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Determination of Reactor Operational Mode for Concentrated Fungicide Production

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Abstract: An aqueous solution of phosphorous acid (H_3PO_3) and potassium hydroxide (KOH) is an example of a phosphonic acid neutralization reaction where potassium salts are formed. This study aimed to design the reactor and the process, which is a problem encountered in the industry, arising from the need to develop a safe and efficient production process. It was found that the production process carried out under exothermic conditions should be in a temperature-controlled reactor at optimum acid/base initial concentration ratios and feed flow rates as 1/1.5, 22800 mL/min, respectively. Due to the thermal safety and risk accidents at the batch reactor, a tubular flow reactor (TFR) has been designed to increase the controllability and the yield of the products produced in the batch reactor having some advantages as to feeding, sampling, and measuring from different points at different times.

Keywords: Fungicide production; phosphorous acid potassium salts; tubular flow reactor; neutralization reaction; mono and dipotassium phosphite.

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INTRODUCTION

Fungal microorganisms are the structures that cause plant diseases and need to be kept under control. The most effective method to keep these structures under control is the application of fungicides to the plant (1). Phosphite compounds in the HPO_3 structure are widely used in agriculture as fertilizer or fungicide. For an accurate hazard identification and risk assessment, it is crucial to recognize the toxic effects of fungicides and increase the benefits of fungicides while reducing toxic effects (2). Fungicides are used in the treatment of fungi that are formed or likely to occur in plants. Additionally, phosphite salts are pH-neutral chemical compounds and can show healing properties like fungicides in plants.

Although phosphite (PO_3^{3-}) and phosphate (PO_4^{3-}) compounds have similar chemical structures, they have different effects on plants and fungi. Phosphorous acid (H_3PO_3) and its salt (phosphite) contain higher P concentrations (39%) than conventional phosphate-based (H_3PO_4) fertilizer (32% P) (3). It is used as a fungicide in the agricultural sector. It also has fertilizer properties and is used to strengthen the immune system of plants and overcome various infections. In addition, it has various uses such as having a direct antifungal effect on plant pathogens, controlling root-borne diseases, increasing yields, and organic farming. The mono-potassium and di-potassium salts of phosphorous acid, the active ingredient, have direct toxic effects on plant pathogens. They also activate the natural defense mechanisms of plants through disease suppression or eradication. Thus, they provide a double effect mode. However, as in the case of all commercial chemicals and especially environmentally friendly chemicals such as fungicides, a much more improved effect with a small amount is aimed, so improved performance is always required at the same dose. Thus, potassium phosphite will be particularly useful as it will provide potassium as a second precious nutrient besides the other two critical plant nutrients. Moreover, a polyphosphite is expected to provide the known slow-release advantages of polyphosphate.

Currently available commercial methods for the preparation of fertilizer grade potassium phosphite (KH_2PO_3 and K_2HPO_3) include a stirred tank (usually a batch reactor) equipped with a stirrer and cooling jacket, using an aqueous solution of potassium hydroxide as seen in Equations 1 and 2.

$$H_3PO_3 + KOH \rightarrow KH_2PO_3 + H_2O \quad \text{(Eq. 1)}$$

$$KH_2PO_3 + KOH \rightarrow K_2HPO_3 + H_2O \quad \text{(Eq. 2)}$$

Batch reactors are widely used and preferred in industry and their type is frequently preferred in the pharmaceutical industry. The performance of a batch reactor can be determined through mass and energy balances. At the beginning of the reaction, the inputs (substrates) are fed into the reactor and there is no material feeding and/or discharging during the reaction. Temperatures and concentrations are observed over time by the differential equations created with the help of mass and energy balances. In a batch reactor system, it may not be possible in all cases to maximize the product yield, as well as determining the reaction rate, which determines the variation of the concentration over time (4). Since the sudden feeding of the inputs to the reactor changes the temperature and reaction rate abruptly, the batch mode is not often preferred for exothermic neutralization reactions. Fed-batch reactors are commonly used for strongly exothermic reactions (5). As the heat of the reaction can be balanced by the existing cooling of the reactor (6). In fed-batch operating modes, some of the inputs are fed to the reactor before the reaction starts, while the rest is fed to the reactor during the reaction (7). In general, the performance of a batch reactor subjected to an industrial-scale volume change is affected by two important factors, kinetics, and dilution (8).

Despite many years of research and experimental results obtained by chemical engineers in the field of reactor safety, it is known that thermal runaway emergencies still occur in chemical reactors today. In the design and operation of batch and semi-batch reactors, it is necessary to determine the correct and optimum operating conditions and to adapt early warning detection systems to prevent thermal runaways and develop a reliable production process. In the case of homogeneous reactions, a thermal runaway problem may rise faster than for heterogeneous ones due to the lack of interfacial mass transfer resistances (9). A reactor design that provides temperature control is extremely important for such reaction systems. For this reason, tubular (plug) flow reactors (PFR) are recommended. The PFR is characterized by the regularity of the fluid flow in it, without any mixing of the fluid with any other element. In fact, there may be radial mixing of fluid in a PFR; however, there should be no mixing or diffusion along the axial flow. A necessary and sufficient condition for PFR flow is that the residence time in the reactor is the same for all elements of the liquid (10). The inputs enter the reactor and are consumed while flowing axially and the reaction conversion increases along the length of the reactor (11). This also increases the yield of the product.

In fungicides with fertilizer properties, it should be noted that the final product concentration value should be between 6.2 and 6.7 in the production of monopotassium phosphite (KH_2PO_3) and dipotassium phosphite (K_2HPO_3) in order not to harm the soil and the plant as well as showing a nutritive feature (12). Since the pH value of phosphorous acid, the active ingredient in the reaction, is approximately 1.5, it cannot directly contact the plant, in order to prevent it from harming the plant (13). The active substance (H_3PO_3) reacts with potassium hydroxide (KOH), a strong potassium-containing base, to produce KH_2PO_3 and K_2HPO_3 in the form of SL (soluble liquid) with fungicidal properties. The pH values of the potassium phosphite salts obtained are approximately 6.5 (3).

Mono and dipotassium phosphide compounds are generally produced by reaction with phosphonic acid (H_3PO_3) by charging an aqueous solution of potassium hydroxide (KOH) in a batch reactor. Potassium hydroxide (KOH) releases heat during the dissolution process (14), while phosphorous acid (H_3PO_3) absorbs heat. The neutralization reaction of a weak acid and a strong base is an exothermic reaction that still has unsolved problems in the production, storage, and analysis results. Acid-base foaming, boiling, explosion, and splashes are highly likely to occur in the reaction during the production. Even when the rate of the reaction is slowed, the reaction reaches 94 °C. Phosphine gas (monophosphan, PH_3), is formed at temperatures of 65 °C and above and has an odor reminiscent of garlic and has an extremely toxic feature for humans, plants, and the environment (15). Unless the formation of phosphine gas is prevented or removed from the product, this gas causes serious poisoning as well as reducing the production efficiency rapidly (16). This situation poses a great risk for not only human health but also for the environment and economy. Occupational accidents resulting in many injuries in production with neutralization are recorded: causes temporary or permanent bodily damage, acid-base burns and splashes, environmental residues, and economic damage (17). In order to control splashes and explosions, a reduction in all these risks by adjusting pH values and maintaining acid-base balance is aimed (18).

