



Removal of Ammonium and Phosphate from Synthetic Wastewater of Complex Fertilizer Industry Through Struvite Crystallization Process

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ABSTRACT

The complex fertilizer industry produces wastewater that contributes to water pollution because it contains high organic nitrogen in the form of urea which can be hydrolyzed to ammonium using the urease enzyme and high levels of phosphate and ammonium concentrations. Struvite precipitation is an effective method for removing and recovering ammonium and phosphate from wastewater. This study aimed to determine the effect of aeration and the enzyme urease in removing ammonium and phosphate in complex fertilizer synthetic wastewater through struvite precipitation. Struvite precipitation was carried out in a batch reactor with a working volume of 0.5 L with variations in aeration rate, aeration time, and the addition of urease enzyme from Jack bean peas (*Canavalia ensiformis*). Residual ammonium and phosphate levels were analyzed, and struvite crystal formation (MAP) was determined using Scanning Electron Microscope (SEM) and X-ray diffraction (XRD). The results showed that the aeration reactor could form struvite crystals and transform the ammonium and phosphate content in the synthetic wastewater of complex fertilizers. The removal of ammonium with a molar ratio of $[Mg^{2+}] : [NH_4^+] : [PO_4^{3-}]$ 1:2:1 reached 61-77% at high aeration rates because much ammonia was released into the air. The phosphate removal reached 99%. The urease enzyme was proven to hydrolyze urea into ammonium, increase the pH value, and affect the shape of the resulting struvite crystals. The precipitate product obtained was struvite crystals, which SEM-EDX and XRD analysis confirmed.

1. INTRODUCTION

The complex fertilizer industry is an industry that produces various fertilizer products, including urea, ammonium sulfate, phosphate fertilizer, NPK, and others. In addition to producing fertilizers, the complex fertilizer industry generally produces non-fertilizer by-products such as sulfuric acid, phosphoric acid, and ammonia. However, the production of complex fertilizers produces wastewater which contributes to the pollution of water bodies because

it contains high levels of phosphate and ammonium concentrations. This waste will pollute the environment if it is directly disposed of without processing (Ikhlas, 2017)

Complex fertilizer wastewater also contains high concentrations of organic nitrogen as indicated by high TKN (Total Kjeldahl Nitrogen). TKN is the sum of ammonium nitrogen and organic nitrogen. The organic nitrogen contained in the complex fertilizer waste is urea, so it is necessary to hydrolyze urea. Urea hydrolysis aims to

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convert urea into ammonium. Urea hydrolysis is known to be carried out enzymatically using the enzyme urease from jack bean urease which works at a temperature of 25°C with a pH range of 4-9 (Fidaleo & Lavecchia, 2003). Enzymatic hydrolysis of urea occurs according to the following reaction:



This reaction produces carbamates which are unstable compounds and will decompose spontaneously into carbonic acid and ammonia. Ammonia production raises the pH during the reaction (Kabdasli et al., 2006). One method extensively studied for removing high concentrations of ammonium and phosphate from wastewater is the struvite precipitation method or Magnesium Ammonium Phosphate (MAP) (Yu et al., 2012). Struvite precipitation produces crystals with an orthorhombic structure formed from magnesium, ammonium, and phosphate in the same molar concentration.

Struvite is an ideal alternative fertilizer because it has the characteristics of an odorless, clean crystal that releases nutrients slowly to the soil and has low solubility, thus avoiding the eutrophication problems that may arise from other phosphorus fertilizers (Kataki et al., 2016). The most popular method of producing struvite from sewage is the chemical precipitation method by adding a magnesium salt and adjusting the pH to a base (Kruk et al., 2014). The reaction for the formation of struvite can be expressed in the following equation (Doyle & Parsons, 2002):



The formation of struvite crystals occurs in 3 states, namely unsaturated, metastable, and supersaturated (Ye et al., 2014). This struvite crystallization method has been widely used in various types of wastewater, such as urine wastewater (Aguado et al., 2019), (X. Liu et al., 2014), palm oil mill effluent (Ngatiman et al., 2021), swine wastewater (Cai et al., 2020), municipal sewage sludge (Zin et al., 2021), dairy wastewater (Numviyimana et al., 2020).

