

CONTROL OF pH IN NEUTRALIZATION  
REACTOR OF A WASTE WATER TREATMENT SYSTEM  
USING IDENTIFICATION REACTOR

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Approval of the Graduate School of Natural and Applied Sciences.

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## **ABSTRACT**

### **CONTROL OF pH IN NEUTRALIZATION REACTOR OF A WASTE WATER TREATMENT SYSTEM USING IDENTIFICATION REACTOR**

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A typical wastewater effluent of a chemical process can contain several strong acids/bases, weak acids/bases as well as their salts. They must be neutralized before being discharged to the environment in order to protect aquatic life and human welfare. However, neutralization process is highly non-linear and has time-varying characteristics. Therefore, the control of pH is a challenging problem where advanced control strategies are often considered.

In this study, the aim is to design a pH control system that will be capable of controlling the pH-value of a plant waste-water effluent stream having unknown acids with unknown concentrations using an on-line identification procedure. A Model Predictive Controller, MPC, and a Fuzzy Logic Controller, FLC, are designed and used in a laboratory scale pH neutralization system. The characteristic of the upstream flow is obtained by a small identification reactor which has ten times faster dynamics and which is working parallel to actual neutralization tank. In the control strategy, steady-state titration curve of the process stream is obtained using the data collected in terms of pH value from the response of the identification reactor to a pulse input in base flow rate and using the simulated response of the identification reactor for the same input. After obtaining the steady-state titration curve, it is used in the design of a Proportional-Integral, PI, and of an Adaptive Model Predictive

Controller, AMPC. On the other hand, identification reactor is not used in the FLC scheme. The performances of the designed controllers are tested mainly for disturbance rejection, set–point tracking and robustness issues theoretically and experimentally. The superiority of the FLC is verified.

Keywords: pH Control, Non–linear Control, Identification, Model Predictive Control, Fuzzy Logic Control.

## ÖZ

### TANIMLAMA TEPKİME TANKI KULLANARAK, ATIK SU ARITIM SİSTEMİNİN NÖTRALİZASYON TANKINDA pH DEĞERİNİN DENETLENMESİ

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Kimyasal bir sürecin atık suyunda çeşitli kuvvetli ve zayıf asitler/bazlar ile bunların tuzları da barınmaktadır. Atık sular, suda yaşayan canlıların ve insan sağlığının korunması amacıyla çevreye deşarj edilmeden önce nötralize edilmelidir. Ancak, nötralizasyon noktası civarında titrasyon eğrisinin eğimindeki ani deęişmeler nedeniyle, pH süreçleri doğrusal olmayan ve zamanla deęişen bir dinamięe sahiptirler. Bu nedenle, zor olan pH deęerinin denetiminde genellikle gelişmiş denetim teknikleri düşünölmektedir.

Bu çalışmada, bilinmeyen derişimde ve çeşitlilikte asit içeren bir süreç atık suyunun pH deęerini, hat-üstünde tanımlama metodu kullanarak denetleyebilecek denetim sistemleri tasarlanmıştır. Bir Model Öngörömlü Denetleç, MÖD, ve bir Bulanık Mantık Denetleç, BMD, tasarlanmış ve laboratuvar ölçekli bir nötralizasyon sistemine uygulanmıştır. Atık suyun karakteristikleri, ana nötralizasyon tankına paralel olarak çalışan ve yaklaşık olarak ana tanktan on kat daha hızlı bir dinamięe sahip küçük bir tanımlama tankından elde edilmiştir. Denetim stratejisinde, süreç akımının yatışkın–durum titrasyon eğrisi, tanımlama tankının pH deęerinin bu tanka giren baz akımına uygulanan bir vuru etkiye verdięi yanıt ile aynı vuru için süreç modelinin benzetimle elde edilen tepkisinin birlikte kullanılmasından elde edilmektedir. Yatışkın–durum titrasyon eğrisinin elde edilmesinden sonra, Oransal–Entegral, PI, ve Uyarlamalı Model Öngörömlü Denetleç, UMÖD, tasarımlarında kullanılmıştır. Öte

yandan tanımlama tankı Bulanık Mantık Denetleç, BMD, tasarımında kullanılmamıştır. Tasarlanan tüm denetleçler öncelikle bozan etkeni uzaklaştırma, ayar noktası takibi ve gürbüzlük performansları açısından incelenmiş ve Bulanık Mantık Denetleç'in en iyi performansı verdiği belirlenmiştir.

Anahtar Kelimeler: pH Denetimi, Doğrusal Olmayan Denetim, Tanımlama, Model Öngörümlü Denetim, Bulanık Mantık Denetim.

To my family, for their love and encouragement...



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## NOMENCLATURE

a	Step response coefficient
A	Dynamic matrix
b	Total ion concentration in the titrating stream (mol/L)
c	Total ion concentration in the process stream (mol/L)
C	Control horizon
d	Disturbance
e	Error between output of the pH process and its target value
E	Closed-loop prediction error
E*	Open-loop prediction error
f	Move suppression factor, ratio of diagonal elements of input and output weighting matrices
F	Process stream flow rate (L/min)
g	Gradient vector
H	Impulse response coefficients
H	Hessian matrix
K	Equilibrium constants
K <sub>p</sub>	Process transfer function gain
M	Model horizon
P	Prediction horizon
pK	Negative logarithm of dissociation constant, $-\log_{10}(K)$
r	Set-point vector
t	Time (s)
u	Titration stream flow rate (L/min)
V	Volume (L)
W <sub>1</sub>	Weight of the error
W <sub>2</sub>	Weight of the input changes
x	Total ion concentration in an acid/base mixture (mol/L)
y	Process output
Y	Strong acid equivalent (mol/L)

**Greek Letters:**

$\alpha$	Gain multiplication factor
$\lambda$	Weighting matrix diagonal element
$\tau$	Process transfer function time constant
$\Delta$	Increment

**Subscripts:**

i	$i^{\text{th}}$ component
id.	Identification reactor
main.	Main reactor
max	Maximum
min	Minimum
sp	Set-point

**Superscripts:**

'	Deviation form
*	Current measurement
-1	Matrix inverse
past	Effects of known past inputs on the predicted outputs of the plant

**Abbreviations:**

AMPC	Adaptive Model Predictive Control
BEP	Basic Evolutionary Programming
DFAC	Dynamic Fuzzy Adaptive Controller
DMC	Dynamic Matrix Control
FIS	Fuzzy Inference System
FLC	Fuzzy Logic Control
IACC	Integral of the Control Change
IACE	Integrated Absolute Control Effort
IAE	Integral of the Absolute Error
MF	Membership Functions
MPC	Model Predictive Control
PI	Proportional Integral
WMPC	Wiener Model Predictive Control



## CHAPTER I

### INTRODUCTION

Many of the chemical processes exhibit non-linear behavior due to changing process gain in different operating points. The control of these processes is not easy due to this non-linear behavior. Control of pH neutralization is an example of such processes and it is classified as one of the most important and difficult control problems in the chemical process industry especially in the category of waste-water treatment processes. In addition to the non-linear behavior, they also have time-varying characteristics.

In waste-water treatment plants influent stream composition changes continuously due to changes in waste streams of different processing units. The non-linearity of these processes is because of the steady-state S-shaped titration curve which also gives variable steady-state gain. Also, the change of influent acid composition results in a change in the shape of the titration curve with time.

During the last two decades, tradition is changed in the control area and model-based controllers are used frequently in process industries. Therefore, to enable such a control algorithm, titration curve of the influent stream has to be known and must be corrected or simply updated with time. "Despite potential problems in continuous model updating, success of pH control relies on the correctness of the titrating curve. Another approach, instead of continuously updating model parameters, is to reparameterize the titration curve when necessary" (Lin and Yu, 1993).

In the literature, different approaches to identify and to control with adaptation of the pH processes can be found. Some of these methods are; reaction invariant control (Gustafsson and Waller, 1983), strong acid equivalent control (Wright and Kravaris, 1991, 2001; Wright et al. 1991), gain scheduled PI control (Lin and Yu, 1993), fuzzy logic control, (Garrido et al., 1997, Adroer et al., 1999), adaptive control (Lin and Yu, 1993; Wright et al., 1998; Sung et al., 1995; Sung et al., 1998), model predictive control (Maiti et al., 1994; Norquay et al.,

1998; Galán et al., 2004 and Lu et al., 2004) Regunath and Kadiramanathan (2001). Actually, to overcome time-varying non-linear character of the pH processes, adaptive controllers must be used.

In this study, an identification reactor is used to identify the characteristics of the influent stream of a neutralization process, which is the steady-state titration curve, similar to the study of Wright et al. (1998). The pH process model developed by Wright and Kravaris (1991) is combined with the suggested identification method to overcome the time-varying dynamics of the influent stream. An adaptive model predictive controller and a fuzzy logic controller are then designed in terms of the strong acid equivalent concept and in terms of pH, respectively, and used for the control of a neutralization process.

The outline of this thesis is as follows. Literature survey on pH process modeling and pH control with advanced control techniques are given in Chapter II. A short review of Wright and Kravaris's (1991) minimal-order realization model for the pH processes with PI controller algorithm is presented in Chapter III which is followed by brief reviews of Model Predictive Control, MPC, (Chapter IV) and Fuzzy Logic Control, FLC, (Chapter V). In Chapter VI, the experimental set-up is described. The next chapter, Chapter VII, is devoted to the results of the simulation and experimental studies together with discussions. Conclusions are given in Chapter VIII.

## CHAPTER II

### LITERATURE SURVEY

The control of pH is recognized as a difficult non-linear control problem and has been extensively treated in the literature due to its highly non-linear and time-varying behavior. Studies related to the development of models for static and dynamic behavior of the pH processes and different pH control applications will be presented in this Chapter.

The general approach for modeling pH processes was first developed by McAvoy et al. (1972) for the dynamics of pH in stirred tank reactors. Mathematical model was derived from material balances, equilibrium relations and electroneutrality condition, and it is generally applicable. Neutralization of acetic acid with sodium hydroxide was examined and the accuracy of the resulting mathematical model was verified experimentally by step input tests.

A general method for developing the dynamic model for acid-base reactions is presented in the work of Gustafsson and Waller (1983). The method is based on the separation of the model into reaction variant and invariant parts. In this way, dynamic model of pH process can be expressed by one linear differential equation as dynamic part and a non-linear implicit algebraic relation as static part. An application of adaptive reaction invariant feedback control of pH is also given. However, the controller requires the detailed model of the acid-base reactions.

Gustafsson (1985) experimentally tested reaction invariant feedback control and adaptive linear pH-feedback control of pH in fast acid-base reactions in stirred tank reactors under varying buffer concentrations. Dynamic model used in the controllers was developed using the earlier presented method of Gustafsson and Waller (1983). Parameters of the dynamic model first obtained using off-line identification experiments and they used in the design of the controllers. Experimental results for sulfuric acid-sodium carbonate-sodium hydroxide system were presented.

A general mathematical model for pH processes was reviewed by Wright and Kravaris (1991). A minimal-order model having same input-output behavior as the full-order model was derived and the strong acid equivalent concept first introduced. The strong acid equivalent is the state in the reduced model and it can be calculated from online pH measurements by knowing only the titration curve of the inlet process stream. pH control problem was reformulated and an equivalent control objective in terms of the strong acid equivalent was proposed. A linear controller in terms of strong acid equivalent was designed and the performance of the proposed controller was evaluated under a variety of concentration disturbances and modeling errors.

The method of strong acid equivalent introduced by Wright and Kravaris (1991) was implemented to a laboratory-scale pH neutralization system by Wright et al. (1991). A proportional-integral (PI) controller in terms of strong acid equivalent was designed to control a neutralization process in a continuous-stirred tank reactor. Neutralization of hydrochloric acid with sodium hydroxide and neutralization of acetic acid with sodium hydroxide were experimentally studied. Experimental results for both of the systems show that the pH control algorithm based on the strong acid equivalent concept can overcome disturbances in the presence of modeling errors.

Gustafsson and Waller (1992) discussed the methods of continuous control of pH in process streams. The paper investigates the need for adaptation and illustrates the advantages and disadvantages of linear and non-linear adaptive control methods for practical pH control. The simulations and experiments show that non-linear control is superior if the characteristic of the process is well-known. On the other hand, since the process characteristics are usually not well-known, linear feedback from pH is often as good as non-linear feedback. The most significant advantages of non-linear control over linear control is that, if the pH control system oscillates for shorter and longer periods of time, which is quite common in practice, non-linear feedback control results in tighter control with smaller amplitudes of the oscillations than that of linear feedback control.

Three-region fuzzy logic controller for controlling a non-linear pH process was demonstrated by Qin and Borders (1994). According to the prior knowledge of the pH process (steady-state titration curve), three operation range was determined and a fuzzy controller that uses pH value as an auxiliary variable was designed. Use of auxiliary enables the controller to make aggressive control action for low gain regions and to make mild control actions for high gain regions. Computer simulations were performed to evaluate the performance of the

proposed controller for controlling the pH value in a continuous–stirred tank reactor (CSTR) where neutralization takes place. Three–region controller was compared with conventional fuzzy controller and it was seen that proposed controller performed well for both set–point tracking and disturbance rejection issues.

Sung et al. (1995) developed a new identification strategy which consists a small identification tank to incorporate both non–linearity and time–varying characteristics of the pH process. They obtained the equivalent titration curve of the process stream from the identification reactor and used this information to transform the measured pH value of the main neutralization reactor to the state variable that is also the controlled variable in a PI controller. Their work includes a simple stability analysis to determine the parameters of the PI controller. Simulation results show that proposed method can handle both the non–linearities and time–varying characteristics of the pH process.

Gustafsson et al. (1995) discussed the chemistry of the acid–base reactions for more complex situations such as formation of metal complexes and precipitation of solids. Static and dynamic model for pH processes were presented. Approximations on the static model were also investigated and it is stated that “most systems can be approximated with fictitious mono–protic weak acids with an accuracy that is satisfactory for pH control purposes”.

Sung and Lee (1995) used a simple set–point change to obtain the two–parameter titration curve model for a pH process. In their proposed method, they used Wright and Kravaris’ (1991) control strategy and Yuwana and Seborg’s (1982) auto–tuning concept. Several simulations were performed to show the performance of the identification method. However, if the identification data were contaminated by the disturbances, the proposed method would not work well.

Predictive control of pH using non–linear fuzzy model was proposed by Kavšek-Biasizzo et al. (1997). A non–linear fuzzy model was used to obtain the step response coefficients of the pH process in an on–line manner. Then these coefficients were incorporated into the dynamic matrix control (DMC) structure in which dynamic matrix of the controller updated at each sampling interval instead of being fixed. Takagi–Sugeno type inference system was used to obtain Non–linear Auto Regressive with exogenous input (NARX) of the pH process. Base flow rate and the pH value were the inputs to the fuzzy inference system. The accuracy of the NARX model was tested by simulations and it was observed that the model can

accurately represent static and dynamic behavior of the pH process. Performance of the fuzzy predictive controller was tested using simulated bench scale pH process for only set-point tracking and smooth response was obtained.

Garrido et al. (1997) presented application of fuzzy logic control to a wastewater neutralization process. Effectiveness of the fuzzy logic was compared, through computer simulations, with other advanced controllers developed by Kurtz (1985), Riggs et al. (1990) and Gaulian et al. (1990). Fuzzy controller inputs were error in pH value and the error velocity which is the difference between actual error and that of the previous sampling time. The output was parameter S and actual flow ratio was adjusted using a power law relation. The pH process studied consists of a single CSTR having an acid wastewater stream and basic titrating stream as inputs. The control objective is to keep the effluent pH value at 7.0 in the presence of disturbances. Comparisons were performed for two subsections; (1) behavior of the controller under different buffering capacity of wastewater; (2) behavior of the controller under changing acid concentrations of the inlet wastewater. It was observed that the settling time of the pH response after a disturbance enters the system increases as the  $pK_a$  value of the acid decreases for all of the controllers tested except fuzzy logic controller. This is due to fact that, the process gain around  $pH = 7.0$  increases for acids with low  $pK_a$  value. The inverse behavior of the fuzzy logic controller showed that the controller was better for low buffering conditions (wastewater containing strong acids) and therefore, for unbuffered wastewaters fuzzy logic controller gave the best results for various step changes in acid concentration.

