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Heat transfer system and feedback temperature controller design for safety process operation of phosphorous acid potassium salts production

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ABSTRACT

The phosphorous acid salts are widely used in the industry because of the effective treatment against various fungal diseases encountered in plants. The production process is exothermic and, with high temperatures around 94°C achieved, significant risks were observed in terms of occupational health and safety. Therefore, the aim is to design a control system that will make this production process reliable for human health, economic and ecological damage. For this purpose, studies were carried out to determine the optimum operating mode, heat transfer system, and temperature controller design to prevent a sudden temperature rise. First, the overall heat transfer coefficient between the reactor and the jacket was determined as 51.0930 W/m^2°C and, the refrigerant was chosen as cooling water with 1.271 g/s flow rate which is relatively more economical and accessible. The model parameters of the system were determined with a detailed dynamic analysis by giving positive and negative step inputs to the cooling water flow rate and then obtaining model parameters through reaction curve and linear regression methods. By using the obtained model parameters theoretical P, PI and PID parameters were calculated by Cohen Coon and, Ziegler-Nichols approaches, and the success of controller parameters was tested, simulated with the MATLAB Simulink program and lastly, successful temperature control was achieved in the experimental system.

1. Introduction

Ensuring the nourishment of humanity is an issue that has been studied for centuries. Pesticides are being developed as an alternative solution for this situation. Today, about 80% of pesticides used in agriculture are applied in developed countries [1]. Pesticides having an important role in food production are in use to keep crops healthy against insects, fungi, weeds, and other pests. They are not only known to maintain and/or increase crop yields, but they also indicate how many time crops can be grown on the same field per year. This is particularly important in countries experiencing food shortages [2]. However, there are dangers in the use of pesticides. Pesticides affect the health of thousands of people worldwide each year [3, 4]. This toxic effect of pesticides to humans can cause both acute and chronic health problems, depending on the exposure time and type [2, 5, 6].

Compounds whose active ingredient is phosphorous acid (H_3PO_3) show pesticidal properties in many plants. However, for phosphorous acid products to provide a nourishing effect to the plant, must be produced under suitable conditions, continuously and sustainably. Since the pH value of the phosphoric acid, which is an aqueous solution of phosphorous acid, is approximately 1.5, it should not be directly contacted with the plant, as it can cause permanent damage to the plant [7]. Therefore, for efficient and effective product formation, the active ingredient and weak acid, phosphorous acid (H_3PO_3), need a neutralization reaction with a strong base. Mono and dipotassium phosphide (KH_2PO_3 and K_2HPO_3) are formed because of the exothermic reaction of phosphonic acid, which is an aqueous solution of phosphorous acid, and potassium hydroxide. These products, which have a liquid soluble (SL) form, are expected to be pH-neutral solutions

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(preferably between 6.2-6.7) to provide nutritional benefit while not harming the plant to which they are applied [8]. Potassium phosphide salts both have a direct toxic effect against plant pathogens and activate the natural defense mechanisms of plants by reducing the effects of diseases and/or eliminating diseases. Thus, they make a double effect. Systemically up and down movements of the plants in the vascular system of the plants, including the root system, which is quickly absorbed by the plant and roots to ensure maximum and yielding usage [9]. In addition to acting by walling up the pathogen inside the fungus, killing the surrounding cells in disease formation or insect invasion, and preventing further fungal growth, the plant also activates its immune defense system. During this, other fungitoxic compounds accumulations and metabolic changes, resistance inducers are set off to response more. The remaining part of the plant starts to produce other compounds to increase its resistance against possible infection(s) or attack in other parts of the plant [10]. Induced resistance (IR) and systemic acquired resistance (SAR) and are known two responses. To survive against any negative influence from the outside, plants have the ability to prepare their immune systems against microbial pathogens. This results in robust and rapid stimulation of defense mechanisms after a possible pathogen bombard [11–13]. An example of this stimulation is SAR, which is defined as the immune response of plants to pathogen attacks [13]. Activation of SAR causes broad-spectrum systemic resistance evolution [14–17]. Determining the biochemical changes that cause the resistance state is important for the development of plant protection chemicals, SAR, which is a new model of genetically developed disease resistance [16]. Induced or induced resistance (IR) is the mechanism by which plants deliberately meet specific microbes. This system aims to provide resistance against infection by pathogens in treated plants. Stimulated plant defenses are expected to involve the lignification of cell walls. This is achieved by the addition of chemical cross-links to cell wall peptides. Thus, infection in the plant becomes hard [18]. During the production of potassium salts of phosphorous acid, which is an efficient and effective pesticide, heat release occurs. In other words, the potassium phosphide production reaction is exothermic. When this situation is not taken under control, it contains risks such as reactor explosion, formation of toxic by-product(s), decrease in product yield, and significant ecological and economic damage[19–22]. The rapid depletion of the input concentrations and thus the rapid change in the transformation not only increases the reaction temperature but also prevents the formation of the chemical kinetic model of the reaction. It is essential to design a system that will control the temperature to eliminate possible hazards and to develop an efficient production process. Due to this need, the type and optimum flow rate of the cooling fluid that can absorb the heat released in the reactor during the production of mono and dipotassium phosphide salts were determined and a PID (Proportional Integral Derivative) controller was designed to provide temperature control with the selected refrigerant. In industrial applications, PI and PID controllers are favorably and broadly employed [23]. While most of the PID controllers were used as analog in the past, controllers used with digital signals and computers are frequently encountered today [24]. In recent years; system identification and adaptation schemes are being developed to create PI and PID controllers that are optimal for the design objectives studied [23, 25–27]. The design of PI and PID controllers for linear systems and different design techniques for nonlinear systems are mysteries [23, 28–30]. Due to the non-linear parameter variations and disturbances, PI or PID controllers design procedures have problems in closed loop system [23, 31]. The lack of clarity regarding the selection of parameters of the sampling period and discrete-time responses for PI or PID control systems is another disadvantage of the current state of the problem [23, 32]. Where a mathematical model of a system is available, it is possible to specify the parameters of the controller. However, if a mathematical model is not created, an experimental method is needed to determine the parameters. In-process control systems, controller setting can be defined as of obtaining controller parameters for reaching the desired output. This operation is an important term for control systems as it allows optimization of another operation and minimizes the error of measured output and the setpoint [25, 33, 34].