Several studies have shown that the struvite precipitation method can efficiently remove ammonium and phosphate from wastewater. Formation of struvite from

wastewater with high ammonium and phosphate content can remove up to 90% phosphate content (Aguado et al., 2019; Chripim et al., 2019; Xavier et al., 2014) and ammonium content up to more than 80% (Daegi Kim et al., 2017) and (Siciliano et al., 2013). Thus, complex fertilizer wastewater with high ammonium and phosphate content can be processed by forming struvite as a new product line of a slow-released fertilizer.

Recent studies have not studied how adding urease enzymes and aeration affects struvite formation from complex fertilizer wastewater. Therefore, this study aims to remove high ammonium and phosphate from complex fertilizer wastewater by forming struvite crystals. The effect of adding urease enzymes to wastewater and the presence of aeration must be done to determine the amount of struvite yield and the form of struvite obtained.

2. METHODS

This research consisted of preparing synthetic wastewater; reactor preparation; struvite precipitation operation; and the analysis stage of the residual wastewater content and the characteristics of the structural crystal deposits formed.

2.1. Preparation of Synthetic Wastewater

The wastewater used is a synthetic waste. The composition of the waste has a high level of ammonium. Ammonium chloride (NH_4Cl) was used as a source of ammonium. Monopotassium phosphate (KH_2PO_4) was used as a source of phosphate, and magnesium was added as a precipitate to form struvite (MAP). The stoichiometric $[\text{Mg}^{2+}] : [\text{PO}_4^{3-}]$ molar ratio to form struvite is 1:1. This wastewater also has a high content of organic nitrogen in the form of urea. This urea cannot form struvite, so it needs to be hydrolyzed to be converted into ammonium with the enzyme urease. KF was used as a source of fluoride.

In ammonium base wastewater, the ammonium contained is 7705 ppm, and there is no phosphate content. It shows that the molar ratio of $[\text{NH}_4^+] : [\text{PO}_4^{3-}]$ is 1:0, so adding wastewater from a phosphate base is necessary to achieve a more balanced molar ratio. Phosphate source from phosphate base wastewater contained 624 ppm ammonium and 2264 ppm phosphate.

The ammonium and phosphate base wastewater were mixed until the $[\text{NH}_4^+]:[\text{PO}_4^{3-}]$ did not differ much. Based on the calculation of the phosphate requirement to achieve the best $[\text{NH}_4^+]:[\text{PO}_4^{3-}]$ molar ratio, 0.1 L of ammonium-based wastewater and 3.9 L of phosphate-based wastewater are required to achieve a waste volume of 4 L.

The results of mixing ammonium-base wastewater and phosphate-based wastewater obtained ammonium and phosphate concentrations of 801.2 mg/L and 2207.4 mg/L with a molar ratio of $[\text{NH}_4^+]:[\text{PO}_4^{3-}]$ 2:1. The addition of the required amount of magnesium is adjusted to the molar ratio of PO_4^{3-} which is 1: 1. So the ratio of the molar ratio $[\text{NH}_4^+]:[\text{Mg}^{2+}]:[\text{PO}_4^{3-}]$ is 2: 1: 1.

The characteristics of the treated wastewater (mixed between ammonium- and phosphate-based wastewater) used in this study can be seen in Table 1.

2.2. Reactor Preparation

The struvite precipitation device uses a bulk reactor tube with a volume of 1 litre. Wastewater is introduced by adding magnesium according to the calculated molar ratio. The reactor was fitted with a pond pump to perform aeration instead of stirring to increase the pH. The pH value is measured periodically until the specified reaction time is complete.

