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Research Article

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Self-Tuning PID (STPID) Control Of Ph in Recovering Process of Magnesium Hydroxide (MG(OH)$_2$) by Coagulation from Salt

Tuz Çözeltilerinden Çöktürmeyle Magnezyum Hidroksitin (MG(OH)$_2$) Geri Kazanılmasında Kendinden Ayarlamalı Oransal İntegral Türevsel Hareket (Stpid) ile PH Kontrolü

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ABSTRACT

The salt obtained from the salt sources are not desirable purity level and contains some impurities. In the scope of this study, coagulation of magnesium hydroxide (Mg(OH)$_2$) in pH control has been carried out by adding sodium hydroxide (NaOH) to magnesium chloride (MgCl$_2$) and by this way recovery process has been achieved by Self Tuning PID Control. To provide the best coagulation pH 11 has been obtained as optimum pH value in consideration of the performed calculations.

Key Words

Coagulation, Brine, Self-Tuning PID Control, pH control.

ÖZ

Mevcut tuz kaynaklarından elde edilen tuz, istenilen safiyette olmayıp, istenmeyen birtakım safsızlıkları içermektedir. Bu çalışma kapsamında, magnezyum klorüre (MgCl$_2$), sodyum hidroksit (NaOH) ilave edilerek magnezyum hidroksitin (Mg(OH)$_2$) pH kontrollü olarak çöktürülmesi gerçekleştirilmiş, geri kazanımının Kendinden Ayarlamalı PID Kontrol (Self Tunning PID Control) ile sağlanması amaçlanmıştır. Yapılan hesaplamalar ışığında en iyi çöktürmenin sağlandığı optimum pH değerinin 11 olduğu elde edilmiştir.

Anahtar Kelimeler

Koagülasyon, Tuz Çözeltisi, Kendinden Ayarlamalı PID Kontrol, pH kontrolü.

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INTRODUCTION

A large amount of salt production in Turkey is carried out from the crude salt obtained from Salt Lake (Tuz Gölü). In addition to crude salt NaCl, it also contains water-soluble and insoluble impurities.

In the chemical industry, crude salt dissolves in water or brine with impurities [1]. Therefore, the brine should be purified before being processed. The impurities from the crude salt dissolved in the brine are precipitated with chemicals and removed by various processes. The salt produced in the first stage of salt production is called raw salt.

The most important step in the production of salt is to bring the solution obtained from existing salt sources or coarse salt to supersaturation by evaporation, then subject it to purification by crystallizing [2].

The process applied in the purification of solid or solution salts obtained from existing salt deposits is the crystallization of salt solutions brought to saturation at high temperature. The price of the crystal salt obtained from salt mines is very low due to the impurities it contains and there is no chance to be used in many industries without refining. 94-96% of the crude salt obtained from salt sources consists of NaCl.

The remaining 4-6% is $MgCl_2$, $MgSO_4$, $CaCl_2$ and traces of other substances. All of these substances are called soluble or insoluble impurities. Since these impurities remain in the system continuously due to the production technology, time-dependent accumulation occurs with the crude salt added in order to increase the saturation. Although it is not possible to obtain these salts as commercial products, which have low concentrations in the environment, these salts may prevent a feasible production in terms of energy efficiency and production efficiency over time [3].

With chemical purification, it is possible to remove these salts without taking them into the production line, but if it is done continuously, it negatively affects the competitiveness of the companies economically. For this reason, chemical purification is recommended on days when these salts reach certain saturation degrees.

The export of the raw mines and minerals does not create much added value for our country. Our mines need to be purified or converted into products with high added value by making their derivatives. For this purpose, in this study, it is aimed to produce cheap and high-quality salt by providing maximum savings from labor and energy in intensive salt production by developing a serious process control instead of the old technologies used today.

MATERIALS and METHODS

Purification of Brine

Soluble impurities ($MgCl_2$, $MgSO_4$, $CaCl_2$, $CaSO_4$ etc.) in the crude salt accumulate in the system over time and may adversely affect the energy/production efficiency [4]. Calcium accumulating in the system causes crust formation in the circulation line, especially in the heat exchangers, and prevents the instantaneous efficiency at the starting point. Some of these salts, which have a negative impact on plant life and costs, pass into the final product as impurities and may cause problems depending on the purpose of use [5].

For example, salts other than NaCl in the textile industry cause an increase in the total hardness and NaCl containing these salts is not preferred in the textile industry. Impurities that reach the level of negatively affecting energy and production efficiency should be removed by chemical purification processes.

Precipitation of $Mg(OH)_2$

$Mg(OH)_2$, which has a very low solubility in water, is obtained by adding a base to the magnesium ions in the brine [6]. Magnesium is removed by precipitation of $Mg(OH)_2$. In order to calculate the optimum pH value required for effective precipitation to occur, the solubility multiplication of the $Mg(OH)_2$ compound is used (Table 1).

The optimum pH value of approximately 10.52 for realizing the precipitation process was determined using the equations below [7].

$$MgCl_2 + 2NaOH \rightarrow Mg(OH)_2 + 2NaCl \quad (1)$$
$$Mg(OH)_{2\,(k)} \rightarrow Mg^{2+} + 2OH^- \quad (2)$$

Self-Tuning PID and PID Control

By feeding brine (salt solution) solution to the reactor, the mixing speed is adjusted to the optimum value. Self-Tuning PID Control and PID control algorithm are operated depending on system variables [8]. Self Tuning PID and PID feedback control system’s block diagram-

Table 1. Solubility multiplication constants of some compounds of alkaline earth/soil cation Mg^{2+}.

Anion	$K_{çç}$ values for Mg^{2+} compounds.
CO_3^{-2}	3.5×10^{-8}
OH^{-2}	1.8×10^{-11}
$C_2O_4^{-2}$	8.6×10^{-5}
PO_4^{-3}	1.0×10^{-25}

mes of the salt process are given in Figure 1 and Figure 2. In this study, the chemical precipitator fed to the system simultaneously gives the disrupting factor to the system; the control algorithm sends the calculated base amount to the system as an adjustable variable in order to reach the appropriate pH value for precipitation. After reaching the desired pH, it is operated for a while and the reaction is stopped [9].

