Chapter 5

Phosphorus Recovery by Crystallization

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Additional information is available at the end of the chapter

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Abstract

A bubble column, a fluidized bed, and a continuous stirred tank reactor were evaluated as equipment for recovering phosphorus from various wastewaters. Magnesium was added to the solution which contained ammonia and potassium with phosphorus at high concentrations such as livestock wastewater, dehydrated water from a sewage plant, and synthetic livestock wastewater. Magnesium ammonium phosphate or magnesium potassium phosphate could be recovered by adjusting the pH of the solution. Alternatively, calcium was added and then the pH was adjusted to obtain hydroxyapatite solid conditions without seed crystals.

Keywords: magnesium ammonium phosphate, magnesium potassium, phosphate, hydroxyapatite, crystallization, phosphorus, bubble column with draft tube, fluidized bed reactor, continuous stirred tank reactor, seed recycling

1. Background

1.1. Types of crystallization for phosphorus recovery

Crystallization is considered a useful method for phosphorus recovery from various aqueous phases. The technique converts liquid phase phosphorus (usually present as a phosphate-type anion) to solid phase by utilizing the solubility products of a phosphorus-containing salt. Crystals of the relevant reactants are charged to a reactor. When the pH is adjusted to a region where the formation of new fine crystals does not occur, deposition occurs on the seed crystal. There are several dephosphorization methods, the selection of which depends on the composition of the wastewater to be treated. Whether calcium and magnesium salts are chosen depends on the relative concentrations of ammonium or potassium ions and phosphorus already present in the wastewater.

The chemical reaction for recovery of phosphorus by precipitation as magnesium ammonium phosphate (MAP, struvite) and its solubility product pK [1] is presented in Eq. (1):

$$Mg^{2+} + HPO_4^{\ 2-} + NH_4^{\ +} + 6H_2O \rightarrow MgNH_4PO_4 \cdot 6H_2O \quad pK = 13.76 \tag{1}$$

The reaction for the production of magnesium potassium phosphate (MPP) and pK [2] is presented in Eq. (2):

$$Mg^{2+} + HPO_4^{\ 2-} + K^+ + 6H_2O \rightarrow MgNH_4PO_4 \cdot 6H_2O + H_2O \ pK = 11.67 \tag{2}$$

Eq. (3) shows the reaction for the production of hydroxyapatite (HAP) [3]:

$$5Ca^{2+} + 3PO_4^{\ 3-} + OH^- \rightarrow Ca_5\,(PO_4)_3(OH) \quad pK = 57 \tag{3}$$

Phosphorus recovery by MAP and MPP crystallization required high concentration of ammonium and potassium as coexisting species, respectively, and phosphorus reacts efficiently when the solubility products are exceeded by addition of magnesium and adjustment of pH [4–6]. Wastewaters that satisfy these crystallization conditions are limited. To spread and establish this technology, it needs to be applicable to wastewaters with inadequate crystallization conditions and still obtain good results.

HAP techniques at low concentrations are well established. At high-concentration conditions, fine crystals are formed and there is concern regarding their outflow.

1.2. Reactor

In the crystallization process, stable flow conditions and a crystallizer are ideally preferred. Continuous operation makes it possible to achieve large production volumes. Continuous stirred tank reactors (CSTRs), fluidized bed reactors, and bubble column-type reactors can be employed. The basic equipment for achieving phosphorus recovery from higher concentrations consists of a stirred tank and a settling tank. Crystals in the effluent from the stirred tank become fine particles because of high supersaturation. Such particles are not easy to separate from the water treated, so the precipitate slurry is returned to the stirred tank from the bottom of the settling tank to act as seed crystal. Owing to the possibility of fine solids adhering to the return line and stirring propeller in a CSTR, the use of fluidized bed and bubble column reactors becomes preferable.

The fixed bed reactor used for low concentrations involves feeding the influent through the bottom of a rector that is filled with seed crystal and brought into contact with the seed crystal. In a fluidized bed reactor, the reaction surface area is expanded by fluidizing the seed crystal. The residence time is short, so the degree of supersaturation of the inflow is lowered by circulating treated water. In a bubble column with a draft tube, a circulating flow is generated between the inner and outer pipes by air fed to the inner tube. A sedimentation tank is provided at the upper end of the reaction section so that the crystals generated do not flow out. Seed crystals are not used.

1.3. Aqueous solutions containing phosphorus

Waste liquids containing high concentrations of phosphorus available for recovery include urine, livestock wastewater, sidestream, and sludge incineration ash extract liquid.

1.3.1. Urine

Xu et al. [4] prepared synthetic urine and examined conditions for MPP production. Potassium and potassium concentrations were about 960 and 2000 mg/L. Suitable operating pH values for MPP and MAP production were about 11 and 10, respectively. MAP treatment was also performed in a feed solution containing 240 mg/L-P and 4591 mg/L NH_4-N [5].

1.3.2. Piggery wastewater

Livestock wastewater treatment consisted of primary solid-liquid (S/L) separation and secondary biological treatment of the effluent. MAP treatment of the supernatant of primary treatment of a wastewater containing 985 mg/L NH4-N, 161 mg/L PO4-P, and 797 mg/L was reported [6].

For secondary treated water, it is advantageous for S/L separation of crystals generated in the case of membrane separation activated sludge; however, ammonia and phosphorus are reduced by biological treatment, so there are few attempts to apply MAP precipitation. One of the targets of this research is secondary treated water, particularly for operation conditions at lower concentration levels of phosphorus, ammonium, and potassium than ordinarily MAP and MPP treatments [7, 8].

1.3.3. Synthetic wastewater

Potassium chloride, phosphoric acid, magnesium chloride, and ammonium chloride were used to prepare synthetic wastewater.

1.3.4. Sidestream in sewage plant

When anaerobic digestion of excess sludge is undertaken, the concentration of phosphorus and ammonium ions in the dehydrated filtrate increases, and sufficient efficiency can be obtained using the MAP method. However, at intermediate concentrations, the efficiency of MAP decreases. Therefore, in this study, we also aimed to recover phosphorus from intermediate concentration phosphorus wastewaters. The HAP method using a CSTR reactor without seed crystal and the MAP method using a fluidized bed reactor were applied. HAP treatment was appropriate for operating conditions at higher phosphorus concentrations than ordinarily found for secondary treatment [9].

