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Using Chemical Precipitation to Recover Struvite from Household Wastewater for Agricultural Fertilizer Utilization †

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Abstract: Struvite is a substance that can be extracted from wastewater and has the potential to replace conventionally manufactured fertilizers and reduce environmental issues. A slow-release fertilizer can more effectively be used by matching the nutrient requirements of plants through the growing period and gradually supplying N and P for crop growth. Struvite is an ecologically friendly fertilizer because of its gradual fertilizer treatment and high quality. Existing research indicates that the solubility and absorption of struvite by plants are equivalent to those of artificial phosphorus fertilizers such as triple superphosphate or potassium phosphate. Struvite is recognized to be an effective fertilizer for grass, tree seedlings, ornamental plants, vegetables, and flower beds. Struvite precipitation removes phosphorus and nitrogen from sewage water, hence alleviating phosphorus shortages from non-renewable phosphorus sources and water eutrophication. Struvite would also be useful in the grasslands and woods where fertilizers are used. However, the agricultural utility of struvite has not been thoroughly investigated. As a result, this work is reported as a pot experiment designed to assess the fertilizer value of struvite. Experimental settings were created, and pot experiments were conducted to establish the optimal amount of struvite based on two factors. The initial pH for struvite synthesis was 9. The formulated struvite fertilizers were compared to standard phosphorus fertilizers in the pot trials. Fourier-transform spectroscopy and Scanning Electron Microscopy (SEM) with Energy-Dispersive X Ray Spectroscopy (EDAX) were employed to support the quantitative findings. To summarize, struvite precipitation is a desirable and effective method for removing phosphate and nitrogen from domestic sewage water and using them as fertilizers.

Keywords: struvite recovery; domestic wastewater; chemical precipitation; fertilizer; SEM; EDAX

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1. Introduction

Toilet flushing, dishwashing, bathing, cooking, and laundry are all examples of instances in which household wastewater is generated. Human-produced wastewater includes substances retrieved from the kitchen sink, bathroom, dishwashing machines, toilet, and laundry that can be reused as fertilizers. Pollutants, nitrogen, phosphorus, pH, sulfur, chlorides, alkalinity, dangerous compounds, and other inorganic elements can all be found in wastewater. Nitrogen and phosphorus might be recovered inversely from a chemical precipitation technique and be utilized as crop fertilizers [1]. Struvite can develop scale in pipelines and belts, as well as in centrifugal pumps, and clog a system's pipelines and other machinery, including the anaerobic digestion system itself [2]. Anaerobic digestion systems are used in treatment plants. A wastewater treatment plant is an opportunity for using a variety of methods to treat wastewater from factories and reduce its contaminants [3]. However, an enormous quantity of acid is necessary to break and eliminate struvite, adding to the cost [4]. A gradually released phosphorus and nitrogen fertilizer employing struvite could be a solution to non-renewable resource scarcity, eutrophication, and climate change. Struvite seems to be a great substitute supply of mineral phosphate and can be used as an extended-release fertilizer, requiring minimal treatment frequency [5]. Struvite is recovered from a variety of sources, like cooking water [6]. The use of struvite as a fertilizer has attracted international interest because of its efficiency, economic effectiveness, and environmental friendliness. Struvite is a remedy for larger challenges such as the impact of global warming, non-renewable resource scarcity, and the contamination of water [7,8]. A struvite precipitate, produced from the liquid portion of digested matter, is utilized as a multi-nutrient fertilizer in Poland. Crop absorption from struvite may be lower than that from commercialized ammonium phosphate [8,9]. The proposed research aims to create a fertilizer that would aid in the possible saving of resources as follows:

i. To employ the generated struvite as a possible agricultural fertilizer;
ii. To understand the beneficial effects of compounded struvite fertilizers by evaluating crop growth in comparison with conventional nitrogen and phosphorous fertilizers.