An economical and safe way for reactor operation and optimization is inevitably required for the production of a potassium polyphosphite composition with enhanced efficacy as an agricultural fungicide (19–21). This study is an experimental study carried out to determine the optimum experimental conditions and the most appropriate operating mode of the neutralization reaction to produce monopotassium phosphite and dipotassium phosphite salts. For a concentrated fungicide production stoichiometric ratio, operating mode, feed rate, and reactor type are optimized by examining the effects of different operating types (batch, fed-batch and tubular flow operation) on the production process and product yield.

MATERIALS AND METHODS

The effect of feed order and feed amount on product yield is extremely important in acid-base neutralization reactions. As well as product yield, the feeding sequence ensures a safe production process. For this reason, repeated experiments were carried out in order to determine the optimum input conditions and safe production process of this production reaction, which has significant risks. In the neutralization reaction of a weak acid and strong base, acid was fed to the base. The reaction was observed by recording temperature, pH, and conductivity data at frequent intervals throughout the feeding and product formation.

Determination of the Optimum Input Concentration in the Batch Reactor

Repeated experiments were carried out with a batch system to develop a safe and sustainable production process of potassium salts of phosphorous acid. The acid/base inlet concentration (mol/L, M) ratios of mono and dipotassium phosphite salts produced in the batch system were determined as 1/1, 1/1.5, 1/2, 1/3, and 1/4. The pH, temperature, and conductivity data of the salts produced with different input concentrations were recorded at regular intervals before feeding, during the reaction, and in the final product. The effects of the input concentration ratio on the reaction conditions and product yield were observed.

Determination of the Optimum Input Concentration in the Fed-Batch Reactor

In the fed-batch system, repeated experiments were carried out with an acid/base input concentration (M) ratio of 1/1.5. As a result of an exothermic reaction, toxic gas formation is observed due to high temperatures. Phosphine gas (PH_3), which is likely to cause permanent damage to human health, environment, and equipment safety, is an example of these gases. Therefore, it is necessary to keep the temperature under control in order to design a safe production process. Temperature control was achieved by cooling the reactor in a cold-water bath. The initial conditions for pH, water bath, and reactor temperature data for the pilot-scale experiments are shown in Table 1. The production process, which was controlled by a pilot scale, was repeated at the industrial scale and the initial conditions are given in Table 2.

Table 1: Initial conditions of the titration experiment performed by adding base to acid in an ice bath in a fed-batch system V_{acid}= 50 mL, V_{base}= 100 mL

	pH_{acid}	pH_{base}	T_{acid} °C	T_{base} °C
Experiment 1	1.05	15.4	18.3	25.1
Experiment 2	1.2	15.4	15.2	23.5
Experiment 3	1.1	15.5	16.2	22.5

Table 2: Initial conditions of the titration experiment performed by adding base to acid in an ice bath in a fed-batch system in industrial scale molarities.

Property	Acid	Base
Molarity, mol/L	7.31	10.96
pH	0.01	15.8
T, °C	20.8	23
Base, mL	50	100

Determination of the Optimum Input Concentration in the Plug Flow Reactor

It has been observed that the experiments carried out in the fed-batch reactor as an alternative to the batch reactor do not create a safe and sustainable production process according to the temperature increase. Therefore, once the optimum input concentration ratio of 1/1.5 M (A/B) has been determined, an optimum generation system and reactor design are required as the reaction conversion is completed in a short time and the reaction carries significant risks. For a chemical kinetics study, a system should be designed in which samples can be analyzed at certain time intervals. A 12 m long tubular flow reactor with a 10 mm inner and 12 mm outer radii is designed to realize uninterrupted production. There are 5 bases and 1 acid feeding point on the system. Sampling points were made parallel to the feeding points along the reactor. A jacket with a diameter of 14 cm and a length of 120 cm was used to control the high temperature resulting from the reaction around the designed tubular reactor. There is a cooling water inlet at the bottom of the system and a cooling water outlet at the top. The schematic view of the experimental system for tubular flow reactor is given in Figure 1.

The pilot-scale experiments were carried out repetitively with the TFR, which was designed on an industrial scale. As a result of the pilot-scale experiments, the industrial-scale repeated experiments were carried out, as the condition for the sustainability of the system was ensured. In the first stage, 82 g/L acid and base liquid solutions were stored in 5 L volume acid and base tanks for the pilot-scale experiments. Considering the molecular weights of the inputs, it can be calculated that the prepared solutions have 1/1.5 (A/B) molar concentrations. As a result of the flow rate calibration experiments, the optimum flow rate was determined, and the system was expected to come to a steady state for the first feeding. Acid was fed to the TFR at a flow rate of 370 mL/min. About 12 minutes after the acid was fed, the base was fed from point B1 of the reactor. While the base is fed, the volumetric flow rate of the product, which is formed simultaneously, is recorded and after the feeding, the base flow rate was increased periodically at the 4th, 7th, and 10th minutes. After about 30 minutes, the acid feeding was completed, and the pH data of the product were recorded simultaneously. Industrial-scale experiments were designed, because the experiments completed repeatedly with a pilot scale. 400 g/L acid and base solutions were stored in the 10 L volume acid and base storage tanks. The reactant concentrations were 4.9:7.1 M. In this system, where the optimum input concentration ratio (1/1.5) was provided, repeated experiments were carried out. The base was fed to the designed tubular reactor from point B1 at a flow rate of 150 mL/min and simultaneously from point B2 at a flow rate of 250 mL/min. The acid was fed from point A1 at a flow rate of 400 mL/min. 5 minutes after the system became stable, the base feeding was continued from B1, B2, and B3 points. After 12 minutes, two samples were taken at 15-second intervals, and temperature and pH measurements were made. To control the reaction temperature, cooling water was sent at a flow rate of 17400 mL/min for approximately 18 minutes. After 3 minutes, the volumetric flow rate of the cooling water was set to 22800 mL/min. The base fed from the B3 point at the 22nd minute of production continued to be fed from the B4 point until the almost 31st minute. The base feeding was completed, and the product pH value was recorded.

