Chapter

Simultaneously Recovery of Phosphorus and Potassium Using Bubble Column Reactor as Struvite-K and Implementation on Crop Growth

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Abstract

Struvite-K, similar to NH_4-struvite with a composition of Mg:K:P (1:1:1). It is called struvite-K because the K replaces the NH_4 in struvite. The composition usually used as fertilizer and can be recycling from wastewater including livestock wastewaters. In addition, Struvite-K which tends to form scale on surfaces of equipment which problem in many industries. The present study was used bubble column reactor which simple and efficient. In addition, the process can be implementation in wastewater industry which low-tech processes. Then, the struvite-K precipitate was implementation on crop growth which compared with coffee husk compost. The results show the removal of P via struvite-K showed 98.5% with the precipitation Mg:P of 0.7 and K:P of 1 with yields of 11.28 gram. Increases of magnesium dosage which decreases of P removal rate and affected of crystal size structure. Compost and struvite-K have similar positive impact on crop growth of (radish and komatsuna) were compared than control. In the other hand, the struvite-K is more effective than compost. This might be indicated that struvite-K is more slow-release nutrient than compost and higher macro nutrient supplied on soil which crop needed.

Keywords: Struvite-K, precipitates, soil, recovery of wastewater, fertilizers

1. Introduction

1.1 Bubble column reactor for struvite-K precipitation

Magnesium potassium phosphate hexahydrate, commonly known as struvite-K, is a sparingly crystallize consisting of equal molar amount of magnesium potassium and phosphate and six water of hydration, hence the chemical formula is $MgKPO_4.6H_2O$. Struvite-K is formed according to the following reaction:

$$Mg^{2+} + K^{+} + PO_4^{\ 3-} \rightarrow MgKPO_4.6H_2O \quad (1)$$

Struvite-K has been synthesized to show its viability for phosphorus and potassium removal from wastewater [1–4] and naturally occurring. Tanaka [5] reported that phosphorus and potassium from effluents of livestock wastewater contains (5.5 mM) and (63.9 mM), respectively. This is a new resource for potassium and phosphorus demand for use as fertilizers which following global population in every year. It was estimated the world population to reach between 9 and 10 billion by 2050 [6, 7].

Struvite-K precipitates as a white powder needle-like structure, which tends to form scale on surfaces of equipment. Struvite-K scaling is a well-recognized problem in many industrial processes and domestic applications. The problem is that not only from pipes but also pumps, centrifuges and aerators can be blocked by struvite [8–10]. In the other hand, this is not problem but solution to supplying nutrient on crop growth.

Struvite-K, similar to NH_4-struvite has a desired low solubility and chemical composition of Mg:K:P (1:1:1). It also has a high market value in the agricultural area (Sean 2018). It is called struvite-K because the K replaces the NH_4 in struvite ($MgNH_4PO_4.6H_2O$). The present study, we used synthetic wastewater was designed using bubble column reactor to simulate effluent from livestock wastewater to recovery of phosphorus and potassium as struvite-K which used as fertilizers for increasing global food production.

1.2 Implementation on crop growth

Most of the nutrients absorbed by plants come from organic matter. Therefore, the unique fertilizer formula comes from compost or struvite-K precipitate. They provide a rich source of nutrients and can be added to the soil to promote the growth of various crops. Therefore, compost and struvite-K precipitation are intended to serve the society by increasing farmers' incomes to revitalize the soil and increase farm yields. We fertilized the two fertilizers from compost and struvite-K precipitate, in order to evaluate the effects of fertilizer on soil and yields of radish (*Raphanus sativus* L.) and Komatsuna (*Brassica rapa var. perviridis*).

