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Nutrient Recovery From Organic-Rich Wastewater Through Struvite Precipitation using Air Cathode Electrocoagulation Technology

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ABSTRACT: This study developed an innovative method for phosphate recovery contained in wastewater using air cathode electrocoagulation (ACEC) technology. This technique has the advantage of low energy consumption since it can effectively produce the struvite precipitate without any electrical-grid energy consumption. The experiments were conducted under recirculating batch mode by varying the recirculation rate and initial pH of wastewater to investigate their effects on the ACEC reactor performance. SEM-EDS and XRD analyses were performed to examine the morphology and structure of the produced crystals. The result showed that the precipitates exhibited in the form of crystals with irregularly shaped accompanied by a sharp at the surface and composed of Mg (6.67%), P (6.78%), N (1.66%), and O (47.41%). XRD chromatogram showed that the precipitate matched the reference pattern for struvite. The recirculation rate of $2.0\,mL\,min^{-1}$ was determined as an optimum condition with nitrogen and phosphate removal, nitrogen and phosphate removal rate, and struvite precipitation rate was $14.7\% \pm 1.2\%$, $57.1\% \pm 1.1\%$, $22.9 \pm 0.9\,mg\,L^{-1}\,h^{-1}$, $18.6 \pm 0.9\,mg\,L^{-1}\,h^{-1}$, and $498\,mg\,h^{-1}$, respectively. The recirculation rate affects the reactor performance through the magnitude of the hydraulic retention time of nutrients on the reactor and the potential electrical energy generated in the reactor. On the other hand, the reactor with initial pH 8 achieved the best performance with the nitrogen and phosphate removal of $24.6\% \pm 1.6\%$ to $88.4\% \pm 3.8\%$, nitrogen and phosphate removal rate of $24.2 \pm 2.1\,mg\,L^{-1}\,h^{-1}$ and $35.3 \pm 2.1\,mg\,L^{-1}\,h^{-1}$, respectively, and the struvite precipitation rate of $900\,mg\,h^{-1}$. Furthermore, when the initial pH of wastewater increased from 7 to 8, the struvite precipitation rate increased from 499 to $900\,mg^{-1}$. However, increasing the initial pH of wastewater from 8 to 9 caused a decrease in struvite precipitation rate from 900 to $656\,mg\,h^{-1}$. These results demonstrated that the recirculation rate and initial pH of wastewater control the process of the struvite precipitation process in ACEC technology.

KEYWORDS: Struvite recovery, innovative electrocoagulation, nutrient-rich wastewater, renewable energy, energy recovery

Introduction

Nitrogen and phosphorus are essential elements in life, but the continuous release of excess nitrogen and phosphorus into the environment during natural processes and anthropogenic activities leads to eutrophication in natural water systems (Lee & Jones, 1986). Eutrophication is estimated to increase along with increasing population, agricultural intensification, and industrialization. However, eutrophication can be overcome by reducing the nutrient load that enters the water body by reducing the nutrient load discharged from the wastewater treatment plant to the water body. Removal of nutrients from wastewater can be achieved by biological or physical-chemical processes (Bunce et al., 2018). As for the physicochemical category, phosphate removal can be conducted through adsorption (Cheng et al., 2009), membrane separation (Kumar et al., 2007), coagulation (Arnaldos & Pagilla, 2010).

In the last decade, the focus of wastewater treatment has shifted from originating solely to control and prevention of pollution in the environment to the utilization/recovery of resources contained in the wastewater (resource recovery) (Logan, 2008; Logan & Rabaey, 2012). Natural resources such as ammonia and phosphate are important compounds for the human food supply. Ammonia compounds are generally produced from the Haber-Bosch process with high pressure and temperature, which requires 1.0% energy of the total energy needed by the world (Cherkasov et al., 2015). Phosphorus compounds are abundant in nature, but their accessibility and quality are still limited. In the last decade, the price of phosphorus has increased 2 to 3 times and is expected to peak in the next 20 to 70 years (Jacobs et al., 2017), where the supply of phosphorus has begun to decrease. Nearly 100% of the nitrogen and phosphorus consumed by humans is released back into nature in the form of waste either faeces or urine (Spångberg et al., 2014). Wastewater treatment plants are designed to eliminate these nutrients to prevent algae blooming (eutrophication) in receiving water bodies. The most commonly used approach to eliminate nutrients in wastewater is the nitrification/denitrification process and metal precipitation with metal salts, where nutrients are effectively removed (Coats et al., 2011). Nutrient removal by nitrification is a method that requires a substantial amount of energy which reaches 60% of operational costs (Nancharaiah et al., 2016). A more sustainable approach uses close nutrient loops that use/reuse resources (nutrients) in wastewater rather than processing them into unwanted/set aside residues (Chojnacka et al., 2020).