This paper, it is aimed to design the optimum process control system in order to the heat released during the reaction. For this purpose, optimum operating mode and reactant (input), heat transfer coefficient between reactor and jacket, optimum refrigerant type, and flow rate were determined. By using Cohen-Coon and Ziegler-Nichols adjustment methods; P, PI and PID parameters were calculated and their performance and applicability were examined using MATLAB Simulink, MATLAB Simulink Tuning, and VISIDAQ programs and plugins.

2. Materials and Methods

The main purpose of process control is to ensure quality production under safe and efficient operational conditions. Process control is concerned with how to achieve and maintain these goals. Values such as product concentration, temperature and pressure must be controlled for product quality and continuity in large-scale, integrated processing plants such as refineries. Numerous transaction variables can be manipulated for these purposes. Feedback control systems keep controlled variable at the desired value by the adjustment of manipulated variable [35]. Process control can be defined as sequential control, which is a state-based process that follows each other until a process is completed, or continuous control, which includes the observation and adjustment of fixed process parameters. [36]. In this study, it is aimed to determine the model parameters and to create a process control system in the exothermic production process at high temperatures. For this purpose, after determining the overall heat transfer coefficient (U) of the reaction, the system was simulated, and the refrigerant type and flow rate were decided. Since the thermodynamic properties of the production reaction of potassium phosphite salts, which is a neutralization reaction with significant risks, are not encountered in the literature, to determine the heat generated as a result of the reaction, insulation was made around the reactor to be used with 1 cm and 2 cm thick air and elastomeric rubber foam, respectively, and the heat transfer of the reactor with the external environment was minimized. The properties of the insulation material are given in Table 1. After the reactor was insulated, the reaction environment was simulated by sending 39 W of heat to the system for 4000 s with a submersible heater. Thus, 156000 J of heat was given to the system with the help of a submersible heater. The heat generated in the reactor was calculated using Equation (1).

A jacketed and stirred glass reactor with a diameter of 10 cm and a height of 20 cm was used to determine the overall heat transfer coefficient. Experiments were carried out repetitively with two different fluid types, water, and ethylene glycol, at different flow rates. Equation (2) was used for the calculation of the entire heat transfer coefficient (U). Here Q; the amount of heat received by the cooling water (W), U; total heat transfer coefficient (W/m^{2o}C), A; heat transfer area (m^2), ΔT_{ln} (ºC); is the logarithmic mean of the temperature difference between the reactor and the jacket cooling water. The heat transfer area is calculated as 0.063 m^2 by Equation (3).

