Phosphorus recovery from septage treatment plant sludge by struvite formation with alkaline hydrolysis as pre-treatment

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Abstract. Insufficiency of phosphorus due to the limited availability of phosphate rocks is predicted within the next decades. Phosphorus recovery from wastewater sludge was found to be one of the possible alternative sources of phosphorus. Moreover, stringent effluent standards, including that of phosphorus levels, have been newly implemented in the Philippines. Due to these factors, phosphorus recovery from wastewater in Philippine settings was deemed as an important topic to be further studied. In this paper, the feasibility of recovering phosphorus from septage sludge in Metro Manila by the precipitation of struvite crystals was studied. Septage sludge is seen as one of the potential sources and was characterized in terms of the concentration of Mg2+, Ca2+, NH4+ and PO4-3 ions. In the lab-scale experiments conducted, alkaline hydrolysis using sodium hydroxide was performed as a sludge pretreatment before the precipitation proper. Alkaline hydrolysis was done to release the remaining PO4-3 ions from the solids present in the sludge, thereby promoting precipitation after hydrolysis without the need for additional phosphate source. Furthermore, alkaline hydrolysis can effectively release phosphorus without inducing the dissolution of interfering ions such as Ca2+ and other heavy metals. Results show that PO4-3 ions increased by 267.37 mg/L and the total amount of phosphate ions increased from 17.05% to 46.88%, showing the effectiveness of the hydrolysis. For the precipitation of struvite, three parameters were controlled: pH, residence time, and Mg:P ratio. After precipitation, the phosphorus recovery efficiency for the runs were each evaluated and analyzed in order to determine the effects of the parameters on phosphorus recovery. The precipitate of the run with the highest phosphorus recovery obtained was then evaluated using XRD analysis. It was found that the sample with the highest phosphorus recovery is 53.12%, which was achieved at pH 9, a residence time of 1 hour, and an Mg:P ratio of 1.5:1. Therefore, it can be concluded that precipitation subjected at pH 9 has the highest potential for higher phosphorus recovery. Further, the residence time has a minimal effect on phosphorus recovery and higher phosphorus recovery can be achieved at a higher Mg:P ratio with increasing potential for struvite recovery.

1. Introduction
Phosphorus is an essential nutrient for living organisms and is widely used especially in the agriculture industry. In fact, about 90% of phosphorus is used as crop fertilizers in the global food supply chain [1]. Phosphorus is obtained primarily by mining phosphate rocks, which are found only in specific areas, making these rocks a vital and limiting source. Furthermore, studies show that only a 16,000 MT global total supply of these rocks are economical according to the United States Geological Survey (USGS) [1]. This amount becomes crucial problem, as the phosphorus demand, especially as a fertilizer, significantly increases over the world at about 1.2% per year due to the growing population and agricultural yields [1, 2]. For these reasons, a possible phosphate rock depletion by 2050 is predicted as the human population grows towards 9 billion [3]. In the Philippines, it was found that 37% of the total water pollution comes from agricultural practices such as fertilizer, pesticide runoff, animal waste, detergents and untreated sewage in domestic wastewater, where all sources contain a significant amount of phosphorus [4, 5]. Given the benefits of the nutrient phosphorus, excessive amounts of this element in the soil can cause eutrophication, or nutrient enrichment in surface waters. This leads to over
production of toxic algae resulting to green discoloration and deterioration of water, as well as animal kill in the bodies of water [6].

Eutrophication in the Philippines is one of the concerns in the bodies of water, where the nutrient discharge is primarily from agricultural runoff, release of untreated sewage and nutrient-loaded groundwater which is discharged in the coastal ecosystem. It was found in a study by Reyes (2012), that domestic, industrial, agricultural and forest sources heavily cause eutrophication for one of the lakes, Laguna de bay in the Philippines by contributing 39,622 MT of nitrogen and 9506 MT of phosphorus [4]. Effluent from wastewater systems can also be discharged in bodies of water such as in rivers. Phosphate discharges are limited in the range of 1-10 mg/L, yet river streams in the country have been exceeding this phosphate concentration criteria [7]. This may be due to the reason that more than 80% of wastewater remains untreated and released to the environment [8]. Furthermore, in Metro Manila, the problem on nutrient load is intensified as only about 20% of the population has services for sewerage treatment and septic tanks are the main sanitation technology for rural and urban areas [9,10]. Hence, most waters in Metro Manila are contaminated heavily due to lack of treatment for wastewaters and it was found in 2005 that residential sewage contributes to 65-75% of the pollution [11]. Thus, it is of significance to find ways in removing phosphorus from wastewater treatment plants (WWTP) and recover as beneficial products. In WWTP, septage sludge is seen as one of the potential raw materials where phosphorus can be recovered. In general, it has a relatively higher phosphorus content, with a mean value of 4.9 %w/w, compared to other sources, such as urine, manure, crop residues and more [3,11].