*1.2.1 Radish (*Raphanus sativus *L.)*

Radish (*Raphanus sativus L.*) belongs to the genus and species of *Radish* in the *cruciferous* family. Radishes are grown and consumed all over the world and are considered part of the human diet, although it is not common in certain populations. It is one of the most important and popular root vegetables in tropical, subtropical and temperate regions of the world (including Japan). It is grown as an annual and biannual vegetable crop, depending on its planting purpose. Radishes are mainly cold-season vegetable crops. Asian varieties can tolerate higher temperatures than European varieties. In a mild climate, radishes can be grown almost all year except for the summer months [10]. Its young roots can be eaten raw in salads or cooked as vegetables. It has a spicy taste and is considered an appetizer. Young leaves can also be cooked and eaten as vegetables. Radish preparations are useful for liver and gallbladder diseases. Roots, leaves, flowers and pods are active against Gram-positive bacteria, urinary system diseases, hemorrhoids and stomach pain. In addition, the salt extracted from the roots can be dried and burned into white ash, which can be used to relieve stomach problems [11].

1.2.2 Komatsuna (Japanese mustard spinach)

Japanese mustard spinach (*Brassica rapa var. perviridis*) is also called Komatsuna in Japanese. It is a common and popular leafy vegetable in the Japanese diet.

Komatsuna contains low energy and high nutrients, which is very effective in lowering serum cholesterol. On the other hand, Komatsuna has a compound called sulforaphane, which can help our body fight cancer. Sulforaphane actively kills cancer stem cells and slows the growth of tumors [12]. Komatsu greens are rich in calcium and are commonly used in kimchi in Japan and as feed crops in many Asian countries.

In 2019, the production of Komatsuna in Japan increased by 26.5% compared with 2006, and the planting area increased by 29.2%, with a total output of 114,900 tons [13]. Komatsuna can be worn in relatively temperate regions most of the year, but it is usually grown as a cool seasonal crop (spring and autumn). It can tolerate some extreme cold and hot conditions, but it cannot stand for long periods of time [14]. Economically important members of this family include vegetables such as broccoli, cabbage, chinese cabbage, radish, cauliflower as well as the oil crop canola [15].

2. Materials and methods

2.1 Materials

Sodium dihydrogen phosphate, magnesium chloride and sodium hydroxide were obtained from the Kanto Chemical Co. Inc. (Tokyo/Japan), potassium chloride was obtained from Wako Pure Chemical Industries Ltd. All chemicals and reagents were of analytical grade and used without further purification.

The soil was collected at 0–20 cm of depth in the field center of Prefectural University of Hiroshima. The soil was analyzed in the laboratory at the Department of Environmental Science, Prefectural University of Hiroshima, Japan. Elemental characteristics of soil and compost were described previous work [16]. Radish (*Raphanus sativus L.*) and komatsuna (*Brassica rapa var. perviridis*) were collected from market store, which is in the Shobara city, Hiroshima Prefecture.

2.2 Experimental design and treatments.

2.2.1 Bubble column reactor for struvite-K precipitation

The use of different magnesium additives to recover $MgKPO_4.6H_2O$ (MPP) was investigated. The experimental conditions are summarized in **Table 1**. Illustrates the experimental setup of the bubble column used in this study which has capacity of 10 L (**Figure 1**). There was a draught tube structure inside the bubble column. Air was fed using an air pump from the bottom of the tower as instead of mixing for homogeneous of solution. A pH probe was placed directly into the reactor just below the liquid surface. The bubble column was first filled with synthetic wastewater, and then potassium chloride, magnesium chloride and sodium dihydrogen phosphate solutions were fed from the outer reactor to the inner tubes. The pH

Run no.	Mg^{2+} mM	PO_4-P mM	K^+ mM	Mg/P initial
1	5.2	6.4	13.4	0.8
2	6.9	6.4	13.4	1.08
3	7.8	6.4	13.4	1.22

Table 1.
Characteristic of raw water.

