



Recovery of Slow-Release Fertilizer from Tofu Wastewater via Magnesium Ammonium Phosphate (Struvite) Precipitation: Effect of pH and Molar Ratio

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Abstract

Tofu wastewater contains high concentrations of phosphate (PO_4^{3-}) and ammonium (NH_4^+), which can cause eutrophication if discharged without proper treatment. One promising method for nutrient recovery and wastewater remediation is struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) precipitation, which simultaneously removes phosphate and produces a slow-release fertilizer. This study investigates the effect of pH variation (8, 9, and 10) and molar ratios of $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ (1:1:1, 4:1:1, and 1:4:1) on phosphate removal efficiency, product yield, and morphological characteristics of the formed crystals. Experiments were conducted with a reaction time of 60 minutes under controlled pH conditions. The results revealed that both pH and molar ratio significantly affected the phosphate precipitation process. The highest phosphate removal efficiency of 89.94% was obtained at pH 9 with a 4:1:1 molar ratio, indicating that excess Mg^{2+} under moderately alkaline conditions favored struvite formation. In contrast, the highest calculated product yield was observed at pH 8, showing that the condition giving maximum mass-based recovery was not identical to the condition giving maximum phosphate removal efficiency. At pH 10, phosphate removal efficiency decreased, most likely due to the formation of competing $\text{Mg}(\text{OH})_2$ precipitates, which reduced the availability of free Mg^{2+} for struvite crystallization. Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) analysis showed that the struvite crystals exhibited block-like morphologies with irregular surfaces and an average size of 20–50 μm . Although nutrient-release kinetics were not directly measured in this study, the observed crystal size may influence dissolution behavior based on the surface-area effect reported in the literature, supporting the potential application of the recovered struvite as a slow-release fertilizer. Overall, the findings demonstrate that pH 9 and the molar ratio of 4:1:1 represent the optimal conditions for struvite production from tofu wastewater, offering an environmentally sustainable approach to nutrient recovery and contributing to circular economy practices in agriculture.

1. Introduction

The tofu industry plays an important role in the food processing sector in Indonesia [1]. However, alongside its economic benefits, tofu production generates substantial amounts of organic waste, particularly as liquid effluent

[2]. This wastewater, which originates from soybean washing, soaking, boiling, and curdling processes, is rich in organic matter, including proteins and carbohydrates [3, 4]. If discharged directly into the environment without treatment, it can significantly degrade water quality by reducing dissolved oxygen levels, promoting microbial

growth, and releasing unpleasant odors, ultimately threatening aquatic ecosystems [5, 6]. Environmental concerns regarding tofu wastewater have encouraged the exploration of waste-to-resource approaches to mitigate pollution while recovering value-added products [7]. One such approach is the use of wastewater nutrients to produce Slow-Release Fertilizer (SRF) [8].

SRF technology allows for the controlled release of nutrients, aligning with the nutrient uptake pattern of plants, and thereby increasing fertilizer use efficiency, reducing nutrient leaching, and minimizing the risk of environmental contamination [9, 10]. The development of SRF using nutrient-rich tofu wastewater represents a sustainable solution to address both agricultural and environmental concerns [11, 12]. An efficient method for reducing phosphate and ammonium levels in wastewater while generating an eco-friendly and cost-effective slow-release fertilizer (SRF) is through the formation of struvite via the precipitation process [13, 14]. Struvite has the ability to gradually release nutrients, thus efficiently promoting plant growth while minimizing soil and water pollution [15, 16]. Struvite formation is greatly influenced by several operating parameters, including solution pH and the molar ratio of Mg^{2+} , NH_4^+ , and PO_4^{3-} ions [17].

The pH value determines the solubility of ions and the stability of the precipitate, while the molar ratio determines the reaction balance among the three main components that form struvite [18]. It is important to distinguish phosphate removal from struvite formation because the decrease in dissolved phosphate does not always indicate that phosphate is recovered exclusively as $MgNH_4PO_4 \cdot 6H_2O$. Depending on pH and wastewater composition, phosphate may also precipitate as other magnesium or calcium phosphate phases, particularly in the presence of Ca^{2+} , high alkalinity, or competing ions. Therefore, wastewater matrix characteristics can shift the optimum precipitation conditions and affect the purity of the recovered struvite product.