Usage of Struvite as a Fertilizer in India

Struvite derived from urine could potentially be put in fields in the same way that any other fertilizer would. The powder may be converted into granules in a simple granulation drum. A fertilizer in a granular form would make it simpler to use and not rust in damp situations [10,11]. If waste from humans, particularly urine, is removed from the system and transformed into struvite, wastewater treatment can be simplified, meaning that energy consumption would be reduced [12]. With regulatory agencies and regulatory bodies enforcing discharge regulations, a proper sewage treatment is becoming increasingly seen as an obstacle by the owners of companies belonging to the affected sectors [13]. A Wastewater Treatment Plant (WWTP) obtains fewer resources because it is a non-profit element of the industry [14–16]. Struvite extraction using chemical precipitation may significantly boost the pace of performance of this kind of treatment plant, while also adding economization to the whole process, allowing for the synthesis of additional value struvite fertilizers from waste products [17]. Such advancements have the potential to drive waste management emphasis away from disposal and towards recovering contaminants in the form of beneficial waste products [18]. Evaluation works often focus on particular results such as a technique or a procedure, but, in this case, a comprehensive evaluation encompassing all elements of struvite recovery in nutrient-rich residential wastewater has been conducted, encompassing molar ratios and pot experiments [19]. Several sources of sewage include nutrients (N and P) that need to be collected to bring economization to operations for the treatment of wastewater [20].

2. Materials

Ortho-Phosphoric acid, Ammonium Chloride, Magnesium Chloride Hexahydrate, Sodium Hydroxide, and Hydrochloric Acid chemicals were used. Before being processed, a wastewater sample was obtained from the water treatment plant on the university premises. A small amount of the wastewater that had not yet been examined was taken from the university's wastewater treatment center. $Al3^{+}$ ions in the waste product of phosphoric acid had a significant impact on struvite precipitation, and it was possible to achieve an ammonia elimination ratio that was nearly identical to that of pure phosphate salts. H+ in waste phosphoric acid had to be neutralized with a lot of NaOH. The use of both an alkali reagent and a cheap magnesium source in struvite precipitation was suggested as a solution to this issue. Compared to using pure chemicals, using waste phosphoric acid and inexpensive MgO could reduce chemical costs by 68%.

3. Methodology

As shown in Figure 1, the sample contains residence sewage water components that are used throughout the process of research. Because the wastewater had been purified before entering the treatment plant area, it was free of solid particles. A small portion of the material was acidified with strong Nitric acid and mixed with some Ammonium molybdate. The production of a vivid yellow precipitate layer composed of Ammonium Phosphomolybdate, aided with mild heating, revealed the existence of phosphate ions. In the chemical precipitation, a laboratory-scale batch system with five 250mL conical flasks was utilized. A total of 200 mL of domestic wastewater was added to the flasks and kept there at a constant temperature.

(a)

(b)

Figure 1. (**a**) Sample wastewater (KITS Ladies hostel); (**b**) struvite fertilizer.

$MgCl_2$ and $NH_4H_2PO_4$ were mixed to make solutions of varying concentrations. The precursors $MgCl_2$ and NH_4Cl were applied to $PO_4{}^{3-}$. The blended solution was agitated with a magnetic stirrer for 60 min before being tested. The white, solid forms of precipitate that resulted were collected. Two struvite precipitation values were chosen based on the lowest pH above five molar levels, namely 1:1.5:2 and 1:2:4 at a pH of 9.0.

3.1. Struvite Formation Mechanisms

Crystallization water is required for crystal formation and the crystalline characteristics of struvite. It occurs as a result of the following reaction.

$$Mg^{2+} + NH_3 + HPO_4{}^{2-} + 6H_2O \rightarrow MgNH_4PO_4.6H_2O\ (s) \quad (1)$$

Because struvite precipitation decreases the pH, it predicts $HPO_4{}^{2-}$ rather than $PO_4{}^{2}$.

