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KY, Nguyen Minh

PhD Program in Maritime Science and Technology, National Kaohsiung University of Science and Technology (NKUST)

NHUT, Huynh Tan

Faculty of Environment and Natural Resources, Nong Lam University

HIEP, Nguyen Trung

Research Institute for Sustainable Development, Ho Chi Minh City University of Natural Resources and Environment

LAP, Bui Quoc

Faculty of Chemistry and Environment, Thuyloi University

他

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## Investigation of Nitrogen and Phosphorus Recovery from Swine Wastewater by Struvite Crystallization

Nguyen Minh KY<sup>1,2,\*</sup>, Huynh Tan NHUT<sup>2,\*</sup>, Nguyen Trung HIEP<sup>3</sup>, Bui Quoc LAP<sup>4,\*</sup>,  
Nguyen Tri Quang HUNG<sup>2</sup>, Chitsan LIN<sup>5\*</sup>, Tran Thi Minh TAM<sup>2</sup> and Akinori OZAKI<sup>6</sup>

Institute of Tropical Agriculture Kyushu University, Fukuoka 819–0395, Japan

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Swine wastewater is one of the environmental issues that negatively effect on the ecosystem and human health. Developing advanced technologies to treat and recover nutrients from swine wastewater have attracted the attention of scientists. This study investigated the simultaneous removal of nitrogen (N) and phosphorus (P) from swine wastewater by a magnesium ammonium phosphate hexahydrate (struvite,  $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) crystallization process. The optimal condition for precipitation of struvite was determined at pH 9.0, and  $\text{Mg}^{2+}$ :  $\text{NH}_4^+$ :  $\text{PO}_4^{3-}$  ratio of 1.6:1:1.5, and reaction time of 30 minutes, the maximum removal of N and P reached  $98.1 \pm 0.5\%$  and  $98.9 \pm 0.4\%$ , respectively. The highest crystallization mass was obtained at  $14.6 \pm 0.6 \text{ g/l}$ . In addition, characterization of struvite crystal was performed by X-ray diffraction (XRD), morphology, and chemical composition of struvite produced at optimal conditions as analyzed via SEM-EDS. Also, the economic analysis indicated that the struvite crystallization process is cost-feasible and friendly – environmental. These findings illustrated that struvite precipitation is an efficient method for removing both N and P in actual swine wastewater. This paper demonstrates the novel advantages of green technology, contributing to environmental protection towards sustainable development.

**Key words:** Crystallization, Nutrient recovery, Optimal conditions, Struvite, Swine wastewater

### INTRODUCTION

Swine wastewater typically contains a high concentration of nitrogen ( $1257 - 3205 \text{ mg/l}$ ) and phosphorus ( $25 - 182 \text{ mg/l}$ ) that cause eutrophication in the receiving bodies of water such as rivers, streams, lakes, and wetlands (Feng *et al.*, 2020; Shim *et al.*, 2021). In Vietnam, the regulation for livestock wastewater was issued to control the N and P discharge in the environment. On the other hand, N and P are referred to as macronutrients, used as the fertilizer for agricultural activities. The worldwide total N fertilizer consumption has been increased from  $11.3 \times 10^6$  tons in 1961 to  $107.6 \times 10^6$  tons in 2013 (Lu & Tian, 2017). In recent years, global P fertilizers demand is expected to increase rapidly from  $14.5 \times 10^6$  tons in 2005 to  $22 - 27 \times 10^6$  tons in 2050 (Mogollón *et al.*, 2018), whereas the natural sources will severely deplete in the coming decades. Thus, N and P recovery from wastewater sources is

extremely urgent.

Currently, many technologies could be applied to remove N and P from swine wastewater, such as chemical precipitation (Sudiarto *et al.*, 2019), bioremediation (Jiang *et al.*, 2020), physical separation (Sui *et al.*, 2018). However, biological technologies have disadvantages, such as long processing time, large areas, and adaptation period for microorganisms and/or used plant species (Ren *et al.*, 2021). Therefore, aiming to overcome the disadvantages of these above bioremediation technologies, some physical/chemical processes, including struvite precipitation (Kumar & Pal, 2015), ammonia stripping (Zhang *et al.*, 2012), electrodialysis (Ippersiel *et al.*, 2012), reverse osmosis (Lan *et al.*, 2019), and microwave radiation (La *et al.*, 2014) have been suggested to recover and remove N and P in the wastewater. Among these technologies, struvite crystallization has been widely applied thanks to its high removal efficiency for N and P in swine wastewater, short reaction period, and cost-feasibility (Folino *et al.*, 2020; He *et al.*, 2020; Le *et al.*, 2021a; Liu *et al.*, 2011).

Struvite is a crystalline substance involving magnesium, ammonium, and phosphate (e.g.,  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$ ) in equal molar concentrations (Song *et al.*, 2011). It is a high-quality fertilizer due to its slow-release rate of nutrients, which helps to make it friendly environmental (Gong *et al.*, 2018). Struvite crystallization relies on several factors, in which the molar ratio of  $\text{Mg}^{2+}$ :  $\text{NH}_4^+$ :  $\text{PO}_4^{3-}$ , pH value, and reaction time are vital players during the process (Cheng *et al.*, 2019). These factors on struvite precipitation have been examined in previous studies (Nelson *et al.*, 2003; Suzuki *et al.*, 2007). For instance, Bao *et al.* (2011) investigated that the struvite crystal could be reached at pH from 8.0 to 9.0 and Mg:P ratio of 1:2 for nutrient recovery in swine wastewater.