Wright et al. (1998) developed a new identification method for determining the non-linearity of pH processes. In their method, titration curve of the process stream fitted to a two parameter non-linear equation which was derived from the pH process model, assuming unknown species in the process stream are mono-hydroxyl bases or mono-protic acids. Resulting identification method incorporated into the strong acid equivalent control structure presented in previous work of Wright and Kravaris (1991). Several computer simulations and experimental results demonstrated the performance of the proposed identification method. Comparisons between the control systems that uses identification algorithm and fixed titration curve were also provided.

Wiener model predictive control (WMPC) applied to an experimental acid-base neutralization process by Norquay et al. (1998). In addition, a variety of model structures such as Auto Regressive with eXogenous inputs (ARX) and step response model were inspected with their

identification procedures. Due to its highly non-linear gain, the pH process was modeled as Wiener model, composed of a dynamic linear element followed by a cubic spline static non-linearity, and model validation was also performed in the study. It was experimentally shown that Wiener MPC gave better disturbance rejection and set-point tracking performance as compared to both linear MPC and conventional PID controller.

Sung et al. (1998) proposed an adaptive control strategy which uses an on-line recursive estimator for determining the total ion concentration and dissociation constant of a fictitious acid. Using the information obtained from the estimator a non-linear PI controller was designed and used to control the pH value of a neutralization reactor where an acid stream neutralized by a base stream. Several simulations were conducted to evaluate the effect of the feed composition variation, quantization of the actuator, parameters of the estimator and modeling errors and to suggest a guideline in choosing the system parameters. A good control performance and robustness established from the proposed controller under different modeling errors and measurement noise. However, it was assumed that feed stream was composed of a single 1-protic weak acid. Therefore, if a new chemical species enter the system that would change the shape of the titration curve significantly; the control performance would probably be poor.

Adroer et al. (1999) applied the fuzzy controller developed by Garrido et al. (1997) to a laboratory scale neutralization process. They modified the fuzzy controller such that output of the controller was adjusted by a scaling parameter, which is a function of last 15 error measurements, to increase the robustness of the controller for varying buffering capacities of wastewater streams. Proposed controller performance was tested experimentally for changes in the wastewater buffering capacity, changes in the concentration of acid and changes in the type of acid in the wastewater stream. Results revealed that proposed fixed structure fuzzy logic controller with a tuning factor effectively control the neutralization process with variable buffering capacities.

Wright and Kravaris (2001) successfully applied the non-linear controller developed by Wright et al. (1998) to an industrial pH process. The controller was tested for several different set-point pH values and found to be very beneficial to the plant and some of the earlier problems associated with the process were eliminated. Closed-loop performance of the non-linear controller was evaluated under normal operating conditions as well as a number of hardware problems and satisfactory control performance was obtained for each case.

Regunath and Kadirkamanathan (2001) applied fuzzy non-uniform scheduling approach to weak acid-strong base pH neutralization process. Two dimensional Sugeno type fuzzy inference system (FIS) was used to determine proportional and integral gains of a PI controller. Set-point pH and effluent pH values were inputs to the fuzzy inference system and proportional and integral gains of the PI controller were the outputs from the inference system. Six asymmetric Gaussian membership functions used in the standard fuzzy system and they optimized by using basic evolutionary programming (BEP). BEP optimized fuzzy non-uniform grid scheduling control scheme tested via simulations and its performance compared with gain standard scheduled PI, dynamic gain scheduled PI and the standard non-uniform grid scheduling scheme in terms of integral of absolute error (IAE) and integrated absolute control effort (IACE) criterions. It was shown that by using BEP optimized fuzzy non-uniform grid scheduling, IAE score reduced by 67% while having an increment of 0.013% in the IACE score compared to the standard scheduling PI controller.

Babuska et al. (2002) implemented a fuzzy-self tuning PI controller for controlling the pH value of a small scale laboratory fermentation system. The control objective is to keep the pH value of the fermentation medium within the range of  $\pm 0.05$  pH units. Designed control system consists of two parts; an adaptive PI controller and a fuzzy self-tuning mechanism which has two inputs as error and its time derivative. Proportional gain and integral time of the PI controller adjusted on-line by a single parameter updated using the output of the fuzzy inference system. The controller was tested via simulations and then experimentally verified. It was shown that the fuzzy self-tuning PI controller was capable of controlling the pH value of a fermentation process accurately within the required range of pH.

Venkateswarlu and Anuradha (2004) introduced a dynamic fuzzy adaptive controller (DFAC) with a new inference mechanism for the control of pH processes. The DFAC contains a low-level basic control phase and a high-level dynamic learning phase by which the rule base of the basic control phase is modified on-line during the control of the pH process. The DFAC learning mechanism takes error and change of the error as inputs to evaluate the controller performance from the past control trends and then it appropriately modifies the rule base of the basic control phase. Proposed control scheme was applied to a weak acid-strong base pH process using simulations. The performance of the DFAC was tested for regulatory and servo performances under different buffering conditions and compared with conventional PI controller, fuzzy controller and adaptive PID controller. The simulated results show that the



DFAC controller provides improved performance for the control of highly non-linear pH processes.

Linearizing feedforward-feedback control scheme was successfully applied to a pH process by Kalafatis et al. (2004). The pH process was modeled as a Wiener model consisting one dynamic linear element and a static non-linear element which is actually the titration curve of the pH process. Linearization performed using inverse of the exact titration curve and its estimate. Computer simulations verified the superiority of the linearizing feedforward-feedback control scheme over the linearizing feedback scheme. However, an accurate estimate of the titration curve is vital for each controller.

Neutralization of acetic acid with sodium hydroxide in a CSTR was studied by Kumar et al. (2004). The Wiener model consists of a linear dynamic element and a static non-linear element was used to model the pH process. Static non-linearity mapped by a combination of odd-order polynomials, and a non-linear PI controller designed based on the Wiener model. The non-linear PI controller was tuned by pole placement method and further improved by using the method of inequalities. Also, for different operation zones a local linear PI controller were designed and compared with the non-linear PI controller. Several computer simulations and experiments demonstrated the superiority of the non-linear controller over the local linear PI controller and the usefulness of the method of inequalities for designing the non-linear PI controllers was shown.

Galán et al. (2004) successfully applied multi-linear model-based controllers to a laboratory scale neutralization reactor. The pH neutralization process modeled by first-order transfer function models for different operation regions according to the titration curve of the neutralization process. Based on these models, a standard PI, a gain scheduled PI, an  $H_{\infty}$  multi-linear controller and a multi-linear model predictive controller were designed. The controllers were experimentally tested for set-point tracking and flow rate disturbance performances. Excellent closed-loop behavior of the multi-linear controllers was shown

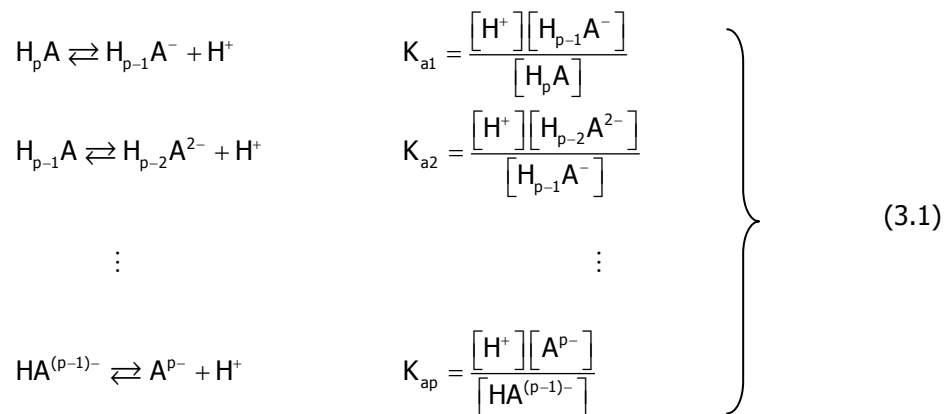
## CHAPTER III

### MODELING STUDIES

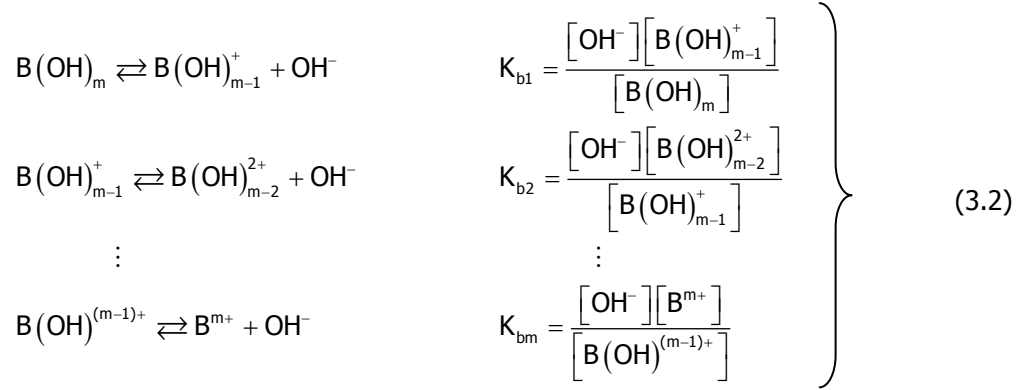
The modeling of single acid/single base pH process was achieved by McAvoy et al. (1972) and their model was generalized by Gustafsson and Waller (1983) and later by Wright and Kravaris (1991) by introducing the strong acid equivalent concept. The model is based on first principles, material balances, and also chemical equilibria. In this chapter, a brief review of mathematical modeling for the pH processes developed by Wright and Kravaris (1991) will be given.

#### 3.1. pH PROCESS MODELING

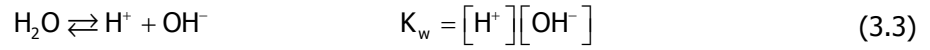
Consider an acid–base reaction process between a p–protic acid and m–hydroxyl base in water. The chemical equilibria and definitions of equilibrium constants for a p–protic acid, where p value usually takes 1, 2 or 3, can be written as:



Similarly for an  $m$ -hydroxyl base, the chemical equilibrium and definitions of equilibrium constants can be written as follows:



The equilibrium of water is:



At this point the total ion concentration of each acid and base will be defined as in Wright and Kravaris (1991): "The total ion concentration of the  $i^{\text{th}}$  species is defined, if  $i$  is an acid, as the sum of all concentrations of species containing the anion of the acid, or if  $i$  is a base, as the sum of all concentrations containing the cation of the base" as:

$$x_i = [\text{H}_p\text{A}] + [\text{H}_{p-1}\text{A}^-] + \dots + [\text{A}^{p-}] \quad \text{if } i \text{ is an acid} \quad (3.4)$$

$$x_i = [\text{B(OH)}_m] + [\text{B(OH)}_{m-1}^+] + \dots + [\text{B}^{m+}] \quad \text{if } i \text{ is a base} \quad (3.5)$$

In addition to the equilibrium stated above, the solution containing acid and base should be electrically neutral. Therefore this electroneutrality condition requires that:

$$\sum_{i=1}^{\# \text{acid}} \{ [\text{H}_{p_i-1}\text{A}^-] + 2[\text{H}_{p_i-2}\text{A}^{2-}] + \dots + p_i[\text{A}^{p_i-}] \} + [\text{OH}^-] = \sum_{j=1}^{\# \text{base}} \{ [\text{B(OH)}_{m_j-1}^+] + 2[\text{B(OH)}_{m_j-2}^{2+}] + \dots + m_j[\text{B}^{m_j+}] \} + [\text{H}^+] \quad (3.6)$$

The chemical equilibria (Eqs. 3.1 – 3.3), definitions of total ion concentrations (Eqs. 3.4 and 3.5), and the electroneutrality condition (Eq. 3.6) can be combined to obtain a single algebraic equation (Wright and Kravaris, 1991) which is called the "pH equation" as:

$$\sum_{i=1}^{\# \text{species}} a_i([\text{H}^+])x_i + [\text{H}^+] - \frac{K_w}{[\text{H}^+]} = 0 \quad (3.7)$$

where the  $a_i([\text{H}^+])$  is defined for an acid as:

$$a_i([\text{H}^+]) = \frac{p_i + (p_i - 1) \frac{[\text{H}^+]}{K_{a_{p_i}}} + \dots + \frac{[\text{H}^+]^{p_i-1}}{K_{a_2} K_{a_3} \dots K_{a_{p_i}}}}{1 + \frac{[\text{H}^+]}{K_{a_{p_i}}} + \dots + \frac{[\text{H}^+]^{p_i-1}}{K_{a_2} K_{a_3} \dots K_{a_{p_i}}} + \frac{[\text{H}^+]^{p_i}}{K_{a_1} K_{a_2} \dots K_{a_{p_i}}}} \quad (3.8)$$

and for a base as:

$$a_i([\text{H}^+]) = \frac{m_i [\text{H}^+]^{m_i} + (m_i - 1) \frac{K_w}{K_{b_{m_i}}} [\text{H}^+]^{m_i-1} + \dots + \frac{K_w^{m_i-1} [\text{H}^+]}{K_{b_2} K_{b_3} \dots K_{b_{m_i}}}}{[\text{H}^+]^{m_i} + \frac{K_w}{K_{b_{m_i}}} [\text{H}^+]^{m_i-1} + \dots + \frac{K_w^{m_i-1} [\text{H}^+]}{K_{b_2} K_{b_3} \dots K_{b_{m_i}}} + \frac{K_w^{m_i}}{K_{b_1} K_{b_2} \dots K_{b_{m_i}}}} \quad (3.9)$$

considering that if the  $j^{\text{th}}$  dissociation of an acid or base is strong, then  $(1 / K_{a_j}) = 0$  for an acid or  $(1 / K_{b_j}) = 0$  for a base, respectively. Since,  $\text{pH} = -\log[\text{H}^+]$ , Eqn. (3.7) becomes:

$$\sum_{i=1}^{\text{\# species}} a_i(\text{pH}) x_i + A(\text{pH}) = 0 \quad (3.10)$$

where  $a_i(\text{pH})$ 's are given before and  $[\text{H}^+]$  is replaced by  $10^{-\text{pH}}$ , and  $A(\text{pH}) = 10^{\text{pH}} - K_w 10^{2\text{pH}}$ . Thus, the "pH equation" (Eq. 3.10) can be used to obtain the titration curve of any acid–base neutralization process.

Consider a solution with initial liquid volume  $V_1$  and containing a mixture of acids and/or bases of total ion concentrations  $x_{1i}$ . This solution will be titrated with a solution having a volume of  $V_2$  and total ion concentrations of  $x_{2i}$ . The total ion concentrations,  $x_i$ , after mixing becomes:

$$x_i = \frac{V_1 x_{1i} + V_2 x_{2i}}{V_1 + V_2} \quad (3.11)$$

Thus  $x_i$  (Eq. 3.11) can be substituted into Eq. (3.10) to obtain the pH value after mixing process as:

$$\sum_{i=1}^{\text{\# species}} a_i(\text{pH}) \frac{V_1 x_{1i} + V_2 x_{2i}}{V_1 + V_2} + A(\text{pH}) = 0 \quad (3.12)$$

rearrangement of Eq. (3.12) yields:

$$\frac{V_2}{V_1} = - \frac{A(\text{pH}) + \sum_{i=1}^{\text{\# species}} a_i(\text{pH})x_{1i}}{A(\text{pH}) + \sum_{i=1}^{\text{\# species}} a_i(\text{pH})x_{2i}} \quad (3.13)$$

for a given  $V_1$  value, plot of pH versus  $V_2$  gives the titration curve. Thus, if the right hand side of Eq. (3.13) is defined as  $T(\text{pH})$  as:

$$T(\text{pH}) = - \frac{A(\text{pH}) + \sum_{i=1}^{\text{\# species}} a_i(\text{pH})x_{1i}}{A(\text{pH}) + \sum_{i=1}^{\text{\# species}} a_i(\text{pH})x_{2i}} \quad (3.14)$$

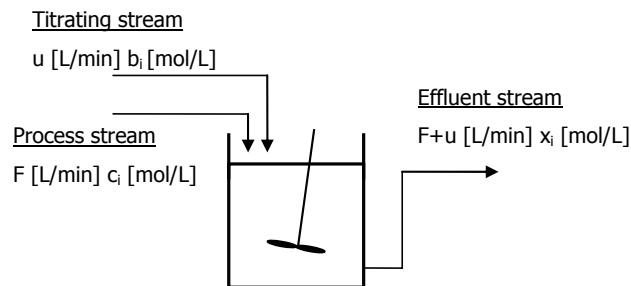
Then, Eq. (3.14) corresponds to the titration curve rotated by  $90^\circ$  and can be used to obtain the titration curve of any acid–base system with given total ion concentrations.

