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Optimal PID control with anti-windup in neutralization process

ZeynepYilmazer Hitit [a,*], ***Ismet Kocer*** [a], ***Gokce Kus*** [a], ***Nermin Zeynep Arslan*** [a], ***Elif Pinar Dal*** [a] **and** ***Habipcan Koz*** [a]

[a]*Ankara University, Faculty of Engineering, Department of Chemical Engineering; 06100 Tandogan, Ankara; Turkey*

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ABSTRACT

PID control, which is a type of automation, was used to ensure that neutralization takes place in a controlled manner. To determine the PID parameters of the system with the Cohen-Coon tuning method, two different dynamic experiments were carried out for pH and temperature in the first stage, and the transfer function and model parameters were found. In the experiment carried out for the pH variable; time constant (τ) is 59 s, dead time (Θ) is 261 s and steady state gain (K) is read from the graph as 14,72, while PID parameters are calculated as K_C =0.0375, τ_I =315.759 s and τ_D =52.601 s. Likewise, while τ=1402 s., Θ= 88 s, and K=-6 were read for the temperature variable, the PID parameters were calculated as K_C =6.196, τ_I =47.23 s, and τ_D =-19.20 s. The determined controller parameters were used as initial parameters and simulated using the S-function block via MATLAB (2007b). The pH set range was coded as 6.5-8.5 and T_{set} for temperature was coded as 22 °C. As a result of the oscillation observed due to the nature of the PID control parameters that are intended to be controlled, the safe operation of the process and the desired set values are ensured. When the obtained PID controller parameters were applied to the neutralization reaction, the PID control successfully controlled the reactor temperature and pH and eliminated possible hazards in operation. Anti-windup provides better control rather than traditional PID control method.

1. Introduction

Acids are substances that give hydronium or hydroxyl ions to their solutions when dissolved in water. Bases are substances that increase the hydroxide ion concentration in their aqueous solutions. When acids and bases come together, they give neutralization reactions [1]. Acids are classified according to their strength. Acids that have a high tendency to ionize in their aqueous solutions and are assumed to be 100% ionized in theory are called strong acids. Similarly, bases that can ionize close to 100% in water in theory are called strong bases [2].

pH is the negative logarithm of the hydrogen ion concentration in the aqueous solution. Accordingly, while pH is 7 in neutral solutions, pH is less than 7 in acid solutions and pH is greater than 7 in base solutions [3]. The molecular structure of Hydrochloric Acid (HCl), an example of a strong acid, is quite simple. Hydrochloric acid has a pronounced effect on bases [4]. This effect can be seen as the temperature rises rapidly as a result of the reaction of acids and bases. Hydrochloric acid can be used to lower the basicity or increase the acidity in a solution. For example, if hydrochloric acid is dropped into a solution containing OH- ions, the base in the solution is replaced by water and H+ ions [5]. Hydrochloric acid should not be released directly into the environment. Inhalation of even a small amount of gaseous hydrochloric acid poses a great risk to the entire respiratory tract for human health [6, 7].

The formation of water and salt as a result of the reaction of the H+ ion in the acid with the OH- ion in the base is called the neutralization reaction. Not every acid and base can be used as a reactant in the reaction [8]. The main properties sought in acids and bases to be used for this purpose are as follows; they must dissociate into ions to a great extent in the aqueous medium, are not volatile in their dilute solutions, dilute solutions are not easily affected by air and light, dilute solutions must not be strongly oxidizing or reducing, and the salts produced