Several studies have shown that a moderately alkaline pH (8–9) and excess Mg^{2+} ions can increase the efficiency of phosphate precipitation, but the optimal conditions can vary with waste characteristics and system ion concentration [19, 20]. Unlike synthetic wastewater, which usually has a more tightly controlled ionic composition, tofu wastewater contains substantial amounts of organic matter, including proteins, lipids, carbohydrates, and colloidal particles. These organic constituents may influence struvite crystallization by complexing Mg^{2+} ions, reducing the availability of free magnesium for $MgNH_4PO_4 \cdot 6H_2O$ formation, or by adsorbing onto the surface of growing nuclei and inhibiting crystal growth.

In addition, suspended and colloidal organic matter may interfere with mass transfer and produce irregular crystal surfaces, resulting in different precipitation behavior compared with synthetic wastewater systems. Therefore, optimization using real tofu wastewater is necessary because the presence of organic compounds can alter nucleation, growth kinetics, and the purity of the recovered struvite product. Thus, in processing tofu

liquid waste, these parameters need to be evaluated to determine the most efficient reaction conditions for removing phosphate while maximizing struvite fertilizer formation. This study was conducted to investigate the effects of pH variation and the $Mg^{2+}:NH_4^+:PO_4^{3-}$ molar ratio on the efficiency of phosphate removal, yield, and characteristics such as morphology and crystallinity of the struvite product formed.

2. Experimental

2.1. Materials

Tofu wastewater was collected from a tofu factory located in Krueng Geukuh Subdistrict, North Aceh Regency, Indonesia. The chemicals used in this study included magnesium chloride ($MgCl_2$, 99%, Merck), ammonium chloride (NH_4Cl , 99.5%, analytical grade, Sigma-Aldrich), potassium hydroxide (KOH, 85%, analytical grade, Merck), sulfuric acid (H_2SO_4 , 95%, analytical grade, Merck), phenolphthalein indicator (99%, indicator grade, Sigma-Aldrich), and laboratory-grade distilled water.

2.2. Experiment

The initial phosphate concentration in tofu wastewater was determined using a UV-Vis spectrophotometer based on the molybdenum blue colorimetric method. After reagent addition and color development, the absorbance was measured at 880 nm. A calibration curve was prepared using standard phosphate solutions in the range of 0–20 mg/L. Based on this analysis, the initial phosphate concentration of the tofu wastewater was 18 mg/L. All phosphate concentrations in this study are reported as mg/L PO_4^{3-} , not as PO_4 -P or total phosphorus.

The initial NH_4^+ concentration naturally present in the tofu wastewater was also determined and included in the stoichiometric calculation; therefore, the amount of NH_4Cl added was adjusted by considering the baseline NH_4^+ already present in the wastewater to obtain the target $Mg^{2+}:NH_4^+:PO_4^{3-}$ molar ratios of 1:1:1, 4:1:1, and 1:4:1. The phosphate from the wastewater was then reacted with $MgCl_2$ and NH_4Cl solutions. In this study, varying concentrations of these two compounds were used to determine their effect on crystal formation and reaction pH. The reaction was carried out by adding 250 mL of wastewater to an Erlenmeyer flask, then adding $MgCl_2$ and NH_4Cl solutions at $Mg:NH_4:PO_4$ ratios of 1:1:1, 4:1:1, and 1:4:1. KOH solution was added to maintain the pH at 8, 9, and 10. The mixture was stirred using a motorized stirrer at 120 rpm for 60 minutes.

The mixture was filtered to obtain crystals resulting from the precipitation reaction from the solution and other components. The crystals are then dried at 50°C to remove any remaining moisture content. During the kinetic experiment, 5 mL aliquots were withdrawn every 10 minutes for 60 minutes from the initial 250 mL reaction volume. The withdrawn volume was not replaced. Since the samples were taken from a well-mixed suspension and phosphate removal efficiency was calculated from concentration values rather than from a total mass balance, no volume correction was applied.