2. Phosphorus recovery characteristics using magnesium ammonium phosphate (MAP) methods from actual water with a suitable crystallizer

2.1. Magnesium ammonium phosphate treatment of secondary treatment effluent of membrane bioreactor: effects of ammonium ion concentration on phosphorus removal

In many cases of livestock wastewater treatment, the membrane separation activated sludge method is selected in preference to sewage treatment. Because the treated water does not contain suspended solids, it is not necessary to separate the recovered phosphorus and this method is therefore effective for recovering phosphorus. The precipitate of the crystallization treatments can be used as received; however, if a nitrification process is used, the ammonium concentration decreases, which is disadvantageous for MAP. Raw urine was purchased from a livestock farmer and placed in the 2 m^3 urine sump at Kumamoto Prefectural Livestock Research Institute. The solution was progressively moved into the aeration tank at 100 L/day and then treated with a tower-type membrane-activated sludge methods. The treated water was received in a 1 t tank and used as the raw water for MAP treatment experiments. **Figure 1** shows MAP experimental apparatus, which consisted of a reaction region with a draft tube structure and precipitation region for S/L separation, both volumes of 5 L.

The reaction region was 15 cm in diameter and 28 cm in height. A double pipe was installed at the bottom, through which circulating flow was generated by injection air. The hydraulic retention time was set to 10 h and activated sludge-treated water was continuously supplied to the reactor bottom. The pH was maintained at 7.5–8.5 via a pH controller dosing a 0.5 N NaOH solution and aeration at 12 L/min carried out using an air pump. The equipment was located outdoors without temperature control. The experiment was carried out for about 100 days. Sampling was conducted every 2–3 days. Changes in water properties are shown as raw and treated waters in **Figure 2**. We try to remove residual concentration by adsorption. Because the phosphorus concentration in the effluent was not adequately decreased by MAP treatment, further treatment was conducted using adsorption methods. Phosphorus-adsorbent pellets (Limonite Co. Ltd., Kumamoto, Japan) were made from a special soil. The soil was compressed to pellets of 11–13 mm diameter and 10–30 mm length with a binder. The main component of the soil was 62% iron oxide, which exhibited phosphorus adsorption characteristics. The pellets were placed in column of 30 cm height and 10 cm diameter, and MAP-treated water was supplied from the bottom at 20 L/day.

The total phosphate (T-PO_4) content of the raw water approximately 100 mg/L up to 80 days after which decreased to 80 mg/L. The treatment characteristic could be categorized into

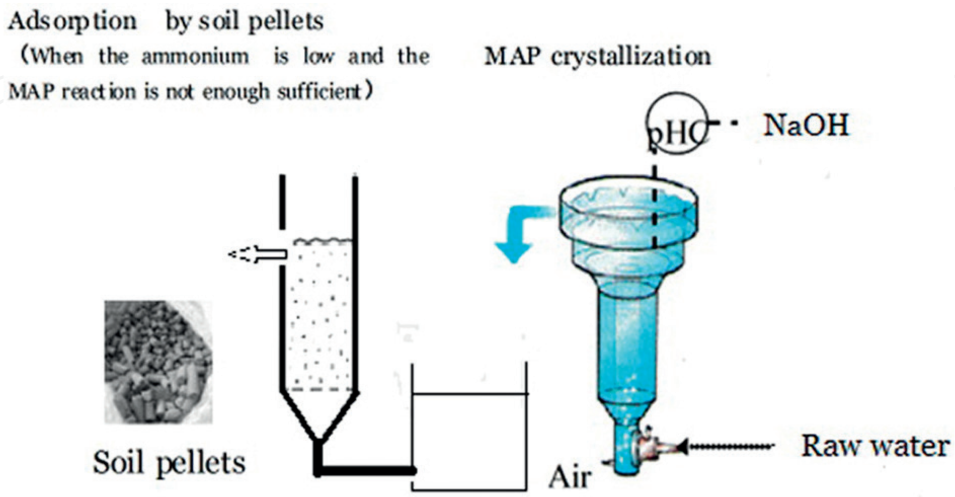


Figure 1. Bubble column with draft tube for magnesium ammonium phosphate (MAP) (or magnesium potassium phosphate, MPP) treatment.

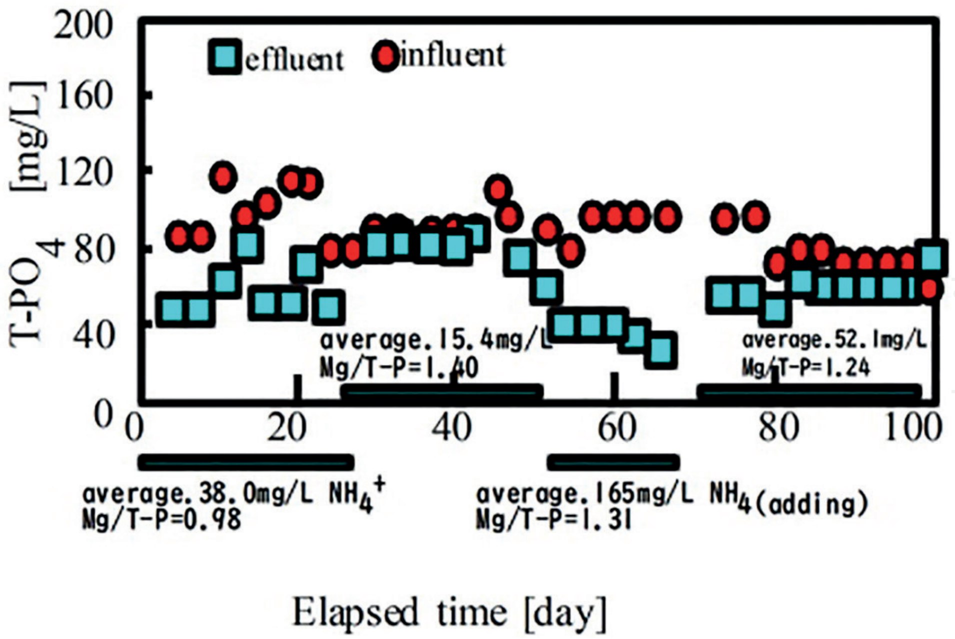


Figure 2. Magnesium ammonium phosphate treatment of secondary livestock wastewaters.

periods: 0–24, 25–54, 50–64, and 71–100 days. The average ammonium concentrations during these periods were 38, 15, 165, and 52 mg/L, respectively. The higher concentrations (165 mg/L) were due to addition of ammonium chloride. The Mg/T-P ratio was maintained at 0.98–1.53. When the raw water phosphoric acid concentration was 100 mg/L, the extent removal increased in relation to the ammonium concentration. Even if the ammonium ion concentration increased, the phosphorus removal decreased when the phosphoric acid concentration decreased to 80 mg/L. Weijia et al. [7] also considered the addition of magnesium and ammonium to phosphorus recovery using a bubble column, because the phosphorus and ammonium concentrations of the activated sludge secondary treatment are reduced by nitrification and biological uptake. The results showed about 50% phosphorus removal and that remaining treatment was required. Attempts have also been made to add magnesium and ammonium to increase the MAP production when the ammonium concentration is low [7]. The chemical reaction for recovery of phosphorus by precipitation as magnesium (MAP, struvite) is shown in **Figure 3**.