Phosphorus can be recovered in the form of phosphates (PO$_4^{3-}$), which enables the recovered product to be used as fertilizer. Fertilizer containing phosphates may be conventional or slow-release, but slow-release fertilizers are often opted for in areas where nutrient release to the soil must be limited to avoid problems such as eutrophication [12]. One of the slow-release fertilizers is struvite, a crystalline substance which contains magnesium, ammonium and phosphate (MgPO$_4$NH$_4$·6H$_2$O). Hence, this is of benefit to crops requiring a magnesium source [13]. Struvite is also known to be causing no harm in growing crops even when used in high dosage. Due to its being a slow-release fertilizer, it may be used in specific lands once every few years. Since Philippines is known to be an agricultural country, the recovery of phosphorus from septage sludge by struvite formation would therefore propose an alternate way in which phosphorus can be recovered and used [14]. However, struvite precipitation is still being investigated at pilot and laboratory scales with studies primarily concentrated on the reduction of the dissolved phosphate concentration to meet the effluent standards in WWTPs [15].

This study aims to recover phosphorus from septage sludge by struvite formation in laboratory scale. The septage sludge raw material is obtained from a septage treatment plant (STP) in Metro Manila, where the obtained sample did not undergo any chemical pretreatment process. After obtaining the sludge sample, alkaline hydrolysis of the septage sludge is done prior to the precipitation process. This is to further release the phosphorus in the form of phosphates from the solid matter in the sludge and increase the potential recovery of phosphorus. Studies show that phosphate content significantly increases as pH increases and that sodium hydroxide (NaOH) was the most preferred alkali for its better solubilization efficiency among other alkali. It was also found that PO$_4$-P in the total phosphorus (TP) was dominant as pH reaches 11, thereby, making this process an effective method [16,17]. This is for the reason that phosphates are being released or separated from metals, such as Iron (Fe) or Aluminum (Al), that it was initially bound to. The metal-bound inorganic phosphates were mostly considered as only a fraction of the TP is from organic phosphates [16]. Among other hydrolysis methods such as thermal and acid hydrolysis, alkaline hydrolysis also gives the advantage of easy operation, simple devices and a high efficiency [17]. Precipitation of struvite is then done at different conditions, depending on the parameters pH, Mg:P ratio and residence time as these are the ones known to influence the precipitation of struvite [18].

2. Materials and Methods

2.1 Materials and Reagents

The chemicals used within the experiment were analytical reagent grade. Stock solution of 12M sodium hydroxide (NaOH) in deionized water was utilized for pH adjustment for alkaline hydrolysis and struvite precipitation. Ammonium chloride (NH$_4$Cl) was added as ammonium source and
magnesium chloride hexahydrate (MgCl₂·6H₂O) as Mg source. High Range Ammonia Nitrogen by the AmVer™ Salicylate Test 'N Tube™ Method of HACH was used to determine ammonia and ammonium concentration within the range 0.4-50.0 mg/L NH₃-N. Similarly, Ultra High Range TNT 845 Phosphorus, Reactive (Orthophosphate) and Total was utilized to quantify the amount of phosphate in the sample at range of 6-60 mg/L PO₄³⁻-P.

2.2 Septage Sludge Preparation
In the laboratory-scale experiment, the septage sludge feed was collected from a septage holding tank in a local sewage treatment plant in Metro Manila, Philippines, as shown in Figure 1. The feed used in the study was subjected to physical treatments such as rock trapping and maceration. A volume of 16.2L of the homogenized feed was characterized to contain 127 mg/L PO₄³⁻, 243 mg/L total phosphorus (TP), 195 mg/L NH₃-N, and 249 mg/L NH₄⁺. As a form of a pre-treatment, the septage sludge was hydrolysed at a pH of 12.5. The alkaline hydrolysis was performed in a Lovibond Jar Tester (ET 750) at 250 rpm for 1.5 hours.