Nevertheless, the cumulative sampled volume was approximately 30 mL, or about 12% of the initial volume, and this limitation has been acknowledged in the experimental description. Phosphate removal efficiency and product yield were calculated using Equations (1) and (2), respectively. In Equation (1), C_0 and C_f represent the initial and final phosphate concentrations, respectively. Product yield was calculated as the ratio between the experimentally recovered dry precipitate mass and the theoretical struvite mass calculated from the limiting reagent based on the stoichiometry of $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ formation. In Equation (2), m_p is the total mass of MgCl_2 and NH_4Cl added as precursor materials.

$$\text{Efficiency (\%)} = \frac{C_0 - C_f}{C_0} \times 100 \quad (1)$$

$$\text{Yield (\%)} = \frac{m_p}{m_p} \times 100 \quad (2)$$

3. Results and Discussion

3.1. The Effect of pH, Molar Ratio, and Reaction Time on the Decrease in Phosphate Concentration

Based on observations across all molar ratio variations, the phosphate concentration in the solution decreased as the reaction time increased from 0 to 60 minutes. This behavior indicates that the phosphate precipitation reaction proceeds rapidly at the initial stage, then slows due to depletion of available phosphate ions or attainment of equilibrium. The quick precipitation reaction in the early phase and the drop in phosphate content at all pH changes demonstrate that the process taking place is the precipitation of dissolved phosphate. The phosphate concentration decreases rapidly at first due to the high initial ion activity and fast nucleation, then slows as reactants are depleted and equilibrium is established [21].

The effect of pH and reaction time on phosphate concentration at different $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratios is shown in Figure 1. Among the tested conditions, pH 9 consistently produced the lowest residual phosphate

concentration throughout the reaction, indicating the highest phosphate removal efficiency. This trend is attributed to the pH-dependent solubility and supersaturation of struvite. Moderately alkaline conditions (approximately pH 8.5–9) favor struvite precipitation by minimizing its solubility while providing sufficient supersaturation for nucleation and crystal growth. At pH 8, phosphate removal was lower because the driving force for struvite formation was less favorable. In contrast, increasing the pH to 10 reduced phosphate removal, likely due to the conversion of NH_4^+ to NH_3 under highly alkaline conditions and the formation of competing magnesium precipitates, both of which decrease the supersaturation necessary for $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ crystallization.

All things considered, the reduction in phosphate concentration shows how successful the precipitation process is at a 1:1:1 molar ratio. The pH 9 condition yields the best results for rapidly and steadily lowering the phosphate concentration, with an ideal reaction time of 40–60 minutes, at which point the phosphate concentration achieves its lowest value and exhibits stability. The superior phosphate removal efficiency observed at pH 9 is attributed to the optimal balance of ion availability and struvite solubility, which promotes rapid nucleation and stable precipitation compared to the less favorable conditions at pH 8 and 10 [22, 23].

In general, all ratios exhibited a decreasing trend in phosphate concentration over the 0–60-minute reaction period, indicating that phosphate removal progressed continuously throughout the process. At the 1:1:1 molar ratio, the initial phosphate concentration of 17.76 mg/L gradually decreased to 4.76 mg/L after 60 minutes. The reduction occurred at a relatively slower rate compared to the other ratios, suggesting that a stoichiometrically balanced composition of reactants may not provide sufficient excess ions to accelerate phosphate precipitation effectively.