* Corresponding author. Tel.: +90 312 203 3433; Fax: +90 312 212 7464
E-mail addresses: zyilmazer@ankara.edu.tr *(Z.Y. Hitit,* issmetkocer@gmail.com *(I. Kocer),* gokceks09@gmail.com *(G. Kus), neparslan@outlook.com (N.Z.Arslan),* pinardal47@gmail.com *(E.P.Dal), kozhabipcan@gmail.com (H. Koz)*
ORCID: 0000-0001-9078-191X *(Z.Y. Hitit), 0000-0001-8319-5437 (I. Kocer), 0000-0001-9931-3899 (G. Kus), 0000-0001-7221-8058 (N.Z. Arslan), 0000-0001-5060-1275 (E.P.Dal), 0000-0002-1054-6993 (H. Koz)*
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during neutralization should dissolve well in water. In this respect, HCl and NaOH acid-base pairs (Equation1) are suitable chemicals for neutralization [9, 10].

$$HCl + NaOH \rightarrow H_2O + NaCl + Heat \quad (1)$$

For neutralization to occur, the number of moles of hydrogen or hydronium ions from the acid must be equal to the number of moles of hydroxide ions from the base [11]. Accordingly, 1 mol of HCl can neutralize with 1 mol of NaOH. Depending on the strength of the acid and base used, the quality of the salt formed as a result of the reaction changes. The salt obtained (NaCl) is a neutral salt, since HCl and NaOH are strong acid-base pairs [12].

HCl, which is generally obtained as a by-product (production of common organic chemicals such as chlorination in the chemical industry, sodium hypochlorite, Teflon, polyvinyl chloride, and perchloroethylene) may not be at a suitable concentration or purity for use as an input in any process. Therefore, it has a destructive feature on the environment and must be disposed of or converted to another harmless form [13]. Based on this information, one of the best methods that can be applied is to obtain salt (NaCl) by performing a neutralization reaction with sodium hydroxide (NaOH). However, it is known that it is difficult to control the reaction parameters by manual methods due to the high rate ($1x10^{-14}$ L/gmol.s) of this reaction [14]. For this reason, neutralization systems are supported by PID control, which is a type of automation [15, 16]. Automatic controllers which are used to control important parameters such as pH, temperature, level, and keep process variables at desired design values in order to maintain the entire chemical process efficiently.

In this study, it was aimed to keep the pH and temperature parameters within the targeted value range throughout the reaction. PID control method, which provides a safe, environmentally friendly and automatic process, is used to keep the parameters within the desired range. Process control systems provide the opportunity to make changes on other process variables while keeping a process variable constant in order to control the above-mentioned variables. In this way, PID control method ensures that the process works in line with the desired values [17, 18].

Since the HCl-NaOH reaction discussed in this study is an exothermic reaction, it is aimed to determine the system parameters at the laboratory scale and design the process control system accordingly. Process control includes reaching the target and maintaining the current situation after reaching the target in the process extending from the input of the desired target to the result. Control parameters vary according to the type of process considered. In order to ensure product quality and continuity, parameters affecting the course of the process such as product concentration, temperature, pH and flow rate should be controlled. Thus, neutralization is achieved successfully [19, 20].

2. Materials and Method

2.1. Experimental Setup

In this study, it is aimed to maintain the process in maximum efficiency and safe conditions while removing HCl. In the system, a reactor made of glass, with a volume of 2 L and a jacket volume of 1.5 L, was used in continuous operation. In order to ensure homogenization in the system, a mechanical agitator was used. A thermocouple was added into the reactor to determine the temperature changes that will occur due to the nature of the reaction used in the experiment. So as to prevent the heat that will be released during the reaction from affecting the process negatively, cooling water is passed through the jacket around the reactor with the help of a peristaltic pump. In order to monitor the pH and temperature desired to be controlled, a pH probe and thermocouple was placed in the system. Temperature, conductivity, pH, and pump flow rates which are desired to be recorded throughout the experiment are provided using a computer and electronic circuit equipment. The HCl that should be neutralized was chosen at 0.014 M as waste at a constant flow rate of 0.193 L/min. To realize neutralization properly, a 1:1 (mol/mol) ratio of acid and base should react. Varying flow rates of NaOH based on controller output value was used to neutralize HCl. Therefore, 0.014 M HCl and 0.025 M NaOH were prepared in beakers and fed to the reactor by peristaltic pumps. The NaCl salt to be obtained during this reaction was removed from the system in the form of anaqueous solution. In Figure 1, PID control experimental setup is given.