After stopping the reaction, the polyelectrolyte is added by slowing the mixer speed. Thanks to the added polyelectrolyte, precipitation becomes more observable. After agglomeration is achieved, a certain amount of sample is taken from the reactor and the precipitation amount (%) is calculated by analytical methods. In control experiments, 20% HCl and 10% NaOH solutions were used. While 20% HCl solution is sent to the system at a constant flow rate of 24 ml/min, 10% NaOH solution is fed into the system simultaneously as an adjustable variable.

In the experiments of brine samples with a pH value of 8.51 at the beginning, using self-adjusting PID and PID control system, three values were considered as pH set value. These are 7, 9 and 11. The experiments were carried out at room temperature and the same brine samples were used for each experiment.

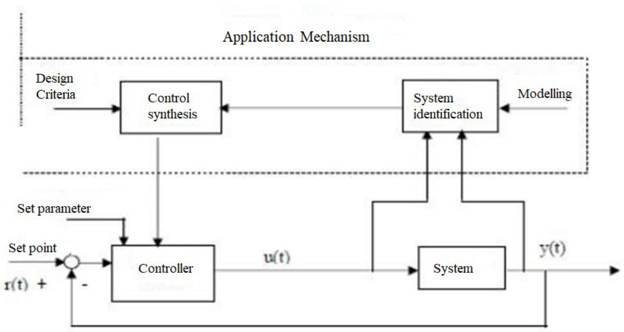


Figure 1. Self Tuning PID feedback control system's block diagramme.

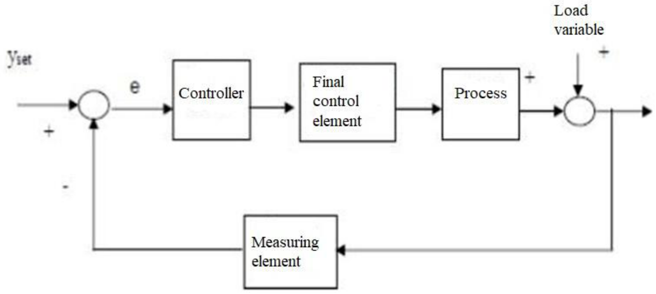
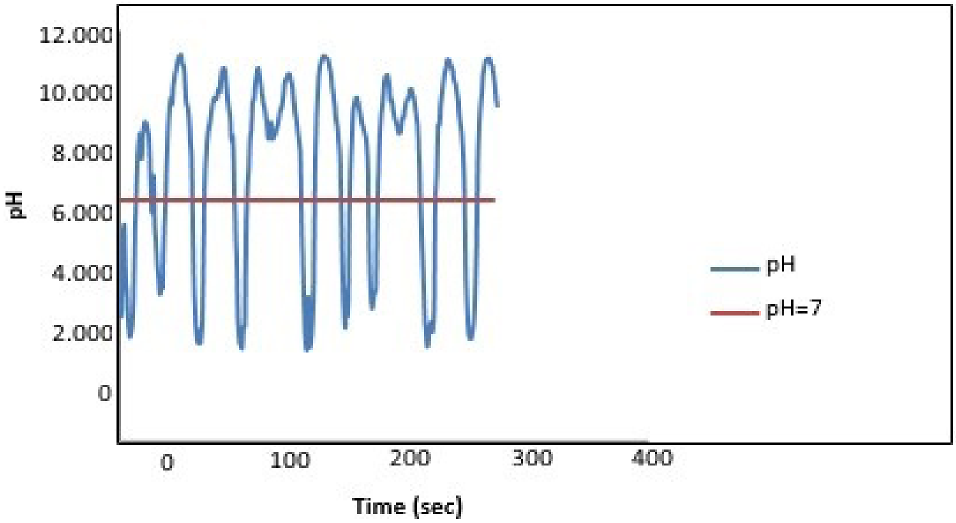


Figure 2. PID feedback control system's block diagramme.

RESULTS and DISCUSSIONS

pH control of a semi-batch reactor was carried out with self-adjusting PID and PID control systems [10]. The system was stabilized at pH 7, 9 and 11, respectively, and negative and positive step effects were given to the HCl flow rate. In order for the control system to work effectively, the optimum value of the t_1 setting parameter was selected as 0.5, while the coefficients of the ARMAX model were used in accordance with the system [11]. In both Self Adjusting PID and PID control, for pH 7, 9 and 11 respectively, while sending 20% HCl solution at a constant flow rate of 24 ml/min, 10% NaOH solution was used as adjustable variable.

In the Self Adjusting PID control, the pH=7 setpoint was retained, although it was noisy (Figure 3). As it is observed, it is difficult to control at pH=7. In the PID control, an offset was observed at pH=7 and it was quite noisy, the PID control was insufficient (Figure 4).



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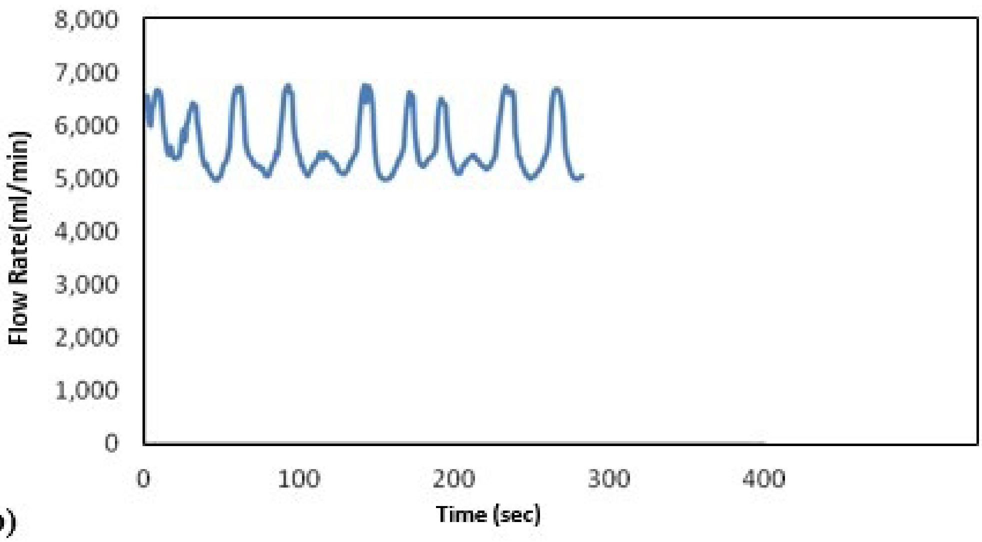

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Figure 3. Self-adjusting PID control results with brine sample for pH=7. a) Time variation with pH. b) Variation in time of the adjustable variable.