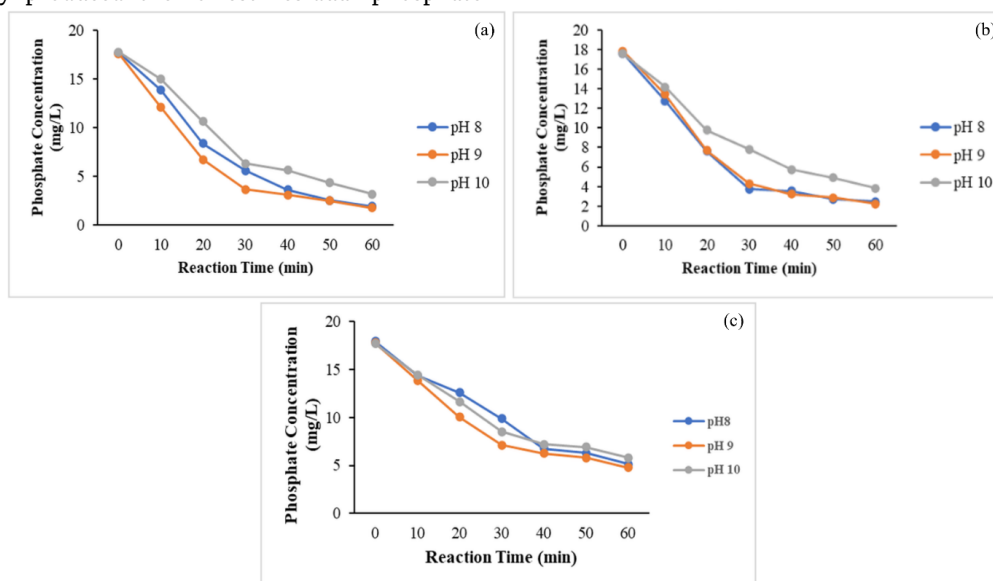


Figure 1. Effect of pH and reaction time on residual phosphate concentration at different $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratios: (a) 1:1:1, (b) 4:1:1, and (c) 1:4:1

In contrast, the 4:1:1 molar ratio demonstrated a more rapid and significant decrease in phosphate concentration. The initial value of 17.59 mg/L dropped sharply to 1.77 mg/L after 60 minutes, indicating that the higher proportion of Mg^{2+} ions substantially enhanced the rate of phosphate removal. This improvement is likely attributed to the increased number of available active sites for phosphate binding and the greater potential for forming insoluble magnesium phosphate species. The 1:4:1 ratio also showed relatively high phosphate removal efficiency, with phosphate concentration decreasing from 17.88 mg/L to 2.24 mg/L within 60 minutes. Although this ratio was effective, its removal rate was slightly slower than that observed at the 4:1:1 ratio. This suggests that while an excess of NH_4^+ ions can still facilitate MAP formation to some extent, the presence of additional Mg^{2+} ions plays a more critical role in accelerating the precipitation process and achieving lower residual phosphate concentrations [24, 25].

3.2. Comparison of Phosphate Removal Efficiency at Different Molar Ratios and pH

Figure 2 presents a comparative summary of phosphate removal efficiency at different solution pH values and $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratios. The results show that phosphate removal efficiency increased when the molar ratio was changed from 1:1:1 to 4:1:1, indicating that Mg^{2+} enrichment enhanced phosphate precipitation. However, increasing the NH_4^+ proportion to obtain the 1:4:1 ratio slightly reduced the removal efficiency compared with the 4:1:1 condition, suggesting that excess NH_4^+ did not further promote struvite formation and may have interfered with precipitate nucleation and crystal growth.

Among all tested conditions, the combination of pH 9 and a 4:1:1 molar ratio produced the highest phosphate removal efficiency of 89.94%, demonstrating optimal conditions for MAP crystallization and phosphate elimination from the solution. The 1:4:1 ratio exhibited lower efficiency, particularly at pH 10 (78.26%), reinforcing the notion that excessive NH_4^+ and highly alkaline conditions negatively impact MAP formation. Even though phosphate removal slightly improved at pH 8 (85.92%) and pH 9 (87.47%), these values remained below the performance achieved by the 4:1:1 system. Under these conditions, Mg^{2+} availability is maximal, providing ideal conditions for MAP crystallization, while excessive NH_4^+ or overly alkaline conditions disrupt the precipitation equilibrium and reduce efficiency [24].

The decrease in phosphate removal efficiency at pH 10 is attributed to less favorable struvite crystallization under highly alkaline conditions. At this pH, the $\text{NH}_4^+/\text{NH}_3$ equilibrium shifts toward NH_3 , reducing the availability of NH_4^+ required for $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ formation and lowering struvite supersaturation [18, 22, 23, 24]. In parallel, excess OH^- can favor the precipitation of magnesium-based competing phases, such as $\text{Mg}(\text{OH})_2$ or magnesium phosphate, which consume free Mg^{2+} and limit its availability for struvite crystallization [19, 20, 26].