2.2. Transfer Function and Closed Loop Control System

The dynamic relationship between an input and an output variable in a process can be explained by the transfer function (Equation2). In a system where simultaneous control of the pH and temperature in the reactor is desired, the feedback control loop operates to ensure that the reactor temperature and pH value in the system are controlled at the targeted set values. This control loop

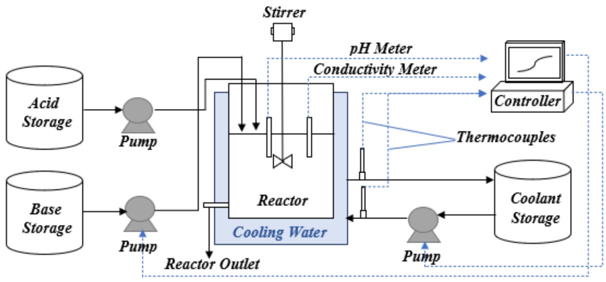

Figure 1. PID Control Experimental Setup

takes place by adjusting the flow rate of the cooling water through the cooling jacket and the flow rate of the base pump for temperature and pH control, respectively [21, 22].

In the feedback control system for temperature, electrical signals are received using a thermocouple of the reactor internal temperature and converted into digital signals by means of a transducer [23, 24]. Thus, these signals can be read with the help of a computer. Then, in order to provide temperature control, the difference between the temperature values measured with the help of a thermocouple and the temperature set value is determined and the error value is calculated. According to this error value, a manipulated variable signal is sent to the cooling water pump by the controller, and temperature control is provided.

For pH control, while the electrical signal is given to the computer by the pH meter, the difference between the measured pH value and the pH set value is calculated in the same way, and the base pump flow rate changes according to the error value.

Considering the data obtained after the system identification with dynamic response analysis, it was concluded that the system is a first order plus dead time model. Accordingly, the transfer function of the mentioned model is given in Equation 2.

$$G(s) = \frac{K(e^{-\theta s})}{\tau s+1} \qquad (2)$$

Where G(s) is the transfer function, K is the process gain, θ is the dead time and τ is the time constant of process. To find the model parameters along with the transfer function the process reaction curve method was used. Negative step effect was given to the flow rate of the cooling water whereas, positive step effect was given to the base pump after the control system became stable. The temperature of the cooling water at the entrance and exit of the jacket with the thermocouples placed in the reactor and the pH data with the pH probe placed in the reactor were continuously recorded. To control the desired neutralization reaction with automation using the information obtained, PID control parameters were calculated by Cohen-Coon tuning method, and these values were used in control experiments using MATLAB Simulink as initial parameters and simulated with the S-function block with the help of MATLAB Simulink.

2.3. PID Control Method and Algorithm

The PID method is widely used in processes with rapid and large load changes. In PID control, the derivative and integral of the difference signal between the set value and the measured value are taken. The proportional signal, integral signal and derivative signal are collected in the adder circuit. In this way, a correction is made in manipulated variable according to Equation (3) [25, 26].

$$m(t) = K_P(t) + K_I \int_0^t e(t)d(t) + K_d \frac{de(t)}{d(t)} + V_o \qquad (3)$$

PID controller parameters need to be adjusted according to process dynamics. For adjustment, firstly the process model must be defined. Process Reaction Curve (PRC) Method was used in order to calculate the PID parameters with Cohen-Coon Method [21].

2.4. Dynamic Experiment

In general, dynamic experiments are known as laboratory-scale measurements performed under excitation conditions that change over time. Since dynamic experiments are performed under physical, economic, and device constraints, they should be applied after excellent preliminary research [18]. After deciding on the parameter to be controlled before the experiment, it should be determined how the variable will be measured. These parameters are the factors that directly form the character of the system. Within the scope of this study, two different dynamic experiments were carried out for two different parameters to be controlled. While the process output variable was defined as pH to determine the pH control, the process output variable was defined as temperature to determine the temperature control. The flow rate of the base pump and cooling water pump was changed to create a load effect (step input) on pH and temperature, respectively. The experimental setup of the PID control for pH and temperature control is given in Figure 2.