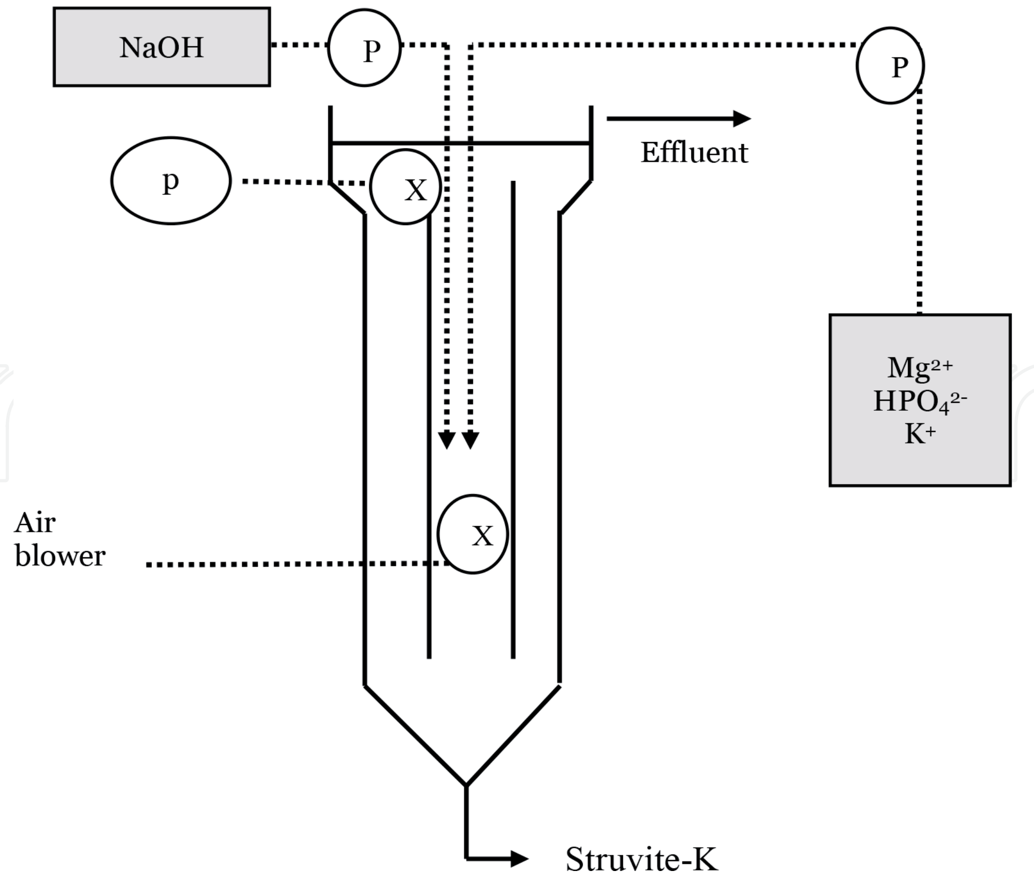

Figure 1.
Experimental set-up.

was adjusted to 12.9 with 0.1 M NaOH solutions. Afterward the solution of potassium chloride, magnesium chloride and sodium dihydrogen phosphate were added continuously with had pH of 3.4 by dose pump until pH of 11 with retention time of 1.98 (defined Eq. (2)).

$$\text{HRT} = \text{Total volume reactor}\,(\text{L}) \,/\, \text{Influent flow rate}\,(\text{L}) \qquad (2)$$

Set points for minimum and maximum pH values defined a narrow band of 0.3 pH units. After filtering the reactor solution, a white precipitation was obtained; it was desiccated at 60°C for 24 h. Samples were taken directly from the precipitation zone. All experiments were done at room temperature.

2.2.2 Pot treatment designs

The experimental design was completely randomized design, with five treatments and three replications were presented by pots. Seeds were soaked in water for 24 h before sowing at a maximum depth of 1.2 cm. The equal proportions of compost samples (150 g and 10 seeds) i,e Radish (*Raphanus sativus L.*) and komatsuna (*Brassica rapa var. perviridis*) were filled and installed in a greenhouse. The LED model (PF15-S5WT8-D with power 5 W) was used as a light source with free space between lamps and a pot about 37 cm. Treatments consisted of C: 100% (control), A1: 0.1% of compost, A2: 0.3% of compost, B1: 0.1% of struvite-K and B2: 0.3% of struvite-K. The pots were watered periodically to prevent drought stress of the plants. The experimental were conducted for 9 days.

