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# Phosphorus Extraction from Waste Activated Sludge using Sulfuric Acid Leaching: Recovery P and Reduction P Leakage

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**Abstract:** Better understand the extractability of phosphorus (P) recoverable made from sustainable activated sludge in order to drastically lessen reliance on non-renewable phosphorus sources. Sulfuric acid ($H_2SO_4$) concentration enhanced P-extraction efficiency. The experimental research design on acid leaching of was defined using the effect of time and concentration. The effect of $H_2SO_4$ concentrations upon P extraction was shown to be significant within that investigation. The best possible leach condition was waste activated sludge with 0.1 M $H_2SO_4$ in 30 mins, which also extracted 98% of a P and was employed for P precipitation. In the connection of pH 9 value and Mg/Ca=2, the efficiency of P recovery 93% substantially improved. The findings of this research are obligated to generate a comprehensive information on the acceptability of P-composite matter recovered from waste activated sludge in order to urge decision-makers to use it. This will also aid in attempting to close the P loop in the human environmental for food cultivation, as well as benefit of reducing P leakage of WAS to landfills.

**Key words:** Phosphorus extraction, Phosphorus recovery, Waste activated sludge

## 1. INTRODUCTION

Phosphorus (P) is an essential mineral for all living species and is used extensively throughout agricultural crops as a soil amendment. Furthermore, P is a constrained and non-renewable resource that is necessary for food production [1]. More than 85% of the phosphate rock (PR) harvested is utilized to make P fertilizer [2]. The demand for P fertilizer for food production is expected to rise dramatically as the global population grows towards 9 billion people by 2050 [2, 3]. Rock phosphate (RP) is quickly diminishing, but it is still available in some parts of the world, including Morocco, China, and the United States [4]. In the next 50-100 years, PR reserves are anticipated to diminish [5]. As a possible consequence, P recovery from any P-rich residue has attracted a lot of excitement.

Waste activated sludge (WAS) is produced in enormous quantities in greywater. It is done in part help reduce phosphorus from sewage before it is discharged into the environment. Due to its high phosphorus concentration, waste activated sludge is widely applied as a fertilizer on agriculture. However, employing activated sludge on agriculture can be difficult due to the presence of additional dangerous compounds (e.g. chromium, nickel, polyaromatic hydrocarbons and others). Furthermore, there could be developing organic contaminants that pose a risk of which we are now unaware. Consequently, the most common approach for recovering phosphorus from WAS is to first release the phosphorus and then recover it from the supernatant [6-8]. Given that the spatial distribution and chemical speciation of phosphorus in WAS impacts their release efficiency, determining the spatial distribution and chemical speciation of phosphorus has become a critical requirement for phosphorus recovery. It is common knowledge that biological phosphorus removal occurs primarily through phosphorus release by phosphorus accumulating organisms (PAOs) under anaerobic conditions and excessive phosphorus absorption under aerobic conditions, resulting in phosphorus primarily existing in the form of polyphosphate (Poly-P) in cells [9]. Phosphorus, on the other hand, has been detected in extracellular polymeric substances (EPS) in multiple investigations [10-12]. Furthermore, a wide number of metals (e.g., Ca, Mg, Al, and

Fe) may precipitate with phosphate (P-precipitates) and exist in inorganic minerals in wastewater [13, 14].

Mg ions impact the struvite particle micro electrophoretic mobility at an isoelectric point of 1.75, according to [15], implying that the higher supersaturation was helpful for crystallization. Mg/Ca 0.2 slowed HAP production, and Mg/Ca > 2 counterbalanced the unfavorable effect of Ca during struvite crystallization, according to [16]. Assuming calcium was removed as calcium phosphate, $Ca_3(PO_4)$ and the remainder of the removed orthophosphate was formed into struvite, calcium phosphates were observed to dominate formed precipitates when the $Mg^{2+}/Ca^{2+}$ activity ratio was approximately 1/0.5 and greater [17].