Phosphorus Analysis

Phosphorus analyses were carried out repeatedly to analyze the chemical kinetics and determine the product yield. A calibration graph was created at a wavelength of 460 nm with the reaction input phosphorous acid standard solution, and then the phosphorous acid concentration of the product was determined through the spectrometric method. In the analysis method, nitric acid (29% HNO_3) solution, 0.25% ammonium vanadate (NH_4VO_3) solution, and 5% ammonium molybdate solution ($(NH_4)_6Mo_7O_{24}.H_2O$) chemicals were used. UV-Vis measurements were obtained with a Shimadzu - UV160 and manual Spectrometer using 1 cm plastic cuvettes (22).

RESULTS AND DISCUSSION

Determination of optimum input concentration ratio and reactant feed order

It is known that the pH values of phosphorous acid, potassium hydroxide, and neutralized phosphorous acid (NPA) water solution at different concentrations are ranging from 1.85 to 2.34, 11.98 to 12.88, and 6.26 to 6.96, respectively (12). The repeated experiments on a pilot scale showed that the reaction proceeded efficiently when the reactants were at an equal mass flow rate. It is seen in Figure 2 that the temperature increase reached uncontrollable values as a result of the gradual increase in the acid and base concentrations. It is essential to prevent the formation of toxic phosphine gas and therefore to control the temperature for both product yield and safe process operation. In this case, regardless of the type of production, the optimum input concentration ratio was determined as 1/1.5 (A/ B). When this ratio was maintained, it is seen in Figure 3 that the pH value of the product was recorded in the range of 6.5-7. This value is the common feature of pesticides that have a nutritive effect when applied to the plants. The effects of feed order to the reactor on temperature and pH on diluted and concentrated systems are given in Figure 4. The test results with the pilot scale obtained proved that feeding the reactor at a low flow rate by keeping the input concentration ratio constant would be effective both in terms of occupational health and safety and product yield. When the input conditions for the effective product were provided, it was observed that the acid-base feeding sequence to the reactor did not have a significant effect on the product and the production process.

Determination of the Type of Reactor and Production

The experiments carried out on a pilot scale and then repeated at an industrial scale, showing that the optimum input concentration ratio should be 1/1.5 for an effective and productive product. After determining the input concentration ratio, the most suitable, reliable, and sustainable reactor type should be determined by considering the production conditions, production time, and product quality of the product. Since the production process of potassium phosphite salts formed by the reaction of a strong base and a weak acid caused acid-base splashes on the pilot scale, it can cause reactor explosion in industrial production. For this reason, errors that might arise were kept under control by conducting repeated experiments with a pilot-scale before designing a safe production process at an industrial scale.

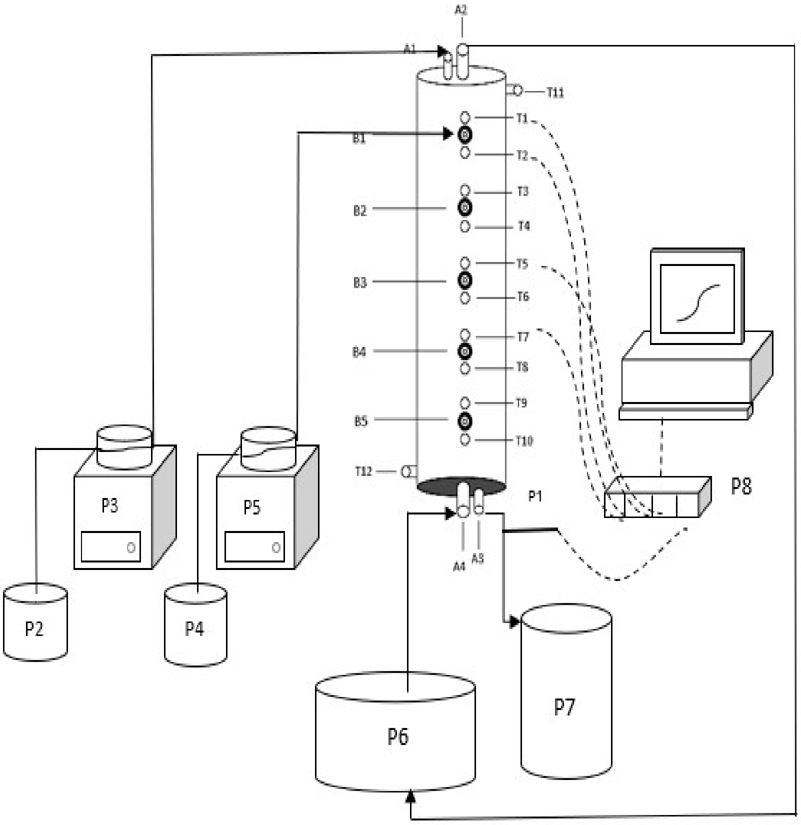


Figure 1: Experimental system for tubular flow reactor (A1: acid feeding, A2: cooling water outlet, A3: product outlet, A4: cooling water inlet, B1-B5: base feeding inlets, N1-N5: sampling points, P1:pH meter, P2: Acid feeding tank, P3: Acid pump, P4: Base feeding tank, P5: base pump, P6:cooling water circulator, P7: product tank, P8: online data module (I/O); T1-T10: thermocouples; T11-12: thermometer.

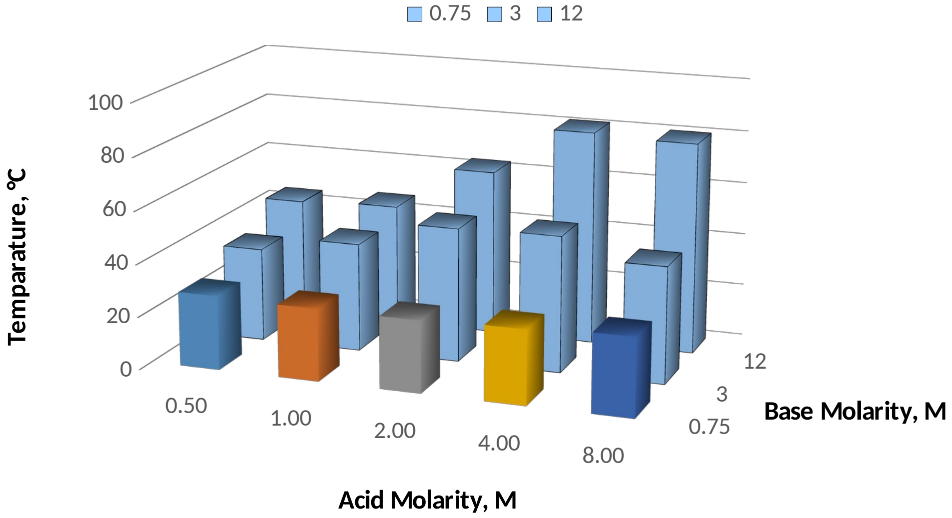


Figure 2: Temperature distribution graph as a result of the reactions with different initial concentrations of acid/base with a constant ratio of 1/1.5.