The aeration rate is varied by adjusting it using a rotameter which functions as a flow meter. The pump's airflow is regulated using a valve to achieve the desired aeration rate. Perforated air stones with many holes are installed at the bottom of the reactor as aeration air holes. Increasing the pH using aeration will be faster using a lot of aerated air holes compared to fewer aerated air holes (Radev et al., 2015). The reactor struvite precipitation apparatus is shown in Figure 1.

2.3. Struvite Precipitation Operations

The experiment was initiated by adding synthetic waste into the reaction zone struvite reactor and magnesium with a predetermined molar ratio of Mg:P 1:1. Aeration is turned on to act as a mixer and pH regulator. The aeration rate was varied into 2 levels, 3.4 L/L/min and 16.6 L/L/min. The length of aeration was varied into 2 levels, 2 and 4

hours. The research was also carried out with the addition of the enzyme urease isolated from Jack bean peas. The addition of urease enzyme is expected to hydrolyze urea into ammonia. The addition of the enzyme was carried out in as much as 50 mL.

2.4. Analysis

The analysis carried out in this study included an analysis of the concentration of residual wastewater resulting from struvite precipitation and characterization of the morphology and crystallinity of the struvite precipitate formed. The analysis of residual wastewater includes the analysis of NH_4 content using the phenate method. PO_4 analysis using the ascorbic acid method. F analysis with SPADNS method.

Morphological analysis of struvite deposits was carried out by Scanning Electron Microscopy (SEM) and struvite crystallinity by X-Ray Diffractometer (XRD).

Table 1. Characteristics of Mixed Wastewater

Parameters	Number	Unit
Phosphate	801,2	mg/L
Ammonium	2207,4	Mg/L
Fluoride	147	mg/L
Urea	3083	mg/L

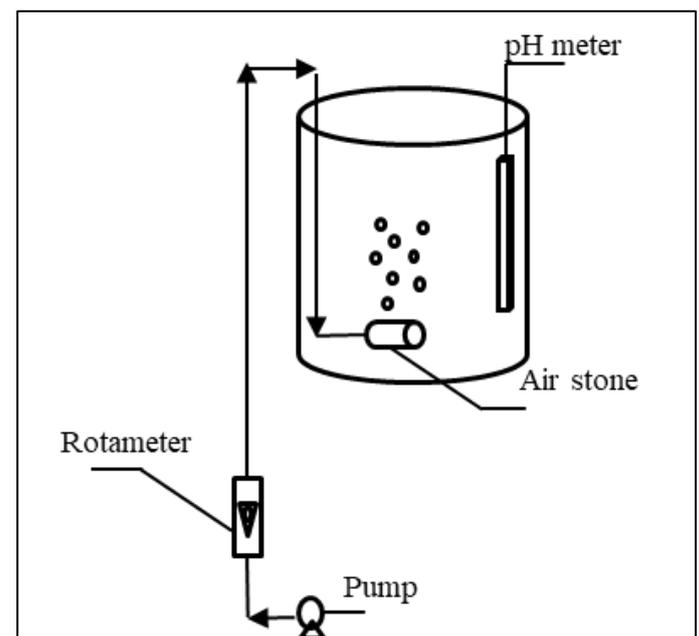


Figure 1. Reactor Struvite Precipitation

3. RESULT AND DISCUSSION

3.1. Struvite Crystals and Ammonium & Phosphate Removal

Aeration carried out in the experiment helped in the process of hydrolysis of the urease enzyme. It is also a substitute for stirring in the formation of struvite. The struvite crystal deposit formed after being observed differs based on the variations. Figure 2 shows the results of the observed struvite crystal deposition. The use of aeration with aeration for 2 hours resulted in lower struvite crystal deposits when compared to aeration for 4 hours. Aeration for 2 hours resulted in a maximum of 2.36 grams of struvite crystals, while aeration for 4 hours resulted in 2.72 grams of struvite crystals.