### 3.2. pH PROCESS DYNAMICS

Consider a continuous–stirred tank reactor where the neutralization process takes place (Figure 3.1). Assuming perfect mixing and constant volume, the material balances around the system can be written as follows (Wright and Kravaris 1991):

$$V \frac{dx_i}{dt} = F(c_i - x_i) + u(b_i - x_i) \quad \text{for } i = 1, \dots, n \quad (3.15)$$

where  $c_i$ ,  $b_i$  and  $x_i$  are the total ion concentration of the  $i^{\text{th}}$  species in the process, titrating and the effluent streams, respectively.  $F$  is the volumetric flow rate of the process stream,  $u$  is the volumetric flow rate of the titrating stream, and  $V$  is the volume of liquid in the reactor vessel.



**Figure 3.1.** A Simple pH Neutralization System

The pH value of the effluent stream can be evaluated using pH equation (3.10) and Eqs. (3.10) and (3.15) form the general mathematical model for the pH process in Figure 3.1.

Wright and Kravaris (1991) introduced the strong acid equivalent concept to reformulate the pH control problem. The weighted sum:

$$Y = - \sum_{i=1}^{\# \text{ species}} a_i (\text{pH}_{\text{sp}}) x_i \quad (3.16)$$

is defined as the strong acid equivalent of a mixture of electrolytes. The strong acid equivalent is related to the amount of base needed to bring the system pH to desired set-point pH value. Thus, instead of pH one can use Y as a controlled variable. Since the dynamics of the process are the same for both pH and Y, an equivalent control objective can be defined as:

“For the system given by (3.15) and (3.16) keep  $Y \approx 10^{\text{pH}} - K_w 10^{\text{pH}}$   
in the presence of disturbances”

requiring that Y can be measured or computed on-line.

As shown by Wright and Kravaris (1991) by measuring the effluent pH value it is not possible to determine the value of total ion concentrations,  $x_i$ . Because all state equations are uncoupled and have same time constants. A minimal order model providing same input-output behavior as the detailed model shown above was also derived by Wright and Kravaris (1991). Assuming the model parameters  $F$ ,  $V$ ,  $c_i$  and  $b_i$  are constant and that the system is initially at steady state, Eq. (3.15) can be rearranged to give:

$$V \frac{d[(c_i - x_i(t))/(c_i - b_i)]}{dt} = u - (F + u) \frac{(c_i - x_i(t))}{(c_i - b_i)} \quad (3.17)$$

for each  $i$ . If the scaled total ion concentration is defined as:

$$X(t) = \frac{c_i - x_i(t)}{c_i - b_i} \quad (3.18)$$

then Eq. (3.15) can be written as:

$$V \frac{dX}{dt} = u - (F + u) X \quad (3.19)$$

Therefore, the inverse of the titration curve of the process stream:

$$T(\text{pH}) = - \frac{A(\text{pH}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH})c_i}{A(\text{pH}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH})b_i} \quad (3.20)$$

can be expressed as:

$$X(t) = \frac{T(\text{pH})}{1 + T(\text{pH})} \quad (3.21)$$

by combining pH equation (Eq. 3.10) with Eq. (3.20). Eqs. (3.19) and (3.21) form the pH process model having exactly the same input-output as the process given by Eq. (3.10) and (3.15). The strong acid equivalent and the scaled total ion concentration,  $X(t)$ , are linearly related with each other as (Wright and Kravaris, 1991):

$$X(t) = \frac{T(\text{pH}_{\text{sp}})}{1 + T(\text{pH}_{\text{sp}})} - \frac{Y - A(\text{pH}_{\text{sp}})}{\left[ A(\text{pH}_{\text{sp}}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH}_{\text{sp}})b_i \right] (1 + T(\text{pH}_{\text{sp}}))} \quad (3.22)$$

by defining:

$$Y' = Y - Y_{\text{sp}} = Y - A(\text{pH}_{\text{sp}}) \quad (3.23)$$

$$u' = u - u_{\text{sp}} = u - FT(\text{pH}_{\text{sp}}) \quad (3.24)$$

reduced pH process model (3.19 and 3.21) becomes:

$$V \frac{dY'}{dt} = -(1 + T(\text{pH}_{\text{sp}}))FY' - \left[ Y' + A(\text{pH}_{\text{sp}}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH}_{\text{sp}})b_i \right] u' \quad (3.25)$$

$$Y' = \left[ A(\text{pH}_{\text{sp}}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH}_{\text{sp}})b_i \right] \frac{T(\text{pH}_{\text{sp}}) - T(\text{pH})}{1 + T(\text{pH})}$$

where  $Y'$  is the state of the model which provides nearly linear dynamics. In most of the industrial processes, titrating streams are much more concentrated than the process streams and thus  $u \ll F$ . Reduced pH process model may be further simplified considering  $X \ll 1$  and  $T(\text{pH}) \ll 1$ :

$$V \frac{dX(t)}{dt} = u - (F + u)X(t) \quad \text{and} \quad X(t) = T(\text{pH}) \quad (3.26)$$

or in terms of deviation variables as:

$$V \frac{dY'}{dt} = -FY' - \left[ A(\text{pH}_{\text{sp}}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH}_{\text{sp}}) b_i \right] u' \quad (3.27)$$

$$Y' = \left[ A(\text{pH}_{\text{sp}}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH}_{\text{sp}}) b_i \right] (T(\text{pH}_{\text{sp}}) - T(\text{pH}))$$

A first order transfer function for the simplified model (Eq. 3.27) can easily be derived:

$$G(s) = \frac{Y'(s)}{U'(s)} = \frac{K_p}{\tau_p s + 1} \quad (3.28)$$

where:

$$U' = \left[ A(\text{pH}_{\text{sp}}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH}_{\text{sp}}) b_i \right] u' \quad (3.29)$$

$$\tau_p = \frac{V}{F} \quad (3.30)$$

$$k_p = -\frac{1}{F} \quad (3.31)$$

Using the transfer function in (Eq 3.28), a linear PI controller can easily be formulated:

$$u' = \frac{K_c \left[ Y' + \frac{1}{\tau_I} \int_0^t Y' dt \right]}{\left[ A(\text{pH}_{\text{sp}}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH}_{\text{sp}}) b_i \right]} \quad (3.32)$$

or in discretized form as in Wright and Kravaris (1991):

$$u'_k - u'_{k-1} = - \frac{K_c \left[ 1 + \frac{\Delta t}{\tau_I} \right] Y'_k + K_c Y'_{k-1}}{\left[ A(\text{pH}_{\text{sp}}) + \sum_{i=1}^{\# \text{ species}} a_i(\text{pH}_{\text{sp}}) b_i \right]} \quad (3.33)$$

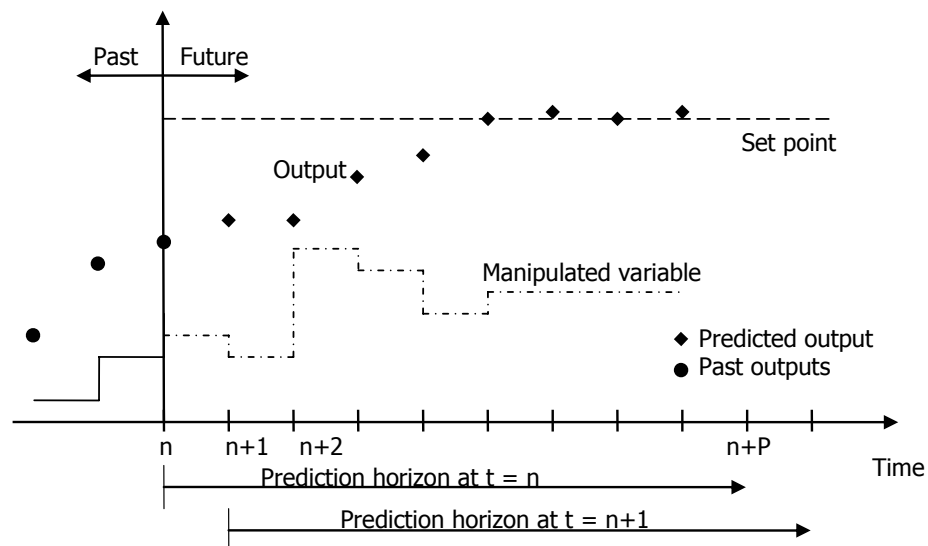
where  $\Delta t$  is the sampling time of the discrete controller.



## CHAPTER IV

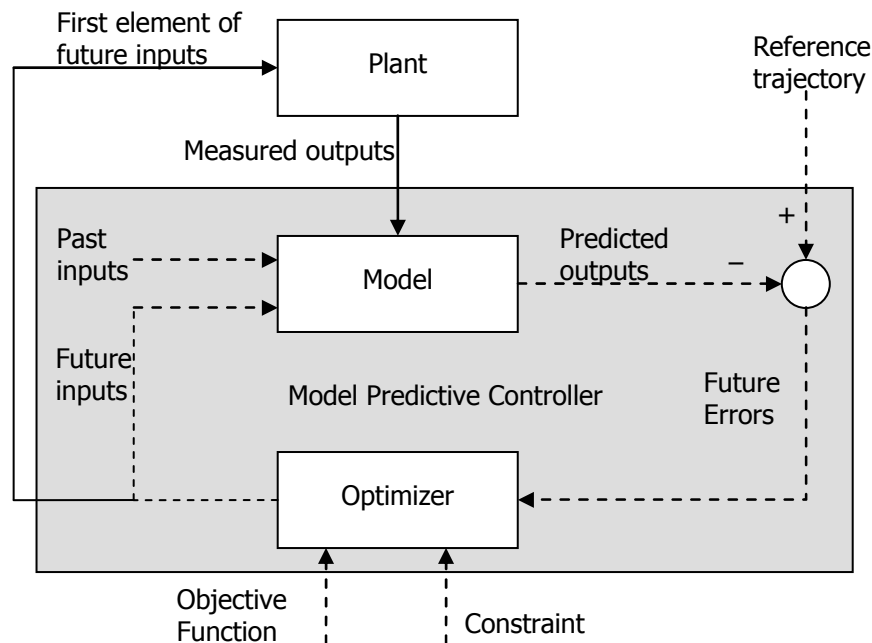
### MODEL PREDICTIVE CONTROL

Model Predictive Control, MPC, is a powerful control technique in which future plant behavior is optimized by the use of an explicit plant model. Also, it is possible to include input and/or output constraints in the optimization problem. Basically, at any sampling interval,  $n$ , the process model is used to evaluate the future plant outputs,  $y(n+k)$  for  $k=1\dots P$ , for a finite prediction horizon,  $P$ , using past plant input–output data and future plant inputs,  $u(n+k)$ , for  $k=1\dots C$ , where  $C$  is the control horizon. Next, plant behavior is optimized, in which future errors minimized, using an objective function that is usually quadratic. After evaluating the optimal manipulated variable profile,  $u(n+k)$ , the first input in this optimal profile,  $u(n)$ , is applied to the plant. The procedure above is repeated for the next sampling instant,  $n+1$ , updating the state of the process with new process measurements. This strategy is shown in Figure 4.1 (Garcia et al., 1989). Since, MPC is a control method based on a model, its success, in fact, depends on the accuracy of the model developed for the process.



**Figure 4.1.** MPC Strategy (Garcia et al., 1989).

MPC constitutes two main parts: Model and Optimizer (Figure 4.2). The model of the plant, e.g. first principle model, black box model etc., is used to predict the plant outputs at future sampling instants. Future error values are then calculated. MPC Optimizer then minimizes the future tracking error using an objective function with available constraints obtaining the optimal future manipulating variable values. Instead of applying all of the evaluated values of these optimal set, only the first element of it is sent to the plant as input and complete procedure is repeated when new plant output measurements are available.



**Figure 4.2.** Basic Structure of MPC (Camacho and Bordons, 2000).

#### 4.1. MPC MODELS

As its name implies, the model of the plant plays the key role in Model Predictive Control. Several types of process models exist ranging from simple linear first principle model to black box model. However, the process dynamics are generally represented by step or impulse discrete convolution models in the MPC structure. These models are simple and can be used to represent any stable plant dynamics. In addition, they can easily be obtained from input–output data of the process. On the other hand, these models have unavoidable truncation errors.

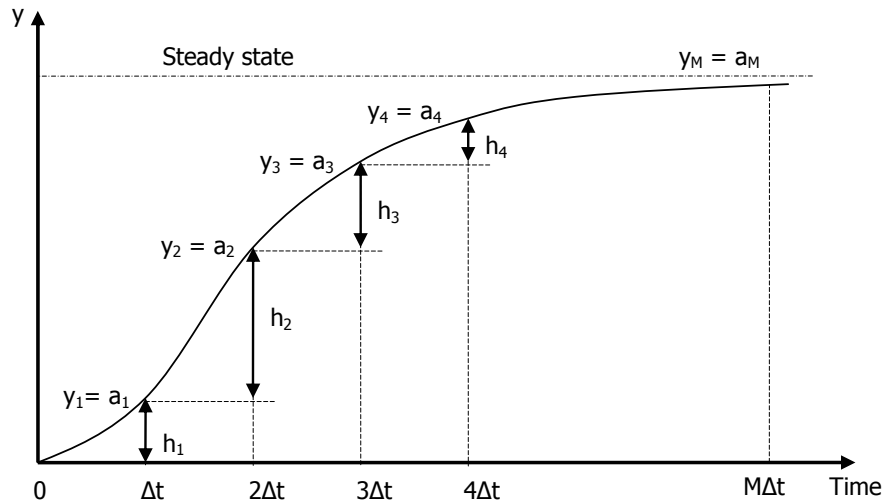
Figure 4.3 shows the open loop response of a linear stable process to a unit step input. From this process response curve step response model of the process can easily be obtained. In Figure 4.3,  $a_i$  values stands for step response coefficients and  $h_i$  values corresponds to the impulse response coefficients. The step response coefficient of the plant at any sampling instant is the summation of the impulse response coefficients up to the this sampling instant, (Seborg et al. 1989), namely:

$$a_i = \sum_{j=1}^i h_j \quad \text{for } i = 1 \dots M \quad (4.1)$$

Eq. (4.1) can be used to obtain the process output for knowing the process input. Thus, a discrete step response model of a single input–single output process can be written as (Peterson et al. 1992):

$$y(k) = \sum_{i=1}^M a_i \Delta u(k-i) + a_M u(k-M-1) + d(k) \quad (4.2)$$

where,  $k$  is the sampling time,  $u$  is the input,  $a_i$  are the step response coefficients,  $d$  is the unmodeled disturbance effects on the output,  $M$  is the model horizon which corresponds to the number of step response coefficients that is required to describe adequately the complete process dynamics, and  $\Delta u$  is the difference between two successive inputs ( $\Delta u(k) = u(k) - u(k-1)$ ).



**Figure 4.3.** An Open Loop Step Response of a Linear Plant (Seborg et al., 1989).



control action on the future output and the last term represents the disturbance effects. Eq (4.4) may be written in more compact form in vector-matrix notation:

$$\mathbf{y} = \mathbf{y}^{\text{past}} + \mathbf{A}\Delta\mathbf{u} + \mathbf{d} \quad (4.5)$$

where  $\mathbf{A}$  is called "Dynamic Matrix",  $\mathbf{y}$  is the predicted output vector,  $\mathbf{y}^{\text{past}}$  is the vector which represents the effects of known past input on the predicted outputs,  $\Delta\mathbf{u}$  is the input vector and  $\mathbf{d}$  is the disturbance vector.

#### 4.2. MPC OPTIMIZATION

The second part of the MPC is the optimization process. Future values of the manipulated variable are calculated in MPC optimization where an objective function is minimized. The objective function may contain any performance criteria such as, product purity, raw material and energy cost, control effort etc. as well as some input and/or output hard or soft constraints. However, in general, the plant output error and manipulating variable change are together penalized to yield a quadratic objective function such as in Eq. (4.6):

$$\min_{\Delta\mathbf{u}} \quad \sum_{i=1}^P \lambda_1 [r - y(k+i)]^2 + \sum_{j=1}^C \lambda_2 [\Delta u(k+C-j)]^2$$

subject to (4.6)

$$u_{\min} \leq u(k+C-j) \leq u_{\max} \quad j = 1 \dots C$$

$$y_{\min} \leq y(k+i) \leq y_{\max} \quad i = 1 \dots P$$

where  $r$  is the set-point,  $\lambda_1$  and  $\lambda_2$  are the weights for output error and manipulated variable change and the variables  $u_{\min}$ ,  $u_{\max}$ ,  $y_{\min}$ , and  $y_{\max}$  are the constraints.