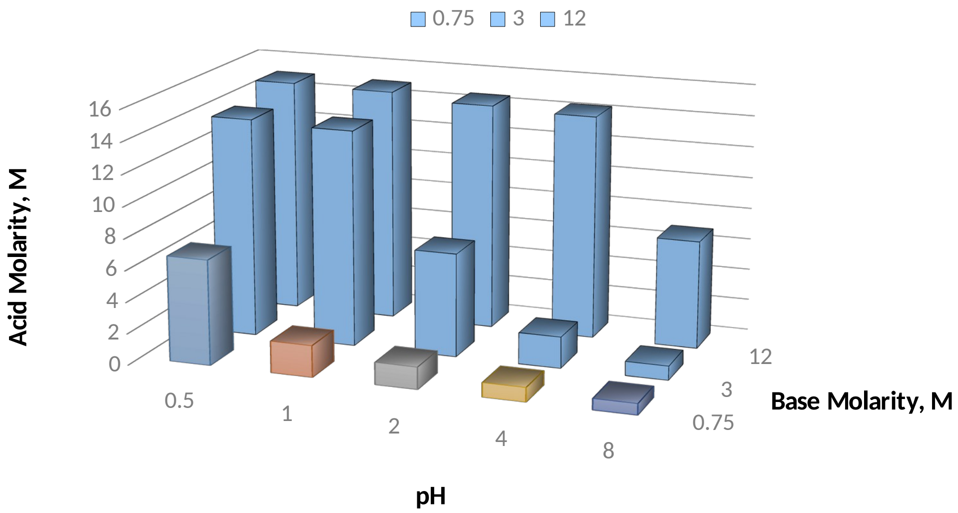


Figure 3: The final pH values of the reaction reached at the initial concentrations of Acid and Base (1/1.5 M).

The exothermic nature of the production reaction poses significant risks. As the reactor temperature rises during the reaction, it is expected that phosphine gas, which is poisonous for humans, plants, and the environment, will be formed (15). In cases where phosphine gas is not disposed of, it causes poisoning as well as reducing product yield (16). Toxic and/or undesirable by-products may occur as well as causing acid-base splashes, explosions, and burns. The repeated experiments in the batch reactor caused sudden temperature increases as seen in Figure 5. Because the reaction was very fast and a strong base and a weak acid were used, it was observed that jumps are more likely to occur in batch operation. It was also known that garlic-smelling phosphine gas was formed during the reaction. The sudden temperature that arises in the reactor can be controlled by the design of a jacketed reactor.

Since the reaction that took place was a neutralization reaction, an 'S' curve was expected when the pH values obtained over time were plotted. When fed-batch and batch processes were compared, this result could not be reached because the reaction was very fast in batch processes. As a result of the repeated productions by keeping the 1:1.5 acid: base input concentration constant, the pH values recorded in the batch reactor during production are given in Figure 6.a, while the pH values recorded in the fed-batch reactor are shown in Figure 6.b.

According to Figures 2, 3, and 6, potassium hydroxide solution was added to phosphorous acid slowly with the help of a pump in order to increase the product selectivity and to control the temperature and operate in isotherm in fed-batch operation.

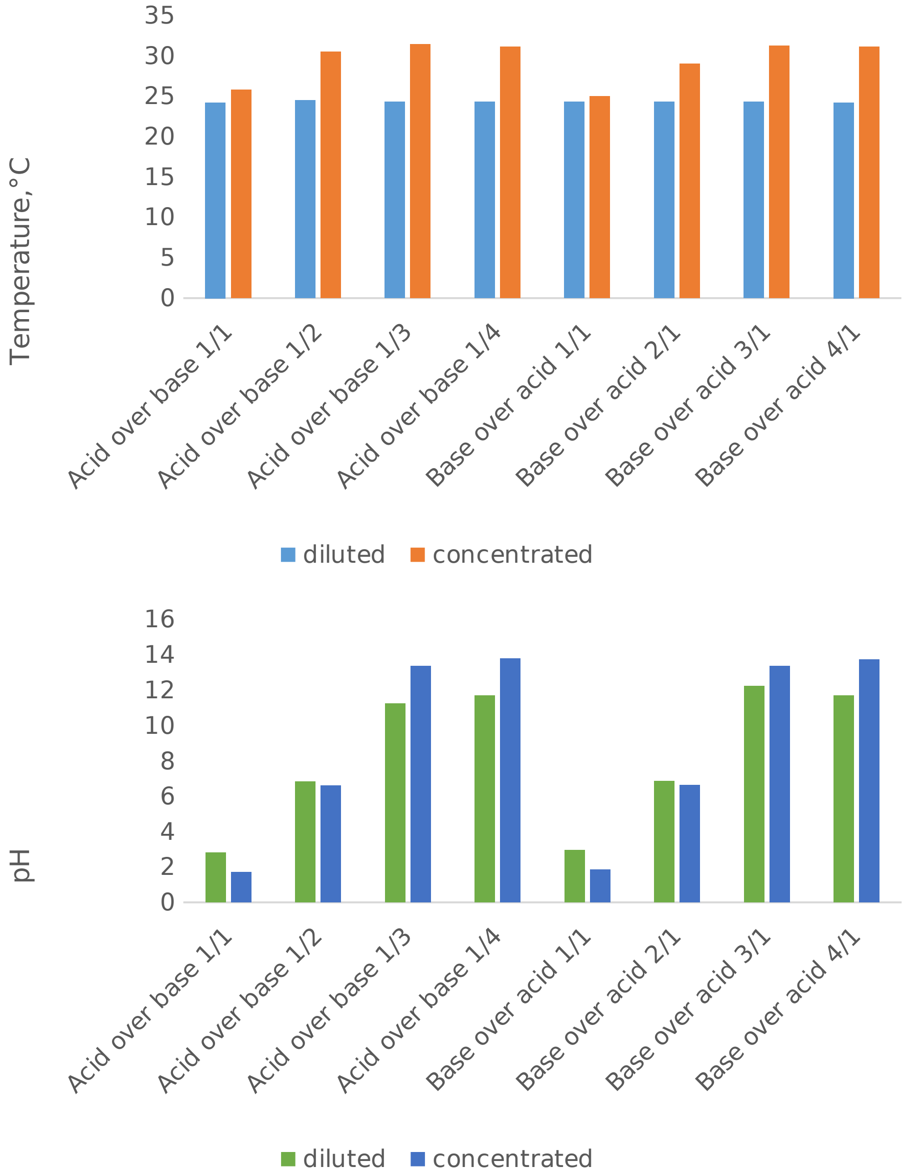


Figure 4: (Top) Base over acid and acid over base comparison with temperature on diluted and concentrated system. **(Bottom)** Base over acid and acid over base comparison with pH on diluted and concetrated system.