<sup>1</sup> PhD Program in Maritime Science and Technology, National Kaohsiung University of Science and Technology (NKUST), Kaohsiung 81157, Taiwan

<sup>2</sup> Faculty of Environment and Natural Resources, Nong Lam University, Ho Chi Minh City 700000, Vietnam

<sup>3</sup> Research Institute for Sustainable Development, Ho Chi Minh City University of Natural Resources and Environment, Ward 1, Tan Binh District, Ho Chi Minh City 700000, Vietnam

<sup>4\*</sup> Faculty of Chemistry and Environment, Thuyloi University, 175 Tay Son Street, Dong Da District, Hanoi, Vietnam. Corresponding Author (Email: buiquoclap@tlu.edu.vn)

<sup>5\*</sup> Department of Marine Environmental Engineering, National Kaohsiung University of Science and Technology (NKUST), Kaohsiung 81157, Taiwan (Email: ctlin@nkust.edu.tw)

<sup>6</sup> Institute of Tropical Agriculture Kyushu University, Japan

\* Joint corresponding authors (Email: buiquoclap@tlu.edu.vn and ctlin@nkust.edu.tw)

# These authors contributed equally to this work.

Mg content is relatively low in swine wastewater, leading to an appropriate Mg source to precipitate struvite crystals.

According to our preliminary survey in Vietnam, the swine wastewater was seriously polluted with high N and P concentrations. Our raw swine wastewater showed that the nitrogen concentration exceeded 13 times, whereas the P concentration was higher than 20 times compared to Vietnam's Regulation of the Effluent of Livestock (QCVN 62-MT:2016/BTNMT). Thus, struvite crystallization was conducted to investigate the efficiency of the proposed simultaneous removal of the N and P processes. This study aims to investigate the optimized conditions for the simultaneous treatment of both N and P in swine wastewater by using struvite precipitation. Based on batch experiments, the factors, which affect the treatment capacity of N and P, including pH, Mg: N: P molar ratio and reaction time, were investigated. In addition, the characterization of struvite crystal was also examined. The obtained results could recommend a basis for examining optimal parameters to simultaneously treat pollutants (i.e., N and P) to yield multi-nutrient products from swine wastewater as nutrient-abundant solutions.

## MATERIALS AND METHODS

### Raw wastewater and chemicals

Swine wastewater was collected at the anaerobically digested liquor, taken from a pig farm in Tay Ninh province, Vietnam, and kept in the refrigerator at 5°C. Before the experiment, the sieve removed coarse particles and suspended solids (0.45 mm pore size 47 mm diameter, Whatman). The physicochemical properties of the swine wastewater are shown in **Table 1**. In this study, 1.5M  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ; 1.5M  $\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$ ; 5M NaOH of analytical grade were used in each experiment.

**Table 1.** The characteristics of swine wastewater and Vietnam discharge standard limits

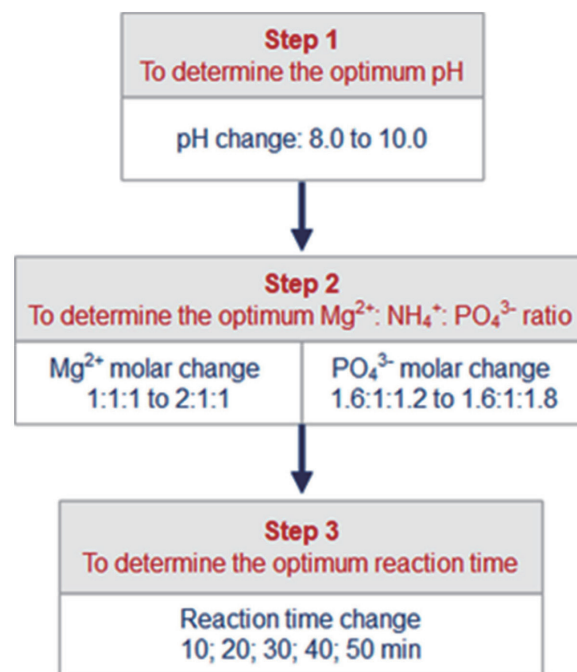
Parameter	Unit	Mean $\pm$ SD (n=3)	Vietnam's discharge standard limits
pH	–	8.3 $\pm$ 0.1	6.0 – 9.0
Temperature	°C	29.0 $\pm$ 1.2	50.0
$\text{NH}_4^+$	mg/l	674.9 $\pm$ 73.0	20.0
$\text{PO}_4^{3-}$	mg/l	149.4 $\pm$ 17.7	4.0
$\text{Ca}^{2+}$	mg/l	29.1 $\pm$ 4.1	N/A
$\text{Mg}^{2+}$	mg/l	113.8 $\pm$ 9.2	N/A

Notes: N/A: Not available, SD: Standard deviation

### Experimental procedure

The schematic diagram of struvite crystallization is shown in **Fig. 1**. Batch experiments of struvite precipitation were examined at different pH values ranging from 8.0 – 10 (at Mg:P ratio of 1, and reaction time of 20 minute), changing Mg:N ratios (1:1.1, 1.2, 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, 1.9, and 2.0); pH = 9, Mg:P ratio of 1:1, reaction time 20 minute), different Mg:P ratios (1.6:1.2,

1.6:1.3, 1.6:1.4, 1.6:1.5, 1.6:1.6, 1.6:1.7, and 1.6:1.8); pH = 9, Mg:N ratio of 1:1, reaction time 20 minute), and reaction time (10, 20, 30, 40, and 50 minute; Mg:P ratio of 1.6; Mg:N ratio of 1.6:1, and pH = 9). For the experiment testing, the concentrations of P and Mg were added to maintain the molar ratio during the process. All these experiments were conducted at room temperature (25°C).



**Fig. 1.** Flowchart of experimentation to select the optimum conditions.

**Fig. 2** shows the struvite crystallization process. In the jar-test experiments, 500 ml actual swine wastewater in each 1000 ml beakers were mixed with salts of 1.5M  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  (Merck, 99%) and 1.5M  $\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$  (Merck, 99%) to ensure the theoretical molar ratio of Mg:N:P of 1:1:1, which was completely mixed using an impeller at 60 rpm. The pH of the solution was adjusted using 5M NaOH (Merck, 99%) and 1M HCl (Merck, 99%). The mixture was stirred at 60 rpm according to the timeline reaction and finally kept for settling for 30 minutes. After each experiment, the supernatant of the effluent was filtered through a membrane filter (0.45  $\mu\text{m}$ ) for component analysis. All experiments were conducted in triplicate.