2.5. Anti-Windup

Actuators always possess both a lower and an upper limit, referred to as saturation limits. When these limits, or saturations, are reached, the dynamics of the process output tend to respond more slowly compared to instances where there's a significant set point alteration or disturbance. Consequently, in such scenarios, the integral component (I) of a PID controller experiences rapid escalation due to the gradual reduction in error caused by saturation. The control output, or actuator, becomes saturated. This occurrence is termed "integral windup" [27].

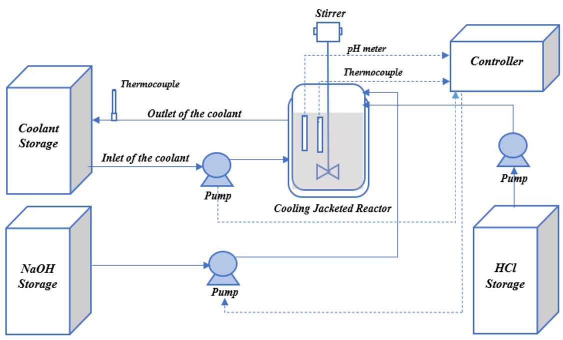

Figure 2. PID Control for the Temperature and pH

As a result of actuator saturation, the integral component, up until the rise time, becomes substantially larger than its eventual value. Consequently, a notable overshoot (i.e., a considerable negative error) becomes unavoidable in order to diminish the already accumulated integral component to the desired final integral value. In this manner, integral windup adversely affects control performance [28, 29].

Numerous techniques exist to mitigate integral windup. One of these approaches is known as "back calculation," while another is referred to as the "Conditional Integration Method." The Back Calculation Anti-windup technique employs an internal feedback loop to guide the convergence of the integral component toward the actuator's limit [18].

The Conditional Integration (CI) Anti-windup method, on the other hand, prevents integral windup by temporarily suspending the integral action when the actuator reaches its limit value[30].

3. Result and Discussion

PID controller parameters were calculated with the help of Cohen-Coon tuning method, so the transfer function of the system was determined with the help of a dynamic experiment.

3.1. Dynamic Experiment for Calculating PID Control Parameters

A dynamic experiment was carried out to determine the PID Control Parameters for the neutralization reaction. Two different dynamic experiments were carried out for two different parameters that were to be controlled. For the determination of pH control parameters, the process variable is defined as pH and manipulated variable is defined as base flow rate, while the temperature for the determination of control parameters, the process variable is defined as temperature and the manipulated variable as cooling water flow rate. To determine these parameters, the step inputs are given to manipulated variables for the dynamic analysis. Figure 3.a shows pH change with time by giving step input to base flow rate. Figure 3.b represents T change with time by giving step input to cooling water flow rate. Accordingly, since the transition from the first steady state to the second steady state was observed, it was concluded that the system has shown first order plus dead time model properties [27, 31]. In addition, a positive step effect is given to the system when the system reaches the first steady state for the purpose of determining the model parameters. This positive charge effect was achieved by increasing the base pump flow rate from 10 mL/min to 40 mL/min at 491 s. It was observed that the pH value increased from 3,1536 to 10.5176 when this effect was given to the system. Model parameters were determined by dynamic process reaction curve method. For this purpose, the "S curve" formed between 0-500 s was examined. In the area covered, the run time (τ) is 59 s, the dead time (Θ) is 261 s, and the static gain (K) is found as 14.72 ◦Cs/mL pH value/mL NaOH flow rate. After the model parameters are determined, the Cohen Coon method is applied to find the PID parameters according to the PID control algorithm. Accordingly, PID parameters calculated for pH were calculated as K_C=0.0375, τ_I=315.759 1/s, and τ_D=52.601 s using Cohen-Coon equations as shown in Table 1.