Struvite precipitation is one of the promising techniques that is commonly used to remove and recover nutrients contained in the wastewater (Siciliano et al., 2020). This technique
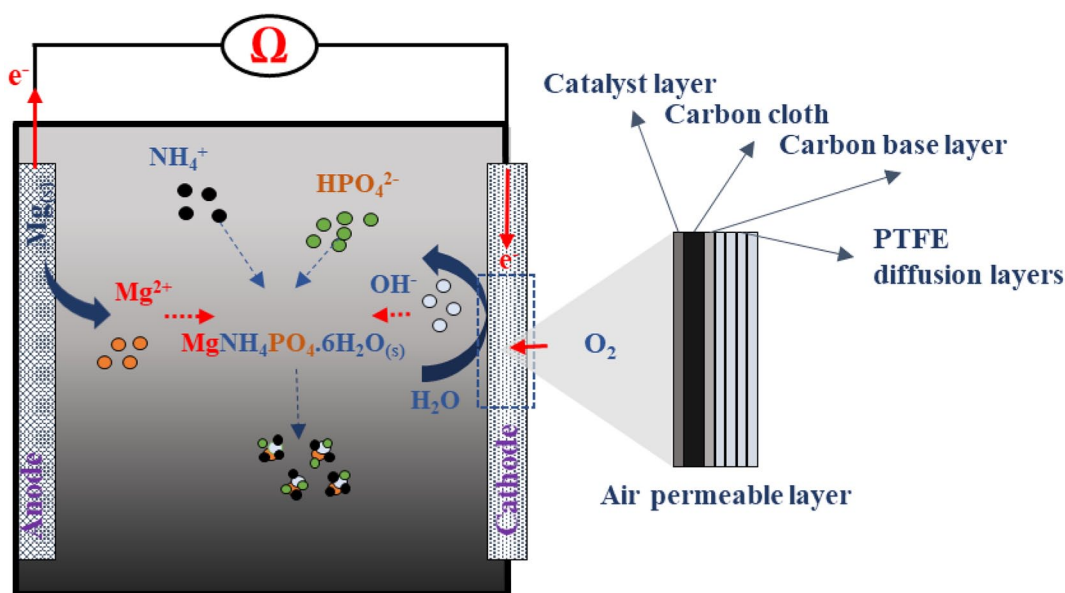
Figure 1. Schematic diagram of air-cathode electrocoagulation.

is preferred because it reduces the environmental impact caused by phosphorus emission and produces the fertilizer as a struvite (Kékedy-Nagy et al., 2022). Although it contains large amounts of nitrogen and magnesium, Magnesium Ammonium Phosphate Hexahydrate (MAP) ($MgNH_4PO_4 \cdot 6H_2O$ or struvite) is phosphate fertilizer; it is an effective alternative source of phosphate rock to maintain agricultural production systems. Nutrient recovery in the form of struvite precipitation can be obtained by electrocoagulation technology (Kékedy-Nagy et al., 2022). However, this conventional technique has the disadvantage of high operational costs because it uses electrical energy as a driving force in the process of forming struvite.

Air cathode electrocoagulation (ACEC) is an emerging technology that can be used as an alternative for struvite recovery from wastewater (Kim et al., 2018). Compared with other techniques (such as conventional electrocoagulation), this technique has the advantages of low energy consumption (requires no additional energy), high efficiency, and relatively low cost (Maitlo et al., 2019). Thus, the use of ACEC technology is an interesting process for nutrient recovery from wastewater by struvite precipitation. In this study, the effect of recirculation rate and initial pH of the wastewater was observed to determine their effect on the ACEC reactor performance. The ACEC reactor performance was then evaluated by the nutrient removal efficiency, nutrient removal rate, and struvite precipitation rate. Subsequently, SEM-EDS and XRD analyses were performed to examine the morphology and structure of the produced precipitate.