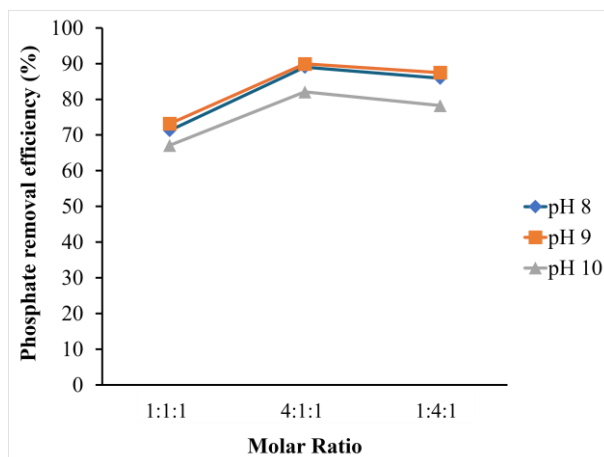


Figure 2. Effect of solution pH and molar ratio on phosphate removal

3.3. Effect of pH and Molar Ratio on Fertilizer Formation and Product Yield

As shown in Figure 3(a), pH 9 produced the highest product weight across the tested molar ratios, indicating greater recovery of the precipitated product. This suggests that moderately alkaline conditions favored the formation of magnesium ammonium phosphate by providing a suitable ionic strength for crystal growth. However, Figure 3(b) shows that the highest product yield (%) was achieved at pH 8, particularly at the 1:1:1 molar ratio. This discrepancy indicates that the condition that produced the greatest precipitate mass did not necessarily yield the highest normalized yield. Therefore, product weight and product yield should be considered complementary indicators of precipitation performance.

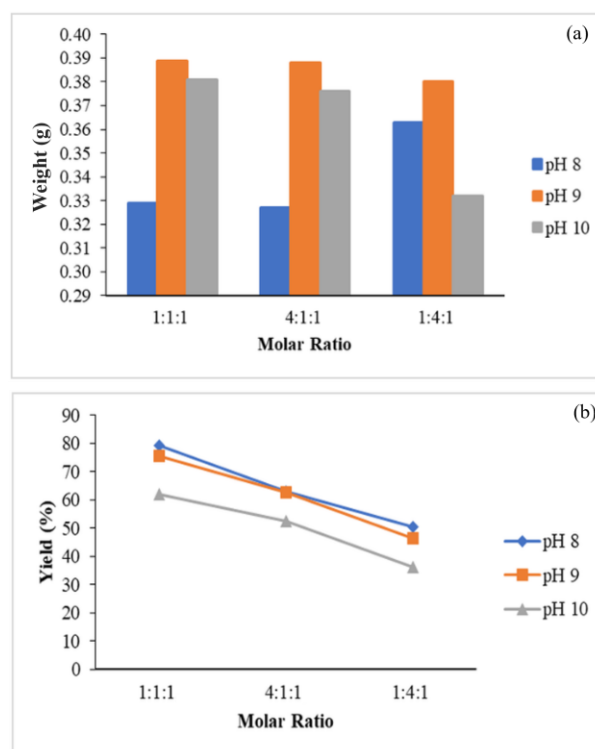


Figure 3. Effect of pH and $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratio on product generation: (a) product weight and (b) product yield

The results presented in Figure 3 reveal that both parameters significantly affect the quantity and quality of the precipitated product, identified as magnesium ammonium phosphate hexahydrate ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$). The difference between product weight and calculated yield may be related to the balance between absolute precipitate formation and reagent utilization. At pH 9, struvite precipitation was more favorable because the solution condition promoted lower struvite solubility and stronger supersaturation, resulting in higher phosphate removal and greater product mass. In contrast, the higher calculated yield at pH 8 suggests more efficient conversion of the initial raw material, although the absolute product mass and phosphate removal efficiency were lower than those observed at pH 9. At pH 10, both product formation and yield decreased, most likely because highly alkaline conditions may shift the $\text{NH}_4^+/\text{NH}_3$ equilibrium and promote competing magnesium-based precipitates, thereby reducing the availability of Mg^{2+} and NH_4^+ for struvite crystallization.