2.2.3 Analytical methods of struvite-K precipitates

The concentrations of PO_4^{3-} were determined by standard method (Japan Industrial Standard method JIS KO 102). The concentrations of potassium and magnesium ions were measured using atomic absorption spectrophotometer (AA-6300, Shimadzu Kyoto, Japan). The white precipitation was dissolved in 0.5 M HNO_3 in 1 h for determination of the crystalline components. Microscope images (Olympus CX-32) was used to observe the surface morphology of the crystals. The percent P removal and P recovery are in Eqs. (3) and (4), respectively.

$$P\ removal\% = \frac{P\ Initial - P\ equilibrium}{P\ Initial} 100 \quad (3)$$

$$P\ recovery\% = \frac{P\ in\ white\ precipitation}{P\ decrement} 100 \quad (4)$$

2.2.4 Analytical methods of soil

The sample was ground by using a coffee mill to pass through a sieve <2-mm before analysis [16]. Cation exchange capacity (CEC) were extracted 1 M NH_4OAc pH 7 via NH_4 with the procedure in below:

$$\text{CEC}(\text{meq}/100\,\text{g}) = 13.7 * \text{NH}_4\,(\text{mg}/\text{L}) * \text{dilution}/3 \quad (5)$$

The ammonium (NH_4) was measured by the phenate spectrophotometric with UV–Vis Spectrophotometer (Jasco V-530) at 640 nm. The detailed procedure in below:

Solution A

1. Phenol 5 gram
2. Sodium nitriferricyonide dehydrate 0.1 gram
3. Homogenized and adjusted with dilution water 500 mL

Solution B

1. 200 ml of NaCLO (assay 5%) = 200/5 = 40 mL
2. NaOH = 15 gram
3. Homogenized and adjusted with dilution water 1000 mL

Procedure:

10 mL from the liquid sample and 5 ml from solution A and B, respectively. Afterward, waiting for 30 minutes before analysis. Standard solution was used NH_4Cl with calibration curve, y = 0.1747x – 0.1465. R^2 = 0.9999.

3. Results and discussion

3.1 Effect different initial of Mg:P ratios on % P removal and precipitate

Bubble column reactor was used in laboratory studies with hopefully, it can be implementation in wastewater industry. From a structural point of view, they are very simple units equipped with added a dose pump as mixing system that allows for the homogenization of the wastewater with the reactants. The mixing condition inside the reactor represents a fundamental aspect because it affects the struvite formation [17]. An effective mixing promotes the crystals nucleation and growth by improving the mass of ions from the solution to the solid phase. Generally, completely mixed reactors can operate continuously or in batch mode. A batch column reactor works according to a series of phase and the struvite-K production and precipitation occur in the same unit (**Figure 2**).

In this study, we used $MgCl_2.6H_2O$ as magnesium source which have advantages of being very soluble allowing to recovery a precipitate with a high purity degree [18]. Due to easy management was used in many studies through which the kinetics of the nucleation process have been assessed [19, 20]. While the pH was maintained in alkaline condition of 11 which following based on [21]. Since the conditions favorable precipitation of struvite-K. Based on **Figure 3** shows that P removal rate was decreased from 98.5–80% which increasing of molar ratios of Mg:P from 0.8 to 1.22 since strong influence of precipitation in many scientist [22]. While for hydraulic retention time we maintained at 1.98 h; since in around of this was much more consistent than the rate reported in the literature and no effect with difference of HRT in precipitation [3, 4]. Furthermore, P recovery of struvite-K we conducted only for Mg:P of 0,8 since these concentrations was effective for % P removal than others with precipitate of Mg:P of 0,7 and K:P of 1 are shown in **Table 2**.

The optimal molar ratios must be assessed case-by-case as it depends heavily on the chemical–physical characteristics of wastewater and the reactor used. On the contrary, other works in which unconventional reagents were exploited, found that greater dosages are necessary. For example, Quintana et al. [23] found a strong influence a Mg:P molar ratio on the abatement of phosphate amount and the major removal was detected when used MgO dosed at molar ratio of 1.5. Moreover, the authors observed that the increase of the molar ratio promoted the removal rate growth (Quintana et al. [24]). In agreement with this consideration, Jaffer et al. [25] affirmed that with Mg:P addition lower than 1.05 the precipitation resulted was greater to avoid the formation of magnesium sodium phosphate [4].