Microbial material is separated into sludge, known as waste activated sludge, in Thailand's municipal treatment. P in wastewater at the end of pipe in Bangkok Metropolitan Administration (BMA) comes from domestic waste, septage sludge, and green waste, according to [18]. Waste activated sludge (WAS) is a significant by-product of the BMA WWTP. WAS, on the other hand, is polluted with human diseases such as feces-borne coliform bacteria, viable helminth eggs, and active parasite cysts. Fresh sewage sludge should not be disposed of on land unless it has been treated to remove pathogens [19]. As a result, applying WAS directly to agricultural areas is forbidden. Besides, WAS through pretreatment of biological and chemical to chemical precipitation processes [20, 21].

Nevertheless, there have been very few research in Thailand to recover P from WAS and reduction of P leakage. The objectives of this study are: (1) to evaluate P extraction efficiency from waste activated sludge by using of sulfuric acid and (2) to evaluate P recovery efficiency, the amount of P solids precipitates and reduction P leakage to landfills from WAS by the addition of magnesium. The study's successful results are intended to pave way for the implementation of an engineering approach for P recovery, which will save money on imported fertilizer while simultaneously protecting the environment from eutrophication caused by excessive P discharge.

## 2. MATERIALS AND METHODS

### 2. 1 Sample preparation

The WAS used in this investigation came from the Nong Khaem water quality control WWTP, BMA, Thailand's secondary sedimentation tank. The properties of WAS were measured following collection, and it was stored in airtight plastic containers at 4°C before usage. The primary features of WAS, which show that the sample comprises essential components of 428 mg P/l, 227 mg Ca/l, and 155 mg Mg/l. To remain consistent prior to actually acids extraction, the total solids (TS) content of WAS was diluted with distilled water to 3 percent solids content (w/v), which is also the average TS concentration observed in common WAS [9].

### 2. 2Acid leaching tests

In chemical leaching studies, 500 ml of WAS (3 percent solids content) was mixed with various concentrations of $H_2SO_4$ (0.01, 0.05, 0.1, and 0.5 M) to achieve four different temperatures: room temperature and 50°C. The mixture was mixed at 200 rpm with a Jar tester at room temperature (25-30°C). P extraction experiments were carried out in triplicate, with reaction times varying (15, 30, 45, 60, 90, and 120 mins). The leaching of WAS is shown in Table 1. After it had been leached, the mixture was separated using 10 m filter paper (Whatman No. 93). The filtrates were then analyzed for P, Mg, and Ca.

### 2. 3 Phosphorus precipitation experiments from supernatant of acid leaching

P stock solution, pH 9, and the Mg/Ca =2 molar ratio represents two factors which could impact the precipitation reactions emphasized throughout this research. The P-stock solutions were prepared up the supernatants from acid leaching procedures. The precipitation tests were carried out in 1000 ml glass beaker with 500 ml P-stock solution. At the beginning of the experiment, the pH of all instances was changed to pH 9 using a dropwise 50 percent sodium hydroxide solution, according to [22]. These tests were performed three times at room temperature in duplicate. For 15 minutes, the stirring speed was set to 200 rpm, then dropped to 75 rpm for 30 minutes, followed by an hour of settling. Using 10 m sheets, the P-containing particles were separated from the liquid (Whatman number.93). Finally, the precipitates formed during this process were filtered and dried in an oven at 103°C for 24 hours. P-containing solids were identified from the dried solids precipitates.