The enhanced product formation at pH 9 can be attributed to the favorable balance between phosphate ion availability and MAP solubility, which promotes efficient nucleation and crystal growth [27]. Although pH 10 also resulted in substantial product formation, the yield was slightly lower than at pH 9. This reduction is associated with the consumption of free Mg^{2+} under highly alkaline conditions through the possible precipitation of brucite ($\text{Mg}(\text{OH})_2$) and other magnesium phosphate phases. These competing precipitates can reduce the availability of Mg^{2+} for the crystallization of $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$, thereby decreasing phosphate removal efficiency and product formation at pH 10 [28].

As a consequence, excessive alkalinity reduces the efficiency of phosphate precipitation and the overall mass of the resulting fertilizer. Conversely, pH 8 produced the smallest amount of fertilizer, suggesting that relatively low alkalinity limits the deprotonation of phosphate species and inhibits effective crystal nucleation [21]. These findings clearly indicate that mildly alkaline conditions are favorable for MAP formation, whereas pH values that are too low or too high diminish product yield.

With respect to molar ratios, the results demonstrate that both 1:1:1 and 4:1:1 ratios yielded relatively higher fertilizer yields than 1:4:1, particularly at pH 9 and 10. At the 4:1:1 ratio, the fertilizer mass increased significantly, indicating that excess Mg^{2+} ions enhance precipitation by providing additional active sites for phosphate binding and crystal growth. However, when the ratio was altered to 1:4:1, the product yield decreased noticeably, especially at pH 10. This observation suggests that excessive NH_4^+ ions may interfere with the precipitation process, possibly by shifting the ionic equilibrium or competing for coordination with phosphate ions [29]. Interestingly, the yield at 4:1:1 was not dramatically higher than that of

1:1:1, implying that beyond a certain threshold, increasing Mg^{2+} concentration does not proportionally increase fertilizer production unless other reactants are present in compatible proportions [30].

The increase in fertilizer yield observed between pH 8 and pH 9 is likely due to enhanced ionic activity in the solution, which facilitates the aggregation of ions into crystalline structures resembling salts, thereby increasing the mass of precipitates. In contrast, the decline in product yield at pH 10 can be attributed to the formation of $\text{Mg}(\text{OH})_2$, which competes with MAP formation and reduces the availability of Mg^{2+} for phosphate binding. Consequently, the combination of pH 9 and the 4:1:1 molar ratio provides the most favorable conditions for MAP synthesis, balancing ion availability, precipitation kinetics, and product crystallinity.

The relationship between pH and $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratio on product yield is presented in Figure 4. The results reveal that both parameters significantly influence the extent of product formation, with distinct trends observed across the various molar ratios and pH levels tested. At the 1:1:1 molar ratio, the highest yield was obtained, particularly at pH 8 and pH 9, suggesting that a stoichiometrically balanced composition of Mg^{2+} , NH_4^+ , and PO_4^{3-} promotes optimal fertilizer formation. Under these conditions, the reaction proceeds efficiently with minimal excess of any ion, resulting in higher conversion and stable crystal growth [31].

When the ratio was increased to 4:1:1, the yield began to decline slightly. This reduction is likely due to an excess of Mg^{2+} ions that did not fully participate in the reaction, resulting in incomplete phosphate utilization and reduced precipitation efficiency. A more pronounced decrease in yield was observed at the 1:4:1 ratio, which can be attributed to excessive concentrations of NH_4^+ ions. The surplus ammonium ions do not significantly enhance MAP formation and may even interfere with phosphate precipitation by shifting the ionic equilibrium or inhibiting crystal nucleation [32]. Consequently, the imbalance in ion availability under this ratio limits the efficiency of the precipitation process [33].

In terms of pH, the highest yield was observed at pH 8, indicating that this slightly alkaline condition promotes the formation of a more stable, compact fertilizer product. At pH 9, the yield was slightly lower but remained relatively high, suggesting that precipitation still occurred effectively. In contrast, pH 10 produced the lowest yield across all molar ratios. This outcome is most likely due to the formation of $\text{Mg}(\text{OH})_2$ under highly alkaline conditions, which competes with MAP formation by reducing the availability of free Mg^{2+} ions. The presence of $\text{Mg}(\text{OH})_2$ not only diminishes phosphate removal efficiency but also reduces the overall yield of the desired product [26].