3.2. Pot Experiments

In India, sterile NPK fertilizer has been categorized as fertilizer-grade material. For all crops, it is the greatest compost available for agricultural use. Sterameal contains key nutrients like N, P, and K. It enhances and maintains soil fertility by promoting the growth of bacteria in the soil. Different fertilizers including struvite and steramaeal NPK fertilizers were tested in pot trials to see how they affected plant development. All the pots were planted with the same type of soil, plant, and experimental circumstances, and all the fertilizers were buried beneath the soil. The pH of the soil was 6.0.

As shown in Figure 2, Sterameal is a compacted organic manure mix of animal and plant origin that has been used for many years in horticultural and agronomical fields to produce plants.

Figure 2. Commercial sterameal NPK fertilizer.

The seeds used in the tests were from a plant species in the legume family called *Vigna radiate* (mung bean), sometimes called green gram, maash, or moong, and are shown in Figure 3.

Figure 3. Vigna radiate.

The Indian subcontinent, Southeast Asia, and East Asia are where the mung bean is mostly grown. It is a component of savory and sweet meals alike. A perennial vine with yellow blossoms and fluffy brown pods, mung bean is also known as the green gramme. In each pot, containing 3 kg of soil, 10 seeds of the *Vigna radiata* were planted in red soil. A total of twenty pot samples were preserved, including six pots each for the struvite and Sterameal NPK fertilizers. As per Table 1, a specific amount of each fertilizer was put in the soil.

Table 1. Pot experiment—the amount of each fertilizer put in the soil.

Name of the Fertilizer	Amount of Fertilizer Put in the Soil (g/kg)					
	P1	P2	P3	P4	P5	P6
Sterameal	1 g/3 kg	1 g/3 kg	3 g/3 kg	3 g/3 kg	5 g/3 kg	5 g/3 kg
Struvite (A)	1 g/3 kg	1 g/3 kg	3 g/3 kg	3 g/3 kg	5 g/3 kg	5 g/3 kg
Struvite (B)	1 g/3 kg	1 g/3 kg	3 g/3 kg	3 g/3 kg	5 g/3 kg	5 g/3 kg
Control	0 g/3 kg	0 g/3 kg	-	-	-	-

3.3. Various Parameters Influencing the Chemical Precipitation of Struvite

When supersaturation is reached, a struvite-containing undersaturated solution (Mg^{2+}, NH_4^+, PO_4^{3-}) starts to precipitate. It is more acceptable to start precipitation by raising the pH of the solution. This is advantageous to streamline the technology and lower the price of residential wastewater. The NH_4^+ ratio begins to drop at higher pH levels, and nitrogen begins to volatilize from the solutions as NH_3 gas [5]. As a result, struvite seldom forms at higher pH levels, i.e., above 10.5.

The proposed study was conducted over six months. After fertilizing the soil, 10 *Vigna radiata* seeds were placed in each pot, and the growth of each seed was tracked. According to Moerman et al.'s 2007 research, calcium ions (Ca^{2+}) might improve the elimination of phosphorus by precipitating $Ca_3(PO_4)_2$. In the meantime, a lot of $Ca_3(PO_4)_2$ powder readily exits with the wastewater, lowering the wastewater quality. The three main feeding sequences for struvite precipitates were the pH, Mg^{2+}, and $PO_4{}^{3-}$. The pH decreased during the experiment, going from 9 to 6. The wastewater had conductivity and alkalinity that were both rather low. As shown in Figure 4, the pot trials were conducted in accordance with the standards of the Tamil Nadu Agricultural University.

Figure 4. A pot experiment using controls, synthetic fertilizer, and commercial fertilizer.

In our studies, the $NH_4{}^{+}$-N removal efficiency achieved 90% when a buffering reagent was administered after the addition of magnesium and orthophosphate. It was discovered that the residual $PO_4{}^{3}$ and TSS concentrations were less than those from prior trials.