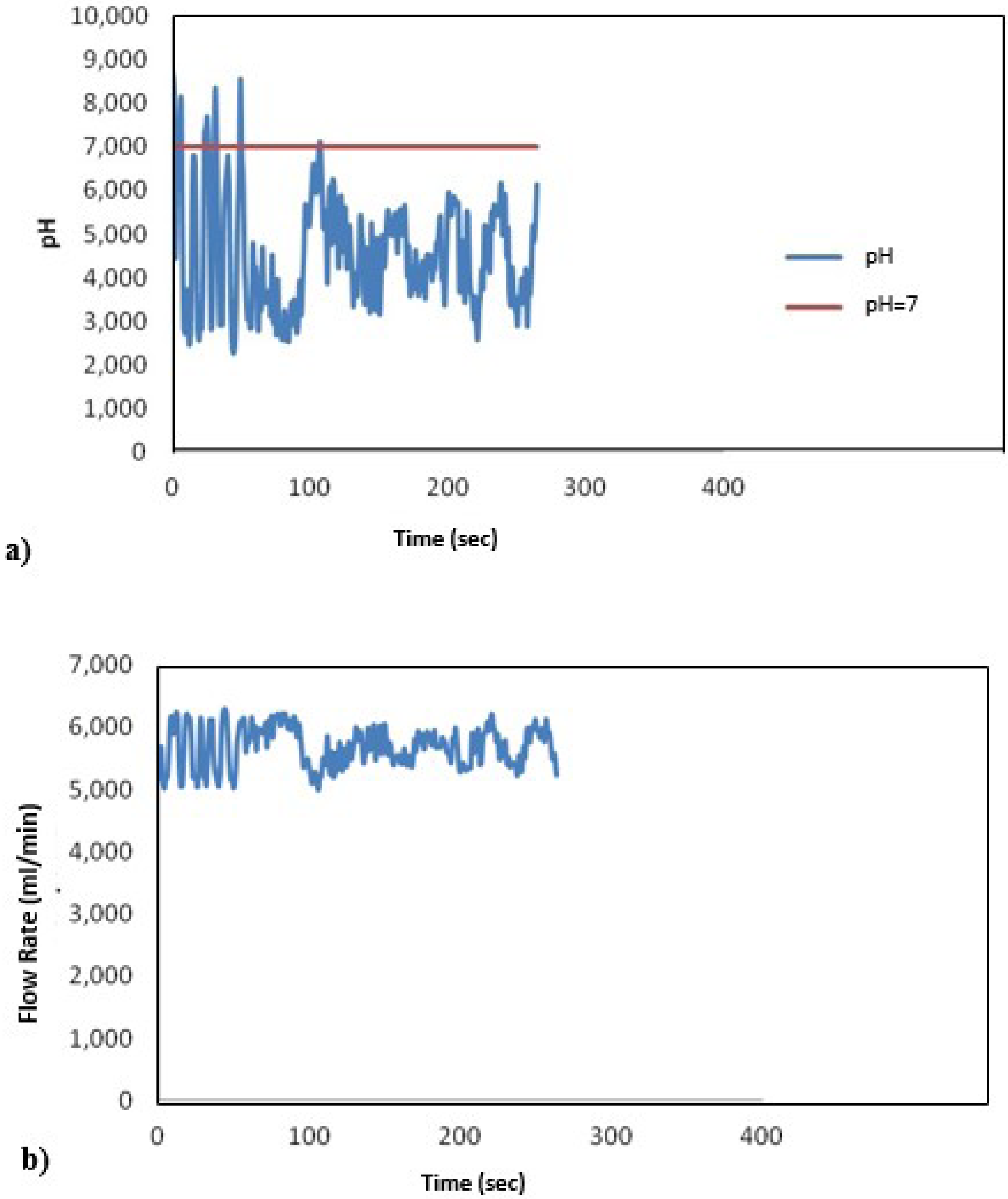
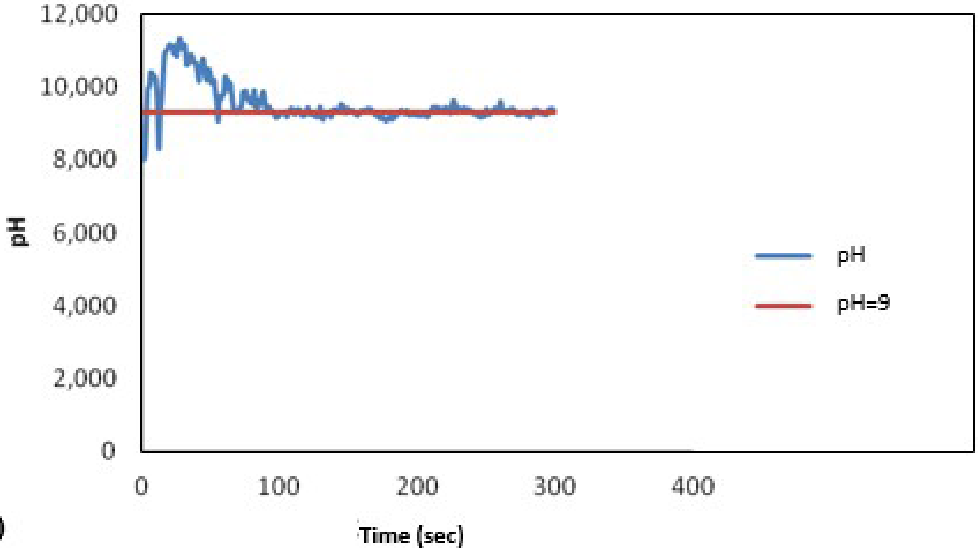


Figure 4. PID control results with brine sample for pH= 7. a) Time variation with pH. b) Variation in time of the adjustable variable.