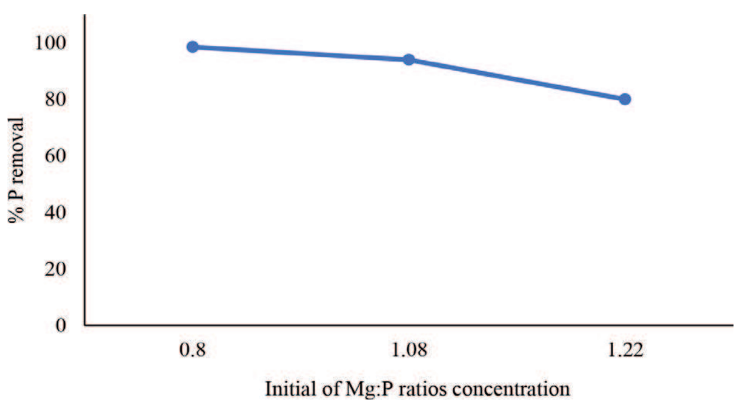

Figure 2.
Percentage of P removal with different of initial of Mg:P ratios concentration.

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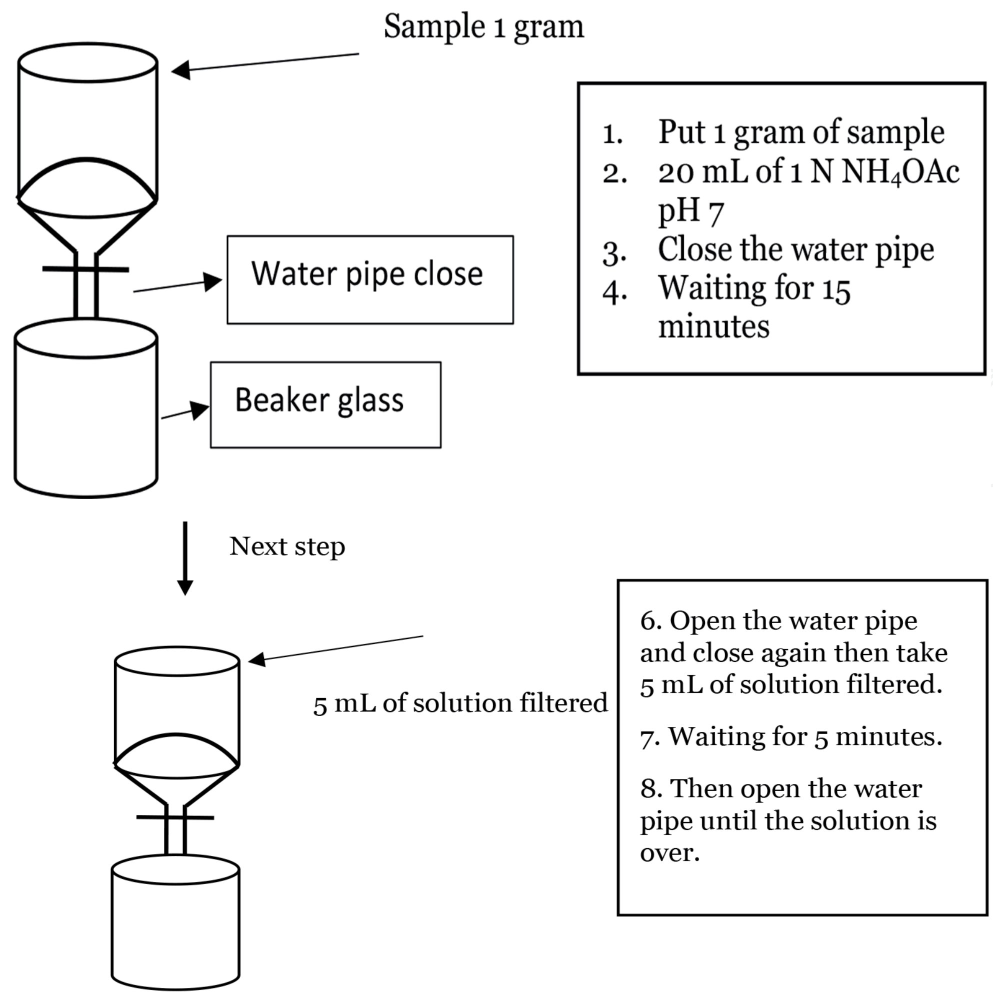

b. Extracted by using 10% NaCL
1. After filtered of solution is over, measure CEC via NH_4
2. Make a solution 10% NaCL