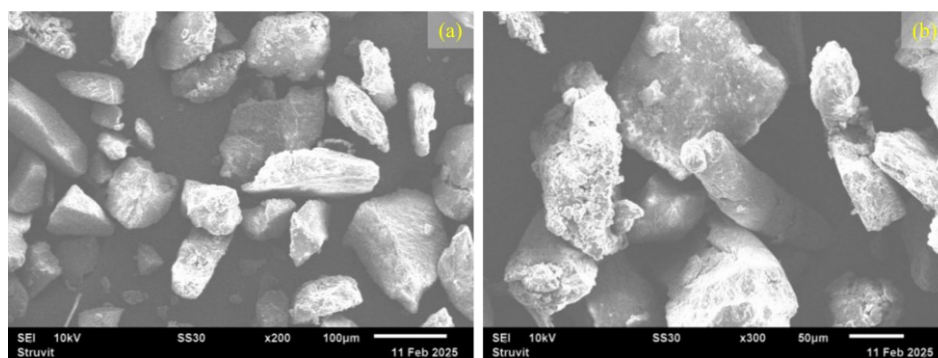


Figure 4. SEM micrographs of the produced fertilizer at (a) 200× and (b) 300× magnifications

3.4. Morphological Characteristics of the Precipitated Fertilizer

The morphological characteristics of the produced fertilizer were analyzed using a Scanning Electron Microscope (SEM), as presented in Figure 4. The SEM micrographs reveal that the precipitated crystals exhibit a block-like morphology with irregular and rough surfaces. Such irregular surface textures are commonly associated with the presence of impurity ions that become incorporated during the crystallization of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$). These impurities may arise from incomplete reactions or secondary components in the reaction mixture that interfere with uniform crystal growth. The struvite crystals formed in this study have an average particle size of 20–50 μm , consistent with the typical microcrystalline structure of chemically precipitated MAP. Larger crystal sizes are often observed in systems with higher magnesium molar ratios, as excess Mg^{2+} promotes more extensive crystal growth and aggregation [34].

However, an excessive addition of magnesium is not necessarily beneficial, as magnesium is classified as a secondary macronutrient in plants, and excessive Mg content in fertilizers may not provide additional agronomic benefits [35, 36]. From a functional perspective, smaller struvite crystals provide a greater total surface area, thereby enhancing dissolution kinetics and facilitating faster nutrient release into the soil solution [37, 38]. In contrast, larger crystals tend to dissolve more slowly, making them suitable for applications that require controlled or slow nutrient release. Although the recovered struvite crystals had an average particle size of approximately 20–50 μm , nutrient-release kinetics were not directly evaluated in this study.

Therefore, the possible influence of crystal size on dissolution behavior should be interpreted with caution as a literature-supported implication rather than as direct experimental evidence. In practice, the dissolution and agronomic effectiveness of struvite fertilizers are also strongly affected by soil pH, granulation, base excess, and application conditions. Further controlled release and plant-growth studies are required to confirm the slow-release performance and fertilizer effectiveness of the recovered struvite [37, 38, 39, 40].

3.5. Struvite crystallinity characterization

XRD analysis was performed to identify and confirm the crystalline phases of the synthesized struvite fertilizer, as shown in Figure 5. Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) possesses an orthorhombic crystal structure formed through crystallization, and its crystallinity influences its performance as a slow-release fertilizer [39]. In this study, synthesis was carried out under pH 9 with a molar ratio of magnesium, nitrogen, and phosphate of 4:1:1. The pH value of 9 was selected because it represents one of the optimal conditions for forming stable struvite crystals, while the excess magnesium in the 4:1:1 ratio promotes efficient precipitation and minimizes the formation of unwanted phases such as ammonium phosphate or magnesium phosphate.

As shown in Figure 5, the XRD pattern exhibits sharp diffraction peaks at 2θ values of approximately 18° , 19.8° , and 20° , which closely match the characteristic diffraction pattern of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) reported in ICDD PDF Card No. 15-0762. The dominant peaks at around 19.8° and 20° indicate a high degree of crystallinity of the struvite phase, while the presence of additional minor peaks further confirms the correspondence with its typical crystal structure. No significant peaks corresponding to other phases, such as amorphous magnesium phosphate or impurity compounds, were detected, indicating that the synthesized product exhibits high phase purity and a well-developed crystalline struvite structure. XRD analysis was performed only on the precipitate obtained under the optimum synthesis conditions, namely pH 9 with a $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratio of 4:1:1.