In the Self-Adjusting PID control, the noises for the pH=9 setpoint were almost non-existent and the pH=9 setpoint was preserved (Figure 5). In the PID control, an offset was observed at pH=9 and it was quite noisy (Figure 6). It has been observed that the PID control is insufficient.

In Self-Adjusting PID control, it was again free of noise for pH=11 set value and reached the set value at pH=11. An offset of 0.5 units was observed in the last seconds. This is at negligible level (Figure 7). In PID control, an offset occurred at pH=11 in PID control, as in pH=7 and pH=9. The control is quite noisy. It has been observed that the PID control is insufficient (Figure 8).



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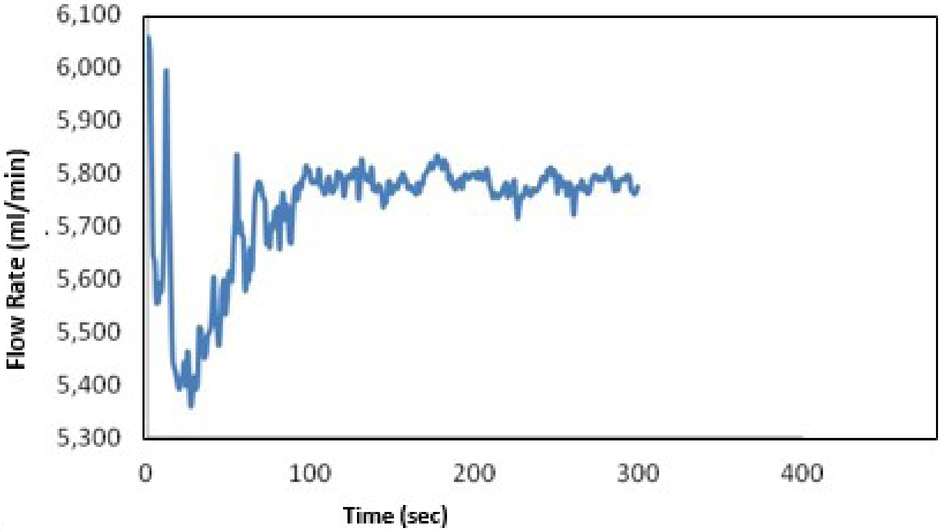

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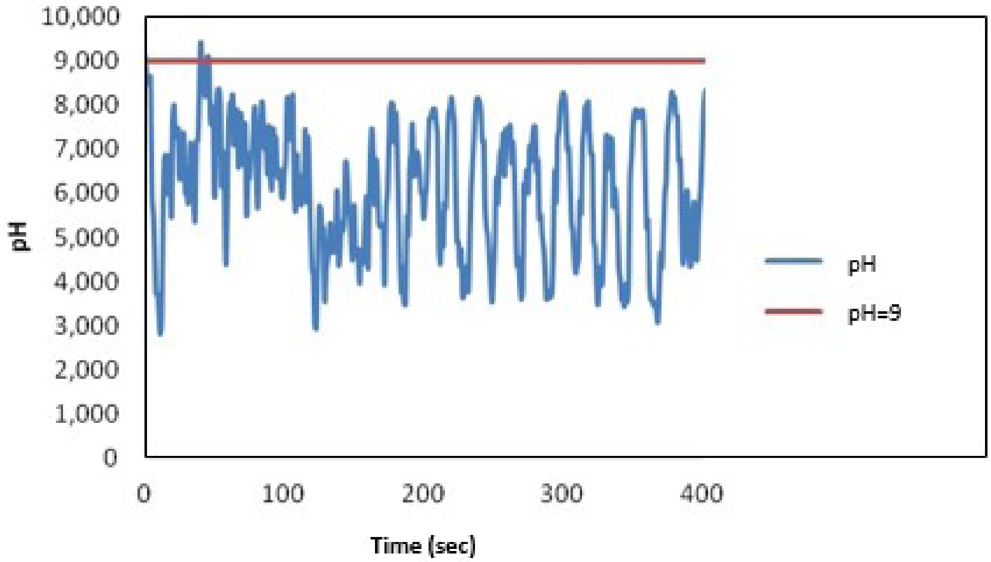
Figure 5. Self-adjusting PID with brine sample for pH=9 a) Time variation with pH. b) Variation in time of the adjustable variable.



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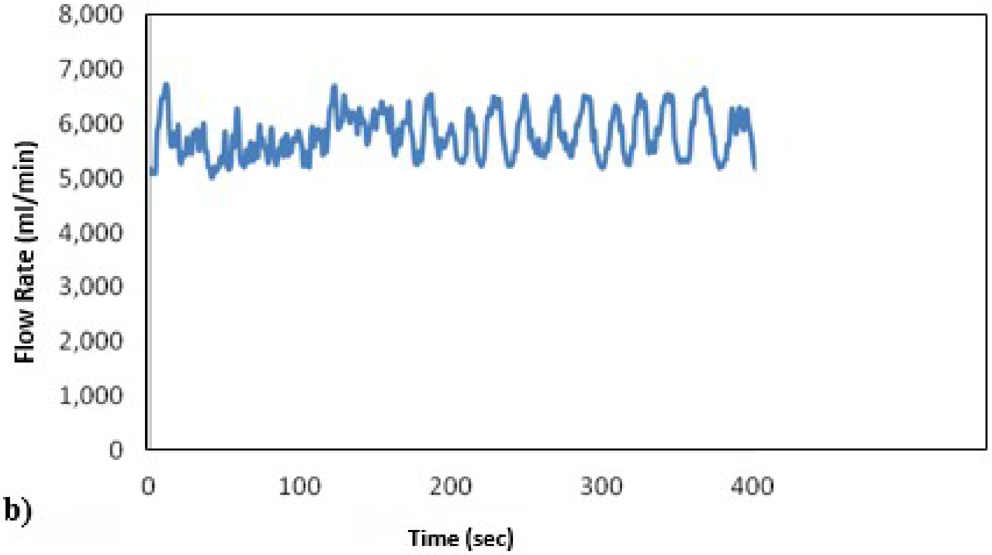


b)

Figure 6. PID control results with brine sample for pH=9. a) Time variation with pH. b) Variation of adjustable variable over time.