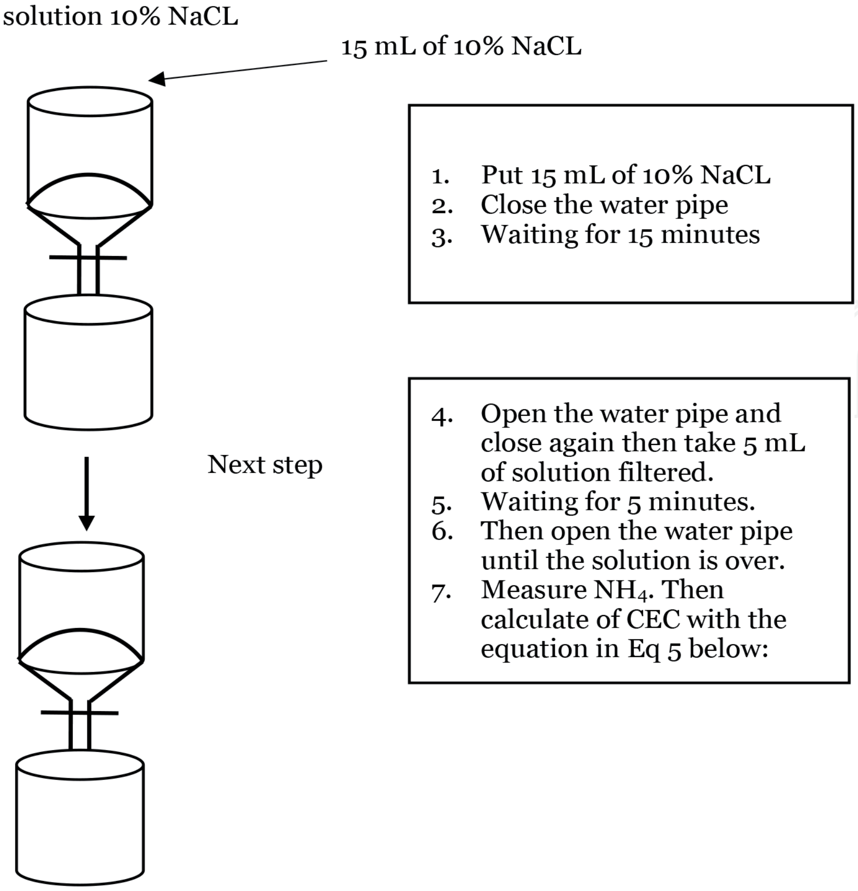

Figure 3.
Procedure for measure exchangeable cation and CEC via NH_4.

pH	Dosage Mg:P ratio	Mg:P (precipitate)	K:P (precipitate)	% P recovery
11	0.8	0.7	1	99.6

Table 2.
Effect of the magnesium dosage on the Mg:P molar concentration ratio in precipitate.

However, an excess of Mg can cause the formation of magnesium phosphate and reducing the precipitation of struvite which following for P removal rate. Korchef et al. [26] observed the phosphate removal caused by precipitation for Mg:P molar ratio under 4 and newberyite ($MgHPO_4.3H_2O$) and cattiite ($Mg_3(PO_4)_2.22H2O$) formation for Mg:P = 5.

3.2 Morphology of struvite-K precipitation

The morphology of struvite-K was observed by using Microscope images (Olympus CX-32). **Figure 4A** shows needle-like structure. This is agreement with some reported [3, 4, 27, 28]. While for **Figure 4B**, the needle-like was mixed with block and hexagonal structure. Furthermore, there are more blocks and hexagons seen in **Figure 4C**. This shows that as the amount of magnesium increases and the removal rate of phosphorus decreases, the structure is affected which mixed with another structure. The increase in the magnesium dosage also reduces the crystal size. The average of crystal size of 22.45 μm (**Figure 4A**). This is agreement with [29] that increase magnesium dosage reduced the average crystal size. Struvite-K forms a white crystalline solid. When dried in 60°C the struvite-K precipitated into appearance of white powder as shown in **Figure 4D**. While for the struvite-K yields of 11.28 gram.