2.1 Determination of Model and Control Parameters

In a system where the reactor temperature is controlled, the feedback control loop operates to adjust the flow rate of the coolant supplied to the jacket to bring the reactor temperature in the system to the set point. In the feedback control system, the reactor internal temperature (T_R) is measured as electrical signals using a thermocouple, and the temperature value with a transducer creates digital signals that the computer can understand. The value of the measured electrical signals is compared with the determined temperature value (reference value) and the error value is calculated. The controller calculates the flow rate of the cooling water (F_{cw}, mL/min) using the calculated error value and sends it to the peristaltic pump, which is the last control element, to transmit to the process. Thus, the cooling water flow rate is adjusted. Depending on the flow rate, the amount of heat transferred from the reactor to the jacket, and therefore the temperature inside the reactor changes. This cycle repeats over and over like this. When the temperature in the reactor (T_R) reaches the set point, the cooling water flow rate and the temperature inside the reactor remain at a constant value unless an external effect is given. In case the system deviates from the setpoint due to any possible factor, the cooling water flow rate is adjusted again to reach the desired set point.

$$Q = m \times C_p \times \Delta T \tag{1}$$

$$Q = U \times A \times \Delta T_{ln} \tag{2}$$

$$A = \pi \times D \times L \tag{3}$$

In this paper, in which the PID controller was selected, firstly the dynamics of the system under the step input was examined and the success of the controller was examined by calculating the controller parameters with the help of the model parameters. It is expected that the system will become stable at a constant temperature value given by the help of a submersible heater at a constant cooling water flow rate. In order to examine the dynamic behavior of the system, positive and negative effects are given to the system that has become stable.

The transfer function of the system which was given depending on the Equation (4) for first order system with time delay was obtained by using the reaction curve and linear regression methods with the help of the recorded in-reactor temperature data. In Euation [4] K_p is the gain of the process, θ is the dead time of process, and τ is the process time constant. P, PI, PID parameters were calculated with Ziegler Nichols and Cohen Coon control parameter calculation methods.

$$G_{(s)} = \frac{K_p\left(e^{-\theta s}\right)}{\tau s + 1} \tag{4}$$

Table 1. Properties of insulation material

Technical Properties	Standard	Unit	Coolflex Plate
Thermal conductivity	EN 12667	W/mK	0.032 (-20°C) 0.034 (0°C) 0.036 (40°C) 0.040 (+60°C)
Water vapor diffusion	EN 12086	-	$\mu \geq 10000$
Fire unit	EN 13501-1	-	B-s3-d0
Temperature strength	EN 14706	℃	Max +110
Soluble chlorine ion level	EN 13468	-	500
pH	EN 13468	-	6-8
Flexibility		-	Perfect
Ozone		-	Good
Strength to chemicals (oil, mineral oil)		-	Good
Mold formation and scent		-	No

The calculated control parameters were tested theoretically in the system simulated with the MATLAB/Simulink program, the results were compared, and their performances were examined. As a result, the model parameters of a batch exothermic neutralization reactor with cooling jacket were found and the controller was designed with the help of the mathematical model.

2.2 Dynamic Behavior of the System

To obtain the model parameters to be used in the design of the process control system, the glass reactor with a reaction volume of 2 L and a jacket volume of 1.5 L is insulated to minimize heat loss. Thermocouples were connected to the inside of the reactor, the cooling water inlet, and the cooling water outlet, and the temperature data were recorded throughout the reaction, the mixer was placed in the reactor to ensure full mixing, the cooling water flow rate was adjusted with the help of a peristaltic pump, and a submersible heater connected to the variac system was placed. All data obtained during the experiment were recorded to the computer with the help of a transmitter and I/O card. To examine the system dynamics, positive and negative load effects are given to the cooling water flow rate of the system. The dynamic regions (S_1-S_3) created by the time variation of the temperature inside the reactor under the positive and negative effects given to the cooling water flow rate are given in Figure 1. S_1 of these dynamic regions; the region where the first steady condition is met for constant cooling water inlet-outlet temperatures and velocities, S_2; the region formed by giving a positive step effect to the cooling water flow rate and S_3 is; represents the region formed by giving a negative step effect to the cooling water flow rate. The step effects on the flow rate, the response of the process, and the variation of the heat supplied to the system with the help of variac over time are given in Figures 2-3, respectively.

3. Results and Discussion

The temperature difference (ΔT_R) inside the reactor was measured and since it was known that there was 1500 mL of water in the reactor, the heat released in the reactor was calculated. Accordingly, since the sum of the heat taken by the water in the reactor and the cooling water in the jacket will be equal to the heat given to the reactor, the heat taken by the cooling water in the jacket should be 92704 J. The mass flow rate ($\dot{m}_{cw}$) of the cooling water was determined with the help of the temperature difference (ΔT) measured in the jacket and the heat values received by the coolant in the jacket.

In jacket-cooled exothermic reactors where water is used as the refrigerant, the heat transfer coefficient of the wall between the outer jacket and the inner tank was calculated with the help of Equation (2). The values used for all calculations and the calculation results are given in Table 2. To determine the most appropriate refrigerant flow rate most accurately, theoretical calculations were made to observe the outlet temperatures of the coolant to be obtained with different flow rates, and the changes in the cooling water outlet temperature (T_{cwo}) at different cooling water flow rates ($\dot{m}_{cw}$) are given in Table 3. For the cooling water in the jacket to take the determined temperature, the liquid should be sent to the jacket at a high flow rate. However, when Table 3 is examined, it is seen that increasing the cooling water flow rate to be sent to the jacket reduces the temperature difference. When working at low flow rates, the cooling water outlet temperature will be high as the water will stay in the system for a longer time, which is a situation that should be avoided as it contains risks.