If we compare the Self-Adjusting PID and PID Control results, while a 20% HCl solution is sent at a constant flow rate of 24 ml/min for pH=7, when 10% NaOH solution is used as the adjustable variable; Self-adjusting PID control experiments and PID control experiments were found to be very difficult to control at set value pH=7, therefore, a very effective control could not be made (Figure 9).

While sending HCl solution at a constant flow rate of 20% for pH=9, using 10% NaOH solution as the adjustable variable, Self-adjusting PID control experiments and PID control experiments are performed at the set value pH=9, the self-adjusting PID controller appears to control nonlinear systems more effectively (Figure 10).

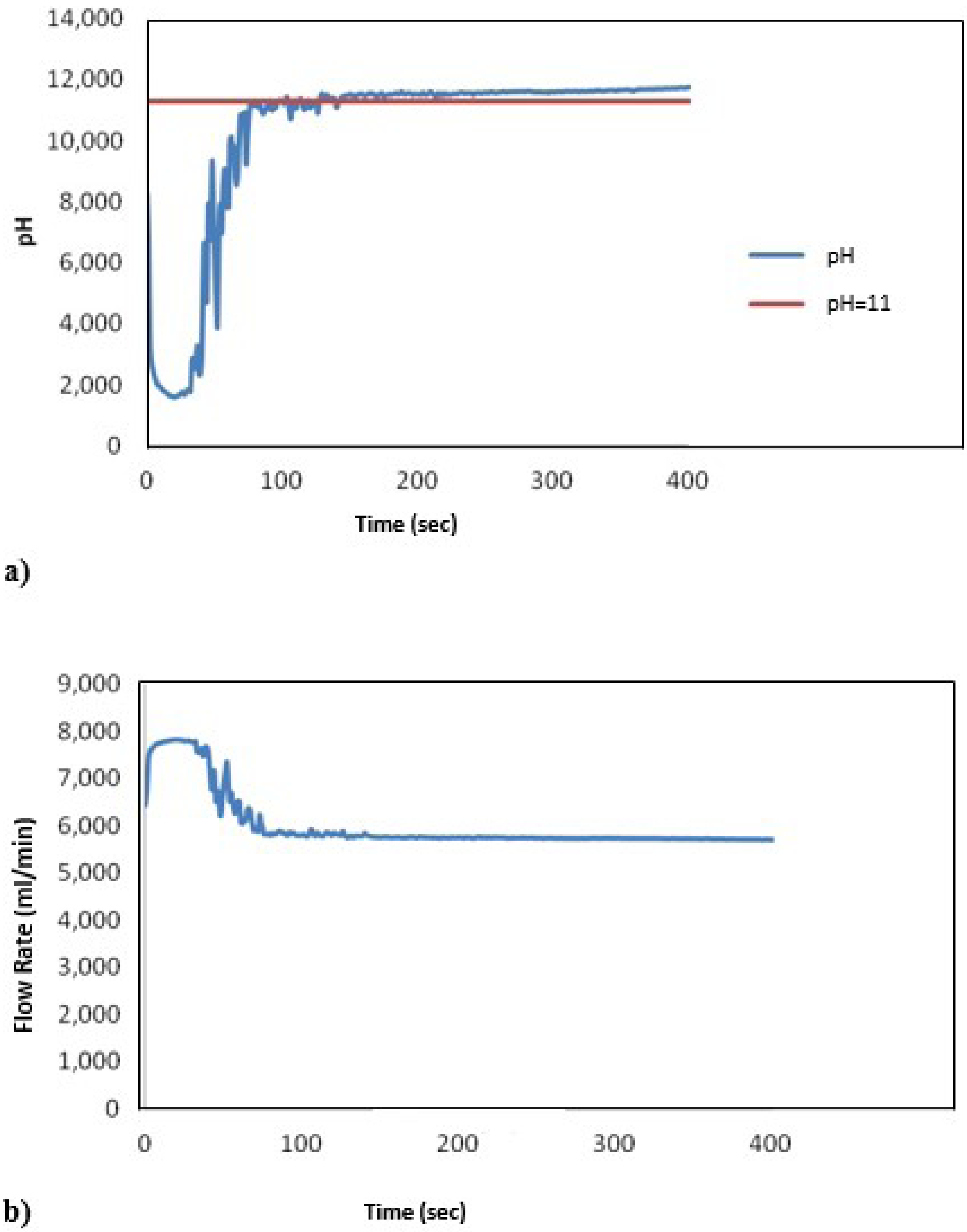


Figure 7. Self-adjusting PID control results with brine sample for pH=11. a) Time variation with pH. b) Variation of adjustable variable over time.