The adsorption results are shown in **Figure 4**. The phosphorus concentration of MAP-treated water was in the range 44–58 mg/L but decreased to 7–21 mg/L because of adsorption. The total average removal efficiency was 74%.

2.2. Magnesium ammonium phosphate treatment of sidestream in sewage treatment: high-speed solid-liquid separation

High concentrations of phosphorus and ammonium ions are contained in the dehydrated filtrate of the sewage, and fine crystals are precipitated unless adequate S/L separation is performed. The average total phosphorus and ammonium concentrations of the dehydrated filtrate at the North Area Kumamoto sewage treatment plant during the test period were 86.4 and 600 mg/L, respectively.

Figure 3. Magnesium ammonium phosphate crystals.

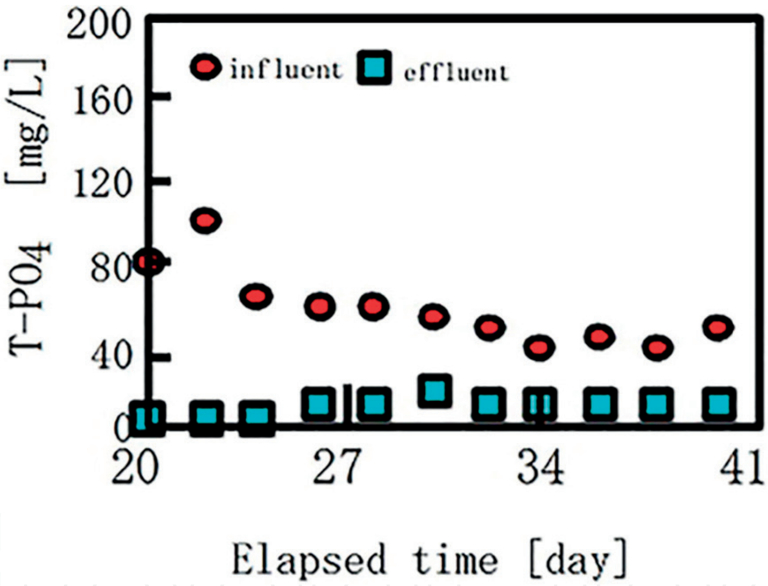


Figure 4. Adsorption treatment of magnesium ammonium phosphate effluent.

2.2.1. Fluidized bed reactor

The fluidized bed reactor apparatus is shown in **Figure 5**. The reactor was acrylic and had a diameter of 2 cm and height of 100 cm. There was no settling region.

The bottom part was filled to a height of 4 cm with glass beads with a diameter of 5 mm to ensure laminar flow. The pH of the raw was around 6.7, and there was almost no change in the phosphorus concentration on adding magnesium. To reduce pH fluctuations, part of the sidestream was diluted with raw water at 1:1. The mixed water was withdrawn and then supplied to the reactor.

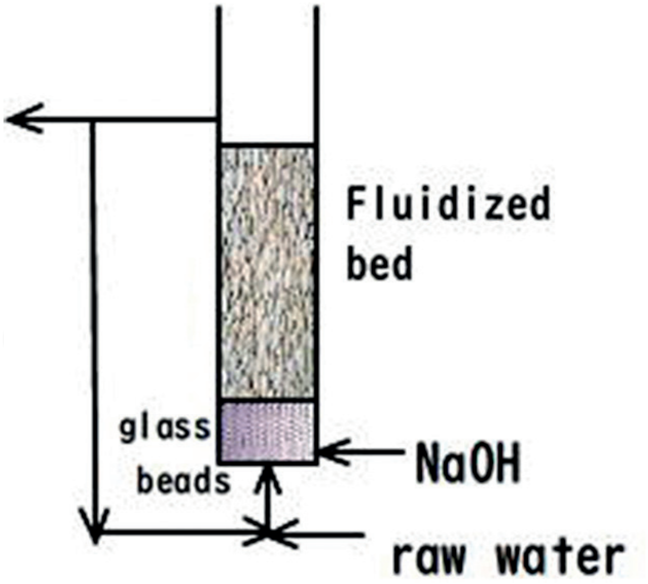


Figure 5. Fluidized bed reactor.

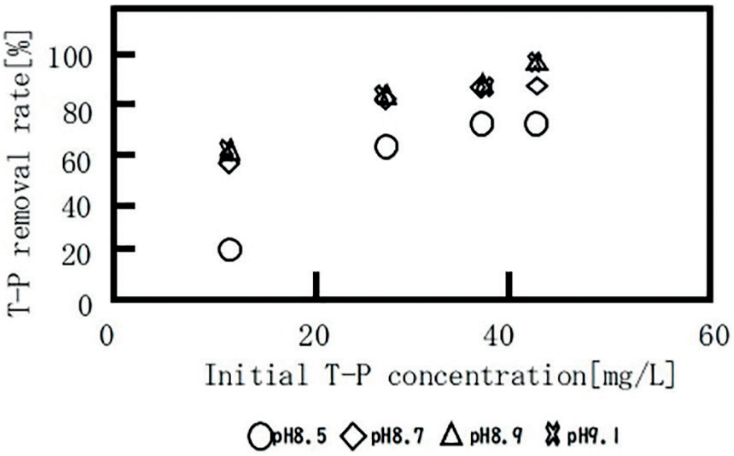


Figure 6. Relationship between initial total phosphorus (T–P) concentration and extent of its removal as a function of pH of effluent.

Solids were suspended by injecting the fluid in an upward direction. Contact between the solid and liquid was large and their superficial velocity was high. Based on these advantages, the MAP rapidly precipitated and deposited on the solid particles, enabling a very short processing time. To lower the degree of supersaturation at the inlet, circulate treated water and mix with raw water to supply. The lack of need for a sedimentation tank also contributed in processing time. **Figure 6** shows the relationship between T-P removal efficiency and pH at different initial P concentrations. At an initial concentration of 11.4 mg/L (PO_4: 34.2 mg/L), the phosphorus removal was about 20% at pH 8.5, but this increased to approximately 60% as the pH increased to 9.1. At an initial concentration 27.2 mg/L (PO_4: 81.6 mg/L), the removal reached 80%. Uludag-Demirer and Othman [8] investigated removal from wastewater containing phosphate 100–1500 mg/L and reported 48% removal at pH 9.0 for water containing