3.1 Determination of Optimum Cooling Fluid

With the test results given in Table 2 and Table 3, optimum working conditions were determined in cases where the refrigerant is water. However, to examine the use of one or several different refrigerants that can be used as an alternative to water, detailed literature research has been carried out. As a result of the research, ethylene glycol ($(CH_2OH)_2$) is known to be widely used especially in the production of polyester fiber; alternative refrigerant was deemed appropriate. To determine the flow rate of this determined refrigerant, just like for water, repeated experiments were carried out. For safe production, the mass flow rate of the fluid to cool the heat released ($\dot{m}_{ceg}$) was determined as 0.95 g/s using the temperature difference (ΔT_R) given in Table 2 and Equation (1).

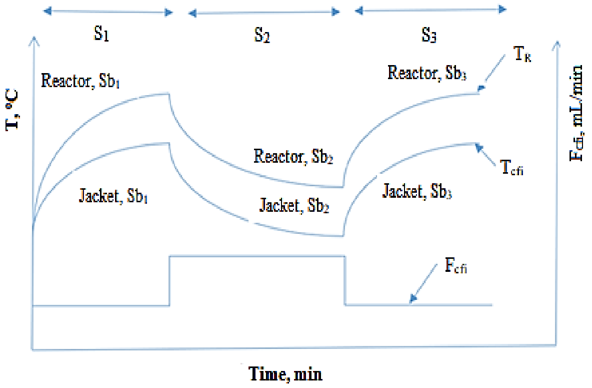


Figure 1. Dynamic zones due to the change in the cooling water flow rate at the reactor temperature; t: time (minute); T: Temperature (ºC); S_1: the region where the first steady condition is met for constant cooling water inlet-outlet temperatures and velocities; S_2: the region formed by giving a positive step effect to the cooling water flow rate; S_3: represents the region formed by giving a negative step effect to the cooling water flow rate; S_{b1}: jacket outlet of cooling water temperature in zone 1; S_{b2}: jacket outlet of cooling water temperature in zone 2; S_{b3}: jacket outlet of cooling water temperature in zone 3; F_{cfi},: cooling fluid flow rate at the inlet (mL/min); T_{cwo}: cooling water outlet temperature (ºC); T_R: the reactor inside temperature (ºC)

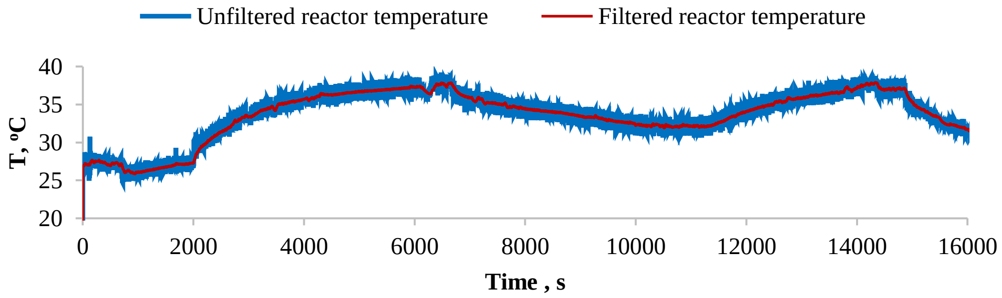


Figure 2. Reactor temperature change over time

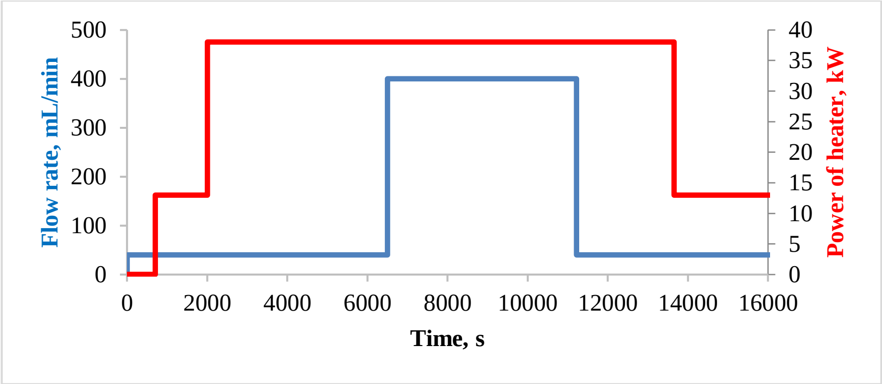


Figure 3. Cooling water flow rate and power of heater over time

Table 2. Results of data used in the wall heat transfer coefficient

T_R		**T_{cwi}**		**ΔT_R**	**ΔT_{cwo}**	**ΔT_L**	**Flow rate**	**Power**	**The heat taken by water in the reactor**	**Heat taken by jacket cooling water**	**A**	**U**
Initial	**Final**	**Initial**	**Final**	℃	℃	℃	**g/s**	**W**	**kJ**	**kJ**	**m^2**	**W/m^2℃**
27.3	37.4	20	30	10.1	13.3	7.2	0.42	39	63.3	92.7	0.06	51.09