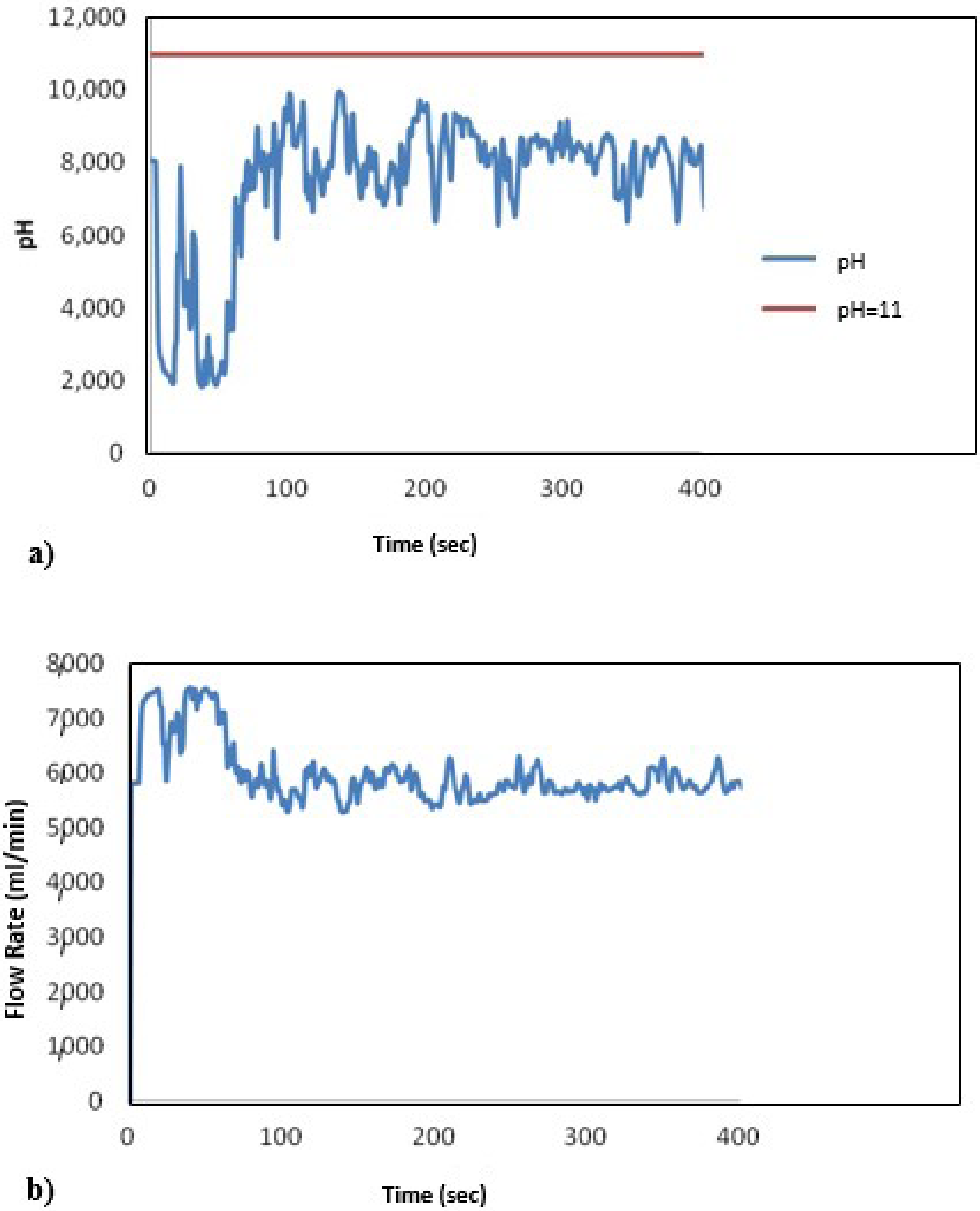


Figure 8. PID control results with brine sample for pH=11. a) Time variation with pH. b) Variation of adjustable variable over time.