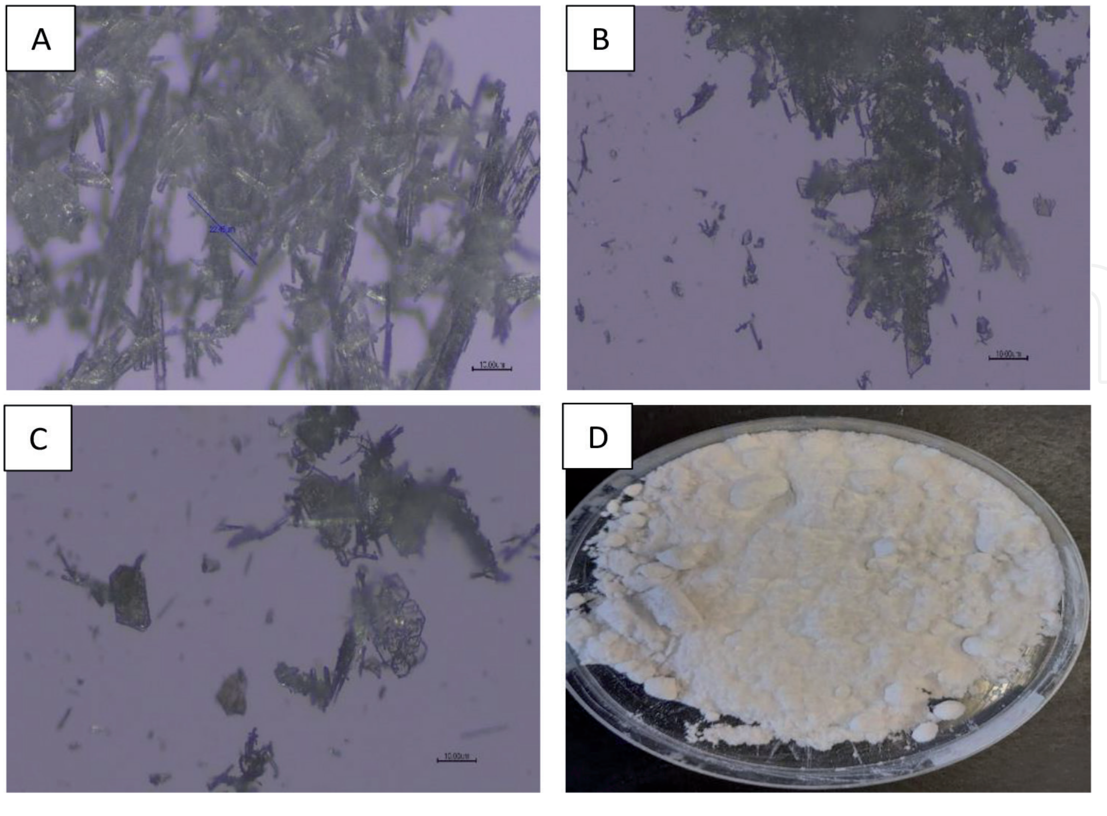

Figure 4.
Struvite-K crystals as seen x10/0.25 of Olympus. Image captured with microscope of Olympus CX-32. (A) Initial Mg:P of 0.8, (B) initial Mg:P 1.08, (C) initial Mg:P of 1.22 and (D) image of struvite-K precipitated dried (crystal of white powder).