Figure 1. Sampling location in local STP for phosphorus recovery

2.3 Phosphorus Recovery as Struvite
The hydrolysed sample was centrifuged to achieve a nitrogen and phosphorus-rich centrate. Prior to the precipitation proper, the resulting solution from alkaline hydrolysis was analysed of its PO₄³⁻, NH₃-N, and foreign ion content specifically the Mg and Ca concentrations. Duplicate experiments were performed. Design-Expert® 7.0 was used to generate an automated sequence of runs based from the following parameters: pH, Mg:P ratio, and residence time. Specific amounts of MgCl₂·6H₂O and NH₄Cl was added to achieve the required Mg:P:N molar ratio for struvite precipitation. The jar tester was used in the precipitation process at constant mixing speed of 200 rpm at varying residence time. Box-Behnken Response Surface Methodology was used to investigate the effects of the parameters on phosphorus recovery using septage sludge. The precipitate with the highest percent P recovery was characterized through X-ray diffraction (XRD).

2.4 Analytical Methods
For the characterization of phosphate and ammonia and ammonium content of the sample, HACH DR 1900 Spectrophotometer was used. Ascorbic Acid method was utilized to analyse the total phosphorus content, according to the standard methods [19]. However, direct measurements of NH₄⁺ is not possible and Equation 1 was used to calculate for the ammonium content of the aqueous sample, given the pH, temperature, and total amount of ammonia and ammonium present through the analysis provided by HACH [20,21]. On the other hand, Mg and Ca contents were evaluated through Flame Atomic Absorption Spectroscopy (FAAS). The precipitates were pressed in a glass sample holder and characterized using an XRD (Shimadzu-binary) at 2° to 80°C with a rate of 2° per minute.

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\frac{(NH₃-N)_{actual}}{(NH₃-N)_{HACH}} = \frac{10^{pH}}{10^{pH+6.7344/T(F)}}
\]  (1)

2.5 Experimental Design
In this study, Box-Behnken was used to determine the significance of the pH, Mg:P molar ratio, and residence time on phosphorus recovery using septage sludge as feed. As shown in Table 1, the effects of pH, Mg:P, and residence time on phosphorus recovery at three levels were analysed.
Given that three parameters were assessed of their influence in phosphorus recovery, Box-Behnken was mainly used as the experimental design to minimize the required number of runs.

| Parameters     | Symbol | Levels |
|----------------|--------|--------|
| Residence time (hr) | A      | -1     | 0     | +1   |
| pH             | B      | 8      | 9     | 10   |
| Mg:P           | C      | 1      | 2     | 3    |

3. Results and Discussion

3.1 Alkaline Hydrolysis of Septage Sludge
After subjecting the septage sludge sample under alkaline hydrolysis, the hydrolysed samples were centrifuged before being analysed for its PO$_4^{3-}$, NH$_4^+$, Mg$_2^+$, and Ca$_2^+$ concentrations. Table 2 below shows the results of the characterization done.

| Parameter | Concentration (mg/L) |
|-----------|----------------------|
| PO$_4^{3-}$ | 394.44               |
| NH$_4^+$  | 0.0772               |
| Mg$_2^+$  | 0.2                  |
| Ca$_2^+$  | 0.3                  |