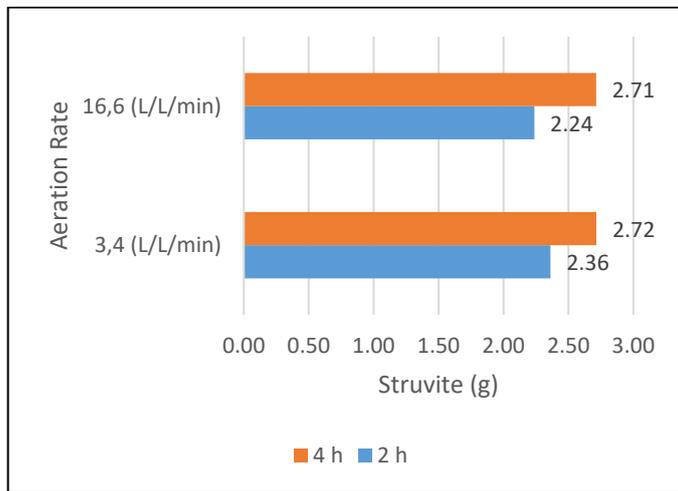


Figure 2. Aeration to Struvite Crystal Comparison

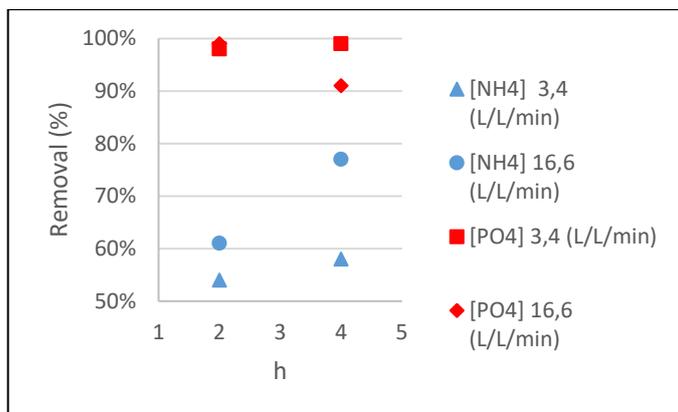


Figure 3. NH₄-N and PO₄-P Removal (%) on Aeration

The results of the struvite crystal precipitate formed are also different when compared based on the aeration rate used. At 2 hours of aeration using an aeration rate of 3.4 L/L/min, it produced a higher struvite deposit of 2.36 grams, while at an aeration rate of 16.6 L/L/min, it produced a lower struvite deposit of 2.24 grams. As for the aeration duration of 4 hours, the resulting struvite crystal deposition results were not significantly different. More prolonged aeration treatment has been shown to affect turbulence factors such as the stirring process.

The removal of ammonium (NH₄-N) and phosphate (PO₄-P) can be seen in Figure 3. Figure 3 shows the results of NH₄-N removal based on variations in aeration rate and aeration time. The use of aeration for 2 hours can remove different NH₄-N compared to 4 hours. NH₄-N removal for 2 hours ranged from 54-61%. The removal of NH₄-N for 4 hours reached 58-77%. The removal of NH₄-N with an aeration duration of 2 hours was lower than that of 4 hours.

When viewed from the variation of the aeration rate, the removal of NH₄-N also looks different. At an aeration rate of 3.4 L/L/min, NH₄-N removal for 2 hours can remove as much as 54%. While for 4 hours, it can remove as much as 58%. The difference looks overly significant. It differs from removing NH₄-N at an aeration rate of 16.6 L/L/min. Removing NH₄-N at an aeration rate of 16.6 L/L/min for 2 hours can remove NH₄-N as much as 61% and 77% for 4 hours. The result of NH₄-N removal at an aeration rate of 3.4 L/L/min was lower than the aeration rate of 16.6 L/L/min.

Figure 3 shows the results of PO₄-P removal based on variations in aeration rate and aeration time. The use of aeration for 2 hours can eliminate 99% of PO₄-P. The removal of PO₄-P with an aeration duration of 4 hours was 91-99%. The PO₄-P allowance was not significantly different. It is influenced by the amount of Mg:PO₄-P 1:1 molar ratio, so the amount of removal produced is high.