In Section 4.1., model for predicting the future plant output is given for a single input–single output plant. By defining  $\mathbf{E}$  as closed–loop prediction of error vector and  $\mathbf{E}^*$  as open–loop prediction error vector (Seborg et al. 1998), Eq. (4.5) can be written as follows:

$$\mathbf{E} = -\mathbf{A}\Delta\mathbf{u} + \mathbf{E}^* \quad (4.7)$$

where  $\mathbf{E} = \mathbf{r} - \mathbf{y}$ ,  $\mathbf{E}^* = \mathbf{r} - (\mathbf{y}^{\text{past}} + \mathbf{d})$ , and  $\mathbf{r}$  is the set–point vector. If it is desired for output to follow the reference trajectory exactly, then  $\mathbf{E}$  must be zero. Thus,

$$0 = -\mathbf{A}\Delta\mathbf{u} + \mathbf{E}^* \quad (4.8)$$

If prediction horizon and control horizon are equal to each other ( $P = C$ ), then solution of Eq. (4.8) is as follows:

$$\Delta \mathbf{u} = -(\mathbf{A})^{-1} \mathbf{E}^* \quad (4.9)$$

However, in Dynamic Matrix Control (DMC), control horizon should be smaller than the prediction horizon and therefore, system of equations in Eq. (4.7) become overdetermined. At this point, there is no unique solution of Eq. (4.7). However, this overdetermined system of equations can be used to obtain the best solution in least-squares sense (Seborg et al., 1989):

$$\mathbf{J}[\Delta \mathbf{u}] = \mathbf{E}^T \mathbf{E} \quad (4.10)$$

and the solution is:

$$\Delta \mathbf{u} = (\mathbf{A}^T \mathbf{A})^{-1} \mathbf{A}^T \mathbf{E}^* \quad (4.11)$$

The control law in Eq. (4.11) has one drawback; if the the matrix  $\mathbf{A}^T \mathbf{A}$  is ill-conditioned or singular, inverse of this matrix produce very large values which results in excessively large control actions. To eliminate this before performing matrix inversion, one can multiply the diagonal elements of  $\mathbf{A}^T \mathbf{A}$  to suppress the large changes in the manipulated variable. Alternatively, one can modify the optimization problem such as in Eq. (4.6) to penalize the manipulated variable change also in the optimization (Marchetti et al. (1983):

$$\mathbf{J}[\Delta \mathbf{u}] = \mathbf{E}^T \mathbf{W}_1 \mathbf{E} + \Delta \mathbf{u}^T \mathbf{W}_2 \Delta \mathbf{u} \quad (4.12)$$

where  $\mathbf{W}_1$  and  $\mathbf{W}_2$  are the diagonal weighting matrices by which one can tune the controller to obtain good control performance and can be defined as follows:

$$\mathbf{W}_1 = \begin{bmatrix} \lambda_1 & & & 0 & 0 \\ & \lambda_1 & & & 0 \\ & & \ddots & & \\ 0 & & & \ddots & \\ 0 & 0 & & & \lambda_1 \end{bmatrix}_{P \times P} \quad \mathbf{W}_2 = \begin{bmatrix} \lambda_2 & & & 0 & 0 \\ & \lambda_2 & & & 0 \\ & & \ddots & & \\ 0 & & & \ddots & \\ 0 & 0 & & & \lambda_2 \end{bmatrix}_{C \times C} \quad (4.13)$$

It must be noted that, in the quadratic objective function given in Eq. (4.12), there are no constraints imposed on the process input,  $u$ , and on the process output,  $y$ . If Eq. (4.12) is solved by inserting Eq. (4.7), the final form of the control law is obtained as:

$$\begin{aligned}\Delta \mathbf{u} &= (\mathbf{A}^T \mathbf{W}_1 \mathbf{A} + \mathbf{W}_2)^{-1} \mathbf{A}^T \mathbf{W}_1 \mathbf{E}^* \\ &= (\mathbf{A}^T \mathbf{W}_1 \mathbf{A} + \mathbf{W}_2)^{-1} \mathbf{A}^T \mathbf{W}_1 [\mathbf{r} - (\mathbf{y}^{\text{past}} + \mathbf{d})]\end{aligned}\quad (4.13)$$

However, in most of the chemical processes there are limitations for the process inputs due to the physical limitations of actuators. Therefore, manipulated variable value may be limited as given below:

$$u_{\min} \leq u(k + C - j) \leq u_{\max} \quad j = 1 \dots C \quad (4.14)$$

Also, the process output may be constrained for several reasons such as purity requirements of the product, downstream process limitations, process safety etc. Thus, there may be constraints on the process output as:

$$y_{\min} \leq y(k + i) \leq y_{\max} \quad i = 1 \dots P \quad (4.15)$$

Thus, when the quadratic objective function in Eq. (4.12) is minimized, subject to constraints given in Eqs. (4.14) and (4.15), it is not possible to find an analytical solution to obtain best estimates of  $\Delta \mathbf{u}$ . However, it is possible to find the best solution by quadratic programming techniques. For this purpose, first of all, the objective function must be converted to standard form of a quadratic problem:

$$\begin{aligned}J[\Delta \mathbf{u}]_{\min \Delta \mathbf{u}} &= \frac{1}{2} \Delta \mathbf{u}^T \mathbf{H} \Delta \mathbf{u} + \mathbf{g} \Delta \mathbf{u} \\ \text{subject to} \quad &\mathbf{A}_{\text{eq.}} \Delta \mathbf{u} = \mathbf{B}_{\text{eq.}} \\ &\mathbf{A}_{\text{ineq.}} \Delta \mathbf{u} \leq \mathbf{B}_{\text{ineq.}}\end{aligned}\quad (4.16)$$

where  $\mathbf{H}$  is Hessian matrix,  $\mathbf{g}$  is Gradient vector,  $\mathbf{A}_{\text{eq.}}$ ,  $\mathbf{B}_{\text{eq.}}$ ,  $\mathbf{A}_{\text{ineq.}}$  and  $\mathbf{B}_{\text{ineq.}}$  are equality and inequality constraint matrices. After converting the standard form Hessian and Gradient matrices takes following values, respectively (Dokucu, 2002):

$$\mathbf{H} = \mathbf{A}^T \mathbf{W}_1 \mathbf{A} + \mathbf{W}_2 \quad (4.17)$$

$$\mathbf{g} = \mathbf{A}^T \mathbf{W}_1 \mathbf{E}^* \quad (4.18)$$

Input and output constraints in Eqs. (4.14) and (4.15) are in the form of inequality constraints and can easily be converted to the matrix form shown in Eq. (4.16) as follows:

$$\begin{bmatrix}
1 & & & & \\
1 & 1 & & & 0 \\
\vdots & & \ddots & & \\
\vdots & & & \ddots & \\
1 & 1 & 1 & \dots & 1 \\
-1 & & & & \\
-1 & -1 & & & 0 \\
\vdots & & \ddots & & \\
\vdots & & & \ddots & \\
-1 & -1 & \dots & \dots & -1
\end{bmatrix}
\begin{bmatrix}
\Delta u(k) \\
\Delta u(k+1) \\
\Delta u(k+2) \\
\vdots \\
\Delta u(k+C-1) \\
\Delta u(k) \\
\Delta u(k+1) \\
\Delta u(k+2) \\
\vdots \\
\Delta u(k+C-1)
\end{bmatrix}
\leq
\begin{bmatrix}
u_{\max} - u_{k-1} \\
u_{\max} - u_{k-1} \\
u_{\max} - u_{k-1} \\
\vdots \\
u_{\max} - u_{k-1} \\
u_{k-1} - u_{\min} \\
u_{k-1} - u_{\min} \\
u_{k-1} - u_{\min} \\
\vdots \\
u_{k-1} - u_{\min}
\end{bmatrix}
\quad (4.19)$$

where right hand side of inequality represents the distance of last process input from the constraints and the terms on the left hand side stands for the summation of manipulation variable change up to corresponding time instant  $(k + i)$ . In this way it is guaranteed that, there will be no violations occurring over the control horizon. In a similar fashion, output constraints can be expressed in matrix form as:

$$\begin{bmatrix}
\mathbf{A} \\
-\mathbf{A}
\end{bmatrix}
\begin{bmatrix}
\Delta u(k) \\
\Delta u(k+1) \\
\Delta u(k+2) \\
\vdots \\
\Delta u(k+C-1) \\
\Delta u(k) \\
\Delta u(k+1) \\
\Delta u(k+2) \\
\vdots \\
\Delta u(k+C-1)
\end{bmatrix}
\leq
\begin{bmatrix}
y_{\max} - (y^{\text{past}} + d)_{k+1} \\
y_{\max} - (y^{\text{past}} + d)_{k+2} \\
y_{\max} - (y^{\text{past}} + d)_{k+3} \\
\vdots \\
y_{\max} - (y^{\text{past}} + d)_{k+P} \\
(y^{\text{past}} + d)_{k+1} - y_{\min} \\
(y^{\text{past}} + d)_{k+2} - y_{\min} \\
(y^{\text{past}} + d)_{k+3} - y_{\min} \\
\vdots \\
(y^{\text{past}} + d)_{k+P} - y_{\min}
\end{bmatrix}
\quad (4.20)$$

where right hand side again is the distance of the process output from the bounds of the constraints and the left hand side represents the forced response of the process.

After defining, Hessian, Gradient and Inequality matrices final form of quadratic optimization problem is obtained and it can be solved using any suitable quadratic programming algorithm or simply using a suitable software package such as MATLAB®.



### 4.3. MPC TUNING

There are several parameters that affect the performance of MPC. These parameters are:

- Model horizon,  $M$
- Prediction horizon,  $P$
- Control horizon,  $C$
- Weighing matrix for predicted errors,  $\mathbf{W}_1$
- Weighing matrix for manipulated variable changes,  $\mathbf{W}_2$

The value of model horizon,  $M$ , should be selected appropriately so that the model adequately represents the process behavior such that  $M\Delta t \geq t_{99}$ . The  $M$  value between 20 and 70 is generally recommended (Seborg et al., 1989). However, in order to avoid the truncation problem its value should be large enough to capture the whole process dynamics.

The value prediction horizon,  $P$ , also affects the performance of the MPC. When its value increases, the system become more stable. On the other hand, since its value determines the dimension of the prediction vector, large  $P$  values requires more computational power.

The third tuning parameter is the control horizon,  $C$ . The value of control horizon also effects the required computational power because, dimension of the future control actions vector is determined by  $C$ . Since, only the first element of the optimal future control actions is applied to the process, large values of  $C$  does not affect the performance of the MPC too much (Seborg et al. 1989). Nevertheless, if  $C$  value increased to very large values, the value of process input will become oscillatory.

The last tuning parameter group is the weighting matrices  $\mathbf{W}_1$  and  $\mathbf{W}_2$ . Generally,  $\mathbf{W}_1$  is selected as identity matrix,  $\mathbf{I}$ , and  $\mathbf{W}_2 = f\mathbf{I}$ . The scalar  $f$  is called as move suppression factor and instead of adjusting the values of the weighting matrices only  $f$  is used as tuning parameter. When  $f$  value is decreased, controller become more aggressive and causes large manipulated variable changes mainly because of the singularity of the matrix multiplication  $\mathbf{A}^T\mathbf{A}$ .

### 4.4. MPC DESIGN

In this study, linearized process model given in Eq. (3.27) is used in the MPC algorithm. For MPC, step response coefficients are obtained by a simulation using the simplified process model. The MPC is designed for the constant disturbance assumption in the algorithm used

which resulted in poor performance in the simulation studies. Therefore, in this study, constant derivative of disturbance is assumed in the algorithm.

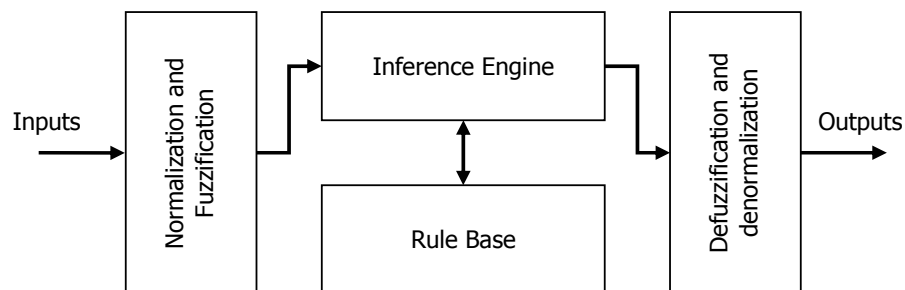
For all simulations and experimental runs, control horizon,  $C$ , is taken as one (1) and kept constant. Prediction horizon,  $P$ , and move suppression factor,  $f$ , are tuned by simulations. It is observed that, according to the identification data, which is actually the identified steady-state titration curve, the value of  $f$  have to be changed appropriately to achieve a good control performance and to obtain mild control input changes. Therefore, the slope of the process gain around the set-point is used to adapt the  $f$  value to the changes in the process gain around the set-point, resulting in an adaptive MPC (AMPC).

## CHAPTER V

### FUZZY LOGIC CONTROL

After the introduction of Fuzzy Logic by Zadeh (1965), different Fuzzy Logic Controller, FLC, designs were developed (Mamdani type, Sugeno type). FLC's incorporate simple if-then (condition-action) rules to solve the control problems without using a process model. They are designed using the operator's knowledge about the process rather than the mathematical model of the process. Therefore, FLC's are suitable to control the processes that have complex nature or that are difficult to model.

Typical structure of a FLC is given in Figure 5.1. It has four main parts; input signal normalization and fuzzification, fuzzy inference engine, fuzzy rule base, defuzzification and denormalization to crisp control input (Passino and Yurkovich, 1998).

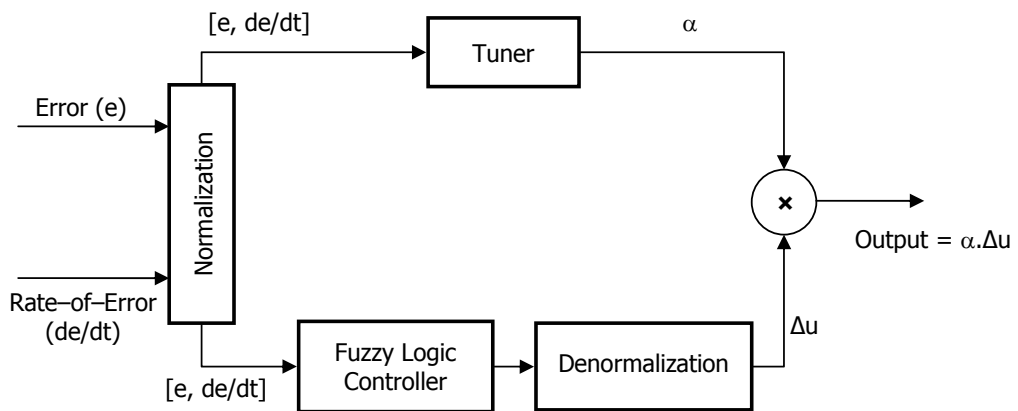


**Figure 5.1.** Block Diagram of a Basic FLC.

Working principle of a FLC is simple. First, crisp inputs values are normalized and are fuzzified by the use of membership functions, MF. After obtaining fuzzified inputs, inference engine makes decisions about the inputs according to the if-then rules incorporated in the rule base. Finally, inferred results are defuzzified and are normalized to obtain crisp output values.

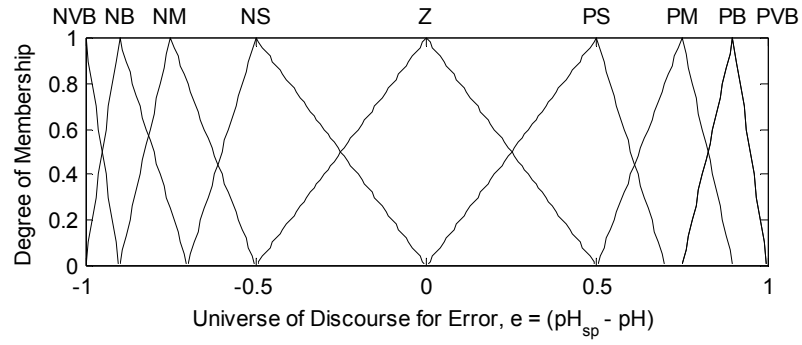
FLC's are tuned by modifying the input and output membership functions, MF's, the rule base and also by changing the input normalization factors,  $N_{e_r}$ ,  $N_{de_r}$ , and output denormalization factor,  $N_{du}$ . Among these, the latter one has a stronger influence on the performance of FLC (Zheng, 1992).