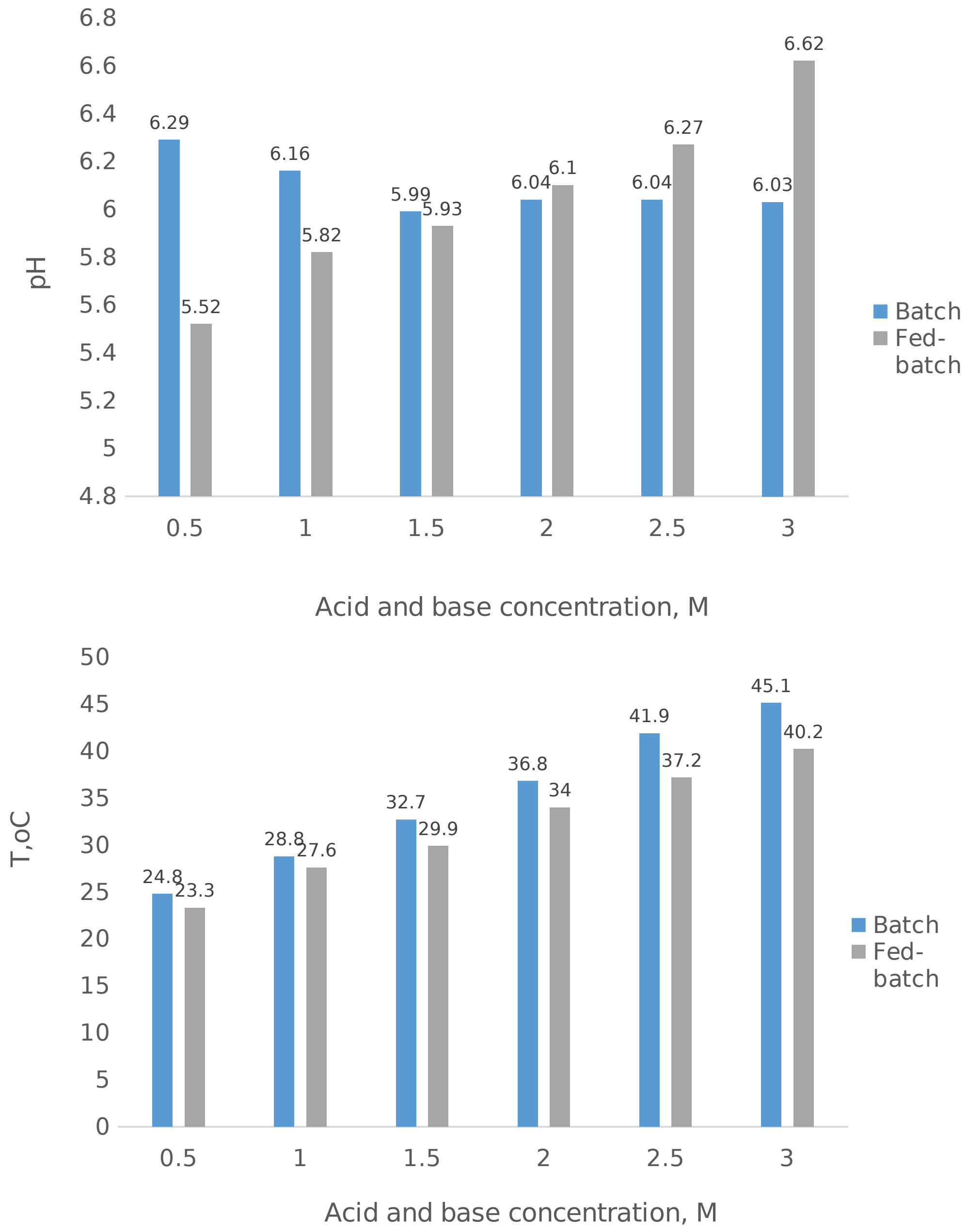
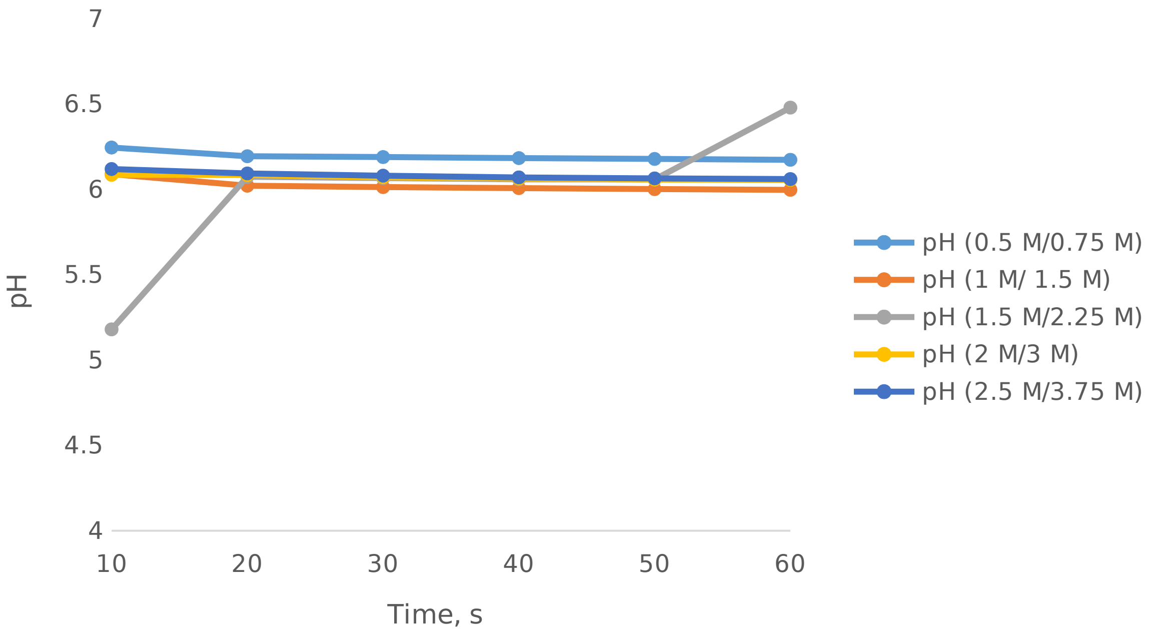


Figure 5: (Top) Comparison of pH values of the product formed in fed-batch and batch operations, **(Bottom)** Comparison of the temperature of the product obtained in fed-batch and batch operations.