Table 3. Investigation of changes in cooling water outlet temperature $\mathbf{T_{cwo}}$ at different cooling water flow rates ($\dot{m}_{cw}$)

$\dot{m}_{cw}$, g/s	0.20	0.30	0.40	0.50	0.54	0.60	0.70	0.80	0.90	1.00	1.10
ΔT, ℃	27.74	18.49	13.87	11.09	10.30	9.25	7.93	6.94	6.16	5.55	5.04
T_{cwo}, ℃	47.74	38.49	33.87	31.09	30.30	29.25	27.93	26.94	26.16	25.55	25.04

Table 4. Investigation of outlet temperatures ($\mathbf{T}_{cego}$) at different cooling ethylene glycol flow rates ($\dot{m}_{ceg}$)

$\dot{m}_{ceg}$, g/s	0.2	0.30	0.40	0.50	0.60	0.70	0.80	0.90	0.95	1.00	1.10
ΔT, ℃	49.11	32.74	24.55	19.64	16.37	14.03	12.27	10.91	10.33	9.82	8.93
T_{cego}, ℃	69.11	52.74	44.55	39.64	36.37	34.03	32.27	30.91	30.33	29.82	28.93

To determine the most suitable refrigerant flow rate, theoretical calculations were made to observe the outlet temperatures of the coolant to be obtained with different flow rates, and the results are given in Table 4.

Since the total heat that the refrigerant in the jacket will take for 4000 seconds is 92704 J, it is seen in Table 4 that the temperature difference decreases with the increase in the flow rate of the ethylene glycol to be sent to the jacket to take this heat. When operating at low flow rates, ethylene glycol will stay in the system longer, just like when water is used as the fluid, so the outlet temperature of the coolant will be high. This should be avoided as it involves risk.

Figure 4, created with the data shown in Table 3 and Table 4, gives an idea about the range of flow rates according to the outlet temperatures of different fluids sent to the jacket. According to this, it is seen that they have approximate exit temperatures because of sending water to the jacket at a flow rate of 0.5-1.1 g/s and ethylene glycol at a flow rate of 0.9-1.1 g/s. At the same time, it is seen in Figure 4 that the water flow rate sent to the jacket is less than the ethylene glycol flow rate to obtain the same outlet temperature in the coolant. For this reason, when water is used to cool this system, it has been seen that approximately ½ less substance can be used than ethylene glycol and considering that it is relatively more economical and not a toxic chemical, it has been decided that the use of water as a refrigerant will be more appropriate. After determining that water is relatively more suitable among the two different refrigerants compared, the optimum flow rate for water should be determined by considering the cost calculation. As a result of the assumption that all 39 W of heat sent to the reactor is taken by the jacket, the exact flow rate of the cooling water was determined as 0.906 g/s. Since the change of the flow rate changes the heat transfer area and thus the overall heat transfer coefficient, the new heat transfer area is calculated as 0.066 m^2. While the height of the jacket to receive this heat was determined as the same as the reactor height, the diameter of the jacket was calculated as 0.1050 cm and the jacket capacity in this direction was calculated as 1.7 L. As a result of all these studies, considering the cost and environmental impact as a refrigerant, using water at an optimum flow rate of 1.271 g/s was determined as the optimum value.

3.2 Dynamic Behavior of The System

Determination of steady state parameters of the system-S1 zone: The reactor heat transfer system design parameters were investigated by repeated experiments under different conditions. First, it is necessary to determine the steady-state parameters of the process. During the experiment, which was started by keeping the cooling water flow rate and heater power constant, the inlet-outlet temperatures of the reactor and the cooling water were measured, and the results were recorded. This process was continued until the second steady state was observed. With the results obtained, the changes in the temperature in the reactor and the cooling water temperature passed through the jacket over time were graphically created. Thus, the steady-state operating parameters of the reactor were determined. To obtain the response of the reactor, the step input was given to the system at a specified time after the reactor was operated in a steady state for a while. The variation of the temperature inside the reactor with time is shown in Figure 1.

