	0%

Table 6.4: Comparison of two controller's performance

7.0 CONCLUSION AND RECOMMENDATION

7.1 Conclusion

As stated before, there are two objectives that need to achieve by the author. The first objective was to develop a nonlinear pH model represent pilot plant in UTP. Since the result from experimental part and simulation part quite similar, the development of pH neutralization process model for the pH plant has been achieved. However there are still some improvements that can be made to the modified pH in order to make the pH model more accurate. The difference at initial response proposes that the model could be modified further. Additional experimental work need to carry out in order to investigate further more about the efficiency of the mixing process, delay of control valve and also the movement of control valve.

The second objective for this research was to design and developed an advanced controller in this process. The proposed controller that been used in this project is Fuzzy Logic Controller. The designation and the development of Fuzzy Logic Controller have been achieved by the author in 8 months period. The comparison between Fuzzy Logic Controller and Conventional PID Controller can be done by using the pH model that been design before. The result shows that Fuzzy Logic Controller performed better than PID controller. The comparison that been made based on the stability, IAE, overshoot and settling time value. The Fuzzy Logic Controller has better IAE (small), overshoot (small), and settling time (fast) compared to PID controller.

7.2 Recommendation

Further study about this project is to implement the both controllers to the actual pilot plant. The comparison between simulation work and experimental work can be done after the implementation succeed. Further improvement of the controller needs if the performance on the implementation not to be same as in the simulation part. The additional types of advance controller need to design also. It would be interesting to find out the differences between the other types of controller in terms of their performance, robustness and stability