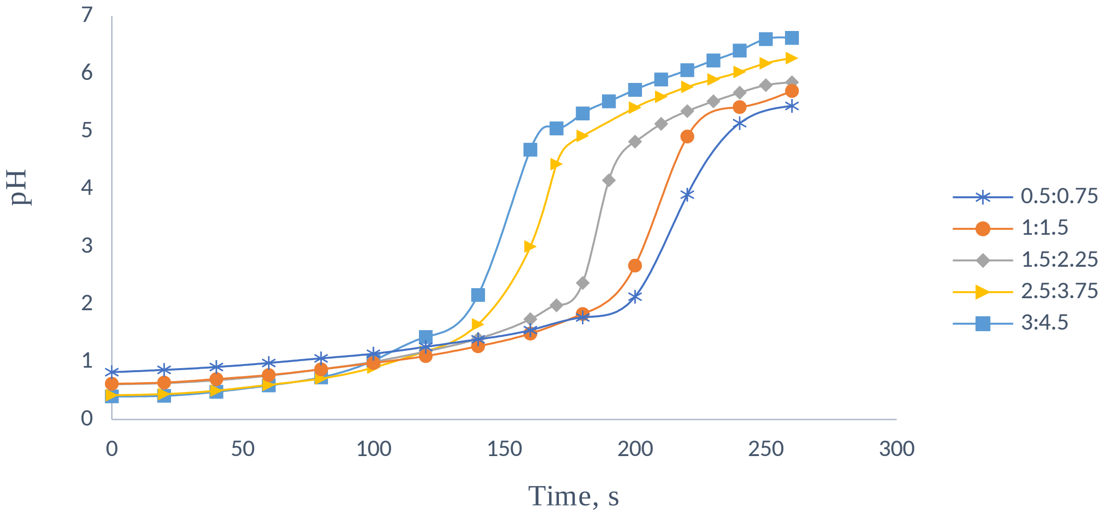


Figure 6: (Top) Graph of pH change over time at 1/1.5 molarity input ratios in a batch reactor **(Bottom)** Graph of pH change over time at 1/1.5 molarity input ratios in a fed-batch reactor.

Tubular Flow Reactor Design (TFR)

The production process of the potassium salts of phosphorous acid produced in the batch reactor causes permanent damage to the workplace, employees, and the environment on an industrial scale. In addition, the low yield of the product was analyzed. A fed-batch and jacketed reactor design were found to be more controllable, but it was still difficult to control the temperature. It was necessary to design a system that can be fed continuously for high product yield and a safe and sustainable production process. It is advantageous to be able to take samples at certain time intervals from the reactor, which are fed continuously and slowly from different points, in order to determine the chemical kinetic model.

In this designed tubular reactor system, a recorder was designed to feed the reagents through the reactor at certain intervals and to measure the values such as pH and temperature in line with these points in a controlled manner. In this way, the variation of the values desired to be obtained throughout the reaction with time was easily measured. Even when the desired values such as pH and temperature couldn’t be reached, it became easier to intervene in the system.

The experimental data were measured with the analog signal modules throughout the production carried out in the designed TFR. The temperature changes with time recorded at the point T2 given in

Figure 7, the change in the empty jacket temperature with time in Figure 7.b, and the pH change at the reactor outlet over time is given in Figure 7.c.

As seen in Figure 7.a, an instantaneous temperature change occurred when the base was sent to the system; that is, the reaction started quickly, and the temperature increased quickly. As seen in Figure 7.b, no temperature change was observed since no cooling water was sent to the jacket.

When Figure 7.c is examined, it is observed that the pH decreased considerably with the acid being sent to the system at the 380th second. With the addition of the base at 1010 seconds, the pH increased over time, and neutralization was observed. It was observed that the base flow rate should be increased when the pH could not reach the desired range. Thus, it was observed that the flow rate was important and therefore the base flow rate was increased until the desired product pH was reached. When the acid stock finished, the pH value was measured as 5.9. With the end of the acid that was being sent to the system, the pH quickly increased to 13 because only the base remained in the environment, as the base feeding continued. The experimental data were measured with analog signal modules throughout the production carried out in the designed TFR. The temperature changes with time recorded at the point T2 is given in Figure 7, the change in the empty jacket temperature with time in Figure 7.b, and the pH change at the reactor outlet over time is given in Figure 7.c.

In the production reaction, the system was expected to reach steady state after about 12 minutes, the pH value of the samples was measured as 6.5 and the temperature values were measured as approximately 55 °C. At the 30th minute of production, sample pH was recorded as 6.6, and sample temperature was recorded as 26 °C. Approximately 1 minute after this measurement, the base feed was completed, and the pH of the final product was measured as 6.7. In Figure 8, the change in the temperature of the samples taken at the point T2 over time (Figure 8.a), the temperature changes in the jacket over time (Figure 8.b), and the pH change of the product over time (Figure 8.c) are given. The pH is acidic because there is no base feeding until the 300th second. As the reaction started to take place with the feeding, the pH value started to increase. During the reaction, acid feed and base feed from two different points continued. When the reaction was completed, it was observed that the pH value of the desired product was reached. The TFR design was proven to be successfully implemented.

Results of Phosphorus Acid Analysis

The stock solutions prepared at different concentrations in the range of 0-500 ppm were prepared for analysis with Shimadzu - UV160 and a manual spectrophotometer to form the calibration line of the amount of phosphorous acid contained in the product. It was observed that the maximum pick values of the spectra were read at a wavelength of 350 nm. Separate absorbance values of the solution at each concentration were recorded for the wavelength of 460 nm and the wavelength of 350 nm given in the literature (23). In Figure 9.a, the calibration graph created at a wavelength of 350 nm, and in Figure 9. b, the calibration graph created at a wavelength of 460 nm is given.

The phosphorus acid concentrations of the products obtained in different reactors were analyzed and the percentages of phosphorus content in the products were determined. The conclusive analysis results with the theoretical values with an acceptable margin of error prove the applicability of the analysis method and the high product yield. When the base is fed to the reactor, it is expected that the volume will double and thus the phosphorous acid concentration will be halved. The product analysis results are consistent with the expected theoretical values.

However, in order for the obtained data to be used in determining the chemical kinetic model, it was expected that the amount of 400 g/L H_3PO_3 in the medium was expected to be completely depleted in time, and when the reaction was completed, the 200 g/L H_3PO_3 concentration was calculated in the medium, and it was concluded that the analysis performed not only measured the phosphorus (P%) in the H_3PO_3 but also the total phosphorus amount in the medium. For this reason, it was concluded that the results of the phosphorus analysis could not be used in kinetic analysis calculations.