### Analysis methods

The anion and cation concentration of the influent and effluent levels were determined according to "Standard methods for examining water and wastewater" (Baird, 2017). The pH value was measured by EZDO 8200 portable pH meter (Taiwan). The struvite crystallization was washed with deionized water three times, dried in an oven at 50°C for 24 h, and then characterized by X-ray Diffraction (XRD, DXIII, Rigaku Co., Japan) under the operational condition: CuK $\alpha$  radiation source,  $\lambda = 1.5406 \text{ \AA}$ , scanning rate  $0.06^\circ \text{ s}^{-1}$  at the angle

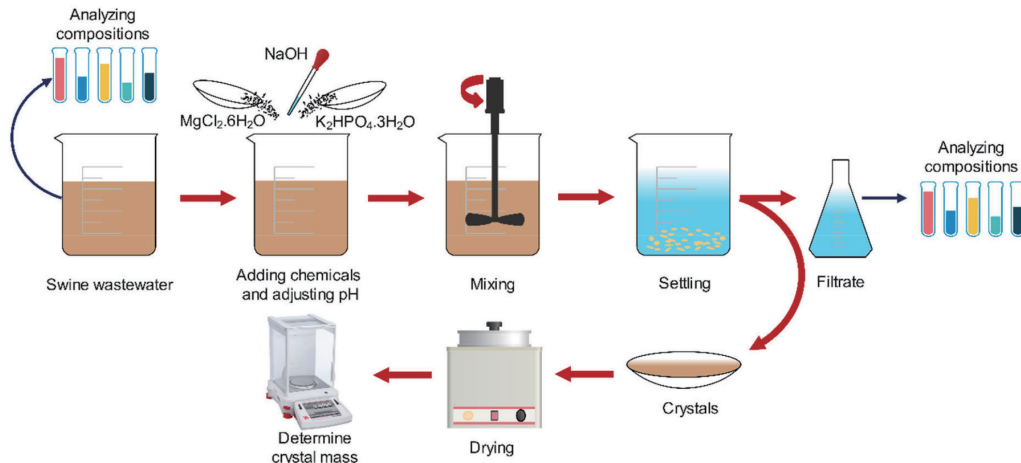


Fig. 2. Diagram of the struvite crystallization process.

of  $10\text{--}70^\circ$  ( $2\theta$ ). The precipitates particles' morphology and surface chemical composition were analyzed using the Scanning Electron Microscopy–Energy Dispersive X-ray Spectroscopy (SEM–EDS, S-4800, HI-9039-0006, Hitachi, Japan). In addition, this research utilized the most common statistical descriptive parameters such as mean and standard deviations ( $\pm$ SD). The data were analyzed by the SPSS 25 software package for Windows (SPSS Inc., Chicago, IL, USA). The removal efficiency ( $H$ , %) of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  were calculated using Equation (1) as follows:

$$H = \frac{C_0 - C}{C_0} \times 100\% \quad (1)$$

Where,  $C_0$  and  $C$  are the initial and final concentration of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  (mg/l) in experiment samples, respectively.

## RESULTS AND DISCUSSION

### Jar-tests for struvite precipitation

#### Effects of pH

The optimal pH for struvite precipitation was investigated through a batch experiment in which the pH range is from 8 to 10. It can be seen from **Fig. 3** that the pH values of the solution have a great significant on  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  removal efficiency. The pH at 10 has the highest  $\text{NH}_4^+$  removal efficiency among the five pH values, up to  $81.5 \pm 3.4\%$ . In contrast, the highest  $\text{PO}_4^{3-}$  removal efficiency at pH 9.0 is up to  $96.2 \pm 0.51\%$ . The  $\text{NH}_4^+$  removal efficiency increases proportionally with the pH value (Le *et al.*, 2020). The difference in the removal efficiency of  $\text{NH}_4^+$  could be consistent with the sequence of pH adjustments in the solution. The previous investigations have illustrated that the removal capacity of  $\text{NH}_4^+$  increases as the pH increases during struvite precipitation (Gong *et al.*, 2018; Kim *et al.*, 2017; Lee *et al.*, 2015; Luo *et al.*, 2019; Siciliano & Rosa, 2014).

On the other hand, when the pH changed from 8.0 to 9.0, the precipitate mass also increased from  $5.48 \pm 1.2\text{ g/l}$  to  $6.58 \pm 1.08\text{ g/l}$ . However, when the pH value was over 9.0, it was observed that the precipitate mass

decreased and reached  $6.17 \pm 1.34\text{ g/l}$  at pH = 10 (**Fig. 3a**). Thus, the results demonstrated that  $\text{NH}_4^+$  treatment efficiency rose with the pH value increase, but the precipitate mass slightly decreased. This was probably due to the dissociation of  $\text{NH}_4^+$ . In the liquid phase, total ammonia–nitrogen (TAN) is in a state of equilibrium between ionized  $\text{NH}_4^+\text{-N}$  and unionized  $\text{NH}_3$  as Equation 2.