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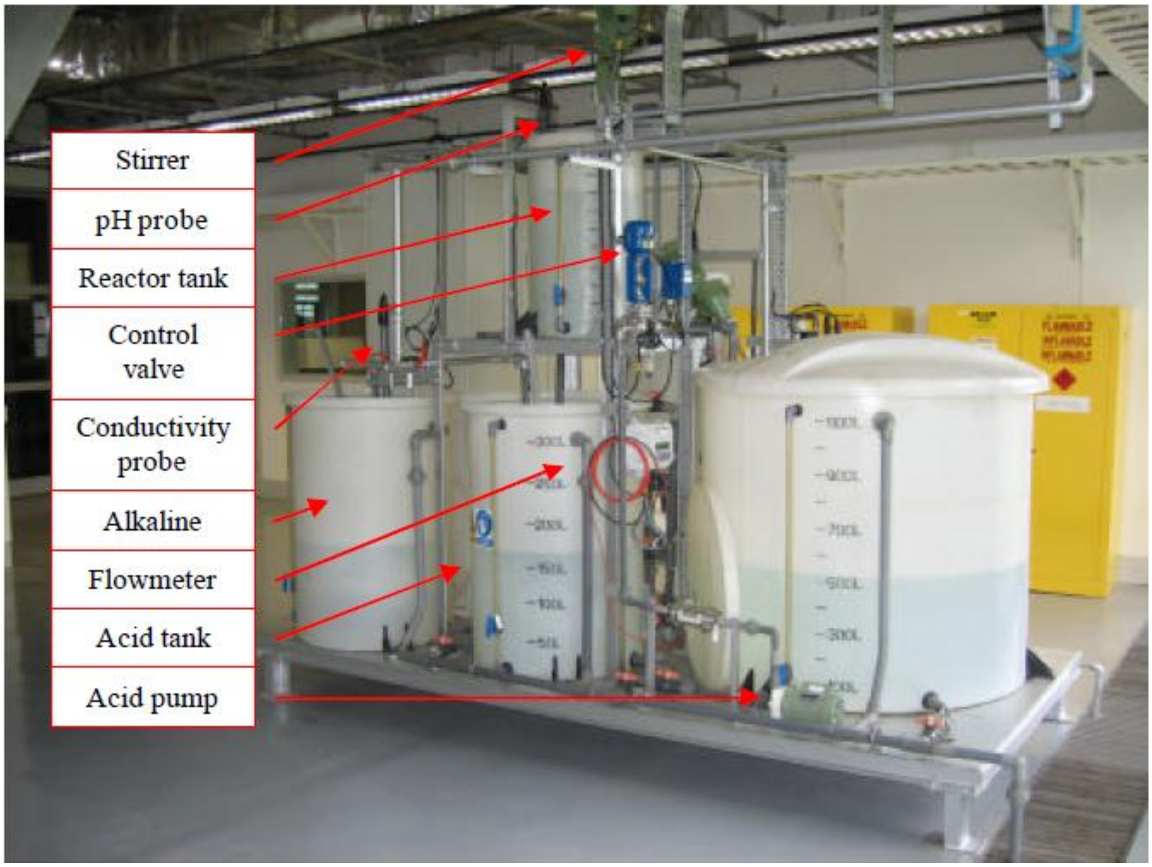
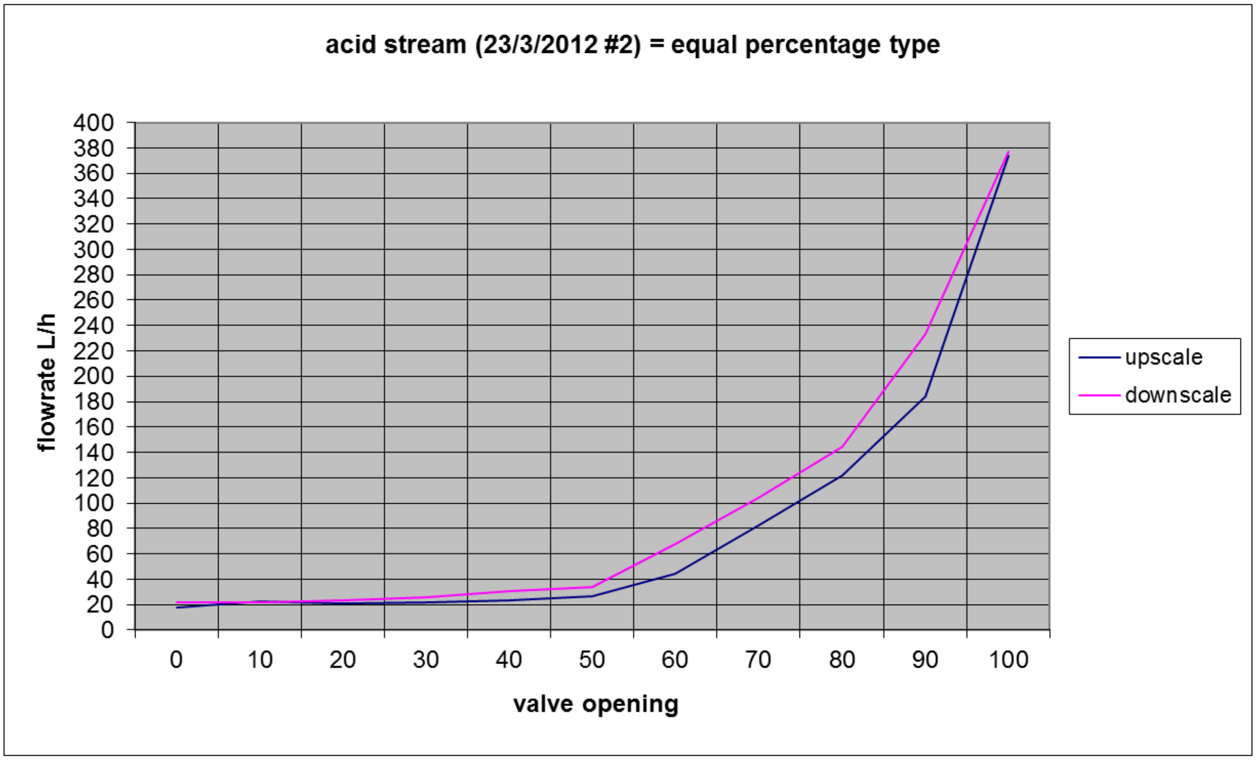
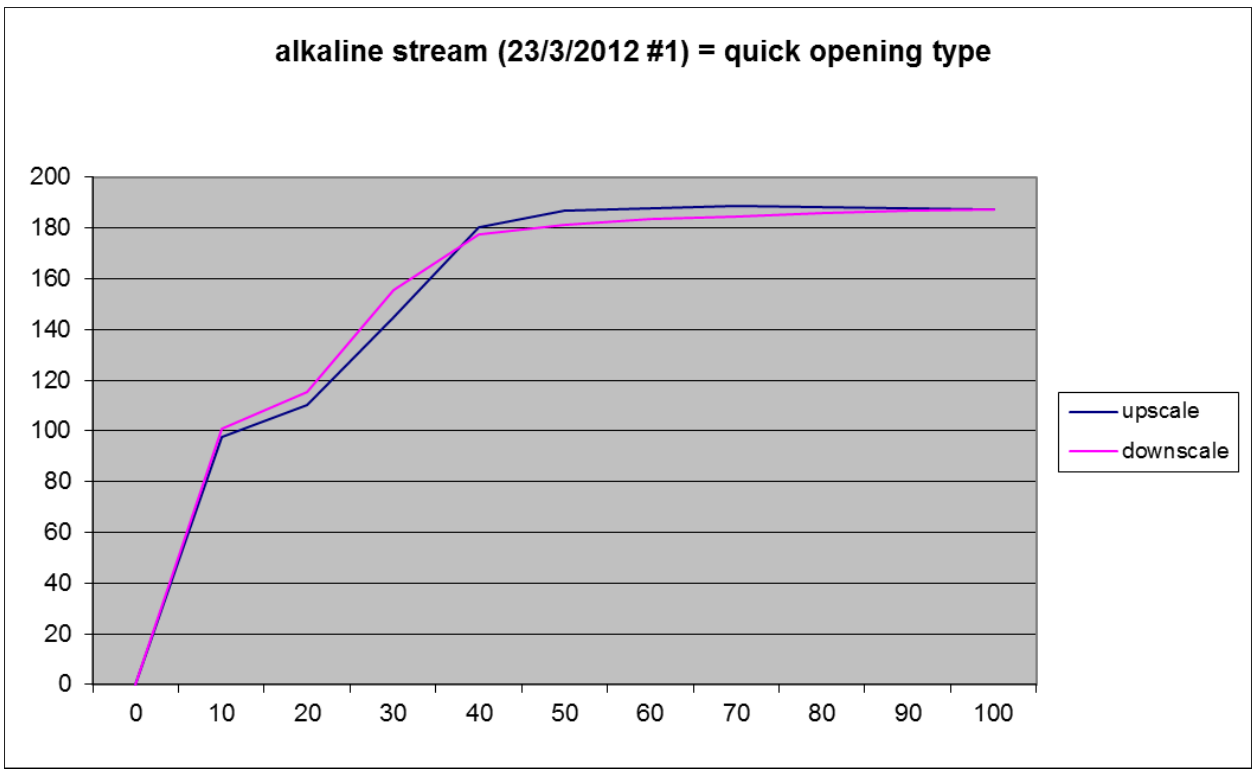
9.0 APPENDICES