Table 1. Operating conditions and parameters used in acid leaching procedure of WAS.

| Run | M of Leaching agent ($H_2SO_4$) | WAS: Acid | Leaching time | Temperature |
|---|---|---|---|---|
| 1 | 0.01 | 1,765:1 | different residence time 15, 30, 45, 60, 90 and 120 mins | Room temperature |
| 2 | 0.05 | 39.5:1 | | |
| 3 | 0.1 | 179:1 | | |
| 4 | 0.5 | 395:1 | | |
| 5 | 0.01 | 1,765:1 | | 50°C |
| 6 | 0.05 | 39.5:1 | | |
| 7 | 0.1 | 179:1 | | |
| 8 | 0.5 | 395:1 | | |

### 2. 4 Analysis and Calculations

The colorimetric approach using vanadomolybdophosphoric acid and ultraviolet–visible spectroscopy was used to determine P and soluble-P. Mg and Ca were measured with AAS (atomic adsorption spectrophotometer). The data were expressed using mean value and standard deviations (S.D.). The properties of the samples (P and total solid (TS)) were determined using Standard Methods (APHA, 2012). If there were any statistically significant differences between the leaching experiments, 16 runs for acid leaching of P recovery and percentage P content in solids and liquids, the researchers performed an independent T test and a one-way analysis of variance (ANOVA). SPSS statistics 18.0 for Windows, a Mahidol University-licensed program, was used to conduct all of the tests.

Phosphorus extraction: The P extraction efficiency defined in Equation (1) was determined by the difference of initial and final $PO^{4-}$concentrations in experiments, as shown in Equation (1).

$$\text{Extraction efficiency (\%)} = \frac{P_{\text{extracted}}}{P_{\text{WAS}}} \times 100 \qquad (1)$$

In which $P_{\text{extrated}}$ is the extracted phosphate concentrations (mg/l) and $P_{\text{WAS}}$ is WAS phosphate concentrations (mg/l).

Phosphorus recovery: The P recovery efficiency defined in Equation (2) was determined by the difference of initial and final $PO^{4-}$concentrations in experiments, as shown in Equation (2).

$$(\%) \quad P_{\text{recovery}} = \left[\frac{[PO_4^{3-}\text{-}P]_{\text{initial}} - [PO_4^{3-}\text{-}P]_{\text{final}}}{[PO_4^{3-}\text{-}P]_{\text{initial}}}\right] \times 100 \qquad (2)$$

In which $[PO_4^{3-}\text{-}P]_{\text{initial}}$ is the initial phosphate concentrations (mg/l) and $[PO_4^{3-}\text{-}P]_{\text{final}}$ is final phosphate concentrations (mg/l).

The mass sense of balance investigation of P, Ca and Mg during the MAP precipitation was approved out summarizing concentration P, Ca and Mg in liquid (supernatant), and magnesium ammonium phosphate. The mass balance was then established with a deviation of less than 10% when compared to the corresponding values of the initial concentration, as indicated in Eq (3):

$$\begin{gathered} M_{in} = M_{out} \\ [M_{in}] = [M_{solid}] + [M_{liquid}] \end{gathered} \qquad (3)$$

In which $M_{in}$ is the liquid phase input of mass P, Ca and Mg, $M_{out}$ is the output of mass P and Mg, $M_{solid}$ is solid mass P, Ca and Mg, $M_{liquid}$ is the precipitation of mass P, Ca and Mg in precipitation.

## 3. RESULTS AND DISCUSSION

### 3. 1 Effect of P extraction using Sulfuric Acid Leaching

A. The effect of acid concentration on leaching P

The leaching process is affected via acid concentration. Figure 1, shows the average results obtained while leaching P from WAS at room temperature (25-30°C) and 50°C for various acid concentrations and times. Whereas environmental leaching has been used, the extraction temperature was determined by the boiling point of the leaching solution. Figure 1, shows the influence of $H_2SO_4$, concentration on P leaching.