In this study, Mamdani type FLC is proposed to control the effluent pH value in the neutralization process. The structure of the proposed FLC is shown in Figure 5.2. In the proposed FLC, error ( $e = \text{pH}_{sp} - \text{pH}$ ) and rate-of-error ( $de/dt$ ) are used as inputs and the manipulated variable change ( $\Delta u = u_k - u_{k-1}$ ) is selected as output. Nine MF's (Negative very big, NVB; Negative big, NB; Negative medium, NM; Negative small, NS; Zero, Z; Positive small, PS; Positive medium, PM; Positive big, PB; Positive very big, PVB) for error, seven MF's (NB, NM, NS, Z, PS, PM, PB) for rate-of-error and seven MF's (NB, NM, NS, Z, PS, PM, PB) for manipulated variable change are defined in FLC structure. Triangular type membership functions are used for both inputs and outputs and they are tuned for their best values by simulations. The graphical representations of membership functions for inputs are given in Figures 5.3 and 5.4 and that of output is given in Figure 5.5.

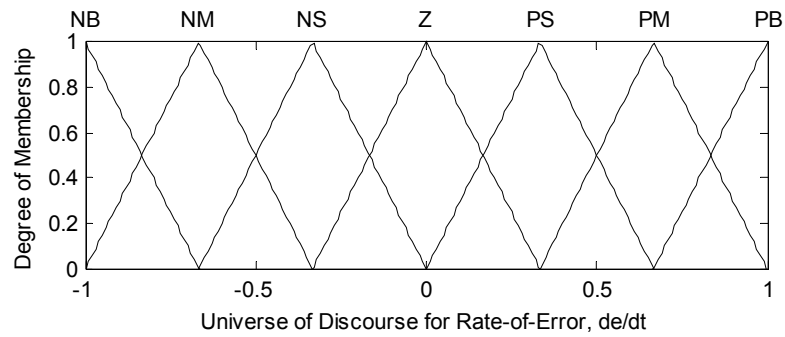


**Figure 5.2.** Block Diagram of the Proposed Fuzzy Logic Controller.

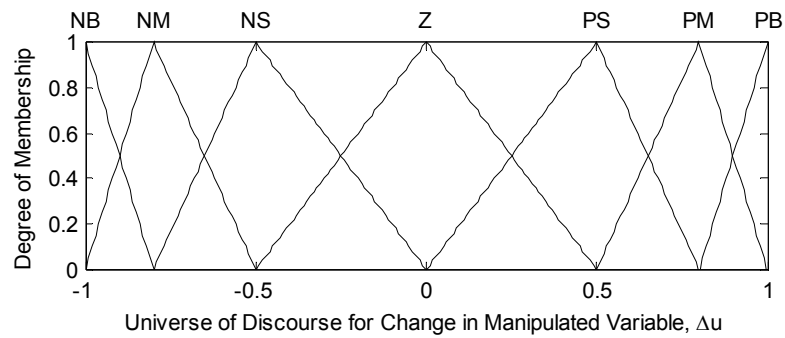
The 9-by-7 rule table is prepared for the FLC by considering that, as base added to the neutralization reactor, pH value in the reactor increases and vice versa. The rule table and corresponding fuzzy control surface are shown in Figure 5.6.



**Figure 5.3.** Membership Functions for Error,  $e = \text{pH}_{\text{sp}} - \text{pH}$ .

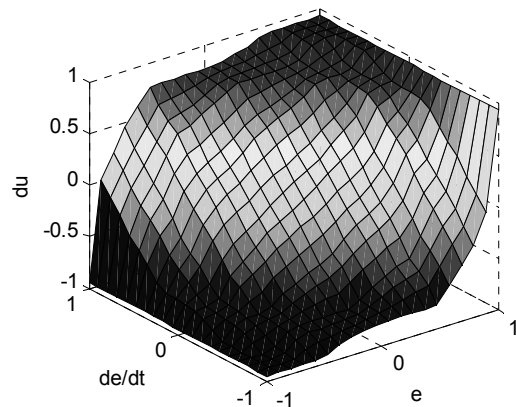


**Figure 5.4.** Membership Functions for Rate-of-Error,  $de/dt$ .



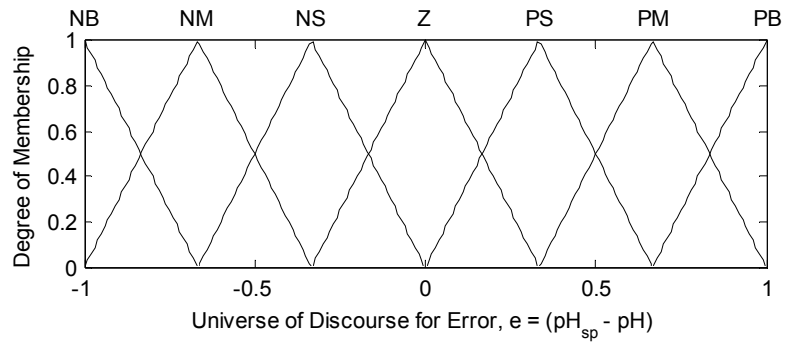
**Figure 5.5.** Membership Functions for Manipulated Variable Change,  $\Delta u$ .

		Rate-of-Error, de/dt						
		NB	NM	NS	Z	PS	PM	PB
Error, e	NVB	NB	NB	NB	NB	NB	NB	NB
	NB	NB	NB	NB	NM	NM	NS	Z
	NM	NB	NB	NM	NM	NS	Z	PS
	NS	NB	NM	NM	NS	Z	PS	PM
	Z	NM	NM	NS	Z	PS	PM	PM
	PS	NM	NS	Z	PS	PM	PM	PB
	PM	NS	Z	PS	PM	PM	PB	PB
	PB	Z	PS	PM	PM	PB	PB	PB
	PVB	PB	PB	PB	PB	PB	PB	PB

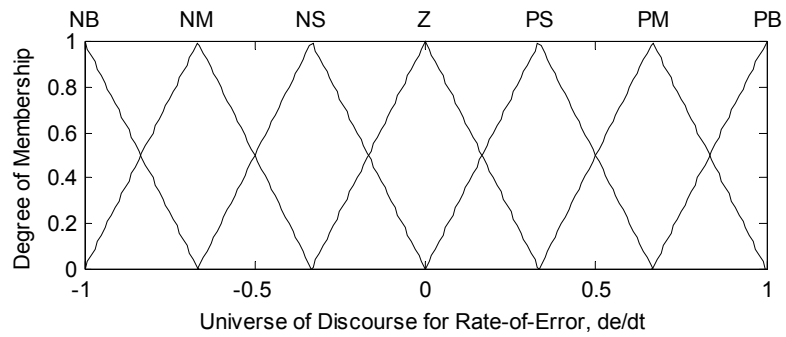


**Figure 5.6.** FLC Rule Table and Fuzzy Control Surface.

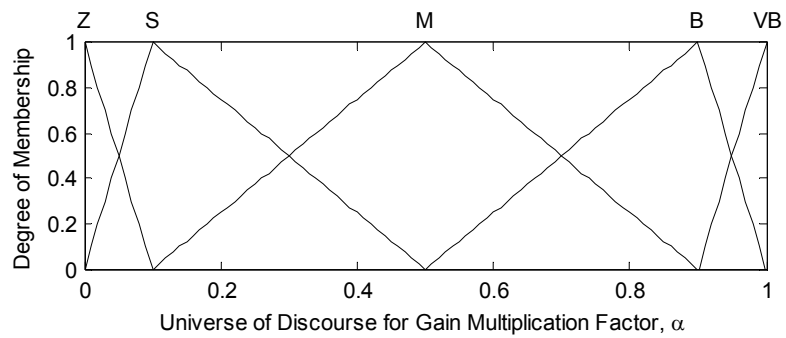
In the FLC algorithm, nonlinear pH process is directly controlled by measuring only the effluent pH value without the use of identification reactor. However, in order to improve the performance of the FLC for high gain regions of the pH process, a tuning mechanism by means of a tuner (Figure 5.2) is added to reduce possible oscillations by multiplying the output,  $\Delta u$ , by gain multiplication factor,  $\alpha$ , (output of tuner). Seven MF's for error ( $e = \text{pH}_{\text{sp}} - \text{pH}$ ), seven MF's for rate-of-error ( $de/dt$ ) and five MF's for the gain multiplication factor,  $\alpha$ , are used in the tuner (Figure 5.7 – 5.9). The tuning module adjusts  $\alpha$  value according to the error and the rate-of-error and increases it when output is away from the set-point and decreases it when output is close to the set-point. The magnitude of this change depends on the value of the second input, rate-of-error. The rule table and the control surface of the tuner are given in Figure 5.10.



**Figure 5.7.** Membership Functions for Error,  $e = \text{pH}_{\text{sp}} - \text{pH}$ .

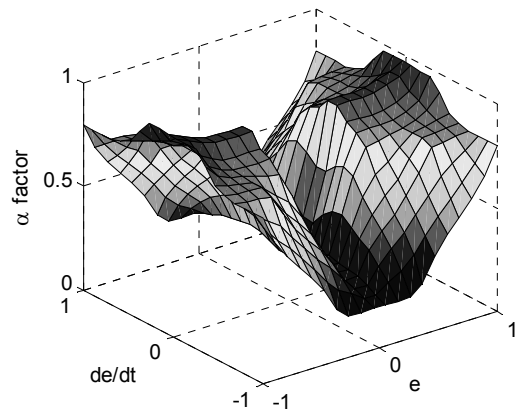


**Figure 5.8.** Membership Functions for Rate-of-Error,  $de/dt$ .



**Figure 5.9.** Membership Functions for Gain Multiplication Factor,  $\alpha$ .

		Rate-of-Error, dt/dt						
		NB	NM	NS	Z	PS	PM	PB
Error, e	NB	B	B	VB	VB	VB	B	B
	NM	M	B	B	VB	B	B	M
	NS	S	M	B	VB	B	M	S
	Z	S	S	S	S	S	S	S
	PS	S	M	B	VB	B	M	S
	PM	M	B	B	VB	B	B	M
	PB	B	B	VB	VB	VB	B	B



**Figure 5.10.** Rule Table and Graphical Representation of the Rules for Tuner Module.



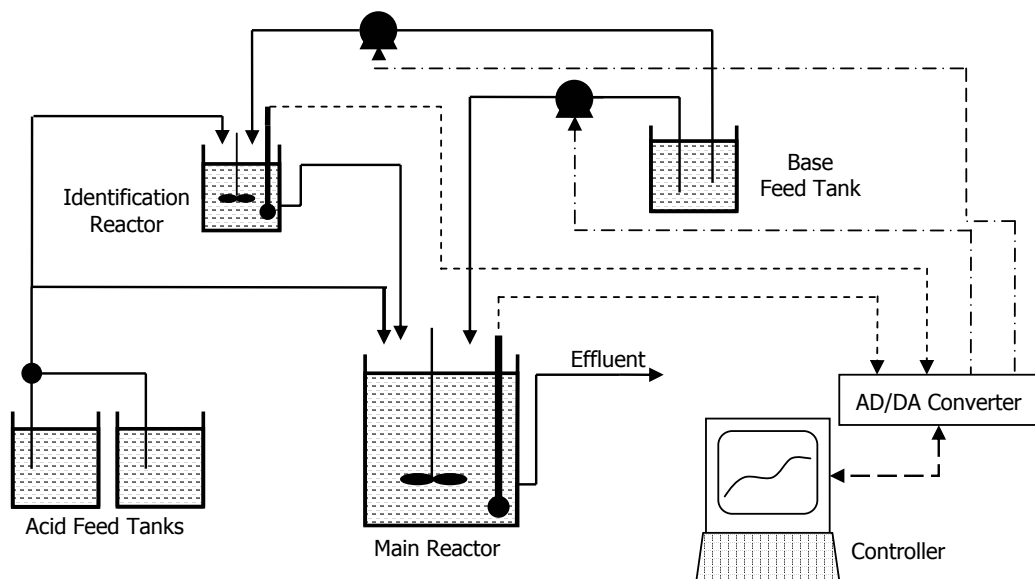
## CHAPTER VI

### EXPERIMENTAL SET-UP

In this section the pH neutralization system constructed for this study will be described in detail and online identification strategy used in the study will be explained briefly.

#### 6.1. THE EXPERIMENTAL SET-UP

In the experimental set-up used in the study (Figure 6.1), two continuous-stirred tank reactors (CSTR's) are used one being as the main reactor and the other being the identification reactor as main equipments.



**Figure 6.1.** Schematic Diagram of the Experimental Set-up for pH Neutralization System.

The control objective is to keep the effluent pH value of the main reactor at a desired set-point value, mostly at  $\text{pH} = 7.0$ , by adjusting the flow rate of titrating stream of the main reactor. Thus, the controlled variable is the pH value of the effluent stream and the manipulated variable is the flow rate of the titrating stream. The major disturbances that can

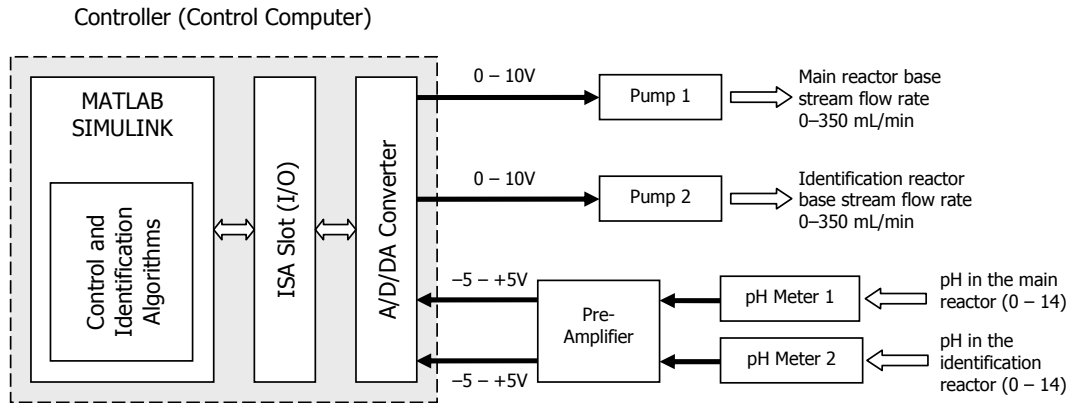
affect the system are the variations of acid composition and the flow rate of the entering acid stream to the main reactor.

The acidic solution (process stream) is taken from the acid feed tanks and is selected by a three-way valve. The volume of the main reactor is 15.0L. Reactor content is mixed with an axial flow type impeller and effluent stream is taken out at the impeller level where the highest efficiency of mixing is obtained. The effluent stream tube with sufficiently large diameter is located to a desired liquid level of the main reactor, to keep the volume constant. In addition, baffles are located in the main reactor to prevent vortex formation.

The identification reactor has similar properties with the main reactor. The volume of the identification reactor is 0.45L. Thus, the time constant of the identification reactor is approximately ten times smaller than that of main reactor. The effluent stream of the identification reactor is returned to the main reactor and it can be viewed as a disturbance (as flow rate/composition disturbances) to the main reactor.

Acid feed pumps are adjusted to constant flow rates of 0.5L/min and 0.15 L/min for the main neutralization reactor and for the identification reactor, respectively. The process streams are neutralized separately with two input base streams. Flow rates of base streams are adjusted with two peristaltic pumps connected to the controller through an AD/DA card. The value of the manipulated variable (the flow rate of the titrating stream of main reactor) is estimated by the controller, by using the measurement of the pH in the main reactor.

The pH values of the solution in both of the reactors are measured with two separate pH electrodes located just at the effluent stream exit locations. Their measurement signals sent to the controller through the AD/AD card. The schematics of the communication interface between controller and the experimental set-up is shown in Figure 6.2. The experimental runs are performed nearly at room temperature around 20–25°C. In the experiments, acid solutions are prepared using tap water to simulate real plant conditions. Sampling time of 5s is used in all controller schemes presented in the study.