Appendix I: pH neutralization process pilot plant



Appendix II: Valve Characteristic





Appendix III: Technical Specification of the pH Meter

i. Controller

Model : alpha-pH1000 1/4 DIN pH/ORP Controller

VIEW ENLARGED IMAGE

Product Features

- :: Built-In Programmable Limit, Proportional (Pulse Length or Pulse Frequency) - ideal for precision process control applications
- :: User-Customization through Advanced Setup Menu offers flexibility in matching the controller's functions to suit individual's specific requirement
- :: Automatic Calibration with Auto-Buffer Recognition e liminates mistakes during calibration
- :: Symmetrical Mode Operation eliminates electronic noise problems when used with solution ground
- :: One-Point Online Calibration without shutting down the line
- :: Hold Relay for use with float switches/flow switches and other controllers as a failsafe function
- :: Two Level Password Protection prevents un authorized tampering with settings
- :: 0 to 2000 Second Time Delay Adjustment on control and alarm delays
- :: Two Galvanically Isolated Scaleable 0-20/4-20 mA Outputs for pH/ORP
- :: Wash Contact Relay controls electrodes cleaning systems at desired duration and frequency
- :: Choice of Glass or Antimony Electrode for general purpose or hydrofluoric acid applications
- :: Adjustable Hysteresis (Dead Band) prevents rapid contact switching near set point
- :: Non-Volatile Memory retains all stored parameters and calibration data even if power fails
- :: Large Dual Display shows pH (or ORP) with temperature simultaneously - features clear multiple icons, set points, and status messages
- :: Choice of Temperature Sensor Pt100/Pt1000 with 2-wire or 3-wire temperature input selection
- :: Easy Installation and Wiring with detachable plug-in connectors

Applications

General: Useful for any batch or on-line type application that requires accurate pH or ORP control.

Water Purification/Treatment: Use for batch and on-line control of incoming process water, rinse water treatment, recirculating system and waste water treatment.

Industrial: Ideal for chemical processing, food processing. aquarium, pharmaceutical, hydroponics and waste control industries.

Regulatory: Hook to recorder to document data for regulatory compliance.

ii. pH Process Electrode

Model: EC100GTSO-05B

Specifications

Product Specification	Description
pH Range	0 to 14
Reference	Annular Teflon, double junction
Reference electrolyte	Saturated KCl, polymerized gel
Operating temperature	0 to 80 °C
Pressure tolerance	6 bars
Temperature sensor	Pt 100
Potential matching pin	Platinum
Material	PPS (Ryton)
Thread	3/4" NPT
Cable	Integral 5m low-noise semi-conductor screened
Connector	BNC
Dimensions: Length (excludes cable)	151 mm
Diameter (external)	26 mm
Weight	650 g

Appendix IV: Layout of User Interface for Experimental Work