3.4. Characterization of the Struvite Formed

Every element has distinctive energy peaks that are typical of it, and they are all well known. Additionally, EDX may be utilized for qualitative and quantitative examinations. Two dried samples of fertilizers made with struvite and designated as A (1:1.5:2) and B (1:2:4) were sent for examination.

4. Results and Discussions

4.1. Characterization of the Synthesized Struvite

SEM EDX and FTIR were used to confirm the development of struvite. The positions of the two synthetic product peaks were identical. Mg_3 $(PO_4)_2$ was detected, according to the SEM EDX graph figures, despite the data.

4.2. SEM-EDX Analysis of the Precipitate

The uneven dendritic form is shown in the precipitate's SEM EDX micrographs. Different ratios of P, N, and Mg may be the reason for the uneven form. The precipitated struvite EDX results from this investigation were in good agreement with the struvite pattern.

The presence of small crystals on the precipitate's outermost layer was confirmed using microscopy, as illustrated in Figure 5.

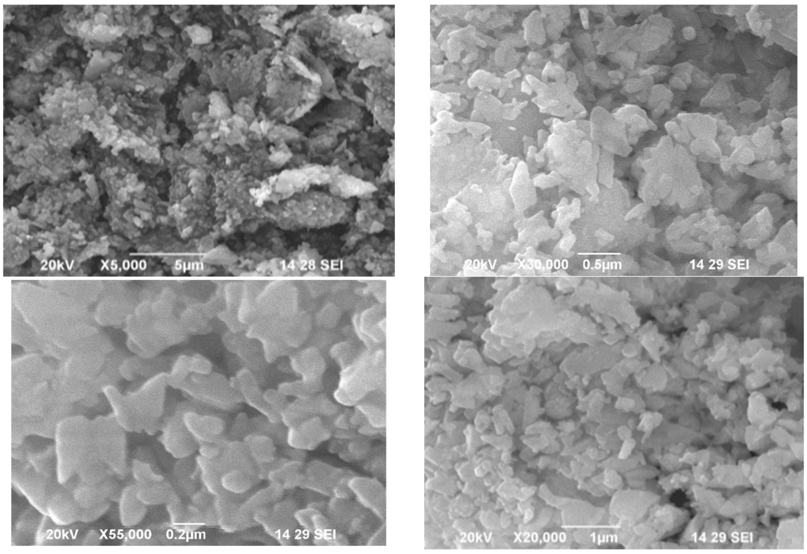


Figure 5. Struvite's SEM pictures with a molar ratio of PO_4^{3-}:Mg^{2+}:NH_4^+ = 1:1.5:2.

4.3. Analysis of the FTIR

The IR spectra of the precipitates are shown in Figure 6. The intensity of the band at 1469 cm is attributable to C-H bending vibrations, while the plateau at 1085 cm is connected to phosphate bonding. Despite, The crystals formed by the molar ratios of 1:1.5, 2:1, and 1:2:4 result in fragmented crystals and distinct clusters of diverse shapes, which are shown in Figures 5 and 6. The phosphate ion's stretching vibration is linked to the band at 1085 cm.