**Figure 6.2.** Communication Interface Between Experimental Set-up and Controller.

## 6.2. IDENTIFICATION STRATEGY

In the experimental studies, experiments are performed to find the set-point tracking, disturbance rejection and robustness issues of the proposed controllers. Three runs are designed for each controller by using a three-acid mixture (HCl, HNO<sub>3</sub> and H<sub>3</sub>PO<sub>4</sub>) similar to the acid ingredients of the industrial waste water of a plant located in Ankara, Turkey, which manufactures aircraft parts.

During the experiments the identification of the incoming stream has to be done as a titration curve to find the time varying characteristics of the input. To achieve this, a portion of the main input stream is fed to the identification reactor and titrated with a base input stream. Sung et al. (1995) suggests using identification of the incoming stream in terms of pH value (titration curve) in intervals of half time constant of the main reactor (0.5V/F). Thus, a pulse input is given by changing the base flow rate entering to the identification reactor to increase the pH value in the reactor to a predefined high pH value. The magnitude of this pulse input is determined arbitrarily to obtain reasonable response. The duration of the pulse input is a dependent function of the acid concentration of the incoming stream.

The identification is done by solving, at the same time, Eq. (3.27) (the model) to find  $Y'$  and  $T(\text{pH})$  for the same pulse input. Thus, for every  $\Delta\text{pH} \geq 0.15$ , the calculated  $T(\text{pH})$  value is matched with the measured pH value and the steady-state titration curve is constructed (Figure 6.3). In order to reduce possible effects of noise on the identification data, raw data is filtered once and cubic spline is applied. Then, for the 0.5V/F time duration the pH

measurements of the main reactor can be used to find the  $T(\text{pH})$  and  $Y'$ , from the identified titration curve, which is further used in the control algorithm for this time interval until the next identification run.

## CHAPTER VII

### RESULTS AND DISCUSSION

The simulations and experimental studies are divided into three sub sections. In the first section, set–point tracking tests are performed for each controller scheme, PI, AMPC and FLC, and in the second part, disturbance rejection capabilities of the controller are tested. Finally, robustness' of the controllers are checked in disturbance rejection case by changing the main neutralization reactor volume. After the simulations and the experiments, performances of the controllers are evaluated according to the Integral of the Absolute Error, IAE, and according to the Integral Absolute of the Control Change, IACC scores, which are given in Eqs. (7.1) and (7.2):

$$IAE = \int_0^{\infty} |e(t)| dt \quad (7.1)$$

$$IACC = \int_0^{\infty} |\Delta u(t)| dt \quad (7.2)$$

The scores above are further normalized by dividing them by the largest corresponding value of each set and the normalized scores are compared. In the simulation studies, trial runs are performed to find the best controller parameters considering the performance scores and these parameters are later used in the experiments.

#### 7.1. SET–POINT TRACKING

In the simulation and experimental runs, input acid mixture (HCl, HNO<sub>3</sub> and H<sub>3</sub>PO<sub>4</sub>) is titrated by a base solution (NaOH). Set-point tracking performances of the proposed controllers are found for different set-point changes. Process parameters, controller parameters and set-point changes for different time intervals are given in Table 7.1 for both the simulation and experimental studies. Only, in the experiments, the second set–point interval given in Table 7.1 is increased by 400s due to the unknown additional buffering effect introduced by the tap water used, which is not possible to be integrated into the simulations.

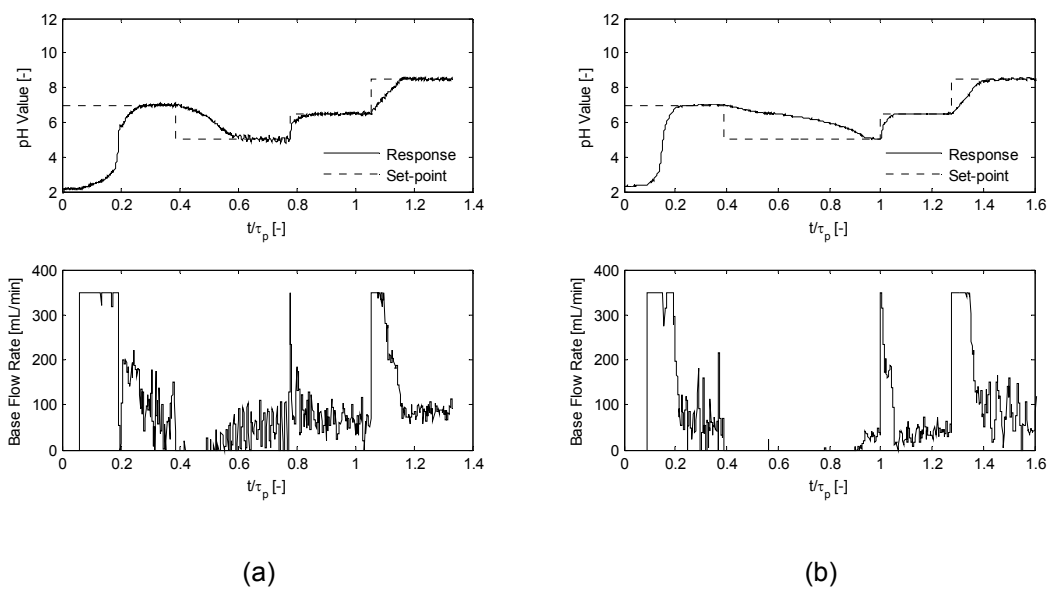
PI and AMPC can only be activated after the first identification run due to the need of the titration curve data to evaluate the strong acid equivalent ( $Y'$ ) from the pH measurements. On the other hand, although FLC does not require this, it is also deactivated until the end of the first identification. Set-point responses of the pH control system are shown in Figures 7.1–7.3 for PI controller, AMPC and FLC, respectively. It is observed that in the experiments response is more sluggish due to the additional buffering effect of the tap water. Considering the normalized IAE scores (Table 7.2), all the controllers have similar performances in simulations and experiments, the lowest belonging to FLC. However, considering the control moves, IACC score, the FLC is better in simulations whereas AMPC is better in experimental runs due to its constraint handling ability and extended buffer effect in the experiments.

**Table 7.1.** The Process and the Controller Parameters for Set–point Tracking.

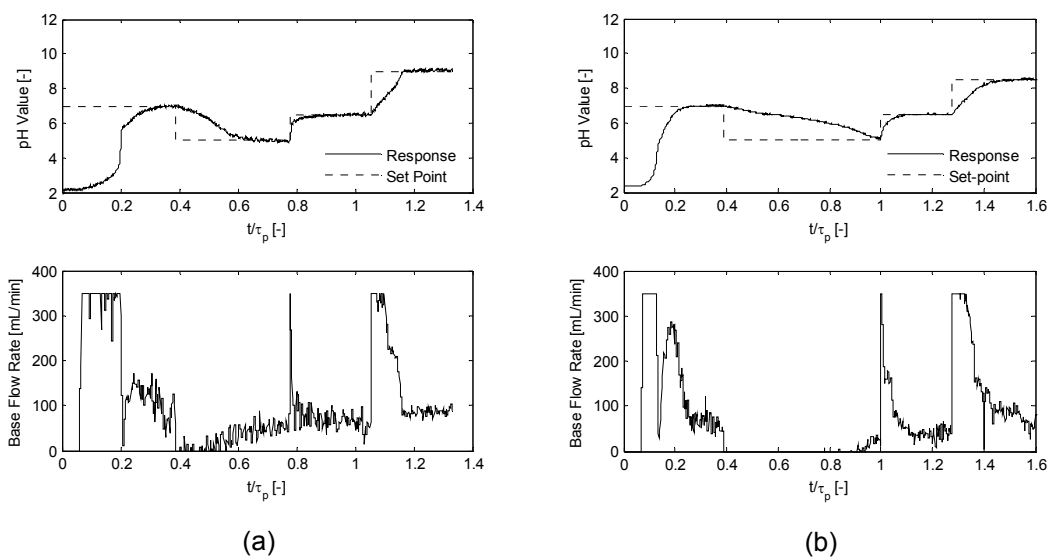
<b>Process Parameters:</b>			
Process stream compositions	:	0.002M HCl – 0.002M HNO <sub>3</sub> – 0.005M H <sub>3</sub> PO <sub>4</sub>	
Titrating stream compositions	:	0.1M NaOH	
$F_{\text{main}}$	:	0.50 L/min	
$V_{\text{main}}$	:	15.0 L	
$F_{\text{id.}}$	:	0.15 L/min	
$V_{\text{id.}}$	:	0.45 L	
Time intervals : pH value (Set-point)	:	1.	0 – 700s : pH <sub>sp</sub> = 7.0
		2.	700 – 1400s : pH <sub>sp</sub> = 5.0
		3.	1400 – 1900s : pH <sub>sp</sub> = 6.5
		4.	1900 – 2400s : pH <sub>sp</sub> = 8.5
<b>Controller Parameters:</b>			
PI	:	$P_1 = P_2 = 0.85$	
AMPC	:	$P = 30, C = 1, f = \text{variable, constrained} : 0 \leq u(\text{mL/min}) \leq 350$	
FLC	:	Error normalization factor	$N_e = 1.354$
		Rate-of-error normalization factor	$N_{de} = 0.026$
		Error normalization factor	$N_{du} = 6.171$

**Table 7.2.** Normalized Performance Scores for Set–point Tracking.

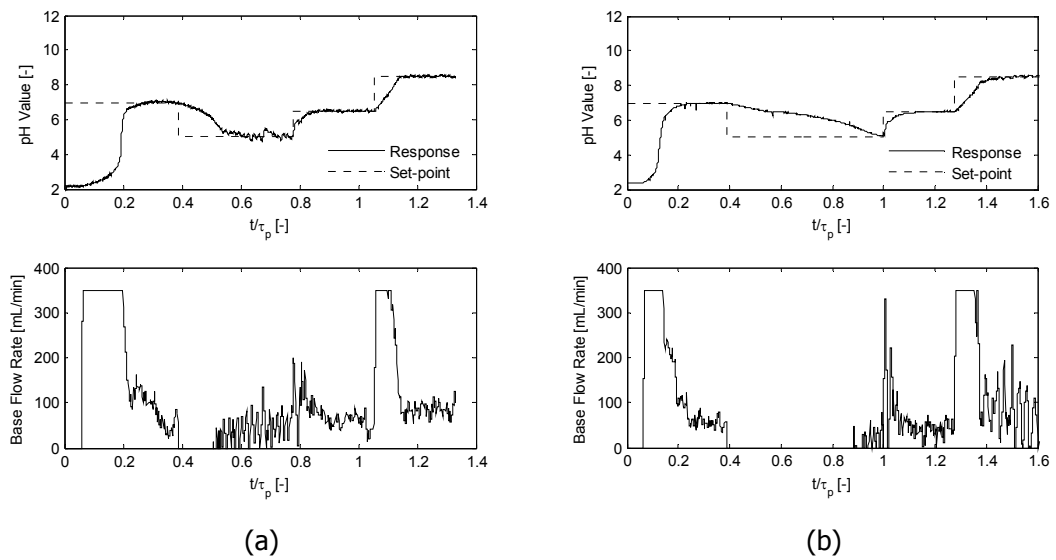
Controller	Simulation Score		Experiment Score	
	IAE	IACC	IAE	IACC
<b>PI</b>	0.911	1.000	0.996	0.945
<b>AMPC</b>	1.000	0.736	1.000	0.756
<b>FLC</b>	0.891	0.675	0.960	1.000



**Figure 7.1.** Set-point Tracking for PI Controller, (a) Simulation, (b) Experiment.



**Figure 7.2.** Set-point Tracking for AMPC, (a) Simulation, (b) Experiment.



**Figure 7.3.** Set-point Tracking for FLC, (a) Simulation, (b) Experiment.

## 7.2. DISTURBANCE REJECTION

The most important problem in the waste water neutralization processes is the variation of the input feed compositions. Therefore, regulatory performance is more important than the servo performance for these processes. Thus, the process stream acid concentrations are changed to find the disturbance rejection performance of the proposed controllers. In Table 7.3, the process and controller parameters used in these runs are given.

During the simulation and experimental runs, two input acid stream contents are used as Type 1 and Type 2, which are buffer dominant and strong acid dominant acid mixtures, respectively. The responses of each control system to acid composition changes are given for simulations and experiments in Figures 7.4–7.6.



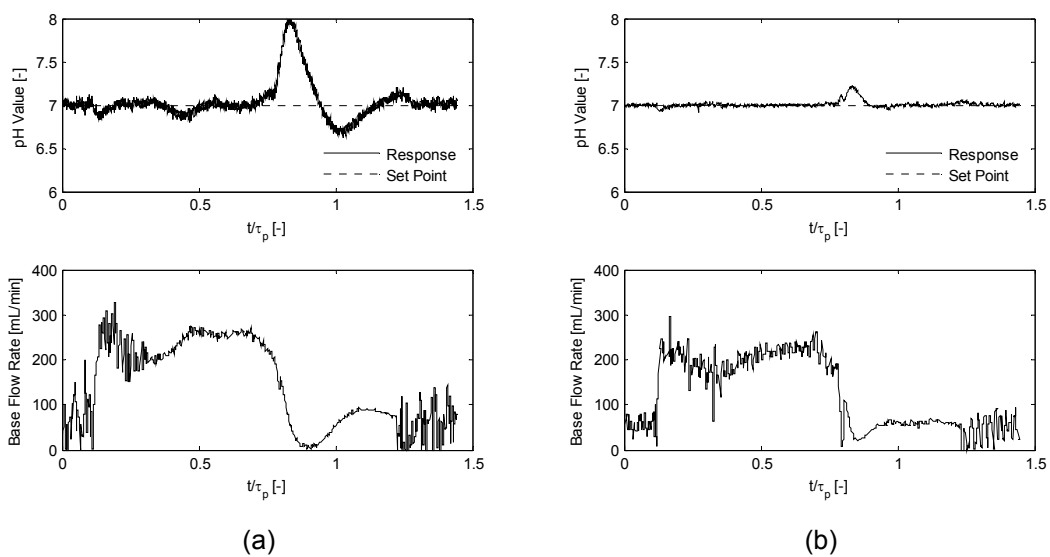
**Table 7.3.** The Process and the Controller Parameters for Disturbance Rejection.

<b>Process Parameters:</b>			
Process stream	: Type 1	→	0.002M HCl – 0.002M HNO <sub>3</sub> – 0.0050M H <sub>3</sub> PO <sub>4</sub>
	: Type 2	→	0.020M HCl – 0.020M HNO <sub>3</sub> – 0.0001M H <sub>3</sub> PO <sub>4</sub>
Titration stream	: 0.1M NaOH		
F <sub>main</sub>	: 0.50 L/min		
V <sub>main</sub>	: 15.0 L		
F <sub>id.</sub>	: 0.15 L/min		
V <sub>id.</sub>	: 0.45 L		
Disturbances	: At time = 200s	→	Change from Type 1 to Type 2
	: At time = 1400s	→	Change from Type 2 to Type 1
<b>Controller Parameters:</b>			
PI	: P <sub>1</sub> = P <sub>2</sub> = 0.85		
AMPC	: P = 35, C = 1, f = variable, constrained: 0 ≤ u(mL/min) ≤ 350		
FLC	: Error normalization factor		N <sub>e</sub> = 1.354
	: Rate-of-error normalization factor		N <sub>de</sub> = 0.026
	: Error normalization factor		N <sub>du</sub> = 6.171

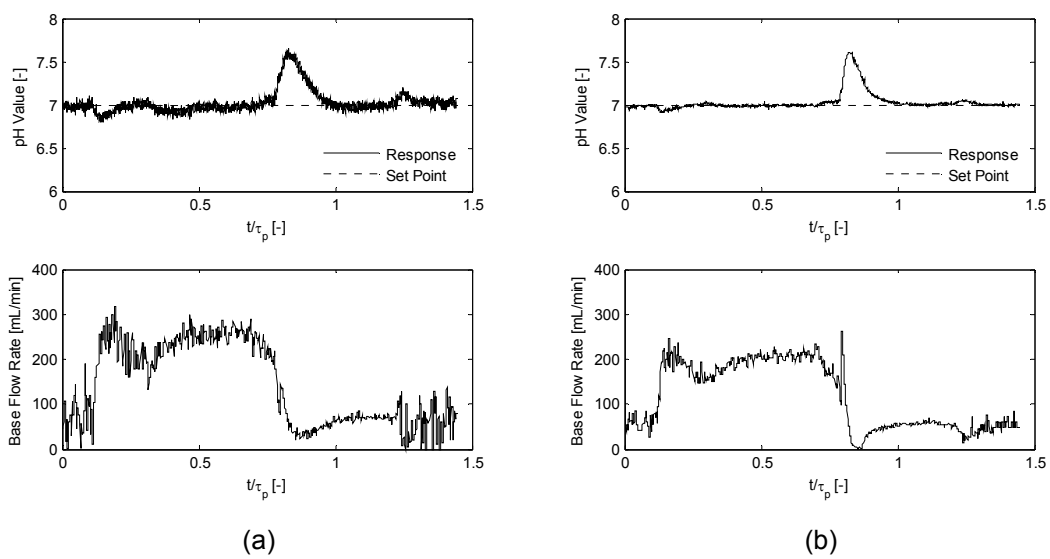
It can be seen that deviation from the set-point is found to be more when input stream is changed from Type 2 to Type 1 than that of from Type 1 to Type 2. This is due to fact that, Type 2 is the strong acid dominant mixture in which the process is much more sensitive to disturbances around the set-point. In the simulation studies, the tuner of FLC is used to eliminate the aggressive control action seen in the trial simulation runs due to the noise added, by introducing the factor  $\alpha$ . However, in the experimental runs, use of the same  $\alpha$  factor resulted in sluggish response. Therefore, the experiments are repeated without the tuner. Considering the IAE scores given in Table 7.4, FLC is found to be better both in simulations (with tuner) and also in experiments (without the tuner), than the other two, although the identification reactor is not included in the FLC scheme. Similar performance as in set-point tracking is also seen for AMPC in the experiments of disturbance rejection resulting in the lowest IACC score for the AMPC for the same reason.