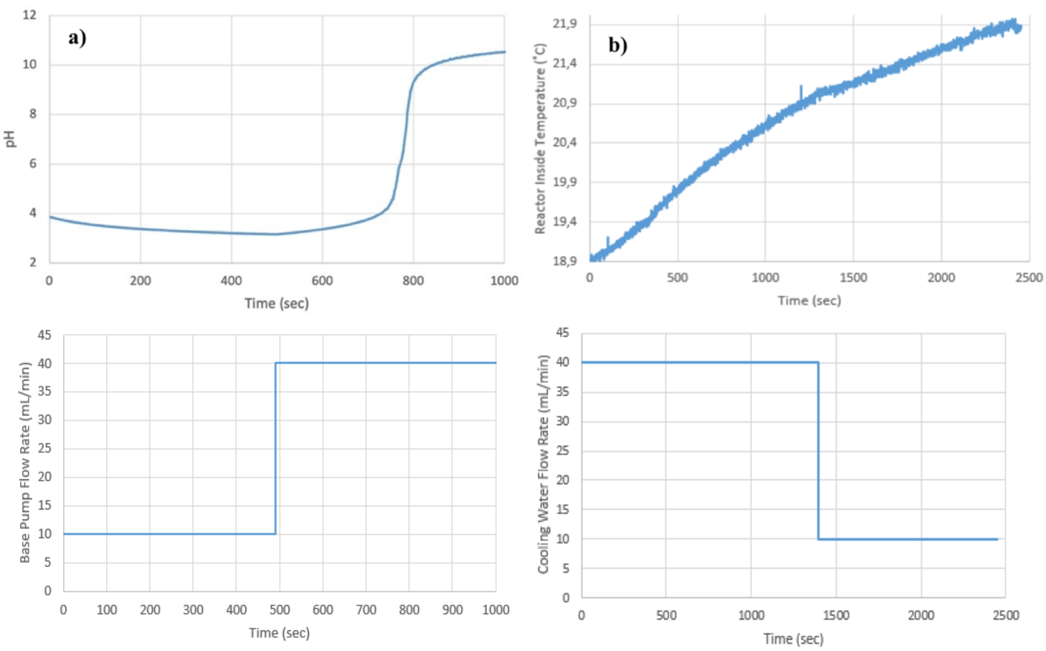

Figure 3. a) pH and base flow rate change b) T and cooling water flow rate change with time during the dynamic experiment

Table 1. Determination of PID control parameters with positive and negative step input given to controlled variables pH and T

Controller	Input	PID Parameters	Controller	Input
pH Controller with PID	Positive step input to NaOH flow rate from 10 mL/min to 40 mL/min	0.0375	315.759	52.601
T Controller with PID	Negative step input to Cooling water flow rate from 40 mL/min to 10 mL/min	6.196	47.23	-19.20

The temperature change with time is given in Figure 3.b, as pH. In addition, a negative load effect is given to the system when the system reaches the first steady state to determine the model parameters. This negative charge effect was achieved by changing the cooling water flow rate from 40 mL/min to 10 mL/min, which is given in Figure 3.b. It was observed that the temperature value increased from 18.87 ^{o}C to 21.87 ^{o}C when this effect was given to the system.

To investigate the model parameters for temperature, between 0-3835 s in the graph were examined. In the area covered, the time constant (τ) is 1402 s., the dead time (Θ) is 88 s, and the steady state gain (K) is found as -6 ◦C s/mL Then, PID parameters are calculated as K_C = 6.196, τ_I = 47.23 1/s, and τ_D= -19.20 s. by using Cohen-Coon equations as shown in Table 1.