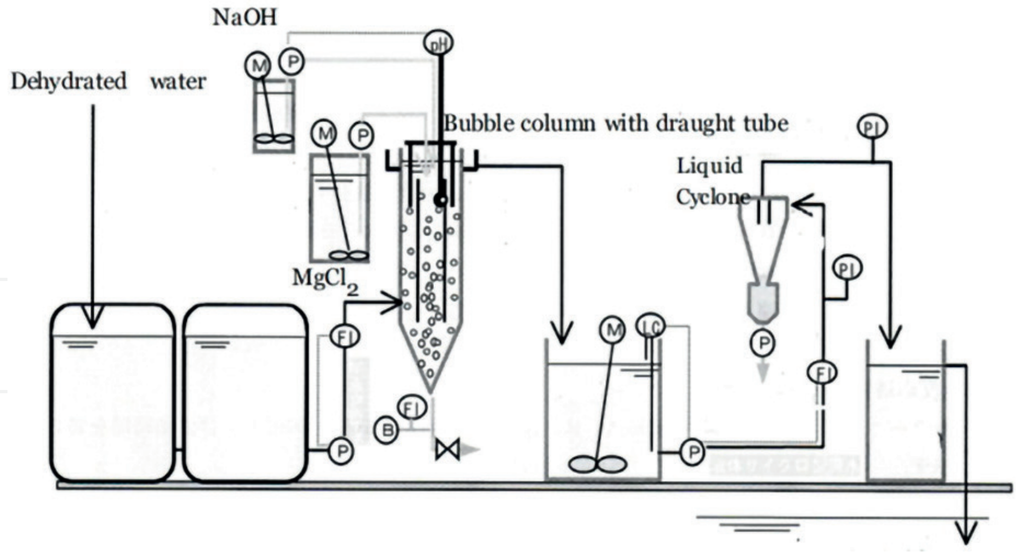


Figure 7. System configuration of mini-plant in sewage treatment center.

100 mg/L phosphate. Guadie et al. [9] also investigated for high (120 mg/L)- and low (12.5 mg/L)-concentration wastewaters. They found that T-P removal at low concentration could be improved by circulation of the treatment water. Su et al. [10] pointed out the effects of seed crystal.

2.2.2. Mini-plant experiment: combination of bubble column with draft tube and liquid cyclone

Unitika Co. Ltd. Central Research Center (Kyoto, Japan) set up a small-scale MAP reactor in a sewage treatment center (Kumamoto, Japan) and applied the technique to the dehydrated liquid of methane fermentation. An overview of the plant is shown in **Figure 7**. The reactor contained an inner cylinder and circulating flow was obtained by feeding air to the inside. A long residence time was necessary to prevent the outflow of solids, so it was not suitable for treatment of large volume. We introduced a hydro-cyclone to minimize the residence time and made the water surface area of the separation section as small as possible. The design influent rate was 5 m^3/day, and the instantaneous supply to hydro-cyclone was 2 m^3/day. The reaction volume was 86 L; the residence time in the reaction and separation regions was 25 and 1.6 min, respectively. The average TP removal efficiency was 53.3% for raw water average concentration 94.7 mg/L (maximum 105 mg/L, minimum 80.7 mg/L). When a hydro-cyclone was applied, the removal improved 77.6%; however, if a longer retention time 4.6 min was applied to the separation, the average TP removal increased to 86.7% in the reactor alone [12].

3. Phosphorus recovery characteristics using hydroxyapatite (HAP) methods from actual water

For experiments under high concentrations, actual dehydrate liquids were collected from the sewage treatment center in Kumamoto Prefecture, Japan.

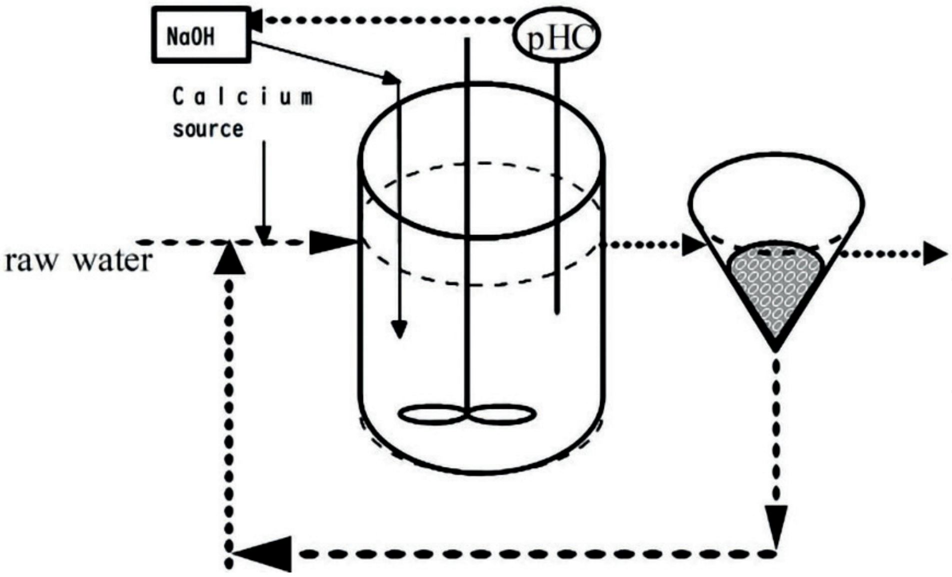
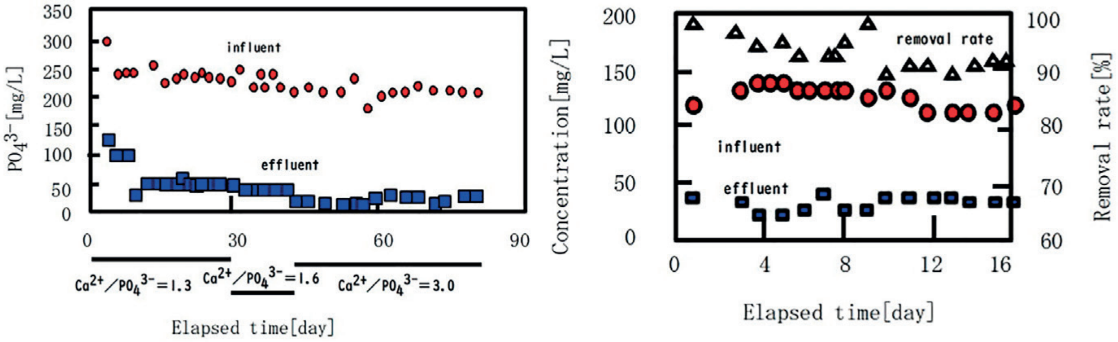


Figure 8. Reactor for hydroxyapatite treatment.