Materials and Methods

Reactor construction

The reactor was a single chamber fuel cell made of acrylic with a distance of inter-electrode and the working volume of 3.0 cm

and 75 mL (5.0 cm × 5.0 cm × 3.0 cm), respectively (Figure 1). The reactor was equipped with a reservoir to hold the electrolyte that recirculated. The 0.30 mm thick plate of magnesium (AZ31 alloy) was used as an anode electrode with 25 cm² of surface area. The wet-proofed (30%) carbon cloth (1,071 HCB, AVCarb®) was used as a cathode electrode with a modification of four polytetrafluoroethylenes (PTFE) diffusion layers on the air-side and a Nafion binder mixed with platinum catalyst on the solution-side. This technique has been demonstrated by previous research (Cheng et al., 2006). The electrodes were then connected with a 1 mm of Titanium wire. The Ag/AgCl reference electrodes were assembled on each of the chambers to measure the electrode potentials.

Reactor operation

The effect of recirculation rate and initial pH of electrolyte were investigated with the circulation of 1 L of artificial wastewater to the ACEC reactor. This artificial wastewater contained ammonium chloride (NH_4Cl) and potassium phosphate ($K_2HPO_4 \cdot 3H_2O$) as nitrogen and phosphate sources. The concentration of ammonia and phosphate in the artificial wastewater was determined at 4,500 mg L^{-1} and 1,000 mg L^{-1}, respectively. This value represented the anaerobic digester effluent of food waste leachate as reported earlier (Ma et al., 2013). In addition, sodium chloride of 0.01 M was added to the reactor as a supporting electrolyte (Kim et al., 2018).

The experiments were conducted under recirculating batch mode by varying the recirculation rate and initial pH of artificial wastewater to investigate their effects on the ACEC reactor performance. Firstly, the reactor was operated with the variation of recirculation rate at 1.0 to 2.0, 3.0, 4.0 mL min^{-1} to 5.0 mL min^{-1}. This variation of recirculation rate was the further elaboration from the previous research (Kim et al., 2018).
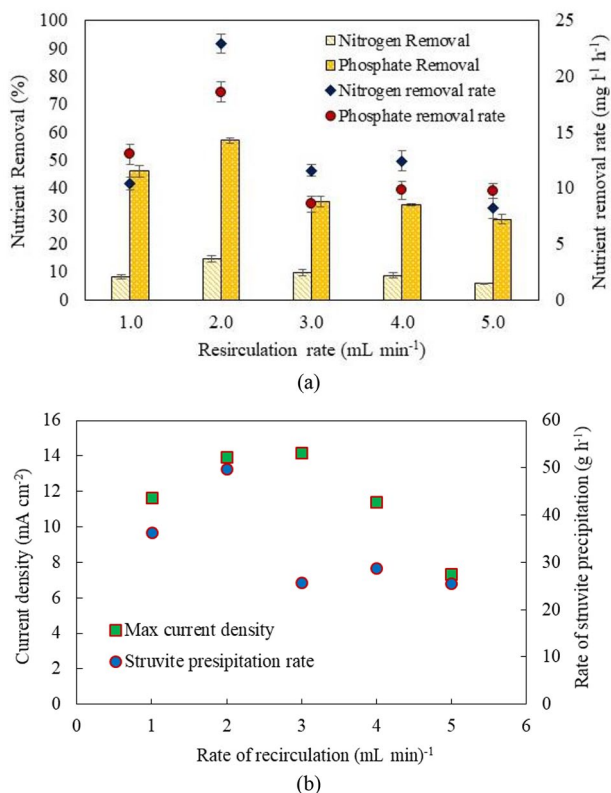
Figure 2. The effect of the recirculation rate on (a) nutrient removal and nutrient removal rate, and (b) potential electrical energy and rate of struvite precipitation.

The reactor was connected to the external resistance of $5\,\Omega$ to observe the current generated by the system. The reactor then operated for 24 hours and the samples were taken at the reservoir for 0, 0.5, 1.0, 2.0, 4.0, 6.0, 8.0, 12, 24 hours. Subsequently, the second experiment was carried out with the variation of initial pH of electrolyte with the optimum condition of recirculation rate from the first experiment. The variation of initial pH was 4, 7, 8, 9, which represent the acidic, neutral, and base/alkali conditions.

Measurement and calculation

While the amount of struvite was determined using a gravimetric method at the end of the cycle, nitrogen (as ammonia) and phosphate (as orthophosphate) concentrations were determined by SMEWW methods. As for magnesium and phosphorus concentration, they were determined using 3500-Mg Magnesium and 4500-P Phosphorus of SMEWW methods, respectively. The standard deviation for all measurements was below 5.0%. The cell voltage was recorded on the data logger to calculate the current density.