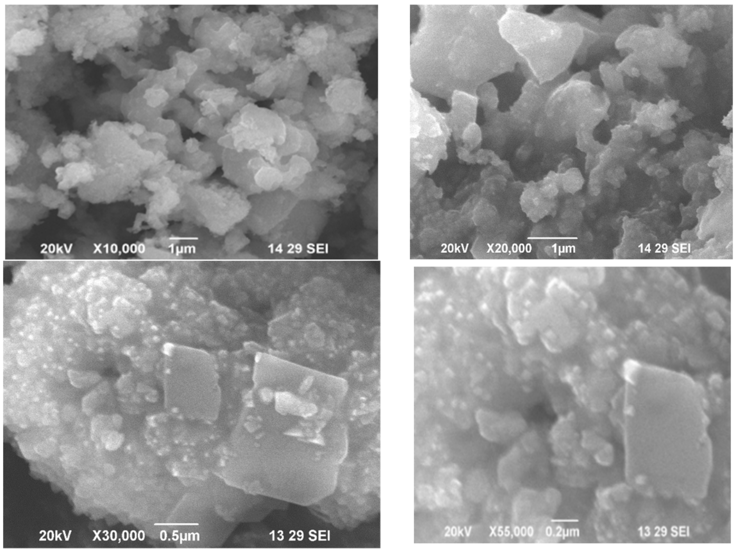


Figure 6. SEM images of struvite with a molar ratio of PO_4^{3-}:Mg^{2+}:NH_4^+ = 1:2:4.

Additionally, the bands identified have been linked to vibrations brought on by the water molecules' deformation at 3415 cm. The intensity at 534 cm is related to the scissoring vibration of the $PO4^{3-}$, whereas the band at 1089 cm is unrelated. The intensity band at 1469 cm is attributable to the C-H bending vibrations, while the plateau at 1085 cm is connected to the phosphate bonding. The phosphate ion's stretching vibration is linked to the band at 1085 cm. The IR spectra of the precipitates are shown in Figures 7 and 8.

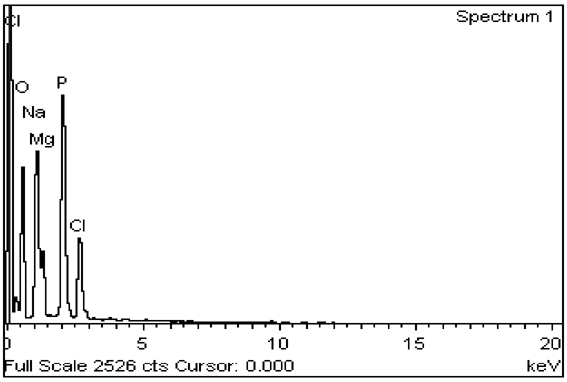


Figure 7. SEM-EDX spectra of the products containing struvite with a molar ratio of PO_4^{3-}:Mg^{2+}:NH_4^+ = 1:2:4 (B) at a pH of 9.0.

Furthermore, the vibrations brought on by the water molecules' deformation is linked to the bands seen at about 3415 cm. The IR spectra of the precipitates are shown in Figures 7 and 8. The intensity at 534 cm is related to the scissoring vibration of PO_4^{3-} whereas the band at 1089 cm is unrelated.

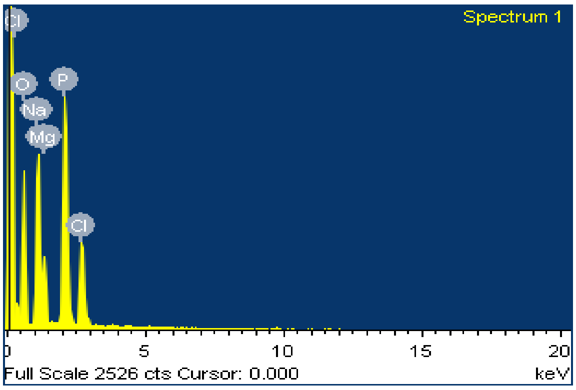


Figure 8. Spectra of products A and B of struvite.

The peak levels in sample A (563.21, 979.84, and 1089.78) and sample B (peak levels 534.28, 883.4, and 1085.92) indicate the presence of phosphorus, and the peak level at 3415.93 indicates the existence of an amine compound class, as shown in Table 2.