3.2. Experimental PID Control of Neutralization Reaction

After the PID parameters were determined for pH, a neutralization reaction of hydrochloric acid and sodium hydroxide was performed with PID control. In the experiment performed in pH control with PID, the determined pH set point must be between 6.5-8.5 according to the water pollution control regulation [32], and pH control with PID is given in Figure 4.a, base flowrate changes with time is given in Figure 4.b and accordingly the Integral Square Error (ISE) change for pH with time is given in Figure 4.c.

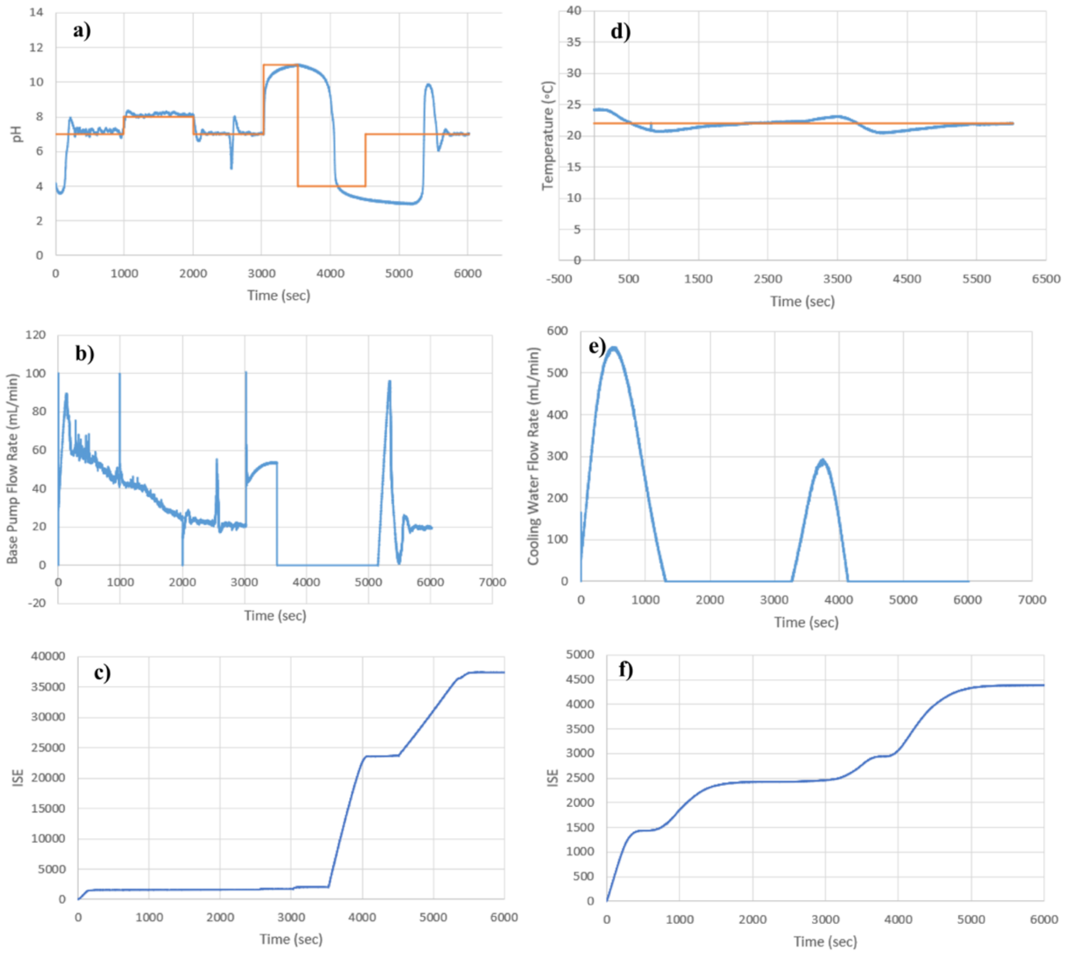

Figure 4. Experimental pH and Temperature Control with PID Control
a. pH control b. Base flow rate c. Integral Square Error (ISE) change for pH d. T control e. B flow rate f. Integral Square Error (ISE) change for T