3.3 Effect of fertilizers on soil cation exchange capacity

The cation exchange capacity (CEC) is a very important soil property for nutrient retention and supply and acts as a bridge between soil and plant [30]. **Table 3** presents the characteristics of soil CEC on two-kind of crop growth (radish and komatsuna). The results shows that all treatment of fertilizer (compost and struvite-K) with two-kind of crop growth were higher than control (soil only). However, the highest results seen in treatment of struvite-K with 0.3% percent ratio of soil for both of crop growth. This is indicated that struvite-K is effective as supplying of nutrient sources which have higher macro nutrient such as phosphate, magnesium and potassium. This is agreement with [31] who found application of phosphate fertilizer increasing of soil CEC after 40 years of application. This can be explained that phosphate, potassium and magnesium is the most important factor affecting on soil CEC.

3.4 Effect of fertilizers on yields of crop growth

As shown in **Table 4** presents the stem height, leaf area of radish. Overall, the treatment of fertilizers was highest than control (soil only).

In the treatment of compost, the highest rate of 0.3% with 4.46 and 0.98 cm on stem height and leaf area, respectively. While for treatment of struvite-K the

Treatment	CEC (meq/100 g)
Komatsuna	
Control	55.3
Compost 0.1%	71.8
Compost 0.3%	74.1
Struvite-K 0.1%	72.5
Struvite-K 0.3%	94.0
Radish	
Control	57.1
Compost 0.1%	67.8
Compost 0.3%	72.3
Struvite-K 0.1%	69.1
Struvite-K 0.3%	86.4

Table 3.
Characteristics of soil cation exchange capacity.

Treatment	Stem length (cm)	Leaf wide (cm)
Control	2.67	0.43
Compost 0.1%	3.08	0.68
Compost 0.3%	4.46	0.98
struvite-K 0.1%	4.38	0.8
struvite-K 0.3%	6.36	1.31

Table 4.
*Radish (*Raphanus sativus L.*).*

highest rate of 0.3% with 6.36 and 1.31 cm on stem height and leaf area, respectively. For images of radish on pot treatment as shown in **Figure 5**. Both of fertilizers were given benefits effect on yields of radish and positive correlation with soil CEC. If we comparison, the struvite-K was given more benefits effect which cause the struvite-K is higher contains of macro content such as magnesium, potassium and phosphate as nutrient sources on crop growth. Furthermore, **Table 5** presents the stem height and leaf area of komatsuna. The same dosage treatment with radish between 0.1% and 0.3%. Overall, the supplying of fertilizers was given benefits effect on yields of komatsuna. However, the more benefits in the treatment of

Figure 5.
Images of radish on pot treatment; (C) 100% (control), (A1) 0.1% of compost, (A2) 0.3% of compost, (B1) 0.1% of struvite-K and (B2) 0.3% of struvite-K.

Treatment	Stem length	Leaf wide
Control	3.43	0.58
Compost 0.1%	4.42	0.87
Compost 0.3%	5.4	1.22
struvite-K 0.1%	4.36	0.99
struvite-K 0.3%	5.74	1.27

Table 5.
Komatsuna (Japanese mustard plant).

Figure 6.
Images of komatsuna on pot treatment, (C) 100% (control), (A1) 0.1% of compost, (A2) 0.3% of compost, (B1) 0.1% of struvite-K and (B2) 0.3% of struvite-K.

struvite-K on dosage of 0.3% with 5.24 and 1.27 cm, on stem length and leaf wide, respectively. This might be indicated that struvite-K is more slow-release than compost and higher macro nutrient supplied on soil which needed on crop. Slow-release fertilizer which releases nutrients slowly over a long release time. Furthermore, it can reduce the number fertilization times and amount of fertilizer applied, which have good impact on fertilizer use efficiency [32–35]. Images of komatsuna on pot treatment as shown in **Figure 6**.

4. Conclusion

The present study used bubble column reactor which simple and efficient. Removal of P via struvite-K showed 98.5% with the precipitates Mg:P of 0.7 and K:P of 1. The yields of 11.28 gram. Compost and struvite-K have positive impact on crop growth of (radish and komatsuna) were compared than control. Which might be caused supplied nutrient source on soil and uptake on crop. However, the struvite-K is more effective than compost may cause contains higher macro nutrient such as magnesium, potassium and phosphate. This is indicated that the recovery process of P and K via struvite-K using bubble column reactor was very effective and efficient to utilization as a fertilizer.

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Conflict of interest

The authors declare no conflict of interest.

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