(a) (b)

Figure 9. Results of continuous hydroxyapatite (HAP) treatment for long periods for different initial phosphorus concentrations when adding calcium chloride. In Run (a), the concentration range was 200–250 mg/L P, and the molar ratio of added calcium was varied. In Run (b), the concentration range was 100–150 mg/L P and the ratio of Ca:P was 3.0

This system has a high degree of supersaturation, so fine precipitates were easily generated, and solid-liquid (S/L) separation was difficult. Therefore, it was desirable to recover as much phosphorus as possible while promoting crystal growth. A sedimentation tank was provided and crystals of the bottom region were returned to the reactor. To examine the effects of calcium source on recovery rate, an experiment was carried out using both calcium chloride and lime (calcium hydroxide). The influence of the addition amount was examined for the calcium chloride system. The HAP apparatus shown in **Figure 8** used a 2.5 L reaction tank and 1 L settling tank. The filtrate of the digested sludge was fed to the reaction tank at 0.6 L/h and 200 mg/L calcium chloride solution was fed to maintain a Ca^{2+}/PO_4^{3-} molar ratio of 3.0. The influence of the calcium chloride addition was examined. The reflux ration from the sedimentation tank to the reaction tank was maintained at 2.0. The pH of reactor was controlled at 8.5–8.7 by adding 0.5 M sodium hydroxide. Run (a) of **Figure 9** shows the relationship

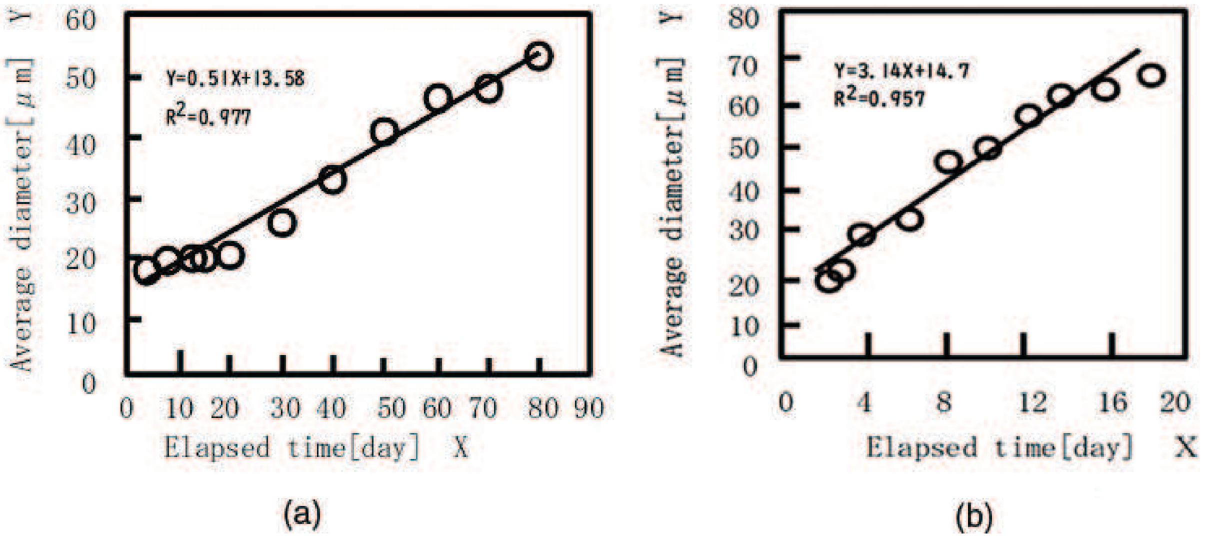


Figure 10. Crystal growth rates accompanying continuous treatment of raw water containing (a) 200–250 mg/L PO_4^{3-} (b) 100–150 mg/L PO_4^{3-}.

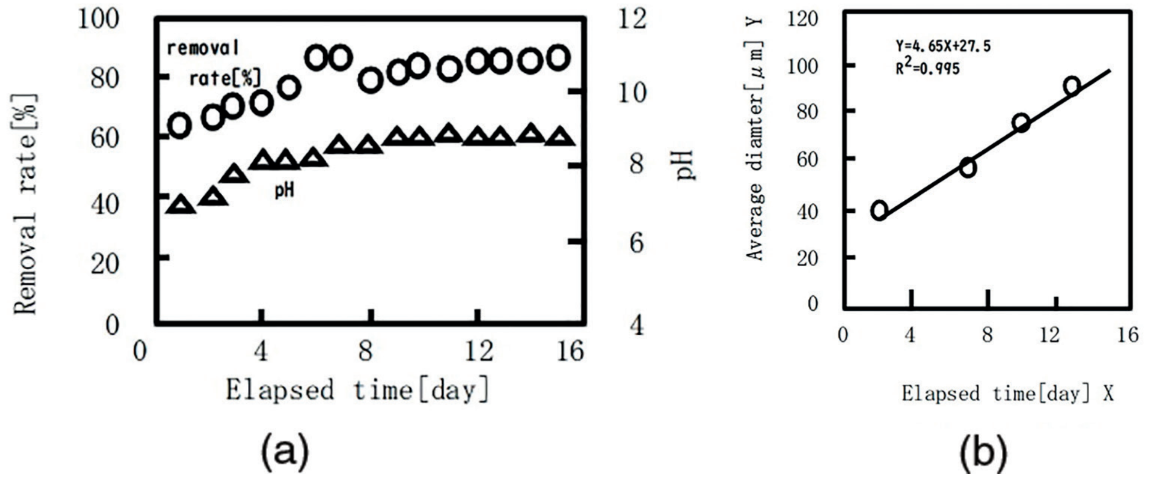


Figure 11. (a) Removal rate and (b) crystal growth during (b) to hydroxyapatite (HAP) treatment with the addition of lime.

between removal of phosphorus and calcium addition. The removal efficiency was 70–90% at Ca/PO_4^{3-} = 1.2, 75–90% at Ca/PO_4^{3-} = 1.6, and 90% or more at Ca/PO_4^{3-} = 3.0. Run (b) of **Figure 10** used a raw water concentration of about 150 mg/L PO_4^{3-}, which was two-thirds of the concentration in Run (a) because it contained wash water. When the calcium chloride addition ratio was 3.0, this gave same removal as Run (a). **Figure 10** shows changes in the average particle diameters: it took 82 days for Run (a) crystals to reach 60 μm, while this size was achieved after 12 days for Run (b). The high initial concentration of Run (a) caused the generation of a large number of primary nuclei (2). Lime feed: dehydrated filtrate was supplied at 0.8 L/h. Lime of 4000 mg/L concentration was fed to the reaction tank at 0.8 L/h and returned from the settling tank at a reflux ration of 2. **Figure 11** shows the relationship between elapsed time, pH, phosphate removal, and average diameter. The pH gradually rose over the 14-day period and reached pH 8.9. The extent of removal increased in proportion to