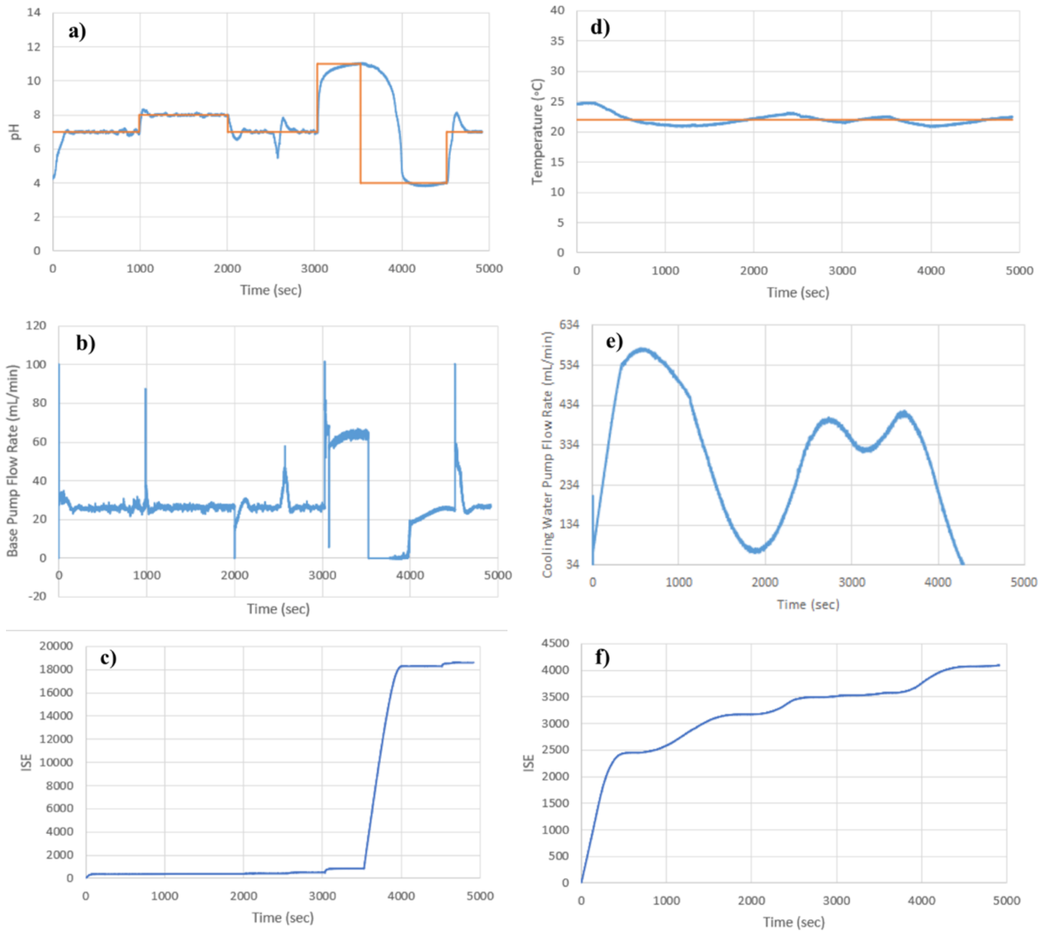

Figure 5. Experimental pH and Temperature Control with PID Control

a. pH control b. Base flow rate c. Integral Square Error (ISE) change for pH d. T control e. B flow rate f. Integral Square Error (ISE) change for T

Similarly, the PID parameters determined for the temperature were used in the neutralization reaction. Thus, temperature in the reactor changes according to time is given in Figure 4.

The flow rate of the cooling water determines the ability to keep the temperature at the desired set value. Temperature control is given in Figure 4.d. The change of cooling water flow rate with a time graph is given in Figure 4.e. Likewise, error signals are given to the cooling water pump while PID controls the temperature. Figure 4.f shows a graph of the evolution over time of the sum of the squares of the integrals of various error signals.