The amount of P extraction increases with increase in acid concentration from figure 1. This trend was only observed with acid concentrations that were below 1 M. Figure 1, represents the time profiles of P extraction with 0.01 and 0.05 $H_2SO_4$ concentration. P extraction reached maximum at 35 % and 44 % (226 and 291 mg P/m$^3$ at room and 50°C) of TP with 0.01 $H_2SO_4$ and concentration of 0.05 M, which P extraction extended at 44 % and 54% (263 and 339 mg P at temperature of room and 50°C). At concentrations of 0.1 M and 0.5 M, the leaching of P extraction is reached >90%. At the concentration (0.05 and 0.1M, 0.05M and 0.1M) are statistically significant ($p<0.05$). However, the concentration of 0.1 M and 0.5 M is not statistically significant. Above results indicated that the concentration of 0.1 M $H_2SO_4$ was more beneficial to P extraction for optimum leaching. High acid concentration reduced the degree of P extraction, this may be due to the metals present which acted as nucleation particles in the centre matrix of the sludge [23, 24]. [25] observed that with acid concentrations that were below 1 M. At high acid concentrations (above 1 M) the amount of phosphorus extraction started to decrease by a factor of about 10%.

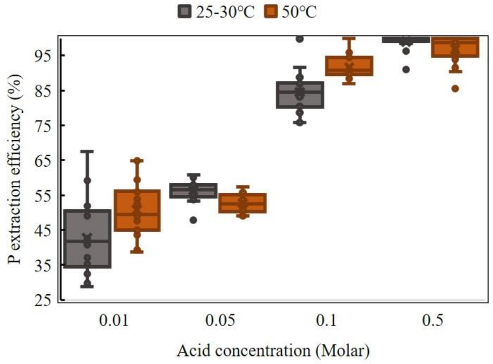

Figure 1. Phosphorus extraction at acid concentration (different time and different temperature).

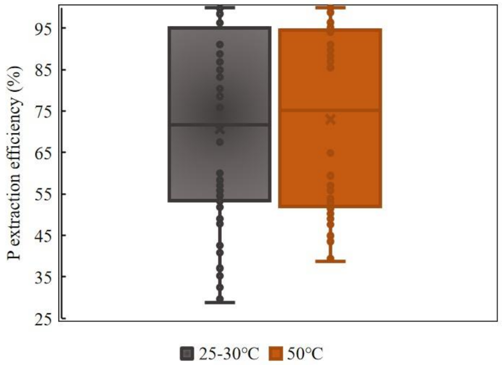

Figure 2. Phosphorus extraction at different temperature (acid concentration lower level 0.01M to upper 0.5M and time 15mins to 120mins).

B. The effect of temperature on leaching of P

Temperature was found to affect the leaching of elements from WAS by using sulfuric acid as illustrated in Figure. 2, which shows the extraction percentage by using 0.01, 0.05, 0.1 and 0.5 M $H_2SO_4$ as a function of room temperature (25-30°C) and 50°C. The leaching percentage increases smoothly increasing from room temperature to 50°C for P at different time and different acid concentration. This result can be explained by the fact that an increase in temperature provides energy necessary for breakdown of the chemical bonds of the metals in the sludge [26]. On the other hand, the leaching percentage for P remains constant at room temperature to 50°C, suggesting that iron compound in WAS needs medium energy (in the form of either temperature or acid concentration) to be broken. As shown in Figure. 2, increasing the temperature from room temperature to 50°C is not statistically significant. Therefore, room temperature was determined to be suitable for leaching.

C. The effect of contact time on leaching of P

Figure. 3, shows the results obtained from the leaching of phosphorus from WAS at different residence times. An increase in residence time increased the amount of P extraction. The percentage of P extraction from 0.01M to 0.5M of WAS were found as 30%-68% room temperature and 39%-65% (50°C), 48%-60% room temperature and 49%-59% (50°C), 76%-100% room temperature and 87%-100% (50°C) 91%-100% room temperature and 85%-100% (50°C) from 15 min to 120 min, respectively. A comparison of the %P with leaching time using a single factor analysis of variance test (ANOVA) indicates that the difference in the mean %P extraction levels obtained at different residence times are not statistically significant. However, the highest amount of P extraction was 98 % at 30 min and its stability thereafter. Thus, the desired time for extraction of P was set to 30 min.