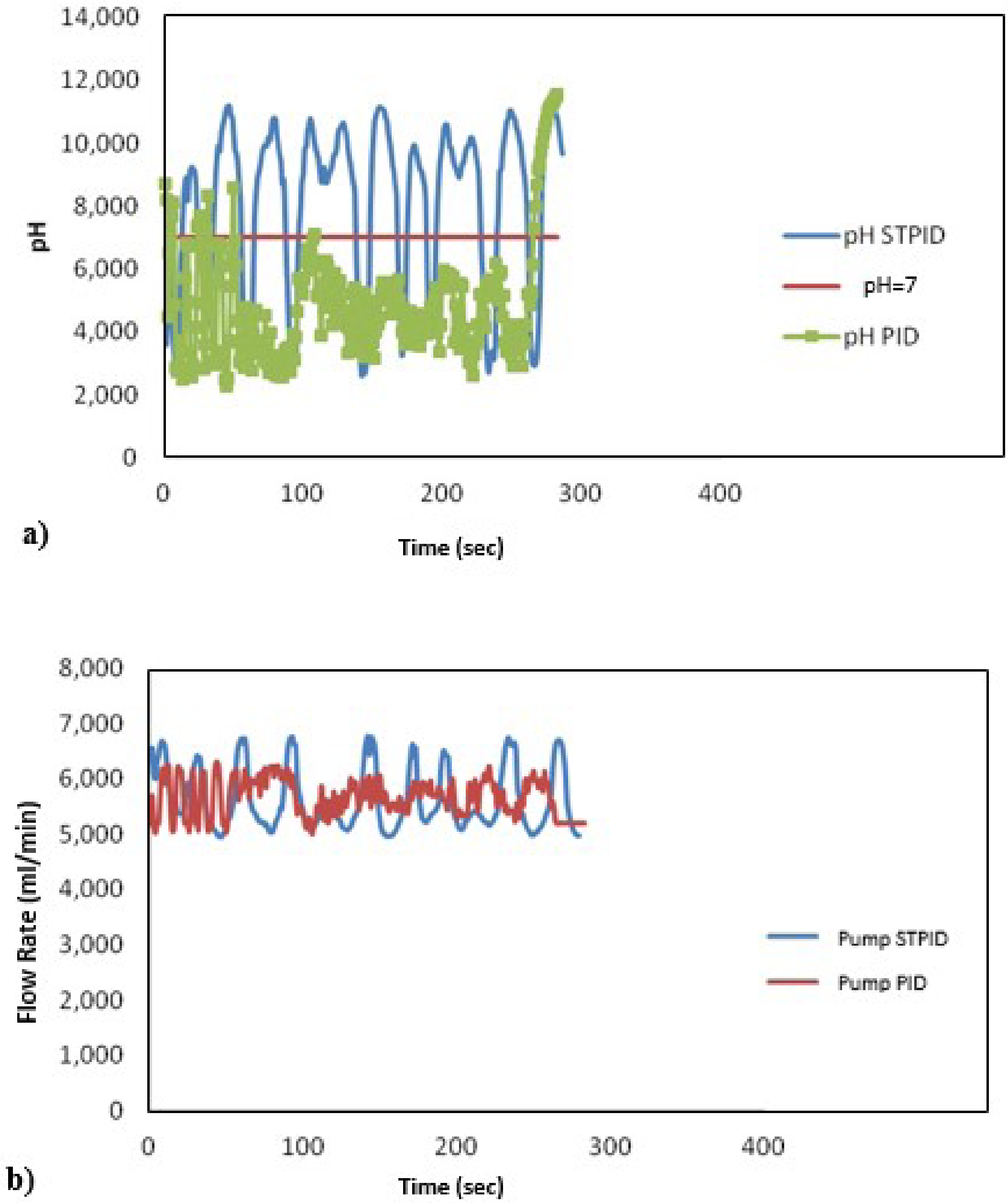
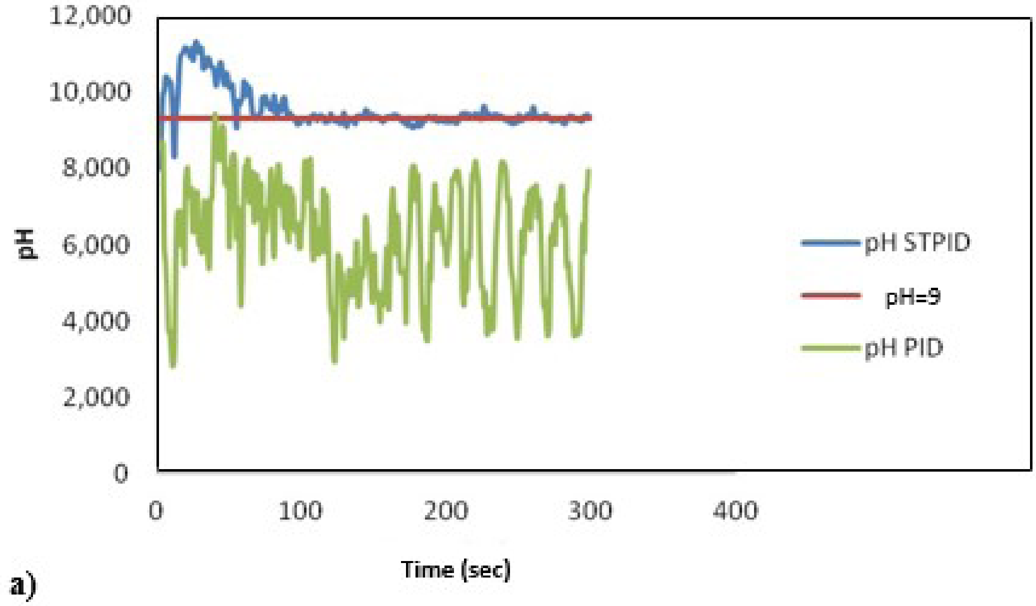


Figure 9. Experiment results with brine using PID and self-adjusting PID control methods for pH=7. a. Time variation with pH. b. Variation of adjustable variable over time.



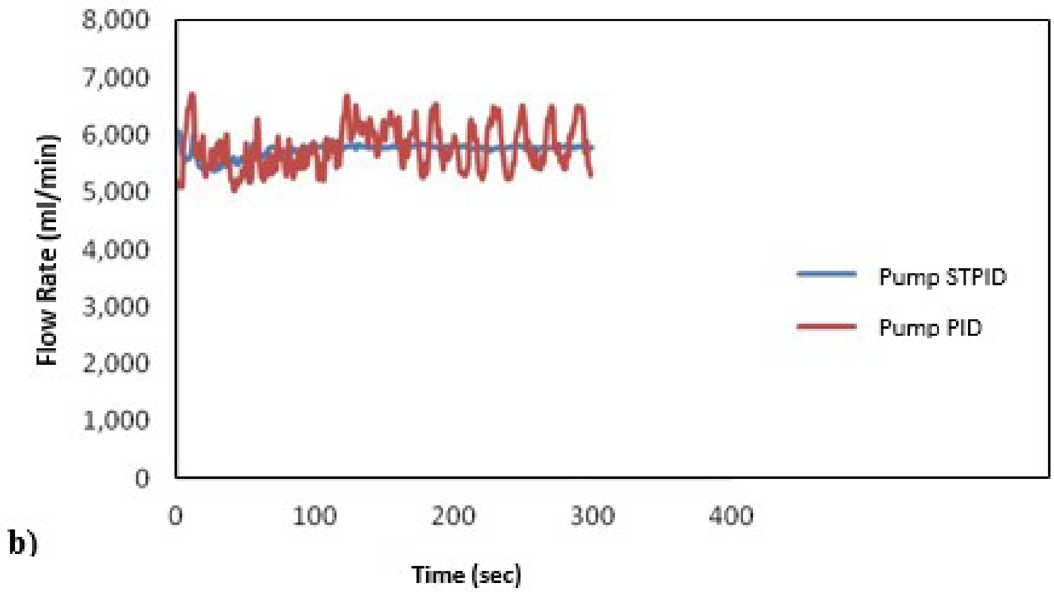


Figure 10. Experiment results with brine using PID and self-adjusting PID control methods for pH=9. a. Time variation with pH. b. Variation of adjustable variable over time.