The efficiency of nutrient removal was determined as $E = ((C_0 - C_t)/C_0) \times 100\%$ (equation (1)), where E (%) is the efficiency of nutrient removal, C_0 (mg L^{-1}) is the initial concentration of the nutrient, and C_t (mg L^{-1}) is the final concentration of the nutrient. The Voltage (V) between anode and cathode was monitored and recorded every 5 minutes using a

voltage recorder (VR-71, T&D Corporation) connected to a personal computer. The current was determined as $i = V/R$ (equation (2)), and the current density was normalized by the cathode projected surface area ($7\,cm^2$).

Results and Discussions

Effect of recirculation rate on nutrients removal and struvite precipitation

In this study, the removal and recovery of nutrients were carried out by recirculating 1.0 l of artificial wastewater into the reactor for 24 hours. The effect of the recirculation rate on the nutrient removal, removal rate, and struvite precipitation rate can be seen in Figure 2a. The figure shows that nitrogen and phosphate removal increased from $8.3\% \pm 0.9\%$, $46.2\% \pm 2.05\%$, $14.8\% \pm 1.2\%$, $57.1\% \pm 1.1\%$, respectively, when recirculation rate increased from 1.0 to 2.0 mL min^{-1}. However, an increase in the recirculation rate from 2.0 to 3.0 mL min^{-1} caused a decrease in nitrogen and phosphate removal to $9.9\% \pm 1.1\%$ to $35.2\% \pm 1.7\%$, respectively. This result shows that the small recirculation rate causes the detention time of wastewater to be longer in the reactor so that a reaction of more struvite formation coincides with a decrease in nutrients concentration contained in the wastewater and vice versa. In addition, this also causes equilibrium concentrations to tend to be more quickly achieved at a relatively large recirculation rate compared to a small recirculation rate. Similar with nutrient removal, the rate of nitrogen and phosphate removal also increased from 10.4 ± 0.6 mg $L^{-1} h^{-1}$ and 13.1 ± 0.9 mg $L^{-1} h^{-1}$ to 22.9 ± 09 mg $l\,h^{-1}$ and 18.6 mg $L^{-1} h^{-1}$, respectively, when the recirculation rate increased from 1.0 to 2.0 mL min^{-1}. Furthermore, nutrient removal rate decreased as the recirculation rate increased from 2 to 3.0, 4.0 to 5.0 mL min^{-1}. These results showed that the performance of the ACEC reactor is likely affected by the recirculation rate.

Table 1 shows that the highest nutrients removal, removal rate, and struvite precipitation rate were achieved at the recirculation rate of 2 mL min^{-1} with nitrogen removal of $14.7\% \pm 1.2\%$, phosphate removal of $57.1\% \pm 1.1\%$, and struvite precipitation rate of 498 mg h^{-1}. It was likely due to the recirculation rate's effect on nutrient loads that should be treated in the reactor. The lower the recirculation rate, the longer the retention time of nutrients in the reactor; thus, the nutrient can optimize the reaction in which magnesium ions are released from the oxidation process at the anode to form struvite. However, in the case of the recirculation rate of 1.0 mL min^{-1}, the removal efficiency, removal rate, and struvite precipitation rate were lower than the reactor with 2.0 mL min^{-1}. This phenomenon is still not clear but it is possible due to the lack of nutrient load into the system since the retention time in the reactor with 1 mL min^{-1} is too long.

It was also shown that the performance of the ACEC reactor is not only affected by the retention time of nutrients in the reactor. The other parameter that should be considered in the ACEC reactor is the current generated from the reactor. Figure 2b shows the effect of recirculation rate on the

Table 1. Performance of ACEC Reactor With Variation of Recirculation Rate on Nutrient Removal, Nutrient Removal Rate, and Struvite Precipitation Rate.