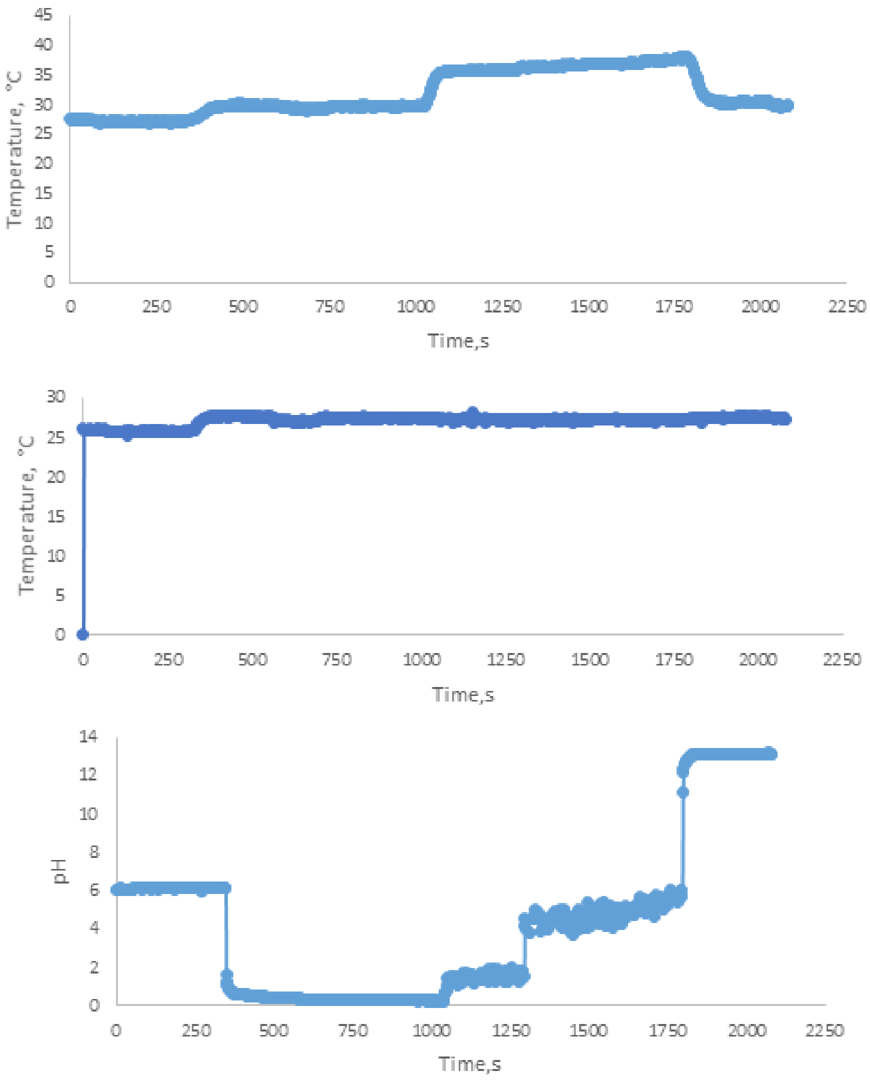


Figure 7: (a) Change of temperature over time at point T2. **(b)** Change of jacket temperature over time (ambient temperature). **(c)** pH change in reactor output over time.

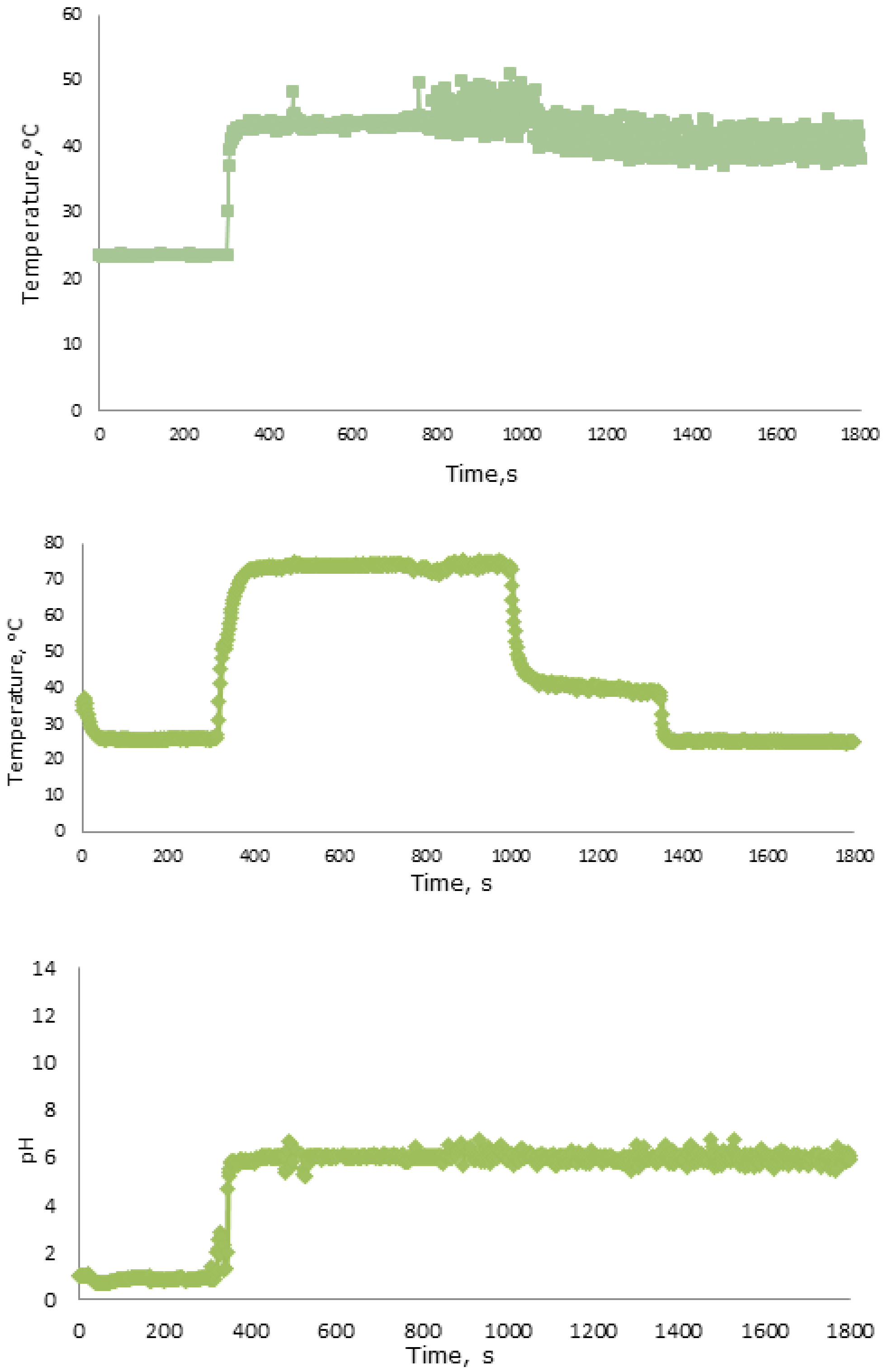
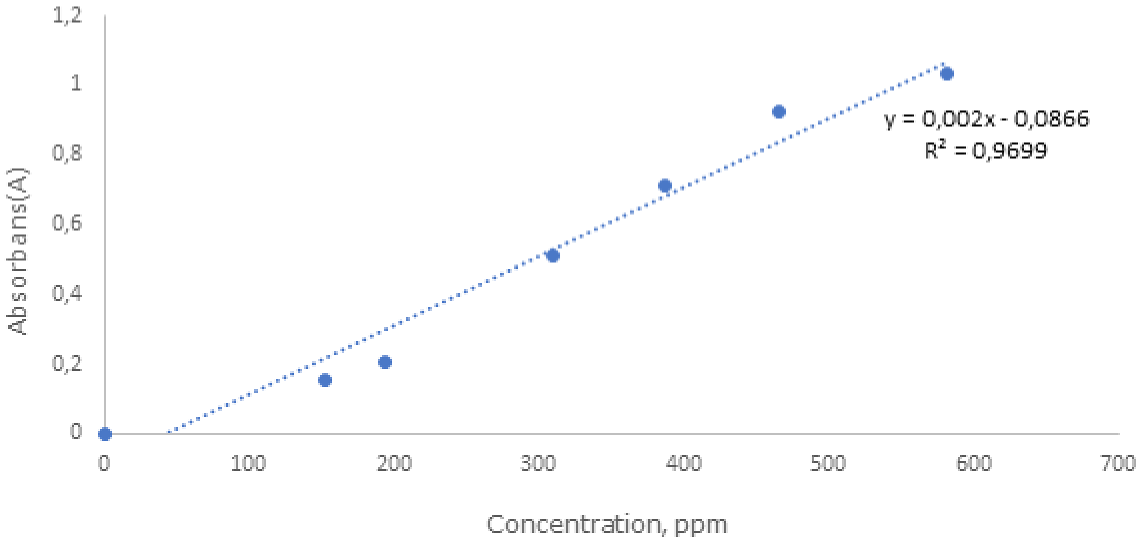