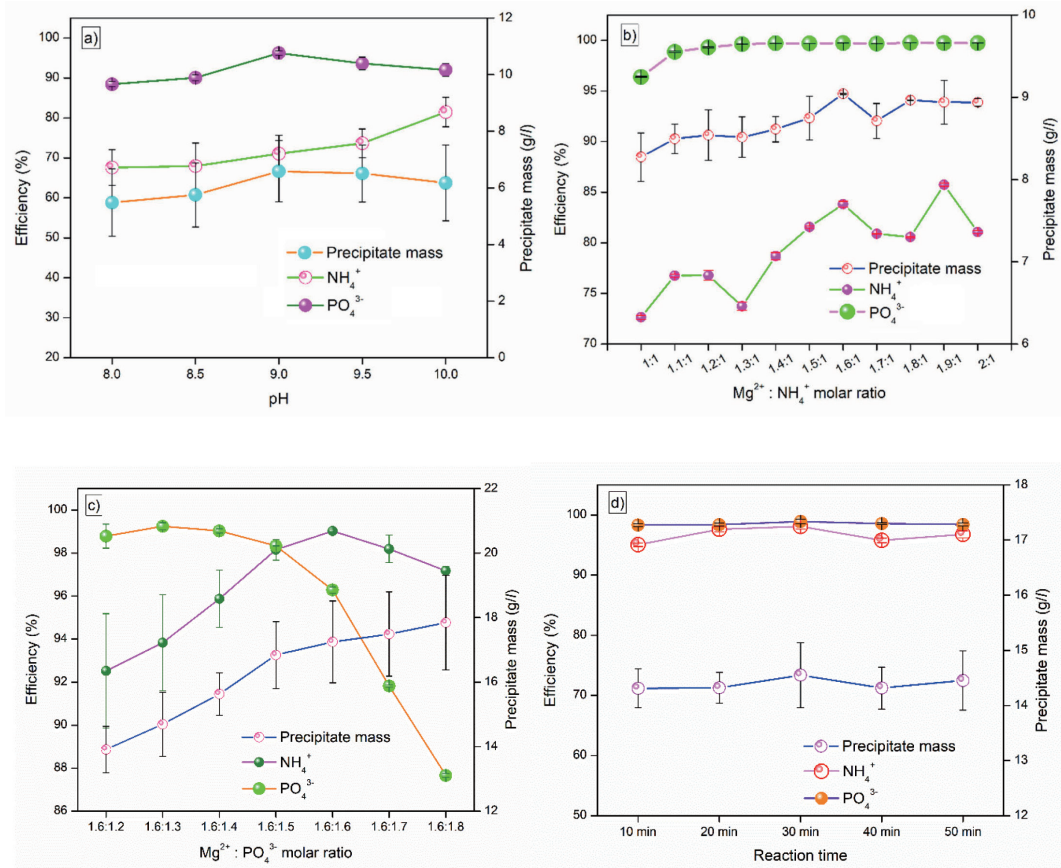


The pH value of the solution influenced this equilibrium. At the pH as alkaline, the unionized fraction increases significantly, and  $\text{NH}_4^+\text{-N}$  is mainly in  $\text{NH}_3$ . Therefore, the struvite crystallization reactions could occur in the wastewater containing  $\text{NH}_4^+\text{-N}$  as a form of  $\text{NH}_4^+$ . Thus, the reaction was difficult to occur, causing the struvite mass to decrease slightly. In addition, the pH value of swine wastewater is a vital parameter for the formation of struvite crystallization, which affects not only the amount of precipitation but also its purity crystallization. Previous studies have reported that a variety of  $\text{Mg}^{2+}$  and  $\text{PO}_4^{3-}$  complex ions patterns in the solution (e.g.,  $\text{MgOH}^+$ ,  $\text{Mg}(\text{OH})_3^-$ ,  $\text{MgH}_2\text{PO}_4^+$ ,  $\text{H}_2\text{PO}_4^-$ , and  $\text{MgPO}_4^-$ ) can be formed when the pH value of the solution is changed (Bouropoulos & Koutsoukos, 2000; Shaddel *et al.*, 2019; Wang *et al.*, 2005; Zhang *et al.*, 2017). Side reactions occur easily at high pH, which reduces  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  reaction. Therefore, pH 9.0 was optimal for struvite precipitation in our experiment.

#### Effects of Mg:N:P molar ratios

Effect of molar ratios (e.g., N/P, Mg/P, etc.) is a critical factor in the struvite crystallization process (Liu *et al.*, 2008; Nelson *et al.*, 2003). Different Mg: N: P molar ratios affected the removal efficiency of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  significantly. To observe the effect of Mg: N: P molar ratio variations on  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  removal efficiency, the experiments conducted at pH 9.0 were chosen in Section "Effects of pH". The  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  removal were improved significantly with an increasing molar of  $\text{Mg}^{2+}$  when the  $\text{NH}_4^+:\text{PO}_4^{3-}$  ratio was fixed at 1:1 (**Fig. 3b**).





**Fig. 3.** Ammonium and phosphate removal, precipitate mass according to optimum operational factors for swine wastewater: (a) pH; (b)  $\text{Mg}^{2+} : \text{NH}_4^+$  molar ratio; (c)  $\text{Mg}^{2+} : \text{PO}_4^{3-}$  molar ratio; (d) reaction time.

However, the removal efficiency of  $\text{NH}_4^+$  decreased when the  $\text{Mg}^{2+}$  molar increased from 1.3 to 1.6. While the removal efficiency of  $\text{PO}_4^{3-}$  was stable and maintained at 99.7%. Therefore, if the molar of  $\text{Mg}^{2+}$  were increased up to a specific value,  $\text{PO}_4^{3-}$  removal would not change. Marti *et al.* (2008) reported that there could be many different kinds of magnesium phosphate precipitates except for struvite at different values of pH, for example,  $\text{Mg}(\text{H}_2\text{PO}_4)_2$ ,  $\text{MgHPO}_4$  or  $\text{Mg}_3(\text{PO}_4)_2$  (Le *et al.*, 2021b). Thus, it could be seen that more Mg does not necessarily mean more struvite. The molar of  $\text{Mg}^{2+}$  chosen for the removal of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  in this experiment is 1.6. Besides, the Mg: N: P molar ratio was kept at 1.6:1:1, the precipitate mass was about  $9.04 \pm 0.007 \text{ g/l}$ , and this value did not illustrate the significant change with increasing  $\text{Mg}^{2+}$  molar.