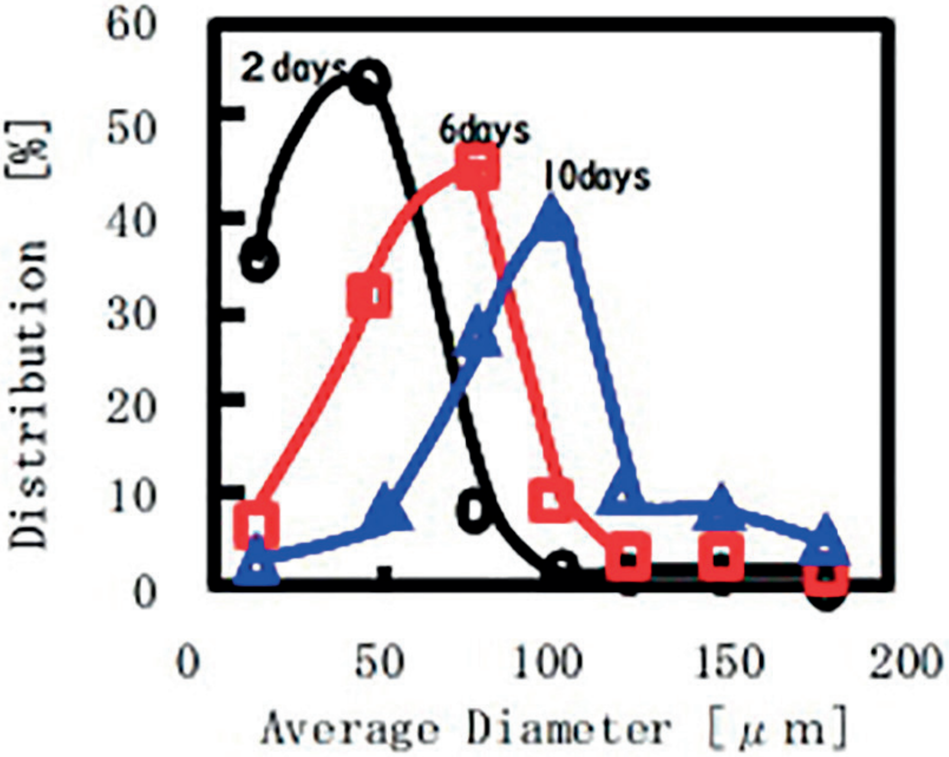


Figure 12. Distribution of diameter.

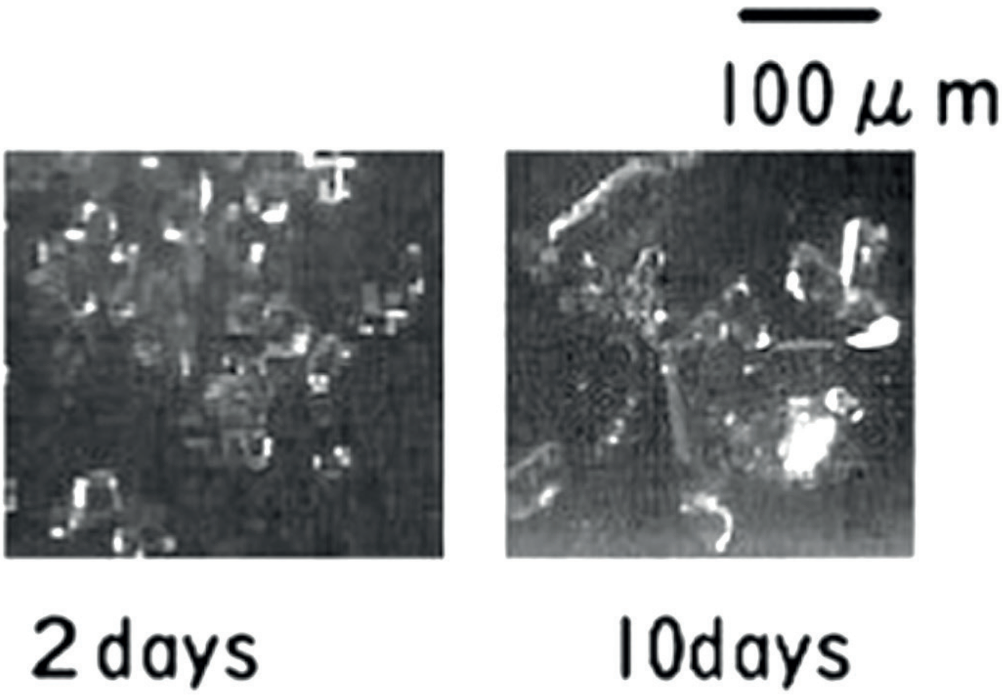


Figure 13. Photographs of crystals.

the pH, reaching approximately 90%. Changes in average particle diameter and distribution as a function of lime dosage and time are shown in **Figure 12**, and photographs are shown in **Figure 13**, respectively. When lime was present as a solid (milk of lime) and acted as a seed crystal, crystals grew rapidly. From these results, it was possible to treat actual wastewater with a phosphate concentration of 200–250 mg/L HAP crystallization. We could grow crystals by circulating precipitated crystals and retaining them for a longer period in the reactor. The appropriate ratio of calcium to phosphate was 1.6–3, and the pH was in the range of 8.5–8.7. Crystal growth was faster with a lower phosphorus concentration. The growth rate increased further with the use of lime as the calcium source.

4. A study of required conditions of magnesium potassium phosphate (MPP) precipitation by using synthetic secondly livestock wastewater

When compared with primary treatment, the phosphorus concentration also decreased because of activated sludge secondary treatment. When magnesium is added to a system in which potassium and ammonium coexist, competition arises in the formation of phosphate salts. MPP production is advantageous if the ammonium concentration is decreased in the secondary treatment. These relationships and the processing characteristics were clarified with respect to continuous processing.

This study was initially performed using batch beaker tests. To determine the influence of the stoichiometric relationship of ingredients, we used a synthetic secondary piggery wastewater (including phosphorus, potassium, and ammonium). **Figure 14** shows the effects of phosphorus concentration on removal and recovery. We had previously confirmed that the optimum pH for MPP production is pH 10–11 [12, 13]. The initial phosphorus concentrations were from 0.65 to 4.6 mM from MAP treatment; the NH_4 concentrations were set at 25.6 and 4.4 mM, respectively. Magnesium was added to be equimolar to phosphorus. Removal of phosphorus refers to a reduction in its concentration in the liquid (expressed as a percentage); recovery refers to the percentage of phosphorus reporting to the solid product. Extents of removal and recovery for an initial phosphorus concentration 0.65 mM were 37.1 and 76.2%, respectively.