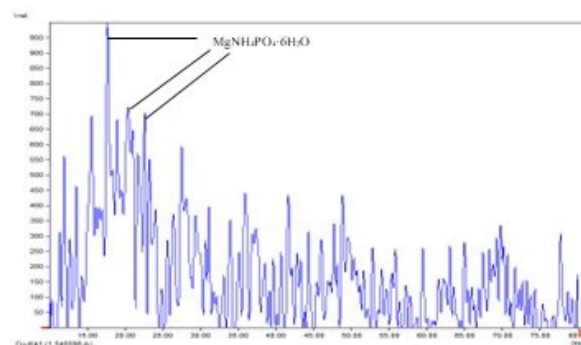


Figure 5. XRD pattern of struvite crystals synthesized at an $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratio of 4:1:1

Therefore, the possible formation of competing phases such as $\text{Mg}(\text{OH})_2$ or $\text{Mg}_3(\text{PO}_4)_2$ at pH 10 was not directly confirmed by XRD. The reduced phosphate removal efficiency and product yield observed at pH 10 may be associated with the formation of these magnesium-based precipitates under highly alkaline conditions; however, further XRD analysis of pH 10 samples is required to verify the presence and contribution of these impurity phases.

4. Conclusion

This study demonstrated that both pH and molar ratio significantly influence the efficiency of phosphate removal, fertilizer yield, and the morphological characteristics of the resulting struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$). The optimal conditions for struvite precipitation were observed at a molar ratio of 4:1:1 ($\text{Mg}:\text{NH}_4:\text{PO}_4$) and a solution pH of 9, under which the phosphate removal efficiency reached 89.94%. At this condition, the reaction system exhibited enhanced precipitation kinetics and greater nutrient recovery. Increasing the pH beyond 9 led to a decline in phosphate removal efficiency and product formation, which can be associated with less favorable struvite crystallization under highly alkaline conditions. At pH 10, the $\text{NH}_4^+/\text{NH}_3$ equilibrium may shift toward NH_3 , reducing NH_4^+ availability for $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ formation, while competing magnesium-based precipitates such as $\text{Mg}(\text{OH})_2$ or magnesium phosphate phases can reduce the availability of free Mg^{2+} . Conversely, at lower pH values (e.g., pH 8), incomplete precipitation occurred due to higher solubility of the constituent ions. The condition giving the highest phosphate removal efficiency was not identical to the condition giving the highest calculated yield. Based on the operational yield metric used in this study, pH 8 showed the highest calculated yield, whereas pH 9 provided the best phosphate removal efficiency and favorable product formation. Excess NH_4^+ at a molar ratio of 1:4:1 slightly inhibited precipitation, while excess Mg^{2+} beyond the stoichiometric requirement did not significantly enhance product formation, indicating that the reaction efficiency depends on maintaining a proper ionic balance. The morphological analysis (SEM) revealed that the precipitated struvite crystals exhibited block-shaped structures with irregular surfaces and an average crystal size of 20–50 μm , typical of struvite formed under optimized laboratory conditions. Although the observed particle size may influence dissolution behavior, nutrient-release kinetics and agronomic effectiveness were not directly evaluated in this study. Therefore, the potential use of the recovered struvite as a slow-release fertilizer should be further validated under controlled soil or plant-growth conditions, considering factors such as soil pH, fertilizer form, base excess, and application conditions. From a practical perspective, the 4:1:1 molar ratio offers the highest phosphate removal efficiency and may be suitable when maximum nutrient recovery is prioritized. However, because this condition requires excess magnesium salt, its economic feasibility for small- or medium-scale tofu factories should be carefully evaluated. In applications where chemical cost is a major constraint, the 1:1:1 ratio may represent a more practical

alternative despite its lower removal efficiency, and further techno-economic assessment is recommended before scale-up implementation.

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