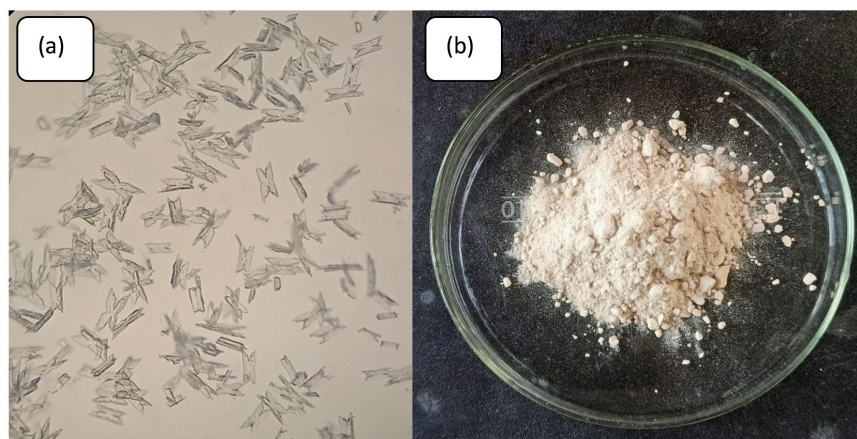
The effect of  $\text{PO}_4^{3-}$  molar variations on removal efficiency of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  was confirmed at a fixed  $\text{Mg}^{2+} : \text{NH}_4^+$  ratio of 1.6:1 (**Fig. 3c**). Higher  $\text{PO}_4^{3-}$  molar improved the removal of  $\text{NH}_4^+$  but when the  $\text{PO}_4^{3-}$  molar increased to 1.5, the removal efficiency of  $\text{PO}_4^{3-}$  tended to decrease. On the contrary, the precipitate mass was raised from  $16.8 \pm 1 \text{ g/l}$  to  $17.8 \pm 1.5 \text{ g/l}$  with the above  $\text{PO}_4^{3-}$  molar augmentation. However, more residual phosphate at higher  $\text{PO}_4^{3-}$  molar (data not shown) was detected in the effluent. Thus, overdosing with the  $\text{PO}_4^{3-}$  level can lead to an adverse process for nutrient products from swine wastewater. The molar of  $\text{PO}_4^{3-}$  chosen

for the removal of ammonium and phosphate is 1.5. Based on the results, we chose the optimum molar ratio of Mg: N: P was 1.6:1:1.5.

#### Effects of reaction time

The period of the reaction among  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  to form struvite has been studied to establish the required optimal time. To observe the influence of reaction time on  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  treatment efficiency, the experiments conducted at pH 9.0 and a molar ratio of 1.6  $\text{Mg}^{2+} : 1 \text{ NH}_4^+ : 1.5 \text{ PO}_4^{3-}$  were chosen in Sections “Effects of pH” and “Effects of Mg:N:P molar ratios”.

**Fig. 3d** shows the variation of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  removal efficiency and the change of precipitate mass with increased reaction time. As the reaction time increased from 10 to 30 minutes, the highest removal efficiency of  $\text{NH}_4^+$  ( $98.1 \pm 0.5\%$ ) and  $\text{PO}_4^{3-}$  ( $98.9 \pm 0.4\%$ ) reached at 30 minutes, and then slightly decreased with the reaction time increased to 50 minutes. It can be observed that the reaction time had a negligible effect on the removal of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$ , but had a light effect on the size of the struvite crystal. It was found from the results reported by Stratful *et al.* (2001) that when the reaction time was increasing (from 1 to 180 minutes), the size of crystals increased (from 0.1 to 3 mm). Although the increasing reaction time could increase the size of the struvite crystal, the excessing reaction time may break the crystallization system, leading to a



**Fig. 4.** The precipitation of (a) crystallization and (b) powder struvite product.

decrease not only the capability of struvite crystallization but also  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  removal. In this study, the highest precipitate mass obtained was  $14.6 \pm 0.6 \text{ g/l}$ , and the mass of precipitate did not change significantly when increasing the reaction time from 10 to 50 minutes. Similarly, Wang *et al.* (2019) investigated the reaction time from 20 to 50 minutes during the struvite crystallization process. These results illustrated that the highest recovery of  $\text{NH}_4^+$  (97.3%) and  $\text{PO}_4^{3-}$  (98.2%) was obtained at 30 minutes. Therefore, the main factors such as pH, molar ratio, and retention time play a critical role that significantly effects on the recovery of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  from wastewater.

### Struvite crystallization

#### *Precipitation of struvite compounds*

According to Bhuiyan *et al.* (2007), the solubility product of struvite ( $K_{sp}$ ) was reported equal to  $10^{-13.26}$ , and the struvite precipitation can efficiently be achieved to remove nitrogen and phosphorus in wastewater. Furthermore, the simultaneous treatment of both ammonium ( $\text{NH}_4^+$ ) and phosphate ( $\text{PO}_4^{3-}$ ) in presenting ammonium from swine wastewater demonstrated efficient struvite precipitation. In this study, struvite product is a white crystal formed in light alkali conditions and slightly soluble in water (**Fig. 4**).

By checking the element's content (O, P, and Mg) showed the purity of recovered struvite compounds was high and was not impacted by  $\text{Ca}^{2+}$  precipitation. Previous studies reported that  $\text{Ca}^{2+}$  could affect striate crystallization due to the integrated  $\text{PO}_4^{3-}$  (Hao *et al.*, 2008; Liu & Wang, 2019; Shen *et al.*, 2011; Song *et al.*, 2007). In the condition of Ca:Mg molar ratios over 0.5,  $\text{Ca}^{2+}$  could inhibit and affect the crystallization reaction as well as its purity (Song *et al.*, 2007). In the present study, however, the Ca: Mg molar ratios varied from 0.28 to 0.67 (at the optimal condition of 0.45), showing a satisfying recovery of the good crystals. Thus, at optimal conditions such as pH = 9.0 and molar ratio of  $1.6 \text{ Mg}^{2+}: 1 \text{ NH}_4^+: 1.5 \text{ PO}_4^{3-}$ , reaction time was determined at 30 minutes in the batch tests. Thus,  $\text{NH}_4^+$  can be combined with other ions such as  $\text{Mg}^{2+}$  and  $\text{PO}_4^{3-}$  to form struvite

crystals. According to the crystallization process, precipitate mass obtained the highest value up to  $14.6 \pm 0.6 \text{ g/l}$ . In addition, the struvite precipitation removed about  $98.1 \pm 0.5\%$   $\text{NH}_4^+$  and  $98.9 \pm 0.4\%$   $\text{PO}_4^{3-}$  in swine wastewater at optimal conditions. Our results showed that over 98% of the  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  are recovered as struvite during the crystallization process. Therefore, it could be seen that the struvite crystallization can efficiently remove both nitrogen and phosphorus from swine wastewater.