Comparing the PO$_4^{3-}$ and NH$_4^+$ concentrations obtained before and after hydrolysis, an increase of 267.37 mg/L in the PO$_4^{3-}$ concentration of the sample was obtained. This shows that the total amount of phosphorus present as phosphate in the sample increased from 17.05% to 46.88%. Thus, alkaline hydrolysis was effective in increasing the phosphate concentration of the sample. On the other hand, a drastic decrease of the NH$_4^+$ concentration to an almost negligible value can be seen after hydrolysis. This decrease in the ammonium concentration is in accordance to theories, which state that at basic pH, more NH$_3$ is present in wastewater as compared to NH$_4^+$. Moreover, at a pH of 11 or higher, all NH$_4^+$ are converted to NH$_3$ [22]. Since NH$_3$ is stable in gaseous form at 25ºC, the increase in pH of the sample may have been accompanied by a release of NH$_3$ in gaseous form. However, not all NH$_3$ is converted since it is still very hydrophobic [23]. Hence, the addition of an NH$_4^+$ source is needed for struvite precipitation to occur. It is also seen that the Mg$_2^+$ and Ca$_2^+$ concentrations after hydrolysis can be considered negligible as compared to the concentration of phosphates. This shows that the addition of Mg$_2^+$ is also needed in order to induce struvite precipitation. Moreover, it can also be seen that Ca$_2^+$ has a low chance of competing with Mg$_2^+$ during the precipitation process.

3.2 Struvite Precipitation
After alkaline hydrolysis of the sample, the struvite precipitation experiment was then conducted. From the precipitation process, %P recovery values from 39.39% to 53.12% were achieved. In order to determine the effects of the pH, residence time, and Mg:P ratios tested on %P recovery, ANOVA was done with the use of Design-Expert® 7.0. From the model summary statistics shown by the software, a 2 Factor Interaction model was chosen. From the ANOVA results, many insignificant terms were found. The interaction between residence time and Mg:P ratio are the only significant parameter found to affect the %P recovery, signified by a p-value of 0.0242. Table 3 below shows the resulting analysis of variance of the reduced model by backward elimination regression.

| Source | Sum of Squares | Df | Mean Square | F-value | p-value | Prov > F |
|--------|----------------|----|-------------|---------|---------|----------|

Table 3. ANOVA for Response Surface Reduced 2FI model
The resulting model graph showing the interactive effect of residence time and Mg:P ratio on the %P recovery when pH is constant is shown in Figure 2 below. From the interaction plot obtained, it is seen that at constant pH and high Mg:P ratio, the %P recovery increases as the residence time decreases. On the other hand, at low Mg:P ratio, the %P recovery increases with residence time. Moreover, the fact that the maximum phosphorus recovery can be achieved at high Mg:P ratio and short residence time is attributed to the fact that higher %P recovery is obtained at higher Mg:P ratio, as the degree of saturation of the solution increases with an increase in the Mg:P ratio [24]. Moreover, the spontaneous nucleation of struvite persists at higher Mg:P molar ratio [25]. As such, at sufficiently high Mg and P concentrations, struvite precipitation can happen instantaneously. The decrease in %P recovery at increasing residence time when the Mg:P ratio is kept high can be attributed to the pH change brought about by the spontaneous precipitation of struvite. The formation of struvite is accompanied by a release of H+ leading to a decrease in pH [26]. Moreover, at increasing pH, the solubility of struvite decreases, also increasing the struvite saturation in the solution [27]. As such, the decrease in pH may have caused the dissolution of struvite and a decrease in the supersaturation of the solution. Moreover, as residence time is increased, the extent of the dissolution of struvite also increases, leading to a decrease in the %P recovered. This phenomenon could have been prevented through the constant monitoring of the pH of the solution. On the other hand, the increase in %P recovery when residence time increases while the Mg:P ratio is kept low can be attributed to the fact that low Mg:P ratio leads to low supersaturation of the solution. Since the induction period preceding the appearance of the first crystal nuclei during nucleation is controlled by supersaturation, the formation of crystals would take a longer time at low Mg:P ratios [28,29]. Moreover, struvite crystallization and growth increase as residence time is increased due to the enhancement of the transfer of mass to the crystals from the solute [24].

### Table

| A – Residence Time | 0.025 | 3 | 0.025 | 2.502E-003 | 0.9612 |
|--------------------|-------|---|-------|-------------|--------|
| C – Mg:P           | 8.78  | 1 | 8.78  | 0.087       | 0.3759 |
| AC                 | 74.09 | 1 | 74.09 | 7.32        | 0.0242 |
| Residual           | 91.04 | 9 | 10.12 |             |        |
| Cor Total          | 173.89| 12|       |             |        |