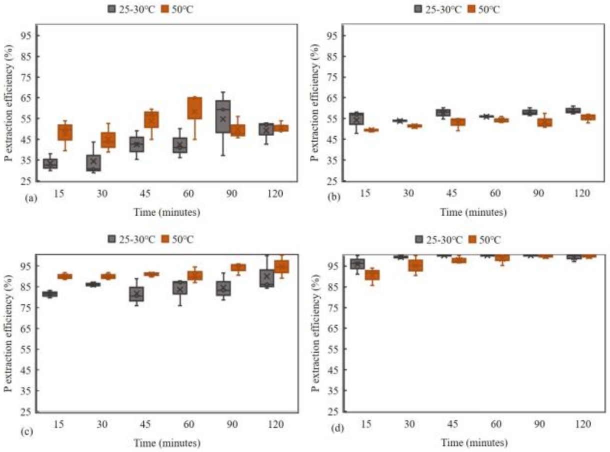

Figure 3. Amount of phosphorus extracted at different residence times (different acid condition (a) 0.01 M (b) 0.05 M (c) 0.1 M and (d) 0.5 M and temperature (25-30°C and 50°C).

A possible reason would be that increasing the contact time leads to more broken bonds and increases the possibility of the occurrence of reactions. In other words, a prolonged leaching time or a higher contact time causes more P compounds to dissolve in the acid. These in turn increase the extraction. However, too long a contact time can decrease extraction efficiency. This can happen due to evaporation of the acid. This dries the leached mass and consequently the desired reactions are prevented. This shows that if the contact time is prolonged, undesirable, and adverse reactions are likely to occur and insoluble compounds may be formed. All this may reduce the efficiency of extraction. Generally, the contact time should not be longer than required [27].

## 3. 2 P recovery via precipitation

The prepared precipitation was carried out at various molar ratios and pH values, as well as the corresponding characteristic P percentages in solid particles and liquid in precipitation processes with pH and starting P concentration variations. For struvite precipitation, a wide range of molar ratios can be utilized. $NH^{4+}$ and $Mg^{2+}$ concentrations below 1 result in poorer N and COD elimination, according to [28]. Most researchers discovered that the most efficient molar ratio for each element should be between 1 and 2, with 80-90 percent recoveries. According to [29] , raising the molar ratio causes greater supersaturation, which leads to increased nucleation and crystal development, but the ultimate pH prevents further precipitation. Chemical precipitation was used to assess the feasibility of recovering P. Two doses of MgO (Mg addition Mg/Ca= 2) at pH 9 improved phosphorus recovery and increased orthophosphate concentrations in the supernatant. In the Studies have shown that affects the precipitation of orthophosphate at two doses of MgO with pH. At two dosages of MgO (Mg addition Mg/Ca=2), the efficiency of P recoveries was investigated to be 93 percent.

## 3. 3 P recovery as different P fertilizer

In this study, extracted P from WAS containing approximately 424 mg P·L $^{-1}$ was used as the P source. The percentage of P content in the solid precipitate is an important measure for developing an alternative P fertilizer. Mg addition (Mg/Ca=2) at pH 9, the percent P content in the solid precipitate was in the range of 12% for all ash types studied. Of these, the percentage of P content in the solid precipitate at the magnesium dose for Mg addition (Mg/Ca=2) at pH 9 was higher to the P content in commercial superphosphate fertilizer at 12 respectively. Therefore, the P containing WAS obtained from both experiments can be used as an alternative P-fertilizer. In addition, the precipitates from other experiments with P content less than 8.80% can be used as soil conditioner due to their good nutrient qualities, especially P [30]. The recoverable P-containing product from urine mixed with other soil amendments could be used as a fertilizer, which contains many important nutrients and contributes less heavy metals to the soils [31]. Mg/Ca=2 can obtain higher P content than commercial P fertilizer, according to the findings (P 8.80 percent). Thailand's food security is greatly helped in sustainable P management. To attain this useful function, recoverable P can indeed be needed in order to make fertilizers in crop production.