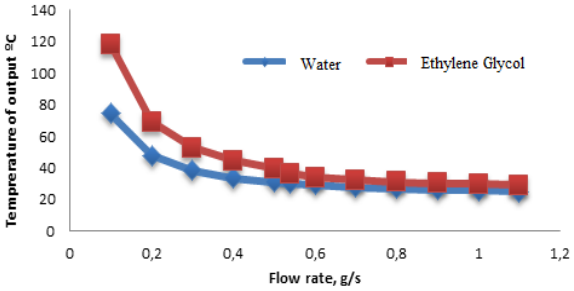


Figure 4. Output temperatures of water and ethylene glycol used as refrigerants at different flow rates

Determination of the variation of reactor temperature with negative/positive step inputs given to the cooling water flow rate (S_2 and S_3 zones): After reaching a steady-state, positive and negative step effects were given to the cooling water flow rate to examine the control efficiency of the reactor. It is necessary to examine the effect of these step effects on the output variable for the open-loop system. In the experiments carried out for this purpose, the temperature of the environment where the reaction conditions were not met was stabilized with the help of variac and positive and negative step inputs were given to the cooling water flow rate while the inlet temperature was constant at room temperature (20 °C). Firstly, the coolant flow rate was 40 mL/min. A positive step input was given to the system when the temperature was steady, and the reactor temperature was approximately 38°C.

As a result of the positive step input, the cooling water flow rate increased approximately 10 times and reached 400 mL/min.

The reactor temperature decreased and reached a steady state again at a temperature of about 33 °C. Similarly, changes in the system were observed by giving a negative step input to the system. Initially, the reactor temperature was steady at 32°C and the cooling water flow rate was 400 mL/min. The cooling water flow rate declined to 40 mL/min after the given negative step input, the temperature inside the reactor dropped to 36 °C.

Determination of process model and control parameters: In this paper, to examine the dynamic behavior of the system, the control parameters were calculated with the help of model parameters by giving positive and negative step effects, and thus the success of the controller was examined. The model parameters formed by the positive and negative effects given to the system by two different methods, namely "Linear Regression Method" and "Reaction Curve Method", were examined.

Determination of Process Model Parameters by Linear Regression Method: The result of the positive step effect given to the cooling water flow rate (S_2 region); the flow rate was increased from 40 mL/min to 400 mL/min. The positive step effect and reaction curve given is given in Figure 5.a. It was observed that the system reached the second steady-state

2400 seconds after the positive step input. Obtained data were used to determine τ, θ, and Kp values as 11.9 min., 1.92 min., 0.016 °C/(mL/min.) respectively.

The data obtained at the end of the positive step inputs were recorded and the system dynamics were examined by giving a negative step effect (S_3 region) to the system. The negative step effect was given by reducing the cooling water flow rate from 400 mL/min to 40 mL/min and the reaction curve was obtained and given in Figure 5.b. It was observed that the system reached the second steady-state 2400 seconds after the given negative step effect. τ, θ, and Kp values were determined as 9.8 min, 5.06 min, and -0.01172 °C/(mL/min), respectively.

Determination of Model Parameters of the System by Reaction Curve Method: The reaction curve of positive step input given to the system in the S_2 region is given in Figure 5.a. By applying the reaction curve method, τ, θ and Kp were found as 2.33 min., 15.1 min. and as -0.0124 °C/(mL/min.), respectively. The reaction curve formed because of the negative step effect given to the system in the S_3 region is given in Figure 5.b. Using this figure, model parameters τ, θ and Kp were determined as 1 min., 17.71 min, and 0.0120 °C/(mL/min).

Determination of PID Control Parameters. P, PI and PID parameters were found with Ziegler-Nichols and Cohen-Coon tuning methods using model parameters obtained by “Linear Regression” and “Reaction Curve” methods. These parameters are given in Table 5. Positive Effect PID parameters found in two different methods were simulated with the computer program which is Matlab-Simulink. Matlab control simulations are given in Figure 6.

Figure 6. a-d were compared, and it was observed that the control parameters found by the Cohen-Coon adjustment method were insufficient to control the system.

The simulation of the PID parameters obtained from the Ziegler Nichols method was found to be the most successful among the results obtained. For this reason, optimum control parameters were determined by the mentioned methods and optimized with the Matlab Simulink PID Tuning program extension. PID control parameters obtained by tuning were used as -154, 0.123, and 0.519, respectively.

Application of Control Action to the System and Experimental PID Control: The reaction was carried out experimentally with the obtained optimum theoretical PID parameters which were given in Table 5. Experimental control parameters that provide the temperature control of the system as a result of the final optimizations with the trial and error method were given in Table 6. The reaction temperature goes up to about 70°C in cases where no control is applied. With the applied PID control, the temperature inside the reactor was kept constant at 33 °C. Experimentally open-loop operation without PID temperature control and operation under PID control was given in Figure 7.