When viewed from the variation of the aeration rate used, the resulting PO₄-P removal did not show any difference. At an aeration rate of 3.4 L/L/min, 99% can be removed with an aeration period of 2 hours, as 99% removal

can be achieved with an aeration period of 4 hours. As for the aeration rate of 16.6 L/L/min, 99% of $\text{PO}_4\text{-P}$ can be removed within 2 hours and 91% with 4 hours of aeration. In the removal results with an aeration period of 4 hours and an aeration rate of 16.6 L/L/min, there may be an error in the measurement so that it is only 91% compared to an aeration rate of 3.4 L/L/min.

The amount of $\text{PO}_4\text{-P}$ removal in the aeration process completely formed struvite crystal deposits. The lower molar content of $\text{PO}_4\text{-P}$, compared to $\text{NH}_4\text{-N}$ molar, makes the amount of $\text{PO}_4\text{-P}$ removal high because almost all of the $\text{PO}_4\text{-P}$ becomes struvite crystal deposits.

3.2. SEM and XRD Results

Identification of struvite was carried out by Scanning Electron Microscope (SEM) test to determine the morphological characteristics and crystal structure. Energy dispersive Xray Spectroscopy (EDX) was performed to determine the chemical components contained in the crystals. X-ray Diffraction (XRD) to identify the crystal

phase. SEM analysis was performed at 1000x and 2000x magnification.

There were 3 samples analyzed; the first one was the ratio of $[\text{Mg}^{2+}] : [\text{NH}_4^+] : [\text{PO}_4^{3-}]$ 1:2:1 with 3,4 L/L/min aeration for 2 hours. This sample has a high amount of precipitation. The second sample was a sample with a ratio of $[\text{Mg}^{2+}] : [\text{NH}_4^+] : [\text{PO}_4^{3-}]$ 1:2:1 with 16.6 L/L/min aerations for 2 hours. This sample has the highest residual ammonium content. Then the third sample was a sample with a ratio of $[\text{Mg}^{2+}] : [\text{NH}_4^+] : [\text{PO}_4^{3-}]$ 1:2:1, which was aerated for 4 hours with 3.4 L/L/min and added enzymes. The results of the SEM-EDX analysis are shown in Figure 4. The results of the EDX microanalysis are shown in Figures 5 and 6.

Figure 4 (a) shows the SEM results of the sample ratio $[\text{Mg}^{2+}] : [\text{NH}_4^+] : [\text{PO}_4^{3-}]$ 1:2:1 with aeration 16,6 L/L/min for 2 hours. The results show the shape of struvite crystals that are long and tapered. The surface of the crystal also looks not rough because of the absence of impurity minerals that allow them to stick to the crystal.