RATE OF RECIRCULATION (mL min^{-1})	NITROGEN REMOVAL (%)	NITROGEN REMOVAL RATE (mg L^{-1} h^{-1})	PHOSPHATE REMOVAL (%)	PHOSPHATE REMOVAL RATE (mg L^{-1} h^{-1})	RATE OF STRUVITE PRECIPITATION (mg h^{-1})
1.0	8.3 ± 0.9	10.4 ± 0.6	46.2 ± 2.1	13.1 ± 0.9	362
2.0	14.7 ± 1.2	22.9 ± 0.9	57.1 ± 1.1	18.6 ± 0.9	498
3.0	9.9 ± 1.1	11.6 ± 0.5	35.2 ± 1.7	8.6 ± 0.7	257
4.0	8.8 ± 1.1	12.5 ± 0.9	34 ± 0.4	9.8 ± 0.8	288
5.0	5.8 ± 0.3	8.2 ± 0.9	28.8 ± 1.7	9.8 ± 0.7	256

potential current density and struvite precipitation rate. The figure shows that the struvite precipitation rate linearly increased as the current density rose. The higher the current density, the higher the struvite precipitation rate. As a result, it was also proved that the performance of the ACEC reactor in producing struvite was affected by the potential current density generated by the reactor.

Effect of initial pH on nutrients removal and struvite precipitation

According to the above results, the reactor with 2.0 mL min^{-1} was selected as the optimum operational condition as the increase in the flow rate to 5.0 mL min^{-1} did not substantially increase the reactor performance. Struvite formation occurs when the concentration of magnesium ion (Mg^{2+}), ammonium (NH$_4^+$), and phosphate (PO$_4^{3-}$) exceed their solubility product (Ksp), which is mainly controlled by the pH (Uludag-Demirer et al., 2005). Therefore, in further experiments, the reactor was operated using 2.0 mL min^{-1} with different initial pH of wastewater (4, 7, 8, 9). Figure 3a shows the nutrient removal and nutrient removal rate of the ACEC reactor with different initial pH. Based on Figure 3a, it can be seen that the optimum initial pH of wastewater was at pH 8 with the nitrogen and phosphate removal of 24.6% ± 1.6% to 88.4% ± 3.8%, nitrogen and phosphate removal rate of 24.2 ± 2.1 mg L^{-1} h^{-1} and 35.3 ± 2.1 mg L^{-1} h^{-1}, respectively, and the struvite precipitation rate of 900 mg h^{-1}. The reactor with initial pH 4 achieved the lowest values for all parameters because the reactor needs the longest time (6 hours) to reach the pH where the struvite formation occurs (Figure 3b). Previous studies reported that the struvite precipitation could take place in the pH range 7 to 11, with the minimum solubility at pH 9 (Buchanan et al., 1994; Siciliano & De Rosa, 2014). The reactor with initial pH of 7 to 8, and 9 achieved relatively high struvite precipitation rate, with the initial pH 8 as the highest among these reactors. The struvite precipitation rate for the reactor was 499, 900, and 656 mg h^{-1}, respectively. These results are likely due to the time that the optimum pH where struvite formation needed at the
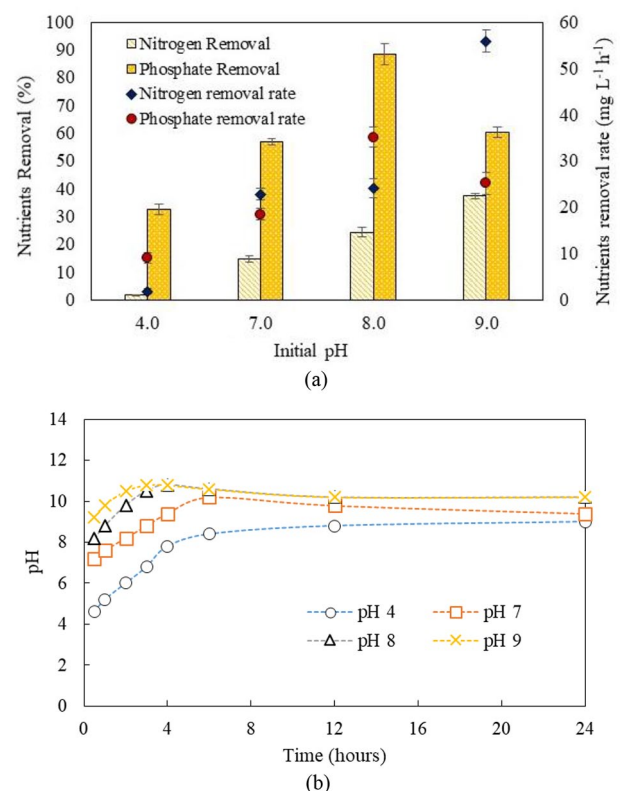
Figure 3. (a) Nutrient removal and nutrient removal rate, and (b) change in pH of the ACEC reactor with different initial pH.

initial pH 8 was greater than that of the other reactor (Figure 3b). This result was consistent with the previous study, which stated that the struvite formation was optimal at the pH values between 8.5 and 9.3 (Siciliano & De Rosa, 2014). The other study reported a rapid rise in the reaction rate of struvite formation in response to pH growth from 8.6 to 9.0 (Huang et al., 2017). Further studies identified that pH values around nine as the most favorable for struvite precipitation (Tomei et al., 2020).