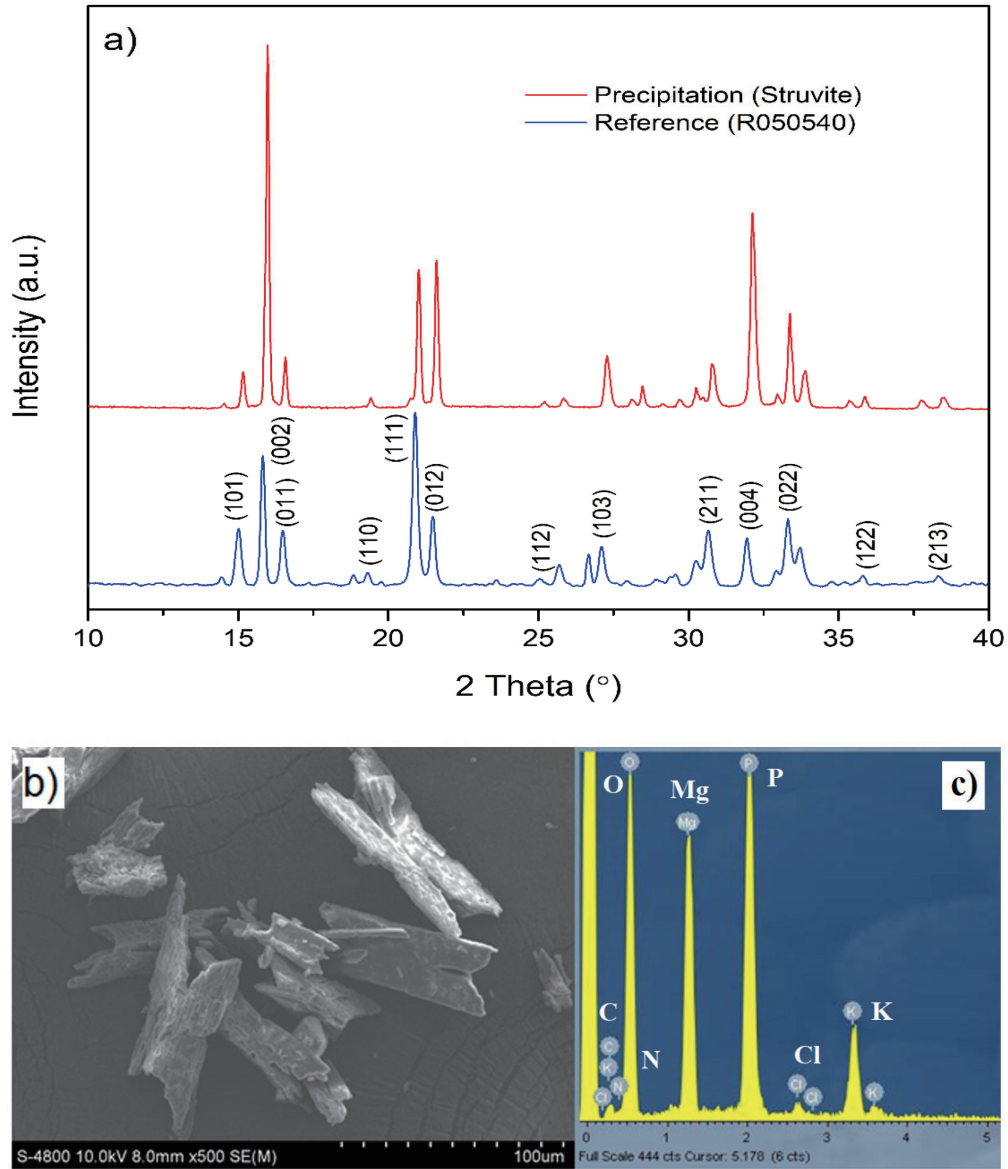
#### *Characterization of struvite precipitates*

The XRD spectrum shown in **Fig. 5a** confirms the crystal phase of particles was struvite with  $2\theta$  peaks at  $15.24^\circ$ ,  $16.03^\circ$ ,  $25.09^\circ$ ,  $31.76^\circ$ , and  $38.09^\circ$  for facets of (101), (002), (112), (211) and (213), respectively, and shows the characteristic peak of the K-struvite crystal ( $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$ ), implying the co-precipitation of K struvite. Therefore, the cation of  $\text{K}^+$  could have come from the high K-concentration ( $>300 \text{ ppm}$ ) of actual swine wastewater that is a minor impurity phase.

**Fig. 5b** shows the SEM images of struvite crystallization collected under optimal conditions (pH = 9.0, Mg:N:P of 1.6:1:1.5, and retention time 30 minute). The struvite precipitates have a butterfly shape with an average diameter of 0.7 mm. Under high magnification, the nuclei had needles, irregular and coarse shape structures.

The EDS spectrum of **Fig. 5c** shows that the main surface elements of particle were Cl, Mg, P, O, N, and K and no Na peak, implying that no magnesium sodium phosphate ( $\text{NaMgPO}_4$ ) was formed, and the atomic percentages of Mg, N, P, and O were 8.59%, 11.84%, 9.80%, and 53.65%, respectively. These results indicated a similar composition to struvite stoichiometry. Furthermore, the atomic ratio of potassium (K) accounts for 2.67%, suggesting that magnesium potassium phosphate ( $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$ ) might co-precipitate with struvite.

In addition, the characterization of the struvite (e.g., size, crystal type) has been examined and compared the results to previous studies (**Table 2**). As a result, it could be seen that the main element content, size, and



**Fig. 5.** (a) XRD patterns, (b) SEM images [ $\times 500$  magnification, scale bar =  $100\mu\text{m}$ ], and (c) EDS spectrum of struvite crystals product. (pH = 9.0,  $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$  of 1.6:1:1.5, and 30 min).