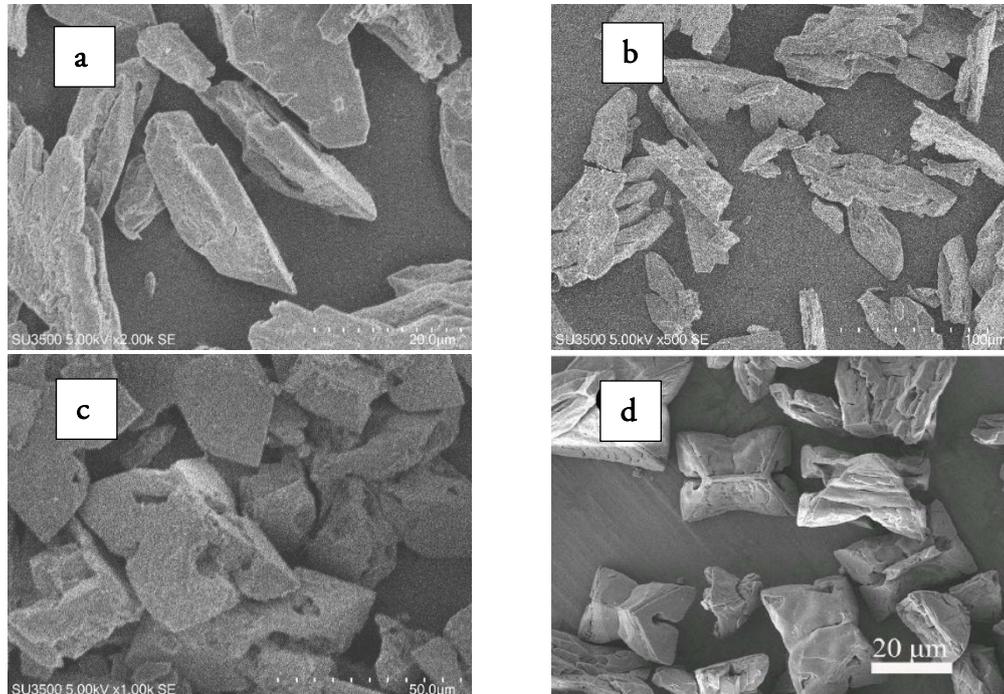


Figure 4. (a) Aeration 16,6 L/L/min for 2 hours, (b) Aeration 3,4 L/L/min for 2 hours, (c) Addition of Enzymes, (d) Biogenic Struvite Sample (Li & Zhou, 2015)

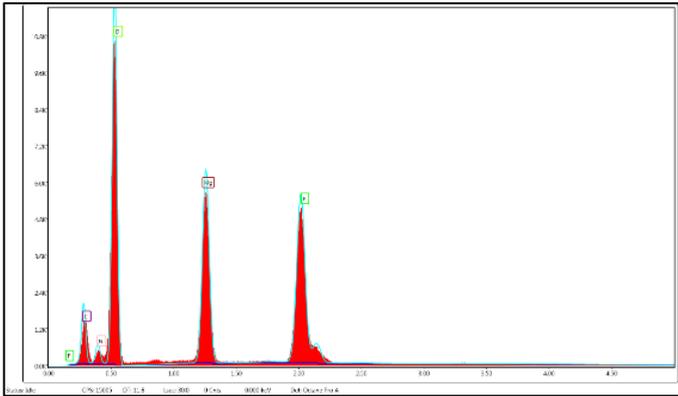


Figure 5. Microanalysis EDX [Mg²⁺] : [NH₄⁺] : [PO₄³⁻] 1:2:1

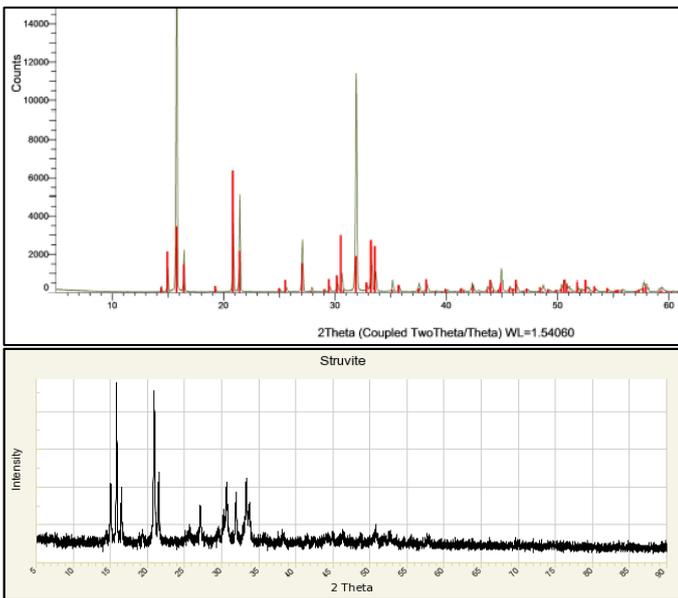


Figure 6. XRD Sample Ratio [Mg²⁺] : [NH₄⁺] : [PO₄³⁻] 1:2:1 with aeration 16,6 L/L/min for 2 hours and XRD struvite (RRUFF, 2019)

Table 2. The Chemical Component of Struvite Crystals in The Sample with The Ratio [Mg²⁺] : [NH₄⁺] : [PO₄³⁻] 1:2:1

Element	Weight %	Atomic %
C K	18.64	25.45
N K	5.81	6.8
O K	52.37	53.67
Mg K	12.53	8.45
P K	10.65	5.64

Figure 4 (b) shows the SEM results of the sample ratio [Mg²⁺] : [NH₄⁺] : [PO₄³⁻] 1:2:1 with aeration 3,4 L/L/min for 2 hours. The crystal results showed that the crystals were long and tapered, similar to those in Figure 4b. The surface of the crystal also looks not rough because of the absence of mineral impurities.