The comparison of reactor ACEC performance with the other technologies is shown in Table 2. The table shows that the ACEC reactor was comparable to the other technologies in terms of nutrients removal. The recent study achieved 88.4%

Table 2. Comparison of the Recent Study with Available Literatures.

NO	TYPE OF REACTOR	TYPE OF WASTEWATER	REACTOR OPERATIONAL	PHOSPHATE REMOVAL (%)	NITROGEN REMOVAL (%)	REFERENCES
1	STR	Raw swine slurry	Batch	80	77	Taddeo and Lepistö (2015)
2	STR	Anaerobically digested manure	Batch	80	n.a	Brown et al. (2018)
3	STR	Synthetic urine	Batch	90		Aguado et al. (2019)
4	Electrolyzer	Synthetic swine wastewater	Batch	78	n.a	Wang, Fu et al. (2019)
5	STR	Anaerobic digestion supernant	Batch	>90	29	Xavier et al. (2014)
6	STR	Anaerobically digested centrate	Batch	>90	n.a	Zhang et al. (2014)
7	STR	Synthetic urine	Batch	97.7	n.a	Xu et al. (2015)
8	STR	Landfill leachate	Batch	n.a	72	Ramaswami et al. (2016)
9	STR	Synthetic swine wastewater	Continuous	98.9	85	Kim et al. (2017)
10	MFC	Swine wastewater	Batch recirculation	70–82	n.a	Ichihashi and Hirooka (2012)
11	MFC	Urine	Batch	82	n.a	You et al. (2016)
12	MEC	Artificial wastewater	Batch	20.1–73.9	n.a	Lei et al. (2019)
13	ACEC	Synthetic Anaerobic digestion effluent	Batch recirculation	98	n.a	Kim et al. (2017)
14	ACEC	Synthetic Anaerobic digestion effluent	Batch recirculation	32.7 57.1 88.4 60.4	1.9 22.9 24.2 56.0	this study*

*with the variation of initial pH.

removal of phosphate, which was comparable to the reactor STR and Electrolyzer from previous studies (Aguado et al., 2019; Brown et al., 2018; Taddeo & Lepistö, 2015; Wang, Fu et al., 2019). In terms of nitrogen removal, the recent study achieved 56.0% which was higher than the STR reactor from the previous study (Xavier et al., 2014). A previous study reported that the struvite precipitation rate (in terms of phosphate removal rate) from the electrolyzer was about 70.46 mg L^{-1} h^{-1} to 396.65 mg L^{-1} h^{-1}. It was higher than the recent study, which achieved 35.3 mg L^{-1} h^{-1} with 900 mg h^{-1} of struvite formation (Wang, Fu et al., 2019). Even though the results were lower than previous study, the reactor ACEC has the potential to replace both the new emerging and conventional electrocoagulation technologies for struvite recovery as it has low energy consumption.

Characterization of struvite precipitates

The struvite precipitates were identified by SEM-EDS and XRD, as shown in Figure 4. The sample of the precipitate was taken from the reactor with initial pH of 8 which was the optimum condition of the experiment. The SEM results showed that the struvite precipitates exhibited in the form of crystals with irregularly shaped accompanied by a sharp at the surface (Figure 4a). This result was similar to the previous studies, which stated that the struvite precipitate might occur in the form of irregular, cubic, and rod-like irregular crystals (Sciarria et al., 2019; Tao et al., 2015; Zou et al., 2017). The EDS analyses showed that the precipitates accumulated in the reactor were that of pure struvite, with the EDS spectra showing the presence of Mg (6.67%), P (6.78%), N (1.66%), and O (47.41%) (Cusick et al., 2014). The XRD results showed a similarity between the specific peaks of the test sample with the struvite (Figure 4b). As shown in the XRD chromatogram, similar specific peaks between the sample and struvite standard have occurred at 2 with 15.83, 16.49, 20.9, 21.48, 27.1, 29.11, 30.2, 30.65, 31.94, 33.3o. This result indicated that the sample test could be identified as struvite ($MgNH_4PO_4.6H_2O$).