**Table 7.4.** Normalized Performance Scores for Disturbance Rejection.

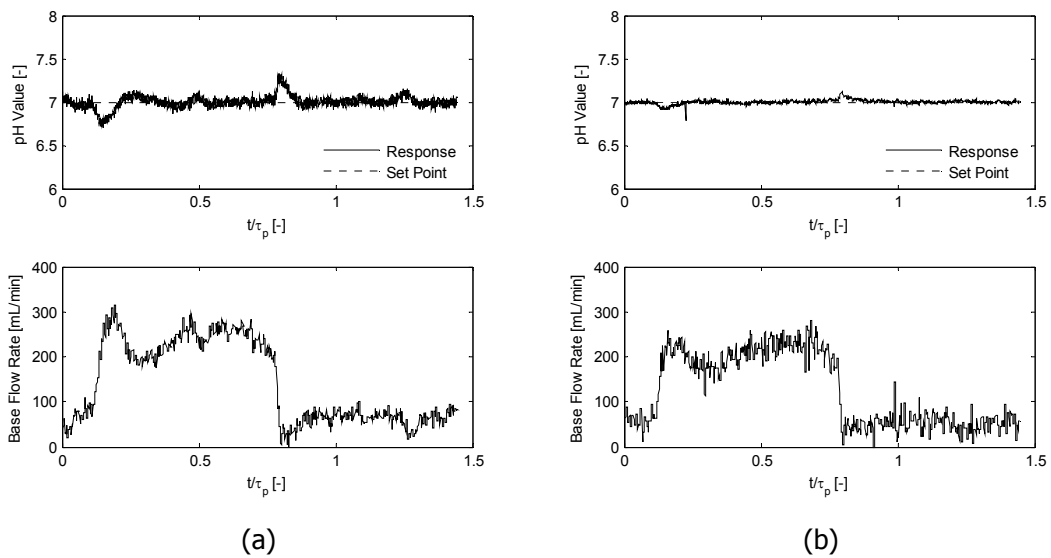
Controller	Simulation Score		Experiment Score	
	IAE	IACC	IAE	IACC
<b>PI</b>	1.000	0.799	0.419	0.745
<b>AMPC</b>	0.643	1.000	1.000	0.481
<b>FLC</b>	0.382	0.686	0.335	1.000



**Figure 7.4.** Disturbance Rejection for PI Controller, (a) Simulation, (b) Experiment.



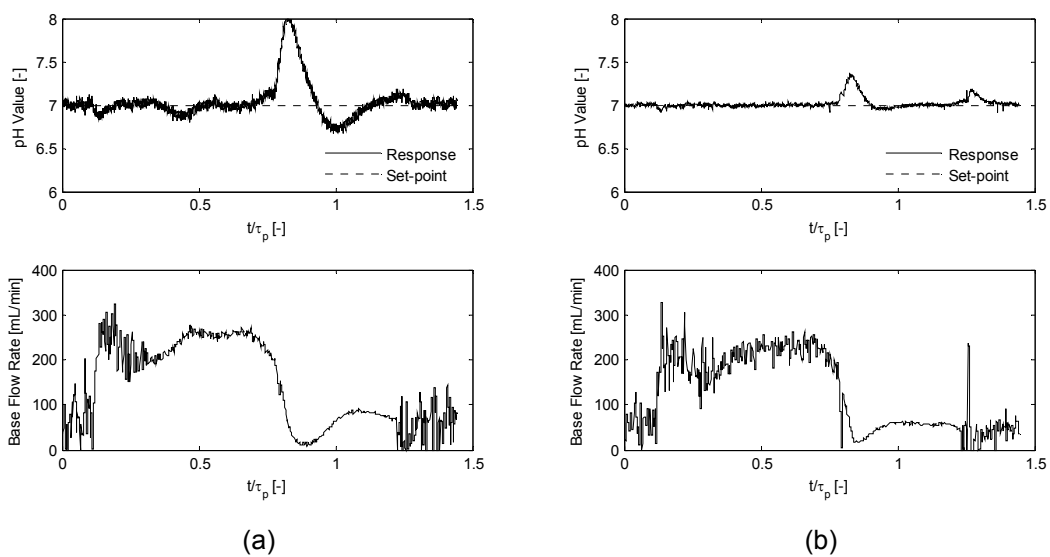
**Figure 7.5.** Disturbance Rejection for AMPC, (a) Simulation, (b) Experiment.



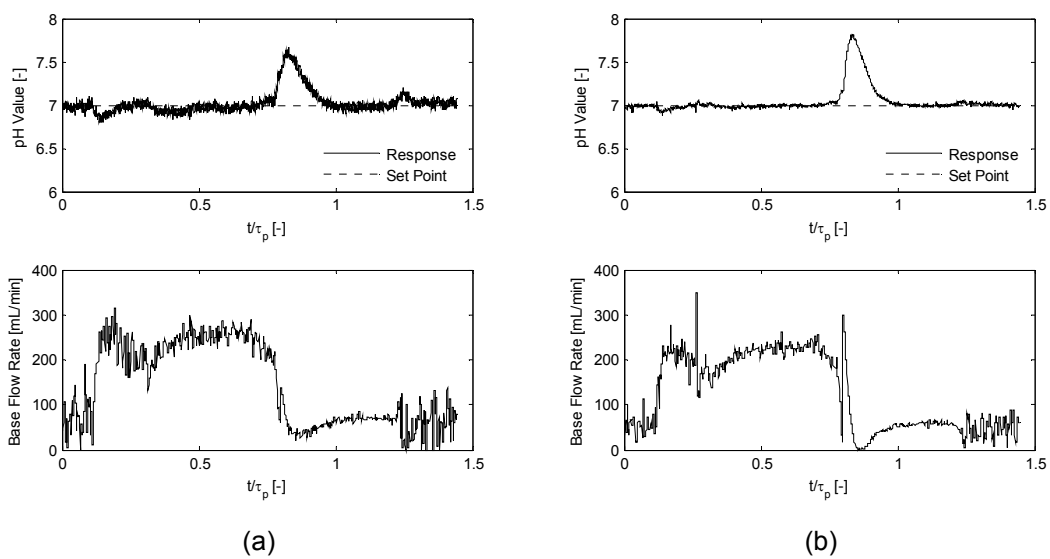
**Figure 7.6.** Disturbance Rejection for FLC, (a) Simulation, (b) Experiment.

### 7.3. ROBUSTNESS

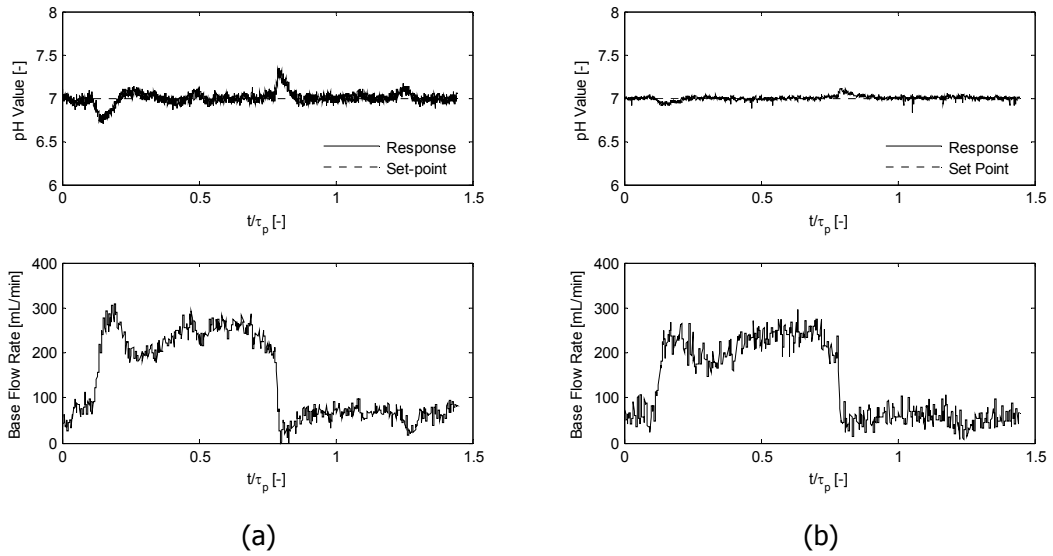
Robustness of the controllers is tested by reducing the volume of the main reactor by 10%. Since, disturbance rejection is more important than the set-point tracking for the neutralization processes, robustness of the controllers are tested for disturbance rejection case only. The process parameters are the same as those for disturbance rejection given in Table 7.3. As indicated in Figures 7.7–7.9, all of the controllers are found robust to modeling errors and there is no significant change in the responses with a 3% increase in the IAE scores in the simulations. In the experiments however, relative changes in the IAE scores are different for the designed controllers and AMPC is found to be more sensitive to modeling errors while FLC has superior performance than the others considering the IAE scores given in Table 7.5.



**Figure 7.7.** Robustness Run for PI Controller in Disturbance Rejection, (a) Simulation, (b) Experiment.



**Figure 7.8.** Robustness Run for AMPC in Disturbance Rejection, (a) Simulation, (b) Experiment.



**Figure 7.9.** Robustness Run for FLC in Disturbance Rejection, (a) Simulation, (b) Experiment.

**Table 7.5.** Normalized Performance Scores for Disturbance Rejection with Modeling Error.

Controller	Simulation Score		Experiment Score	
	IAE	IACC	IAE	IACC
PI	1.000	0.796	0.540	0.966
AMPC	0.662	1.000	1.000	0.725
FLC	0.397	0.682	0.275	1.000

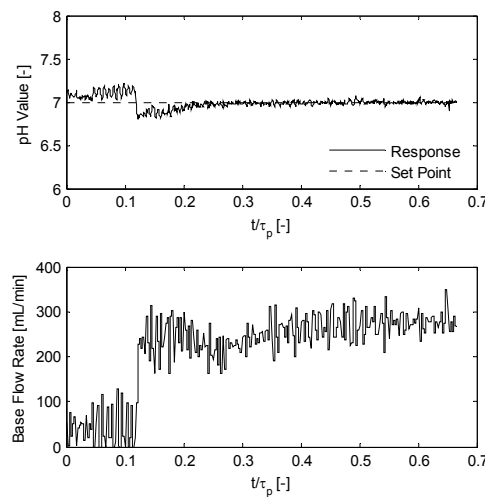
#### 7.4. TEST OF FLC USING ACTUAL PLANT'S WASTE WATER

The performance of the FLC is tested for disturbance rejection case by using actual plant waste water samples obtained from TUSAŞ Aerospace Industries, Inc. (TAI). Two waste water samples whose acid contents are comparatively different were taken at different times in a daily operation of the plant.

In the experiment, the process and controller parameters are taken as in Table 7.3. with a difference in the concentrations of the samples where Type 1 process stream corresponds to the waste water sample which has relatively low acid content and Type 2 represents the

high acid content sample. In addition, instead of the disturbances indicated in Table 7.3., for disturbance test, the reactor process stream is changed from Type 1 (low acid content waste water) to Type 2 (high acid content waste water) and the response of the FLC is obtained. The desired set–point value is taken as pH = 7.0. The NaOH solution used in this run is not same with the one used in previous experiments due to the concentration of acid in the actual plant effluent. Therefore, titrating stream concentration is also increased to 3.0M NaOH.

In Figure 7.10, experimental response of the FLC is shown. As seen form Figure 7.10, the FLC is able to keep the effluent pH value at pH = 7.0 with a short settling time (about 15% of time constant of the main reactor). Thus, the performance of the designed FLC shown by simulation studies and laboratory experiments are also verified by the experiment performed with actual plant waste water.



**Figure 7.10.** Experimental Response of the FLC for Disturbance Rejection Test Performed Using Actual Plant Waste Water Samples.

## **CHAPTER VIII**

### **CONCLUSIONS**

In this study, two advanced control techniques, Model Predictive Control and Fuzzy Logic Control, are successfully implemented to a laboratory scale pH neutralization system containing an identification reactor. After performing a number of computer simulations and experimental runs, following conclusions are obtained:

- An identification reactor can be used successfully in identifying the titration curve of an incoming stream in a waste water neutralization system.
- Experimental responses are found to be more sluggish compared to the simulation responses due to the buffering effect of the tap water.
- As a result of all the evaluations done to find the performances of the designed controllers, it is found that, the system can be modeled accurately and effective model-based controllers can be designed.
- Simulation runs done beforehand are very crucial in the evaluation of the tuning parameters for all the controllers, reducing the time of implementation in experiments.
- The PI controller and AMPC can only be applied by using the identification reactor which is easy to implement and use. On the other hand, FLC does not necessitate the identification reactor and considering the IAE scores it is the best controller design.
- Conventional PI controller is robust and can satisfactorily be used for both set-point tracking and disturbance rejections as good as others.

- The designed AMPC is the best controller for adjusting the control moves due to adaptation done in the  $f$  factor during the experimental runs. However, in the disturbance rejection application, AMPC gave poor results with the constant disturbance assumption in the simulation studies. Therefore, constant derivative of disturbance is assumed in the algorithm for the AMPC and then satisfactory control performance is obtained.
- The testing of the FLC with the actual plant (aircraft parts manufacture plant) waste water for the disturbance rejection case revealed that the designed controller is very good in performance in controlling the pH of the plant effluent as good as the laboratory studies done with the artificial composed acid mixtures.

Finally, in this highly non-linear and time varying process, although all the designed controllers can effectively be used, the FLC which does not necessitate an identification reactor and which shows far better performance than others, is recommended.