The morphology of struvite crystals can be identified based on the growth process of struvite, and their shape can vary (prism type, pyramid type, coffin-like type, needle, or feather-like type) (B. Liu et al., 2013). Pure struvite crystals with equimolar ratios will form a pointed white orthorhombic crystal structure (Daekeun Kim et al., 2009).

Different things are shown in Figure 4 (c), namely the sample with the addition of enzyme and the ratio of [Mg²⁺] : [NH₄⁺] : [PO₄³⁻] 1:2:1 with aeration of 3.4 L/L/min 4 hours. Crystals look more stacked and tapered. The different crystal forms in this sample were caused by adding enzymes to hydrolyze organic nitrogen. Recent studies have shown that the protein selectively binds to the surface of struvite crystals and produces an arrow-shaped morphology, which then evolves into an unusual X-shaped tabular structure as struvite formation progresses. It is the morphological structure of biogenic struvite (Li & Zhou, 2015). Figure 4 (d) is a picture of the morphological structure of biogenic struvite. Even so, the crystal surface on the sample looks not rough because there are no impurity minerals.

Figure 5 shows the results of EDX microanalysis of the sample ratio [Mg²⁺] : [NH₄⁺] : [PO₄³⁻] 1:2:1, and Table 2 shows the chemical components contained in struvite crystals. EDX microanalysis confirmed that the struvite formed consisted of struvite-forming compounds, namely Mg, N, and P. The percentage of chemical components and each component's mass are shown in the table. Figure 5 shows that the components that form the crystals of struvite are magnesium and phosphate, which are dominant in the absence of impurity ions.

XRD analysis required the degree of crystallinity of struvite precipitates. The XRD analysis test can be seen in Figure 6. The results of the test show that the precipitate

formed is struvite crystals when viewed from the peak point that is formed following the peak line in the literature of struvite XRD results. Although the peak line shown is higher than in the literature (RRUFF, 2019), the position of the peak line formed lies at the same theta degree.

3.3. Overall Discussion

The struvite formed from the molar ratio $[Mg^{2+}] : [NH_4^+] : [PO_4^{3-}]$ can be predicted by modelling using the Visual Minteq 3.1 application. Visual Minteq 3.1 software can display the prediction of the amount of component concentration that is deposited at a pH value of 8.5 which is used in this study. Table 3 shows the deposited components based on the Visual Minteq 3.1 software.

Based on Table 3, the pH value data used to predict the precipitated component in Visual Minteq 3.1 Software is pH 8.5. The precipitated magnesium content reaches 98%, while the dissolved reaches 1.99%. The precipitated phosphate content reached 97.96%, while the dissolved reached 2.05%. The ammonium content, which has an excess molar ratio compared to magnesium and phosphate, impacts the amount of concentration being deposited which is lower than that of magnesium and phosphate. The deposited ammonium content is 51.27%, and the remaining 48.73% is still dissolved.

Visual Minteq 3.1 software has predicted the concentration and percentage of components that can be formed based on variations in the molar ratio $[Mg^{2+}] : [NH_4^+] : [PO_4^{3-}]$ 1:2:1. Prediction results using visual minteq 3.1 software showed that the amount of ammonium remaining was lower than the results of the research conducted.

The results showed that the ammonium concentration removed was 54% and 61%, respectively. These results indicate that the ammonium content removed is higher than the predicted results with visual minteq 3.1 software. The results of ammonium removal that occurred in the results showed that ammonium was not only precipitated into struvite crystals but some were released into the air. A nitrogen mass balance then carries this out to determine how much ammonia is released into the air. Figure 7 shows the mass balance of nitrogen flow.