Conclusions
The performance of the ACEC reactor was examined by operating the reactor with the variation of recirculation rate and different initial pH. Struvite precipitates were identified by SEM-EDS and XRD. The results showed that the precipitates exhibited in the form of crystals with irregularly shaped accompanied by a sharp at the surface and composed of Mg (6.67%),
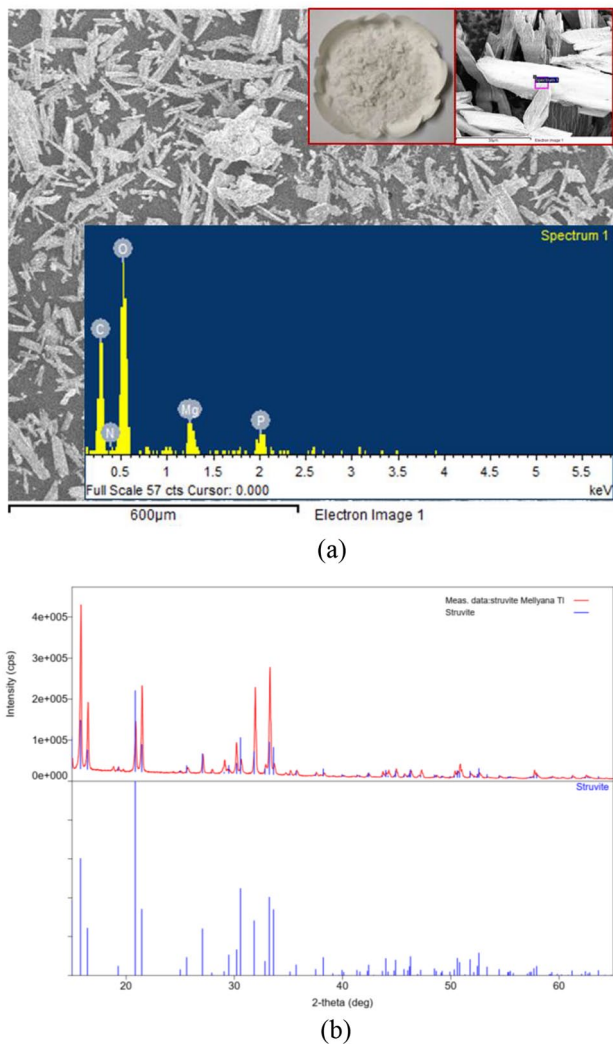
(a)

(b)

Figure 4. (a) SEM–EDS and (b) XRD analyses for struvite precipitates from ACEC reactor.

P (6.78%), N (1.66%), and O (47.41%). XRD chromatogram shows that the precipitate matched the reference pattern for struvite. Based on the evaluation of the reactor performance, the reactor with a recirculation rate of 2 mL min⁻¹ was selected as the optimum condition. In such condition, the nitrogen and phosphate removal, nitrogen and phosphate removal rate, and struvite precipitation rate was $14.7\% \pm 1.2\%$, $57.1\% \pm 1.1\%$, $22.9\% \pm 0.9\,mg/L/h$, $18.6\% \pm 0.9\,mg\,L^{-1}\,h^{-1}$ and $498\,mg\,h^{-1}$, respectively. The recirculation rate affects the reactor performance through the magnitude of the hydraulic retention time of nutrients on the reactor (75, 37.5, 25, 18.8 minutes, and 15 minutes for recirculation rate of 1, 2 mL min⁻¹ to 3.0, 4.0 to 5.0 mL min⁻¹, respectively) and the potential electrical energy generated in the reactor (11.7, 14 to 0, 14.2, 11.4 mA cm⁻¹, and 7.4 mA cm). On the other hand, the reactor with initial pH 8 achieved the best performance with the nitrogen and phosphate removal of $24.6\% \pm 1.6\%$ to $88.4\% \pm 3.8\%$, nitrogen and phosphate removal rate of $24.2 \pm 2.1\,mg\,L^{-1}\,h^{-1}$ and $35.3 \pm 2.1\,mg\,L^{-1}\,h^{-1}$, respectively, and the struvite precipitation rate of $900\,mg\,h^{-1}$ since the value of solubility product was optimum at these

conditions. Based on the results above, it can be concluded that the recirculation rate and initial pH of wastewater control the process of the struvite precipitation process.

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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