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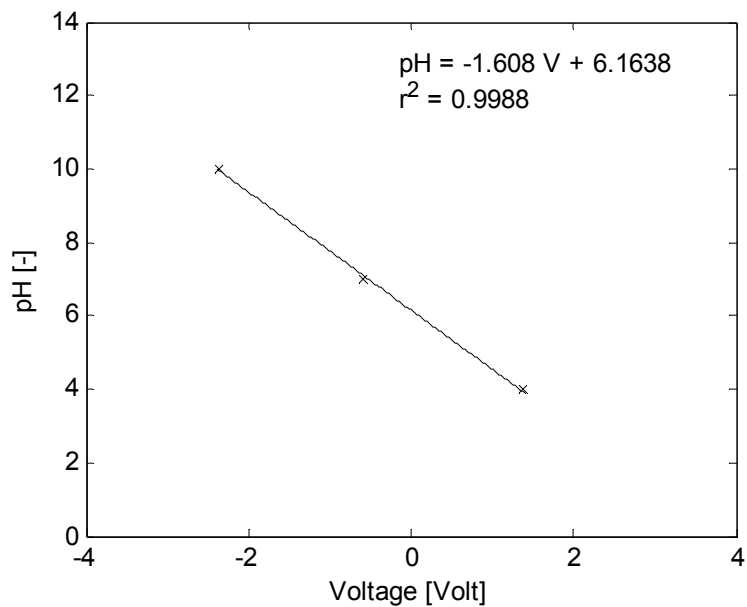
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## APPENDIX A

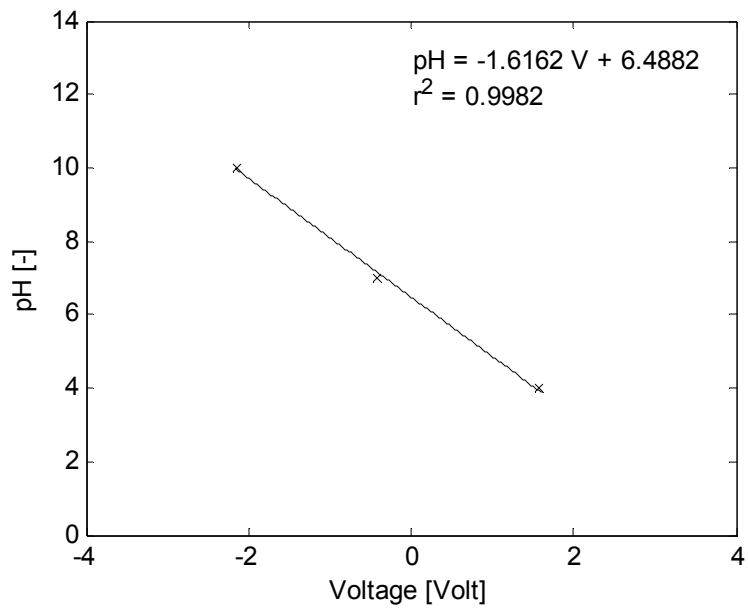
### CALIBRATION DATA

Calibrations of the pH meters were done using three calibration buffer solutions; pH=4.01, pH=7.00 and pH=10.01. Calibration plots for the pH meters are given in Figure A.1 and Figure A.2. Calibration data are plotted as pH versus Voltage, which refers output voltage of the preamplifier, and the data points are fitted to a straight line. Calibration equations and correlation coefficient,  $r^2$ , are shown on the plots.

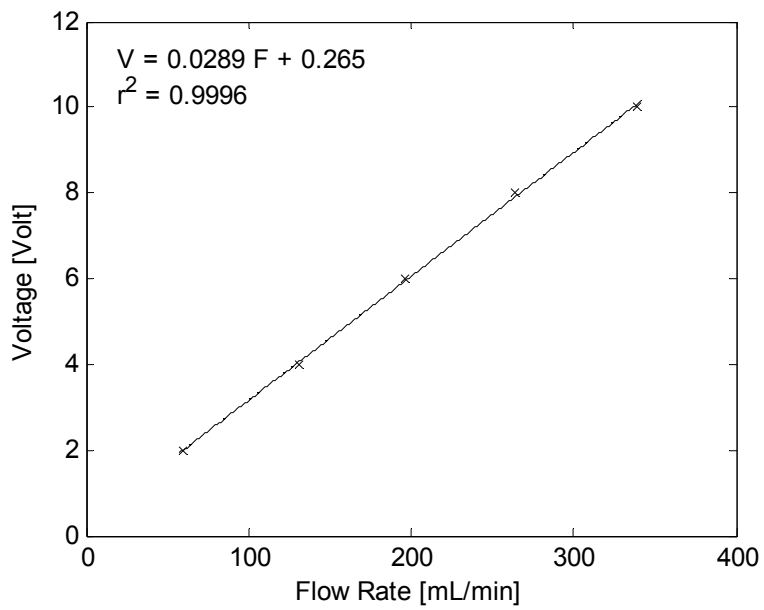


**Figure A.1.** Calibration Plot for pH Meter 1.

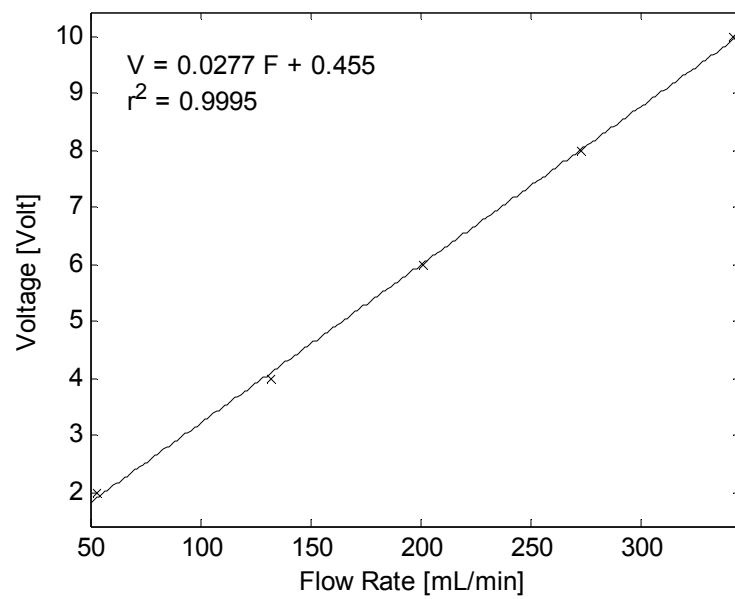
Peristaltic pumps are also calibrated in the range on 0–350 mL/min for selected voltage values of 2, 4, 6, 8 and 10 Volts. AD/AD card output voltage versus flow rate is plotted for each peristaltic pump and curve fitting is applied. Figure A.2 and Figure A.4 show the calibration plots and the resulting calibration equations are also shown on the corresponding figures with their correlation coefficients.



**Figure A.2.** Calibration Plot for pH Meter 2.



**Figure A.3.** Calibration Plot for Peristaltic Pump 1.

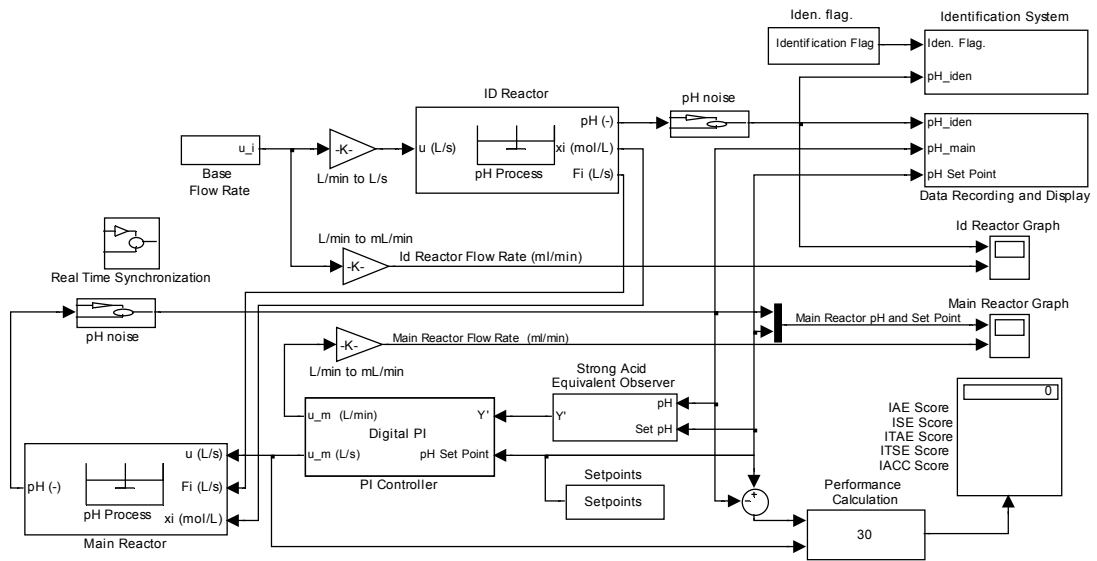


**Figure A.4.** Calibration Plot for Peristaltic Pump 2.

## APPENDIX B

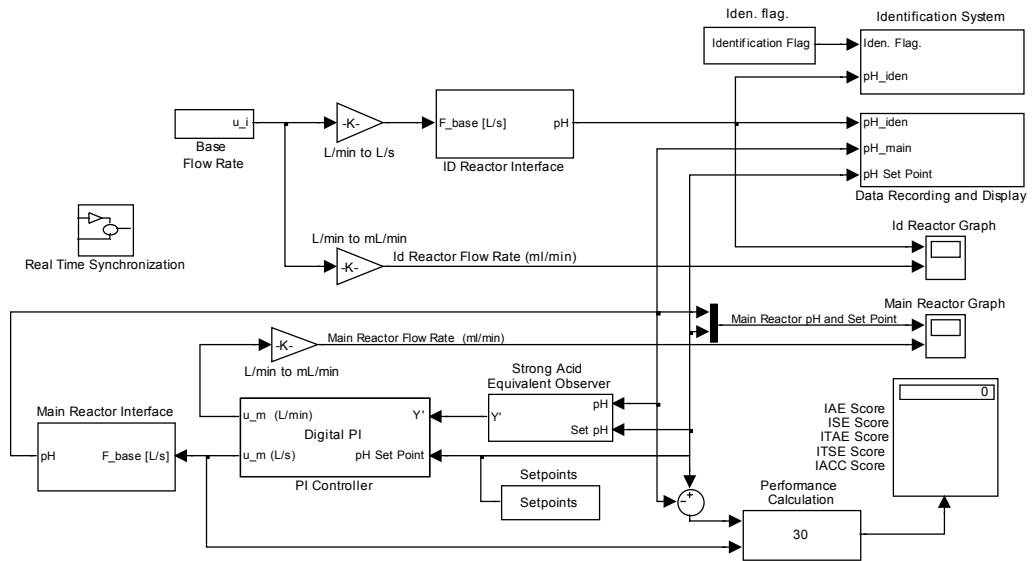
### SIMULINK MODEL OF THE CONTROL SYSTEM

In the study, simulations and experimental studies are performed in MATLAB<sup>®</sup>/SIMULINK<sup>®</sup> software package. SIMULINK model used in simulation studies is shown in Figure B.1 for PI controller only and for other controllers only PI controller subsystem is replaced with corresponding controller subsystem. The SIMULINK model used for simulations is also used in the experimental studies only by changing the pH process model subsystems (Main Reactor and Identification Reactor subsystems in Figure B.1) with input/output interfaces to enable the controller to communicate the experimental system (Figure B.2). All of the subsystems in the models shown in Figure B.1 and B.2 and the controller subsystems can be seen from Figure B.3 through Figure B.12.

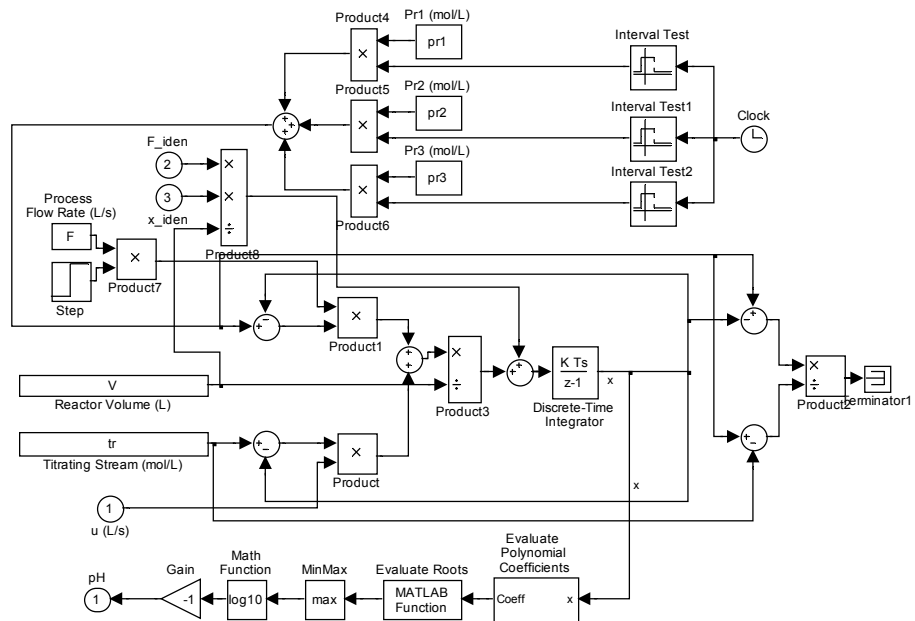


**Figure B.1.** SIMULINK Model Used in Simulations Studies.





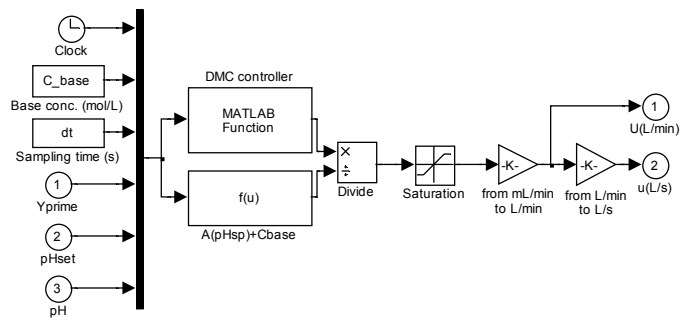
**Figure B.2.** SIMULINK Model Used in Experimental Studies.



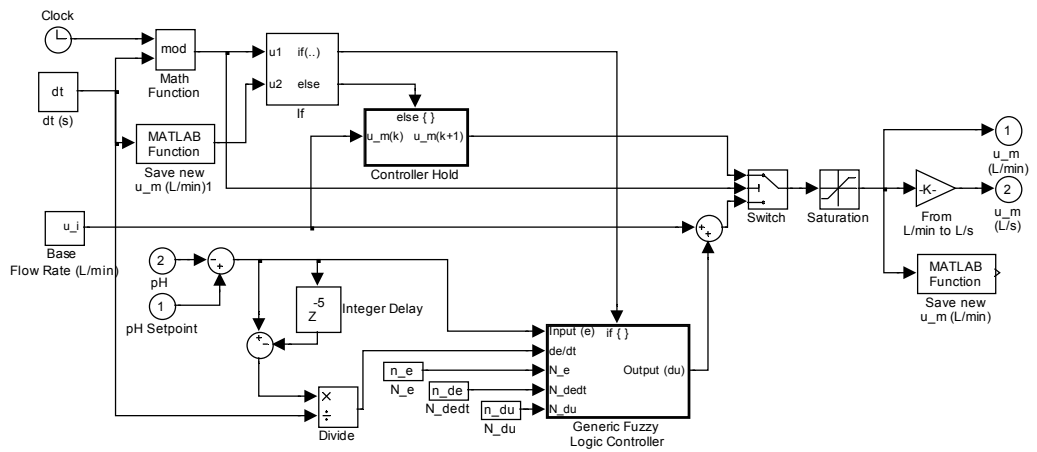
**Figure B.3.** SIMULINK Model for Simulation of pH Neutralization Process.



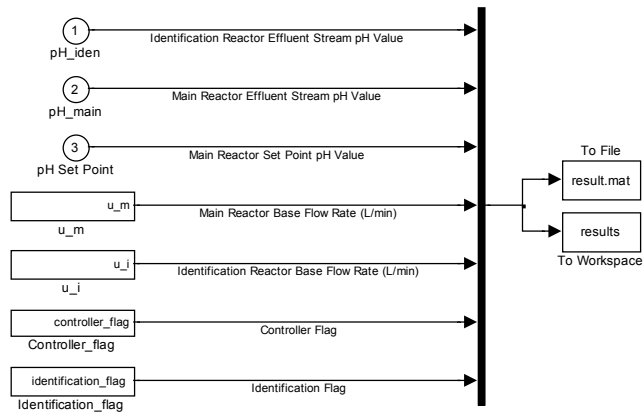




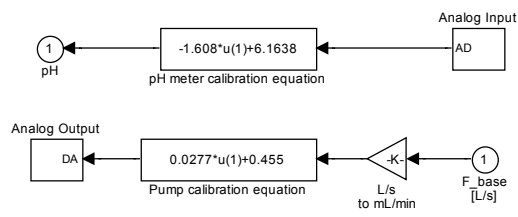
**Figure B.8.** SIMULINK Model for AMPC.



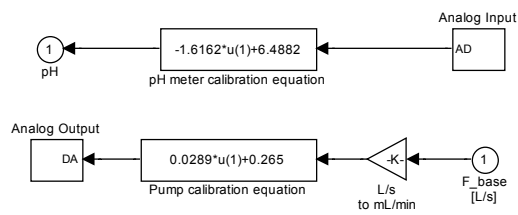
**Figure B.9.** SIMULINK Model for FLC.



**Figure B.10.** SIMULINK Model for Data Recording and Display.



**Figure B.11.** SIMULINK Models for Identification Reactor Interface.



**Figure B.12.** SIMULINK Models for Main Reactor Interface.