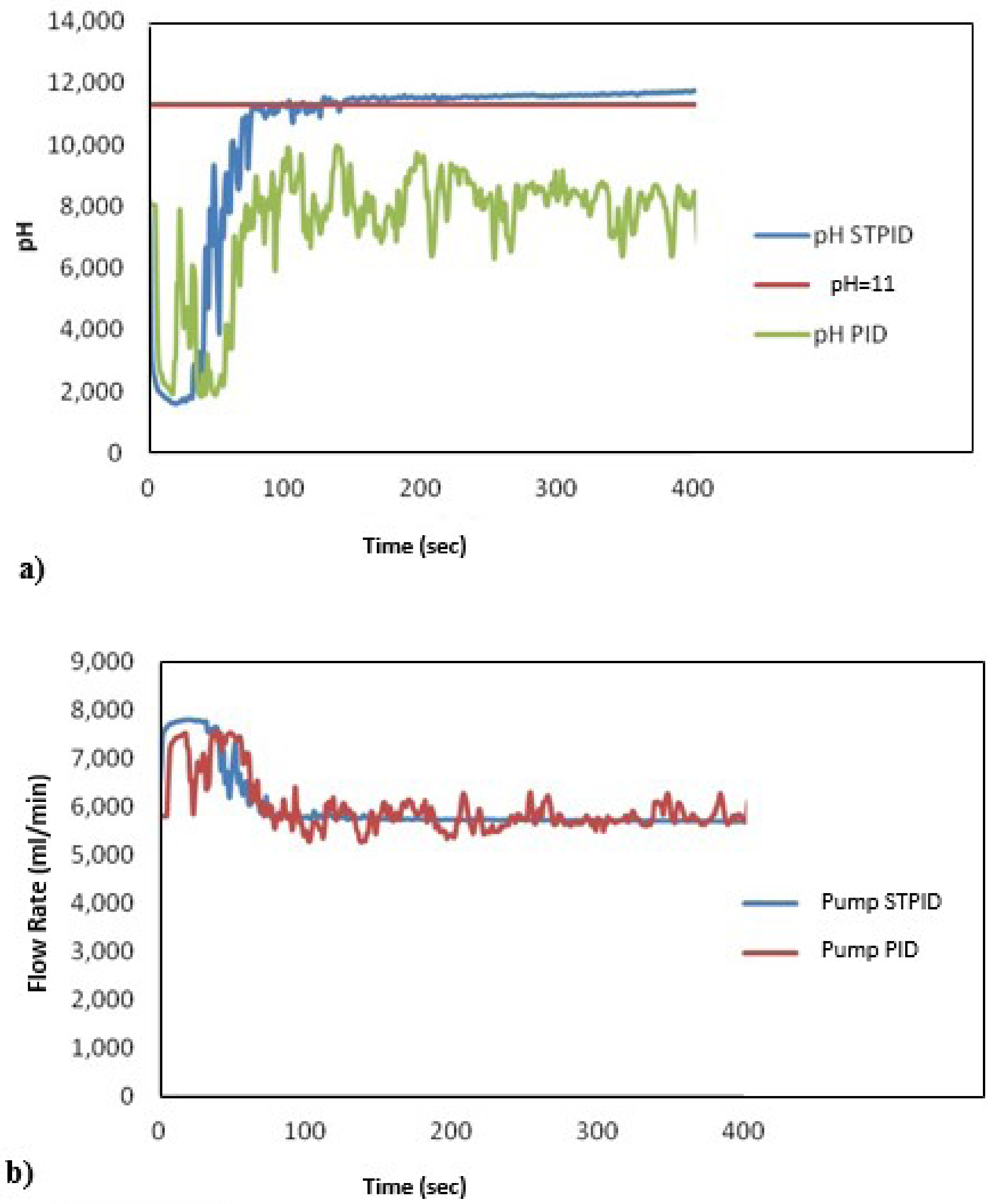


Figure 11. Experiment results with brine using PID and self-tuning PID control methods for pH=11. a. Time variation with pH. b. Variation of adjustable variable over time

While the HCl solution was sent at a constant flow rate of 20% for pH=11, 10% NaOH solution was used as the adjustable variable. When self-adjusting PID control experiments and PID control experiments were performed at set value pH=11, PID control could not be performed effectively (Figure 11).

CONCLUSIONS

In order to carry out the precipitation process, suitable operating conditions were created, and the most suitable pH value was determined. The experiments were carried out at room conditions and the same brine samples were used. In addition, three values were determined as pH value in experimental studies. These are 7, 9 and 11.

As a result of the literature search, after mathematical operations using the solubility multiplication of magnesium hydroxide, it was calculated that the best operating condition for the precipitation of $Mg(OH)_2$ as a result of the reaction of magnesium with NaOH was pH=10.52. As a result of all control studies, it was found that the best operating condition was at the adjusted pH = 11, and as it can be seen, the same result was reached in the literature.

According to the obtained operating conditions, self-regulating PID-controlled precipitation and PID-controlled precipitation were performed in a semi-batch reaction vessel. According to the obtained data, PID-controlled precipitation was insufficient compared to self-adjusting PID-controlled precipitation.
While offset and noise are observed in the precipitation process with PID control, the precipitation process with self-adjusting PID control is free from offset and noise.

The computer control system is connected online to the reactor where the precipitation processes are carried out. The pH measurements are made from the reactor, and the necessary information is transmitted to the computer, where calculations are made, the pump, which is used for pH control and sending the NaOH solution known as the adjustment variable to the system, is being adjusted. A self-adjusting control program is written in VISIDAQ programming language, and the control algorithm is loaded into the online connected computer for control calculations.

The system model was found between the base flow rate and pH used for control purposes under operating conditions. Utilizing the data obtained, the pH of the brine was controlled by a self-adjusting control system, and the base flow rate was used as an adjustable variable. Efficiency was observed in the precipitation process in salt solutions with self-adjusting PID pH control.

In order to ensure the best efficiency in control studies, the control setting parameter in the self-adjusting control algorithm should be selected in the most appropriate way. For this, in the control studies, adjusting the setting parameter, t1, is very important. In the control studies, the optimum value t1=0.5 was taken to observe the control efficiency in the best way.

It is known that the selection of the appropriate precipitator and the pH value are important for the purification of the brine. In this study, the importance of keeping the pH value at the desired value was emphasized in terms of purification efficiency. In addition, it has been determined that the self-adjusting PID control method is more suitable than the PID control method for pH control in this system.

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