Table 6. Experimental PID Control parameters that provide temperature control of the system

Control Parameters	Values
Kc	-10.8800
τ_1	-10.8308
τ_D	-0.7214

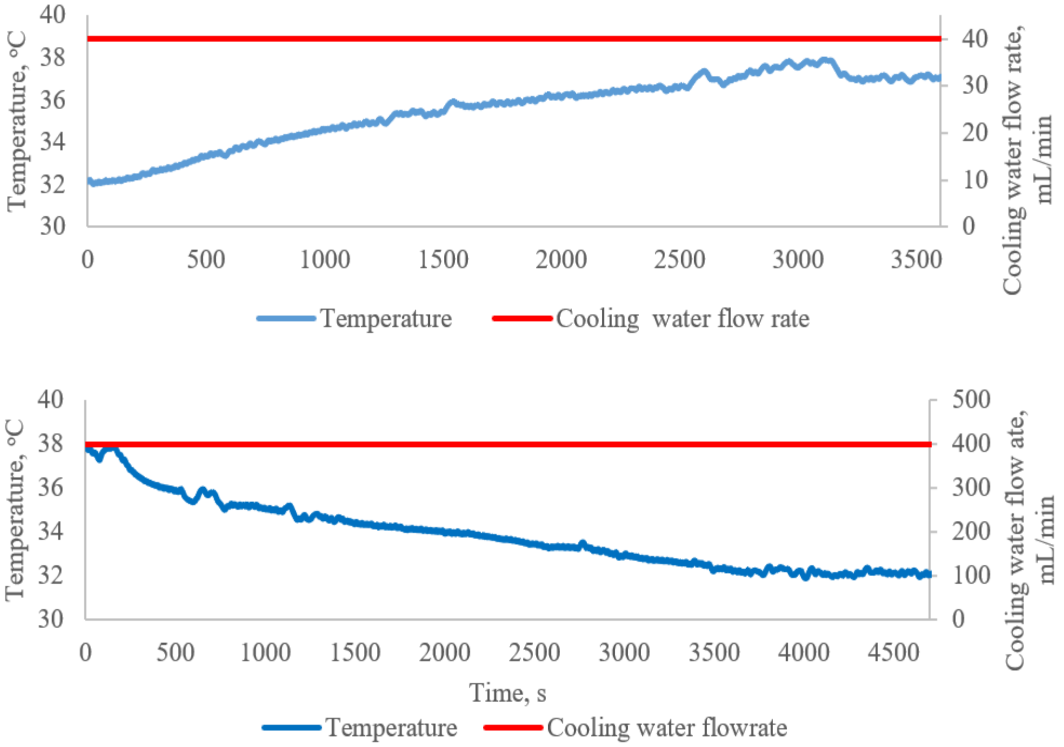


Figure 5. Change in reactor temperature because of negative (a) and positive (b) step effect on cooling water flow rate

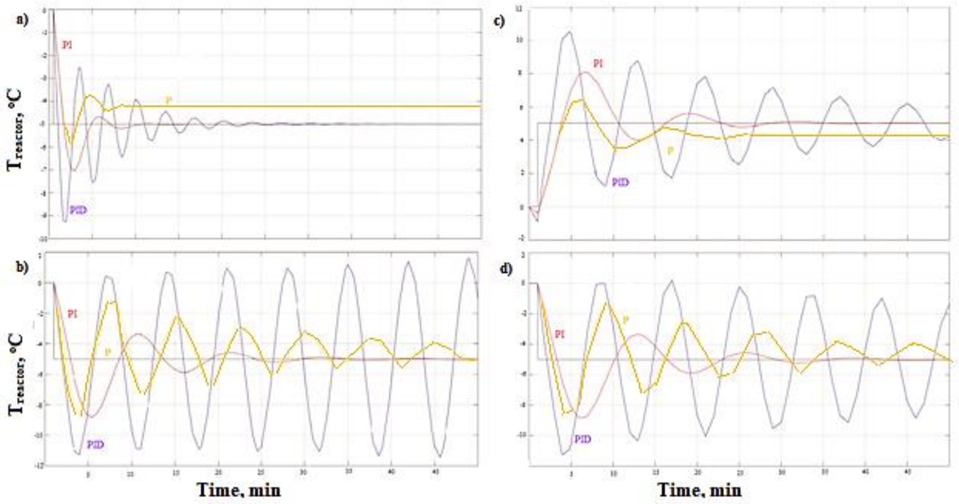
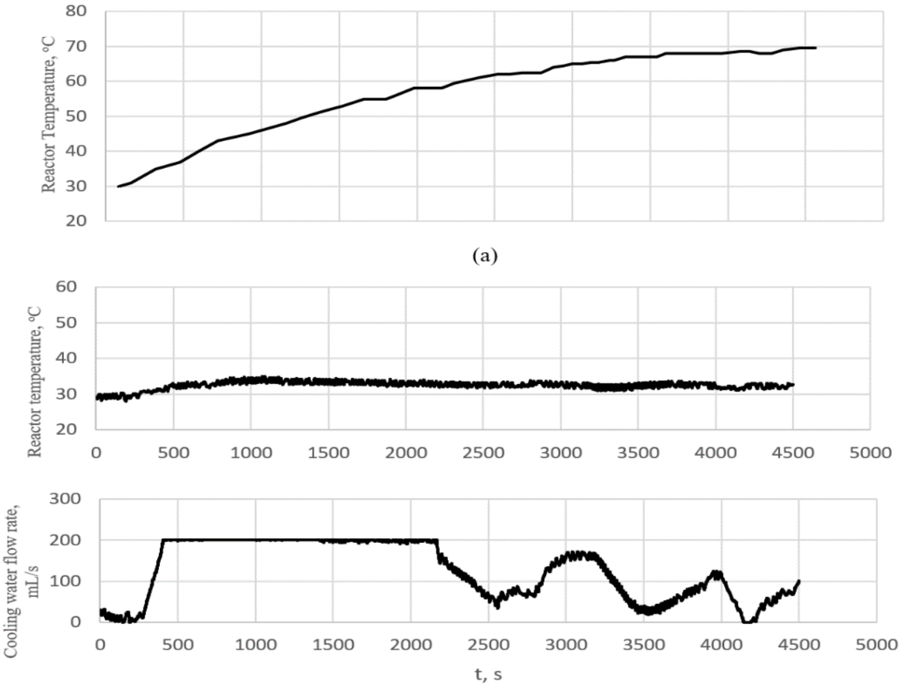