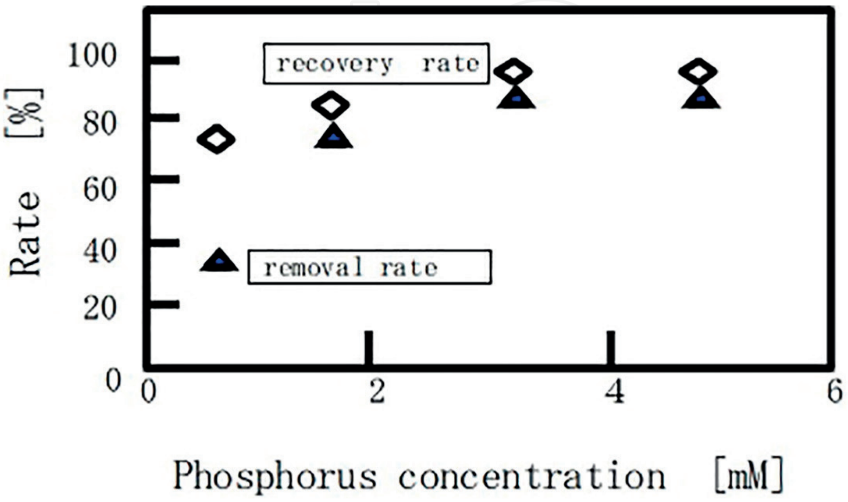


Figure 14. Changes removal rate to removal extent.

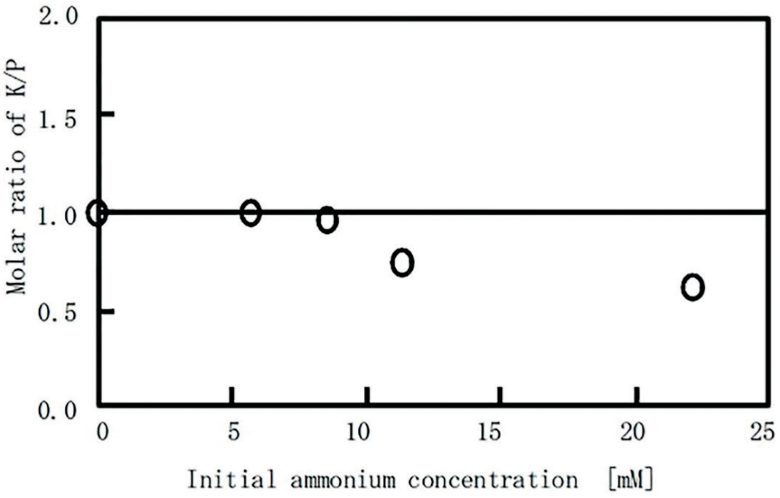


Figure 15. Effects of initial ammonium concentration on K/P [10].

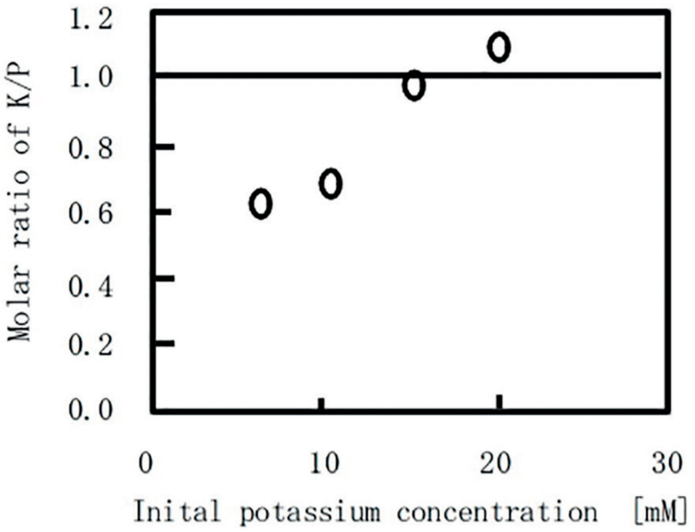


Figure 16. Effects of initial potassium concentration on K/P [10].

Both removal and recovery increased with increasing initial phosphorus concentration; more than 3.0 mM was required to reach approximately 90%. **Figure 15** shows the effects of initial ammonium concentration on the potassium to phosphorus (K:P) molar ratio in the resulting crystals. Phosphorus and potassium were 6.5 and 5.6 mM, respectively, and ammonium concentration ranged from 0 to 22.3 mM. Magnesium was added to be equimolar with phosphorus and the pH was maintained 11. The K:P molar ratio did not change to 11.6 mM Mg and then decreased, which was attributed to competition with MAP production. **Figure 16** shows the effects of potassium concentration on the molar ratio. When the potassium concentration was 17 mM (680 mg/L), the K:P ratio was closed to 1, which showed that at least 680 mg/L K is necessary for MPP production. **Figure 17** shows the influence of the additional molar ratio of magnesium on phosphorus on the component ratio in the crystal pH 11. The input of Mg:P molar ratio was changed. Phosphorus, potassium, and ammonium ion concentrations were

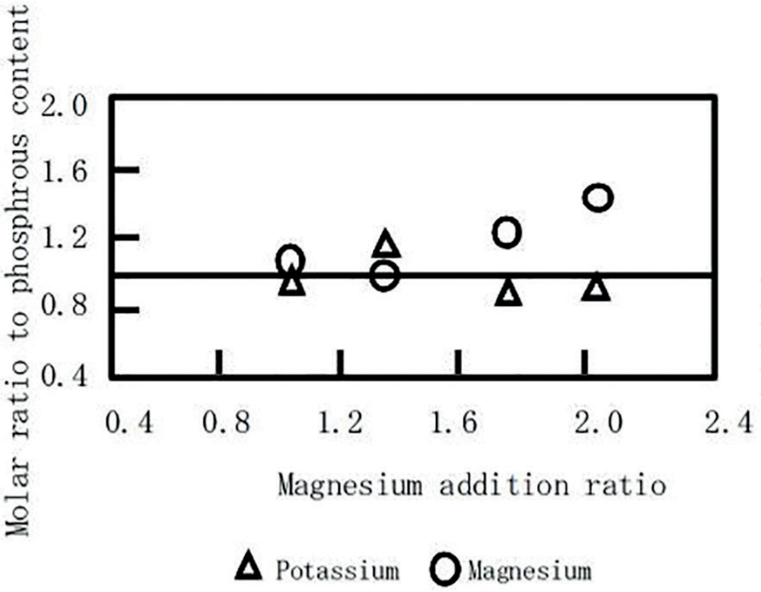
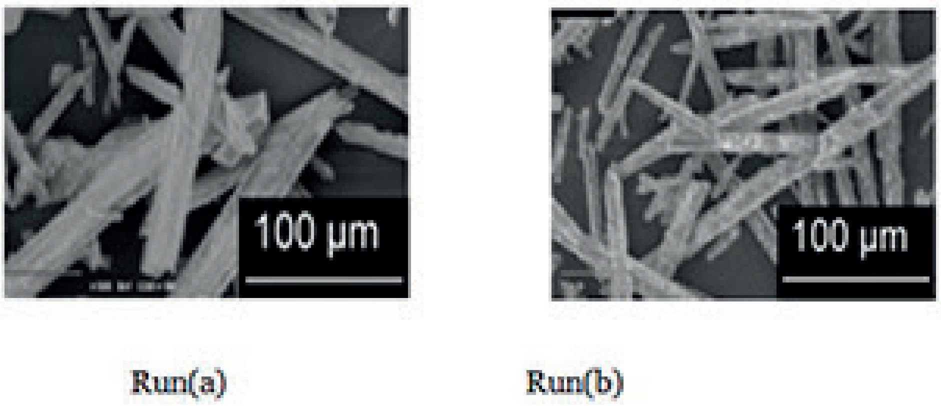


Figure 17. Relationship between the magnesium-to-phosphorus ratio in the wastewater and the potassium- and magnesium-to-phosphorus molar ratios in the resulting crystals.