Different aeration rates affect the removal of ammonia. In Figure 7, the aeration rate of 16.6 L/L/min can release the amount of ammonia into the air up to 12.26 mmol/L. It was higher than the aeration rate of 3.4 L/L/min, which caused 7.82 mmol/L ammonia to be released into the air. The ammonium content in struvite is also different. The aeration rate of 16.6 L/L/min contains ammonium in the form of struvite up to 18.3 mmol/L, while the aeration rate of 3.4 L/L/min reaches 19.5 mmol/L.

The amount of ammonium concentration at the starting point, end point, and struvite precipitated crystals is the result of the measurements that have been made. The amount of ammonium concentration released into the air results from calculating the difference between the initial, final, and ammonium concentrations precipitated into struvite crystals. The amount of TKN, ammonium, and organic nitrogen, has decreased due to ammonium being reduced to struvite and released into the air.

Table 3. Precipitated components based on Visual Minteq 3.1 Software

Component	Total Dissolved (mol/L)	% Dissolved	Total Precipitated (mol/L)	% Precipitated
Mg^{2+}	4,62E-04	1,99	2,28E-02	98,01
NH_4^+	2,16E-03	48,73	2,28E-02	51,27
PO_4^{3-}	4,75E-04	2,05	2,28E-02	97,96

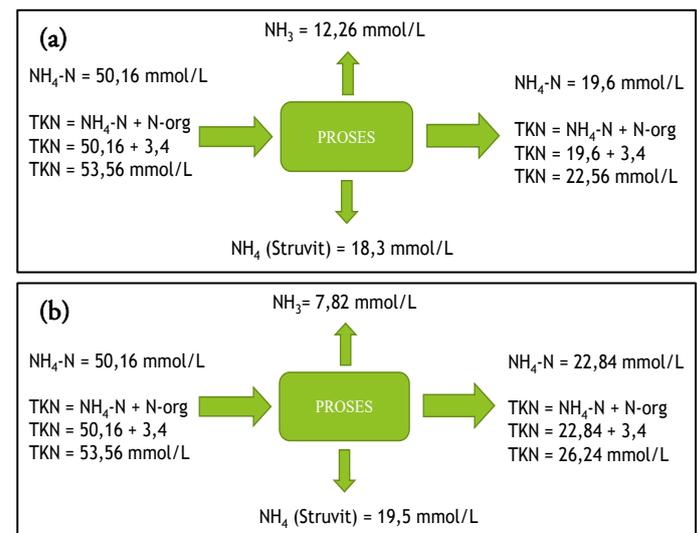


Figure 7. Total Nitrogen Balance, (a) Aeration Rate 16,6 L/L/min; (b) Aeration Rate 3,4 L/L/min

The result of enzymatic urea hydrolysis that produces ammonium has an impact on increasing the ammonium concentration to phosphate molar ratio. The increasing concentration of ammonium molar ratio did not impact increasing ammonium removal results. However, the ammonium removal efficiency will decrease further when the molar ratio of ammonium is higher than the molar ratio of phosphate and magnesium (Ikhlas & Warmadewanthi, 2017).

4. CONCLUSION

The results of this study and discussion indicate that a high aeration rate with an aeration period of 4 hours can remove up to 77% more ammonium concentration as a result of ammonia released into the air. It suggests that struvite precipitation with a higher molar ratio of ammonium than magnesium and phosphate using aeration as a mixing device is not favourable because it leads to air pollution (bad odor, etc). It differs from the prediction results using visual minteq 3.1 software which does not predict the effect of aeration. At the same time, the phosphate removal reached 99% formed into struvite. The results of SEM-EDX and XRD confirmed that the precipitate formed was struvite crystals. The addition of enzymes seemed to affect the morphology of the structures formed. It also differed from those of struvite crystals without the addition of enzymes.

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