**Table 2.** An overview of struvite crystallization from swine wastewater

No.	Additional chemicals	P/Mg molar ratio	pH range	Time	Reactor type	Major elements	Size (length)	Crystal type	References
1	$\text{MgCl}_2$ , $\text{K}_2\text{HPO}_4$	1:1.1	9.0	30 min	Batch	O (49.6%) P (18.5%) Mg (12.8%)	$100\mu\text{m}$	Irregular shaped, coarse	This study
2	$\text{MgCl}_2$ , $\text{K}_2\text{HPO}_4$	1:1.2	8.0–9.5	N/A	Batch	N/A	N/A	N/A	(Lee <i>et al.</i> , 2009)
3	$\text{MgCl}_2$	1: 1.2	8.0–9.0	5 weeks	Continuous flow	N/A	N/A	Irregular shaped, coarse	(Rahman <i>et al.</i> , 2011)
4	$\text{Mg}^{2+}$	1: 0.8–1.0	7.8–8.9	4 hours	Continuous flow	N/A	N/A	Irregular shape crystals	(Liu <i>et al.</i> , 2011)
5	$\text{Mg}^{2+}$	N/A	8.0–9.5	40 min	Batch	Mg, P, O	$10\text{--}30\mu\text{m}$	Tiny needle-shaped, irregular crystals	(Huang <i>et al.</i> , 2014)
6	$\text{MgCl}_2$	1:1.2	8.5	N/A	Batch	P (28.6%) Mg (20.9%)	$\sim 50\mu\text{m}$	Regulated crystals, coarse	(Wu <i>et al.</i> , 2018)
7	$\text{Mg}^{2+}$ , $\text{PO}_4^{3-}$	1:1.2	9.5	N/A	Batch	Mg, N, P	N/A	N/A	(Cai <i>et al.</i> , 2020)

Note: N/A – Not available.

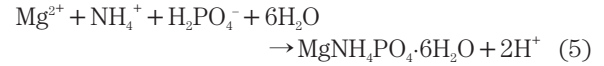
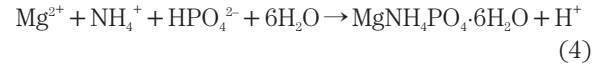
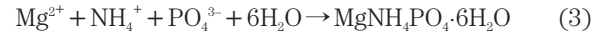


crystal shapes were similar to the struvite crystallization process in the previous studies. Thus, the struvite forms of the precipitates were indicated as the well-detected crystals.

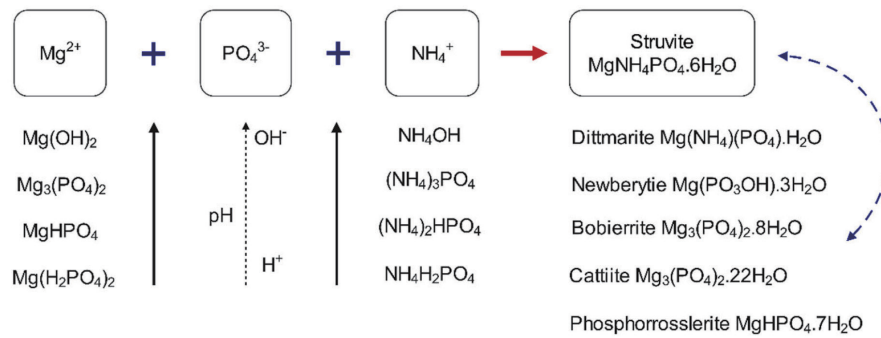
#### Struvite crystallization mechanism

According to the diffusion reaction theory, the crystallization mechanism illustrated that crystal clusters and solute molecules are transported from the fluid phase into the solid surface and precipitating struvite compounds (Myerson, 2002). Also, Rahman *et al.* (2014) showed that the struvite crystal process occurs through two chemical phases: nucleation (crystal birth) and crystal growth. Crystallization has occurred when mixing molecules such as  $Mg^{2+}$ ,  $NH_4^+$ , and  $PO_4^{3-}$  each

other and its contact in clusters at a favorable pH to crystal growth. Thus, struvite is formed when  $Mg^{2+}$ ,  $NH_4^+$ , and  $PO_4^{3-}$  obtained equal molar amounts as the following chemical reactions:



Where the ionic interactions among  $Mg^{2+}$  and  $PO_4^{3-}$  complexes (i.e.,  $PO_4^{3-}$ ,  $HPO_4^{2-}$ ,  $H_2PO_4^-$ ) are seen as the cause of the formation of  $MgPO_4$ ,  $MgHPO_4$  and  $MgH_2PO_4$ . In