After the dynamic analysis, PID control parameters were calculated and coded into the controller. To observe the time-dependent variation of pH and temperature data under PID control, the flow rate of the 0.014 M HCl pump is kept constant at 0.193 mL/min, and flow rate the pH of the 0.025 M NaOH pump is changed by the flow rate controller. pH of the reaction has been tried to be kept in the range of 6.5-8.5. Considering the graphics, it has been observed that the coded set values reached after the oscillation, which is the behavior characteristic of the PID. Input variables were changed to observe the load effect. The load effect is given by increasing the acid pump flow rate from 40 mL/min to 80 mL/min at 2500 s. As a result of this effect, although a sudden increase in pH is observed, it has been observed that PID control is successful in controlling pH at the pH set point.

3.3. PID Control with Anti-windup

Anti-windup, a type of PID control, was applied for both pH parameter and temperature parameter in order to provide better control while performing PID control in the neutralization reaction. Anti-windup control is achieved by using the conditional integral (CI) method. pH change with time by ARW is given in Figure 5.

pH change with time by Anti-windup is given in Figure 5.a. While pH control was achieved with the Anti-windup method, the pH set points were changed. It has been observed that when the pH set point is lowered from 11 to 4 at 3524 s, the control with Anti-windup control provides better control than the PID control without Anti-windup. To keep the pH at set values base flow rate change with time by Anti-windup is shown in Figure 5.b. It has been observed that the sum of the integrals of the error signals decreases thanks to the PID with Anti-windup method, which is used to ensure that integral part of PID controller output the sum of the integrals of the error signals is not above or below the maximum or minimum outputs values of the final control element. The graph of the sum of the squares of the integrals of these error signals for pH versus time is given in Figure 5.c.

Likewise, the temperature control in the reactor was carried out with the Anti-windup method. The results obtained and the variation of the temperature values in the reactor against time are given in Figure 5.d whereas, the change in the flow rate of the cooling water pump against time to keep the temperature at set value is given in Figure 5.e. Finally, the graph of the sum of the squares of the integrals of these error signals for temperature versus time is given in Figure 5.f.

The pH range of the product is 6.5-8.5 in the targeted direction, the experimental designs were made according to this purpose. With the literature search, it was observed that when NaOH and HCl reactants were taken in a 1:1 molar ratio, neutralization was achieved, and the pH value was within the predicted range. The effects of acid and base used in the neutralization process on pH, conductivity, and temperature were investigated by conducting experiments in a continuous type of reactor using constant acid/base concentrations.

4. Conclusion

In order to provide pH and temperature control in neutralization reactions with PID Control Method, PID control without Anti-windup and PID control with Anti-windup were used. PID control method with Anti-windup provides control according to the maximum and minimum working capacities of the working equipment. Based on the data, it has been observed that Anti-windup method, which provides control by adhering to the sudden changes in the nature of pH and temperature parameters in exothermic reactions, where the reaction rate is high, is much more successful in controlling the system parameters that are desired to be controlled. As a conclusion, process control steps were applied systematically to make the hydrochloric acid produced as a by-product in the industry suitable for disposal, and with the help of PID control, the pH of the product formed as a result of the reaction was successfully controlled in the range of 6.5-8.5 and its temperature at 22 °C. A better performance was obtained by using the conditional integral method as an anti-windup technique. In future studies, experiments can be performed using the back calculation method, which is another anti-windup method, and its performance can be compared.

Declaration

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The author(s) also declared that this article is original, was prepared in accordance with international publication and research ethics, and ethical committee permission or any special permission is not required.

Author Contributions

Z.Y.Hitit supervised the study. I.Kocer implemented the controller algorithm, data acquisition and methodology, G.Kus , N.Z. Arslan, E.P.Dal, H.Koz performed experiments and wrote the manuscript together.

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