Run(a)

Run(b)

Figure 18. Scanning electron microscopy analysis of white precipitation [12].

changed to 1, 1.3, 1.7, and 2. pH values were 10 and 11. When Mg:P = 1, the molar ratio of each component was a stoichiometric ratio of almost 1.

These results showed that to maintain the purity of crystals by efficient MAP treatment, it is important to maintain the pH at 11; the phosphorus and potassium and concentrations were required to be 3 mM (54 mg/L) and 17 mM (680 mg/L), respectively, and the ammonium concentration be less 8 mM (120 mg/L).

Figure 18 shows photograph of crystals obtained by continuous treatment in a bubble column using the apparatus of **Figure 1**. **Figures 19** and **20** show the result of X-ray diffraction (XRD) and energy-dispersive x-ray (EDX) analysis, respectively.

It is difficult to distinguish MAP and MPP in photographs because both crystals have a needle-like shape. Considering these results in conjunction with the XRD data, it appears that MPP was formed in Run (b), but not in Run (a). As already mentioned MAP and MPP were generated

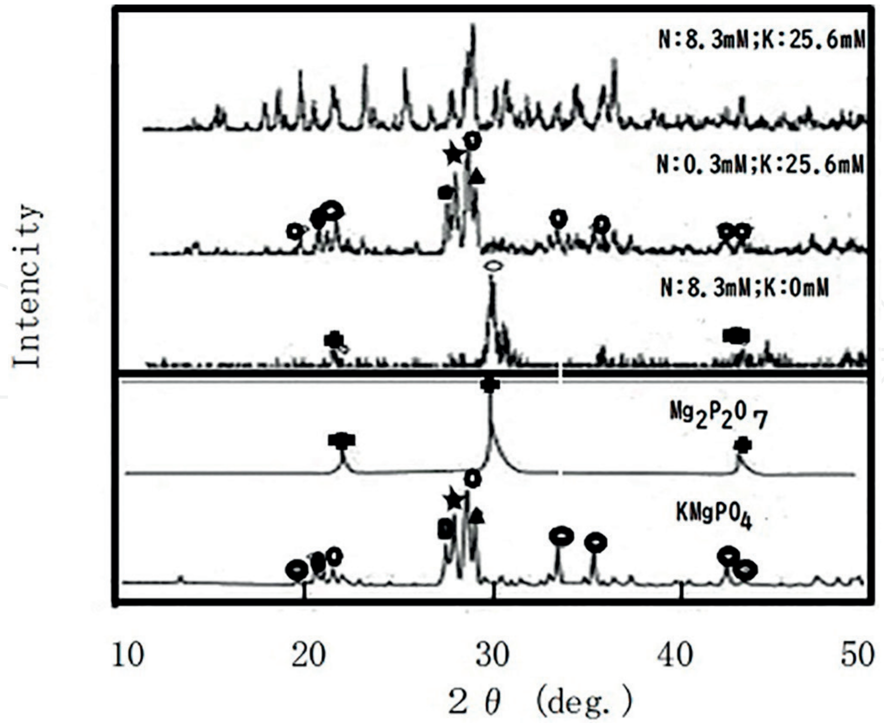
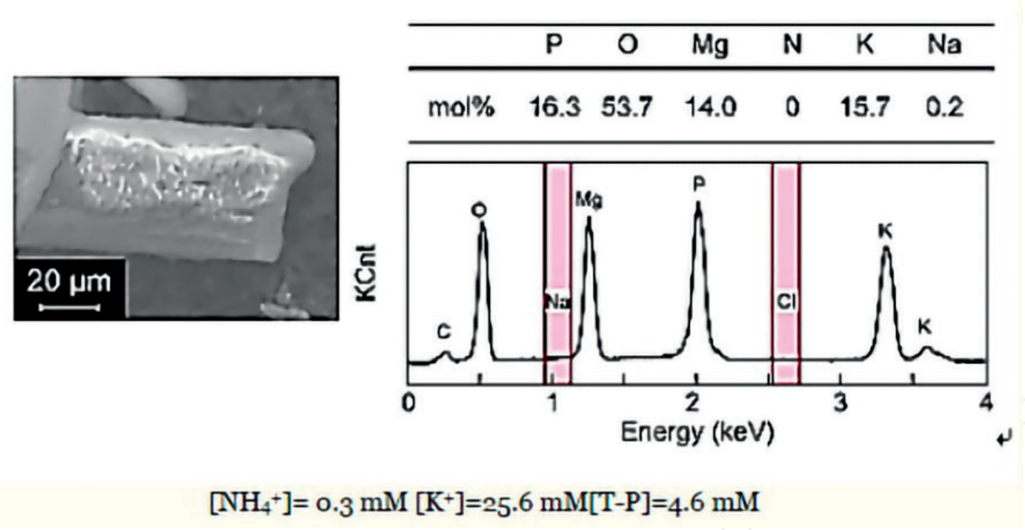


Figure 19. Comparison of X-ray diffraction patterns of sample and standard crystal [12].



	P	O	Mg	N	K	Na
mol%	16.3	53.7	14.0	0	15.7	0.2

$[NH_4^+]$= 0.3 mM $[K^+]$=25.6 mM[T-P]=4.6 mM

Figure 20. Energy-dispersive X-ray analysis of white precipitate [12].

from a concentration of 8.3 mM. The XRD results of Run (a) indicated that the molar percentage ratios of P:Mg:K were 16.3:14.0:15.7, which is close to the target stoichiometry of MPP crystal.

5. Conclusion

MAP treatment for primary treated water of livestock wastewater is handled by immersing a mesh metal in liquid. In the secondary treatment, ammonium and phosphorus ion concentrations are lower by nitrification and bio-uptake, so treatment efficiency is lowered. In the membrane separation activated sludge method, because suspended solid is not mixed, recovery of phosphate is easy. To address this problem, a combination of the MAP and adsorption methods and an HAP method with crystal recirculation and without seed crystal were considered. In addition, competition with MAP precipitation is a problem in the MPP method, so inhibition of the ammonium concentration in the MPP method was studied using artificial water. MPP production was found to be given priority when the concentration of ammonium ions was 3 mM or less and potassium ions were 17 mM or more.

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