**Fig. 6.** Interaction of  $Mg^{2+}$ ,  $NH_4^+$ ,  $PO_4^{3-}$  in struvite crystallization.

**Table 3.** Summary of technologies for removal of swine wastewater nutrients

Technology	Type of reactor	Added chemicals	pH value	HRT/SRT	Removal efficiency	Cost/Benefit	References
Struvite	Batch	$MgCl_2 \cdot 6H_2O$ $K_2HPO_4 \cdot 3H_2O$	9.0	30 min	$98.1 \pm 0.5\%$ ( $NH_4^+$ ) $98.9 \pm 0.4\%$ ( $PO_4^{3-}$ )	0.20 USD/m <sup>3</sup>	This research
Struvite	Batch	$Mg^{2+}$	8.0–9.5	40 min	82.0% ( $NH_4^+$ ) 98.0% ( $PO_4^{3-}$ )	−3.65 USD/m <sup>3</sup>	(Huang <i>et al.</i> , 2014)
Microwave radiation	Microwave oven	None	8.0 – 12.0	5 min	83.1% ( $NH_4^+$ )	N/A	(La <i>et al.</i> , 2014)
Electrocoagulation	Fe electrode	None	8.2 – 9.2	61.8 – 118.2 min	55.0 – 91.0% (TP)	N/A	(Mores <i>et al.</i> , 2016)
Struvite	Batch	$MgCl_2$	8.5	N/A	93.0% ( $NH_4^+$ ) 99.0% ( $PO_4^{3-}$ )	+1.35 USD/m <sup>3</sup>	(Wu <i>et al.</i> , 2018)
Constructed wetlands	SFCW	None	N/A	11 days (HRT)	87.7 – 97.9% ( $NH_4^+$ )	N/A	(Luo <i>et al.</i> , 2018)
Phyto-technologies	Floating aquatic plants	None	5.9 – 7.0	N/A	63.2% (TN) 36.2% (TP)	N/A	(Sudiarto <i>et al.</i> , 2019)
Membrane bioreactor	SMBR	None	7.5	20 days (SRT) 33–51 hours (HRT)	83.0–97.0% ( $NH_4^+$ )	N/A	(Xu <i>et al.</i> , 2019)
Spraying technology	Experimental device	None	>7.0	N/A	88.4% ( $NH_4^+$ )	N/A	(Cao <i>et al.</i> , 2019)
Microalgae cultivation	Batch culture	None	9.0	N/A	45.0% ( $NH_4^+$ ) 70.0% ( $PO_4^{3-}$ )	N/A	(Gracida-Valdepeña <i>et al.</i> , 2020)
Aerobic granular sludge	AGSBR	None	N/A	4 hours	81.2% ( $NH_4^+$ ) 97.4% (TP)	N/A	(Wang <i>et al.</i> , 2020)
Bioelectrochemical systems	MFC	None	$7.5 \pm 0.15$	24 hours (HRT)	$66.6 \pm 1.4\%$ ( $NH_4^+$ ) $32.1 \pm 2.8\%$ ( $PO_4^{3-}$ )	N/A	(Cheng <i>et al.</i> , 2021)
Biochar	Continuous flow	None	9.0	3 hours	$79.0 \pm 6.1\%$ (COD) $84.0 \pm 2.5\%$ (BOD <sub>5</sub> )	N/A	(Lap <i>et al.</i> , 2021)

Notes: N/A: Not available, (−): Cost, (+): Benefit, TP: Total phosphorus, TN: Total nitrogen, COD: Chemical Oxygen Demand, BOD<sub>5</sub>: Biological Oxygen Demand



addition, the reaction kinetics of struvite crystallization which could be observed by the interaction of  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  ions are shown in **Fig. 6**. In these, the struvite crystallization process relies on many important factors such as concentrations of  $\text{PO}_4^{3-}$ ,  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , pH value, and Mg/P ratio, etc. (Desmidt *et al.*, 2013; Kozik *et al.*, 2013; Song *et al.*, 2007).

In this study, experimental results showed the highly  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  recovered efficiency at pH = 9.0. After conducting  $\text{Mg}^{2+}$  and  $\text{PO}_4^{3-}$  changes, we chose the optimal molar ratio of  $\text{Mg}^{2+}$ :  $\text{NH}_4^+$ :  $\text{PO}_4^{3-}$  was 1.6:1:1.5. According to the crystallization process, precipitate mass obtained the highest value up to  $14.6 \pm 0.6 \text{ g/l}$  from swine wastewater. Thus, it could be confirmed that the mechanism of struvite formation is strongly dependent on the interaction of  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  ions and pH value. In addition, based on struvite precipitation, crystals could be considered to recover organic nutrients (e.g., TOC,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$ ) in swine wastewater (Suzuki *et al.*, 2007).

### Comparison of recovery performance of different technologies

As shown in **Table 3**, the struvite technology performed better than the different technologies for the removal of nutrients, and the removal rate can reach as high as 98% in the struvite process.  $\text{NH}_4^+$  is completely removed via chemical crystallization, and the product is applied as a fertilizer for agriculture (Kim *et al.*, 2017). In contrast, the biological methods remove  $\text{NH}_4^+$  from wastewater by metabolism, accumulation in biomass, and cell synthesis (Metcalf *et al.*, 2014). Besides, the treatment time of biological methods is also longer than that of the struvite technology. On the other hand, physicochemical processes can achieve high  $\text{NH}_4^+$  removal efficiency and fast reaction time such as microwave, but the mechanism of  $\text{NH}_3$  removal was suggested as the formation of molecular  $\text{NH}_3$  and the subsequent evaporation of  $\text{NH}_3$  by microwave radiation (Lin *et al.*, 2009). Regarding physicochemical methods such as microwave radian, spraying, and electrocoagulation are difficult to apply in full scale because it requires much energy to operate. This suggests that struvite technology is one of the current and potential treatment technologies for swine wastewater in developing countries.

### CONCLUSIONS

The recovery of nitrogen and phosphorus from the actual swine wastewater was conducted by struvite crystallization. Under pH of 9.0, Mg:N:P of 1.6:1:1.5, and reaction time of 30 minute, the treatment efficiencies of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  were  $98.1 \pm 0.5\%$  and  $98.9 \pm 0.4\%$ , respectively. The formed particles (with an average size of 0.70 mm) have proven to be a struvite phase of  $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$  with a precipitated mass value up to  $14.6 \pm 0.6 \text{ g/l}$ . The particle form of the precipitates was confirmed by XRD analysis and indicated that their struvite crystals could be further recycled as a slow-release fertilizer for agricultural practice in developing countries

(e.g., Vietnam, China, India). Also, cost-analysis (0.20 USD/ $\text{m}^3$ ) indicated that struvite crystallization is a great potential technology for nutrient recovery from swine wastewater.

### AUTHOR CONTRIBUTIONS

Nguyen Minh KY and Huynh Tan NHUT: conceptualization, investigation, methodology, draft manuscript. Nguyen Trung HIEP and Nguyen Tri Quang HUNG: investigation, methodology, reviewing and editing. Bui Quoc LAP and Chitsan Lin: conceptualization, supervision of methodology and experimental activities, revision of the manuscript and played the role of the corresponding author. Tran Thi Minh TAM: methodology. Akinori OZAKI advised methodology and supported to the manuscript arrangement and revision.

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