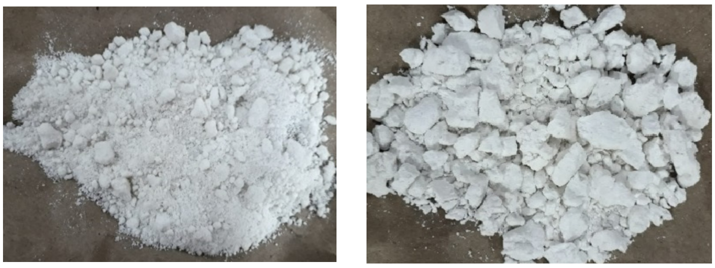
Table 2. IR peak absorption ranges taken from sample A's Merck IR table.

Absorption	Appearance	Group
563.21	Strong	C-Br Stretching P-Cl Stretching
979.84	Strong	P-F Stretching
1089.78	Strong	P-O-C Stretching
1469.76	Medium	C-H Bending
1627.92	Medium	C=O Stretching
1743.65	Strong	O-H Stretching
3419.79	Strong Broad	N-H Stretching N-OH Stretching

4.4. The Influence of P Concentration

Figure 9 depicts many views of struvite aggregates in use. The microphotographs clearly show how aggregates and crystal habits developed at various P concentrations. Low

P concentrations caused aggregates to form with a comparatively more compact shape. There are PO_4^{3-} and NH_4^+ tetrahedral, as well as $Mg[H_2O]_6^{2+}$. Figure 9b depicts the struvite crystal structure with octahedral species.



(**a**) (**b**)

Figure 9. Struvite with the following compositions: (**a**) product A with a molar ratio of PO_4^{3-}:Mg^{2+}:NH_4^+ = 1: 1.5: 2; (**b**) product B with a molar ratio of PO_4^{3-}:Mg^{2+}:NH_4^+ = 1: 2: 4.

4.5. Struvite as a Fertilizer Product

Struvite crystals contain analogs of the mineral, which form when other ions are substituted for struvite in the crystal matrix. Crystals with various morphologies and compositions are formed as a result of these intricate patterns of interactions and imperfections during crystallization. When decomposing by heating, the pace of heating, the presence or lack of water, and the temperature all affect the decomposition sequence, the products, and their quality. In the development and aggregation of struvite crystals, pH plays a crucial role. According to prior studies and the results of the current investigation, rising pH levels for struvite precipitation caused the solution's SI to constantly decrease and caused crystal behaviors to change. The pH of the solution was measured to be between 9.0 and 10.5. Because negatively charged struvite crystals were more electrostatically attracted to one another when the pH was raised, the aggregates' sizes decreased. According to the experimental findings, the ideal starting pH for the production of struvite is 9. Two molar ratio systems had no precipitate formed when the pH fell below 8.0, indicating that the solid phase was under unsaturated conditions. Struvite precipitation may occur throughout an extensive pH range from 9.0 to 12.5 for molar ratios of 1:1.5:2 and from 9.0 to 14.0 for 1:2:4, at which point both SI values peak at pH 10. The greatest $MgHPO4\bullet 3H_2O$ saturation index was less than 0.5. The diverse PO_4^{3-}, Mg^{2+}, and NH_4^+ complex substances were combined to create struvite in an aqueous solution, and the minor initial pH shift produced a variety of chemical compounds. According to the experimental data, the ideal pH for samples A and B was 9.0.

5. Conclusions

An effective method for recovering phosphorus from home and wastewater is struvite precipitation. One potential fix for the issue is a struvite-based fertilizer, which might replace some of the current phosphorous fertilizers. The potential for employing struvite as a base for fertilizer synthesis was assessed in the current study. Domestic wastewater is inherently complicated; thus, bench-scale, "proof-of-concept" research had to be carried out at first. When compared to readily available alternatives, the acquired struvite crystals are a remarkable slow-release fertilizer. Such an extensive treatment plan will accomplish the dual goal of nutrient recovery and an environmentally responsible nutrient-rich wastewater treatment. Due to the possibility of cost reductions and productivity improvements, using a recovered fertilizer like struvite is an environmentally friendly choice over extremely soluble PR-based fertilizers.

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