## 3. 4 Situation of phosphorus flow analysis

In this study, the overall P flows obtained from the P-extraction was emphasized by the Sankey diagram presented in Figure 4. They are represented as vertical input (import) and output (products) flows on the Sankey diagrams. The width of each flow is proportional to its mass and values are reported in grams (g). The phosphorus flow starts on the left-hand side of the diagram with the batch preparation of 428 g P/m$^3$ of waste activated sludge materials. The P extracted in the acid leaching, resulting in intermediate supernatant of 98% (362 g P), due to the decomposition of WAS. This production of about 362 g P is mainly dedicated to exports (about 230 g P or 93%). The rest of the production goes to deposit (about 66 g P or 34% evenly divided between raw and compound feed) from acid leaching and 123 g P from precipitation. Finally, residues of leached and precipitated with 198 g P, the products 230 g P. According to the findings, the research's operation can be P-solid products, as well as benefit of reducing P leakage of WAS to landfills.

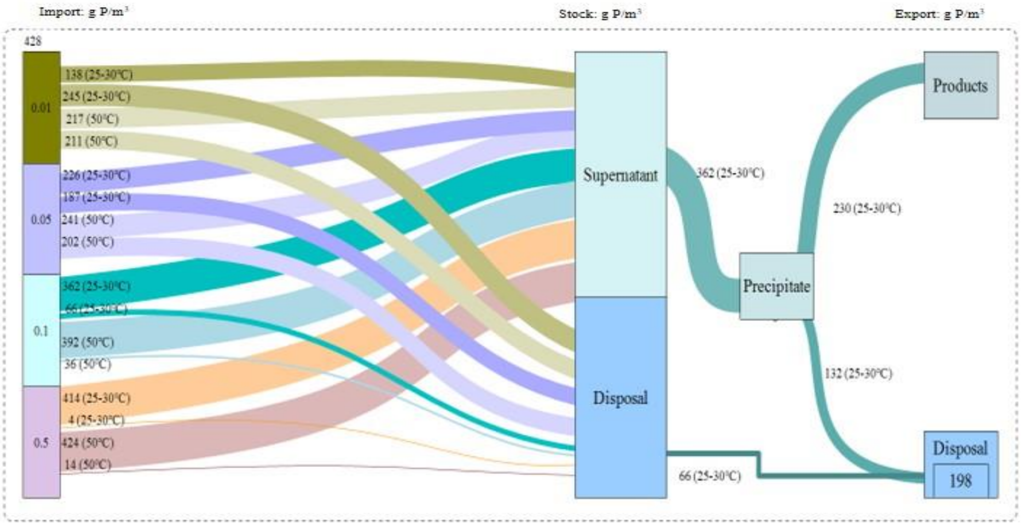

Figure 4. Material flow analysis (MFA) for waste activated sludge in chemical P-extraction through a Sankey diagram at 30 min. All flows are in g P/m3.

## 4.CONCLUSIONS

Acid leaching and sea salt precipitation of P from WAS appears to be a potential strategy for recovering the limited nutrient P. WAS was used to recover 98% of the P, which was mostly made up of calcium and magnesium phosphates. After 30 min of reaction with 0.1 M $H_2SO_4$, optimum conditions for P extraction were formed, necessitating precipitation. Furthermore, the percentage of solid P extracted form WAS was around 93%. P was found to be 12 % in solid precipitates, which is higher than the commercial single superphosphate fertilizer (8.80 percent P). As somewhat of a conclusion, the research adds to our understanding more about how to help decrease the need for chemical P fertilizers thereby also providing a dependable genetic composition for this with the sustainable directions of innovative and sustainable, as well as high-efficiency food production systems. Reduced P discharge of WAS to landfills and lower costs of solid deposits at residential wastewater treatment plants could provide additional benefits.

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