3.3 **Characterization of Precipitates**

Major decrease in the phosphate content of the liquor after precipitation was evident. According to Crutchik and Garrido (2011), the formation of amorphous precipitates is likely to occur in processes involving chemical precipitation. Elemental analysis of foreign ions was only done for Mg and Ca wherein it was found that negligible amounts were present after alkaline hydrolysis [30]. Provided that Mg is the limiting reactant in struvite precipitation, MgCl2·6H2O was added as Mg source to achieve the desired Mg:P ratio. Due to the low Mg:Ca ratio, the formation of calcium phosphate was less likely. Phosphorus recovery in the form of magnesium phosphate (Mg₃(PO₄)₂) is also plausible due to the
decrease in the ammonium content of the sample from the release of ammonia gas [31]. Chemical precipitation of phosphate with other metal ions such as Fe\(^{3+}\) and Al\(^{3+}\) was seen as a possible occurrence. Also, Schaum et al. (2007) found that alkaline hydrolysis causes the release of aluminum in the aqueous phase of wastewater [32]. Similarly, dissolution of amphoteric Fe bound P occurs in alkaline hydrolysis. In comparing the solubility of struvite with other phosphate precipitates, it was discovered that iron phosphate had the lowest solubility which suggests the high probability of recovering phosphorus in this form [33]. Thus, it is possible that phosphorus is recovered in the form of magnesium phosphate, iron phosphate, and aluminum phosphate. Figure 3 shows the XRD analysis for the sample with the highest phosphorus recovery of 53.12%, achieved at a pH of 9, residence time of 1 hr, and Mg:P ratio of 1.5:1. It is evident that amorphous precipitates were obtained, shown by the lack of sharp peaks. In fact, a d-spacing of 4.13Å coincided with the standard d-spacing of aluminum phosphate [34]. Given that appropriate conditions were used in the precipitation process, struvite could be present in trace amounts to be detected in the XRD analysis.

![XRD diffractogram of sample with highest phosphorus recovery](image.png)

**Figure 3.** XRD diffractogram of sample with highest phosphorus recovery

4. **Conclusion**

In this paper, precipitation of struvite from septage sludge as a form of phosphorus recovery was studied. In order to increase the amount of phosphorus recovered in the form of struvite, the septage sludge sample used was subjected to alkaline hydrolysis. An increase in the total phosphorus present as phosphate in the sample from 17.05% to 46.88% was found. After hydrolysis, the NH\(_4\)+ and Mg concentration of the sample was found to be almost negligible. Hence, chemical addition is needed in order to increase the concentration of both parameters to the required levels. After alkaline hydrolysis, struvite precipitation was conducted. Around 39.39% to 53.12% phosphorus recovery percentages were achieved. Thus, the precipitation of struvite as a means of reducing the phosphate concentration of wastewater is a plausible method. Based from the ANOVA analysis, it was found that only the interactive effects of Mg:P ratio and residence time had a significant effect of phosphorus recovery, with the highest %P recovery achieved at maximum Mg:P ratio and minimum residence time. Moreover, it was found that at high Mg:P ratio, %P recovery decreases as residence time increases. On the other hand, %P recovery increases as the residence time increases when the Mg:P ratio is low. These phenomena were closely attributed to the supersaturation properties of struvite. Based from the XRD diffractogram of the sample, amorphous precipitates were assumed to have formed, supported by the presence of minor peaks. From the Mg\(^{2+}\) and Ca\(^{2+}\) analysis after alkaline hydrolysis, it was deduced that phosphorus recovery in the form of calcium phosphate was less likely. Also, since Mg source was added to the sample to increase Mg/Ca ratio and to achieve the desired Mg:P ratio, the formation of magnesium phosphates is also possible. The release of aluminum and iron ions in the aqueous phase due to alkaline pH could also cause the formation of iron and aluminum phosphates. From the d-spacing of the minor
peak with a value of 4.13Å which was obtained from the XRD analysis, it is likely that phosphorus was 
recovered in the form of aluminum phosphates. The presence of struvite in trace amounts is also seen as 
a feasible occurrence since the parameters were adjusted to achieve an appropriate condition for the 
precipitation process. Future works will include metal ion removal process as pre-treatment to the 
hydrolysed sample to eliminate or minimize the formation of other phosphate salts. Further analysis, 
such as elemental analysis, will also be done for the struvite precipitates formed.

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