Figure 6. Linear Regression Positive Step Effect (a-b); (a)Simulation of PID parameters obtained by Ziegler Nichols method; (b) Simulation of PID parameters obtained by Cohen-Coon method; (c-d) Reaction curve Positive Step Effect; (c) Simulation of PID parameters obtained by Ziegler Nichols method; (d) Simulation of PID parameters obtained by the Cohen-Coon method

Table 5. Determination of PID control Parameters under positive/negative step inputs by linear regression and reaction curve method

Method	Input	Controller	Cohen-Coon			Ziegler-Nichols		
			K_c	τ_I	τ_D	K_c	τ_I	τ_D
Linear Regression	***Positive***	P	-518			-491		
		PI	-449	4.786		-442	6.39	
		PID	-675	4.43	0.678	-590	3.84	0.96
	Negative	P	-193			-165		
		PI	-155	8.26		-148	16.84	
		PID	-141	10.367	1.682	-198	10.12	2.53
Reaction Curve	***Positive***	P	-549			-522		
		PI	-477	5.872		-470	7.758	
		PID	-717	5.389	0.824	-627	4.66	1.165
	Negative	P	-1503			-1475		
		PI	-1335	4.786		-1328	3.33	
		PID	-1988	4.43	0.359	-1771	2	0.5



(a)

(b)

Figure 7 (a) Variation of reactor temperature with time in the uncontrolled system (b) Variation of cooling water flow rate and reactor temperature over time in PID Control system

4. Conclusions

During the dynamic investigations of the exothermic neutralization reaction, a heater was used to represent the heat source. The result of the dynamic analyzes carried out in an isolated reactor was evaluated theoretically and experimentally, and a controller design was carried out for the reactor temperature. It was observed that the process model parameters obtained from negative and positive step inputs given to the cooling water flow rate were different. Since the real system should be taken into consideration while creating the control parameters, it is predicted that the PID controller will continuously have positive effects on the cooling water flow rate, and the reaction volume heated during the process was calculated on the positive step input. At the end of the simulation, it was observed that the control parameters found by the Cohen-Coon adjustment method were insufficient to control the system. To find the optimum control parameters, the Linear Regression method, the PID parameters found by the positive step effect Ziegler-Nichols method were used as the initial value and optimized with the Matlab Simulink PID Tuning extension. Before the control application, the last parameters obtained for the final optimization were used as the initial value. Then, the PID parameters were optimized by trial and error methods while the process was in a closed loop. As a result of all these processes, the temperature of the reaction, which rapidly heated up to 70 ^{o}C in an uncontrolled cycle, was kept constant at 33 ^{o}C. In future studies, a comparison between a constant flow rate cooler and a PID controller will help optimization studies. Similarly, performance comparison with controller types such as P, PI, GMP can be another subject of study.

Declaration

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The authors 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 developed the methodology. Z.Y. Hitit and P. Aygener wrote the manuscript together. E. Yorgancioglu and B. Akagun performed the experiments. K. Kesenci, S. Ertunç and B. Akay supervised and improved the study.

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