Figure 8: (Top) Change of temperature at point T2 over time **(Middle)** Change of temperature at point T6 over time **(Bottom)** Change in the pH of the product formed at the reactor output over time.



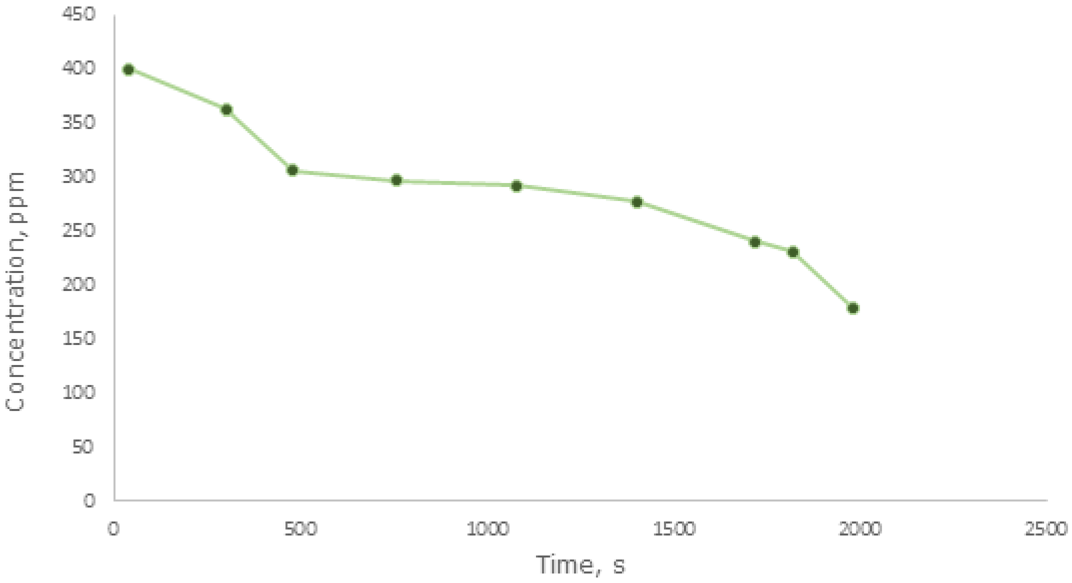


Figure 9: (Top) Calibration graph created at 350 nm wavelength **(Bottom)** Graph of concentration versus time generated at 460 nm wavelength.

In the last decade, usage of continued tubular flow reactors have been seen as promising strategies for industrial production of chemicals. Investigations have been conducted on the various chemical production processes in commercial quantities. Awogbemi et al. studied adaptation of tubular reactor in the production process of biodiesel and found out that using transesterification technique in tubular reactor technologies provide eco-friendly, low-cost, and large-scale production of biodiesel, at a high product yield while generating high-quality biodiesel (24). Burns et al. pointed out the popular usage of tubular flow reactor in the organic chemistry and polymer synthesis and also mentioned advantages of flow techniques including intensified heat transfer, operation under steady state conditions and simplified scale-up with minimal process development (25). Gu et al. investigated heat transfer and storage performance of steam methane reforming in tubular reactor and achieved 34.8% improvement in thermochemical energy storage efficiency by using insulation and coating (26). Körbahti et al. examined tubular flow reactor for the first time in the electrochemical treatment of water-based paint wastewater system (27). In this study, application of the continuous tubular reactor in phosphorous acid potassium salts production was realized for the first time in literature to ensure concentrated fungicide production.

CONCLUSION

The results of the experiments showed that the fed-batch reactor was more controllable and sustainable than the batch reactor. However, since it would be more economical and feasible to provide continuous production on an industrial scale, a tubular flow reactor was designed. The repeated test results conducted to determine the optimum feed flow rates have shown that the safest, most efficient, and sustainable production process is provided by feeding into a tubular flow continuous reactor at a slow flow rate. Considering the temperature and pH data recorded at regular intervals during production, the pH value reached by the final product, and the acid: base feeding sequence did not significantly change the phosphorus concentration of the product because of the analysis. It is necessary to feed slowly the strong base to the reactor from different points of the reactor after feeding the weak acid to the reactor. It is recommended that because it is possible to eliminate possible risks. In future studies, it is suggested that the system will be controlled with advanced control systems to adjust the desired concentration of tubular flow reactors. The temperature inside the reactor was prevented from rising above the desired value with the jacket around the designed tubular reactor and therefore phosphine gas production was prevented. Although it is difficult to control the temperature in the tubular flow reactor, the desired product specifications were obtained are an indication that the reaction can take place in the tubular flow reactor continuously.

CONFLICT OF INTEREST

There is no conflict of interest.

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