

# Recovery of Calcium Phosphates from Composted Chicken Manure

Shigeru SUGIYAMA<sup>1,2\*</sup>, Ryuta KITORA<sup>3</sup>, Haruka KINOSHITA<sup>3</sup>, Keizo NAKAGAWA<sup>1,2</sup>, Masahiro KATOH<sup>1</sup> and Kiyohiko NAKASAKI<sup>4</sup>

<sup>1</sup>*Department of Advanced Materials, Institute of Technology and Science, Tokushima University, Minamijosanjima, Tokushima-shi, Tokushima 770-8506, Japan*

<sup>2</sup>*Department of Resource Circulation Engineering, Center for Frontier Research of Engineering, Tokushima University, Minamijosanjima, Tokushima-shi, Tokushima 770-8506, Japan*

<sup>3</sup>*Department of Chemical Science and Technology, Tokushima University, Minamijosanjima, Tokushima-shi, Tokushima 770-8506, Japan*

<sup>4</sup>*Department of International Development Engineering, Graduate School of Engineering, Tokyo Institute of Technology, 2-12-1, 14-2, Ookayama, Meguro-ku, Tokyo 152-8550, Japan*

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## Abstract

To recover phosphorus from composted chicken manure, a batch method with aqueous HNO<sub>3</sub>, HCl and H<sub>2</sub>SO<sub>4</sub> was used to examine the elution behavior of the aqueous calcium and phosphate contained in the manure. Since the main components in manure are Ca<sup>2+</sup> and K<sup>+</sup> along with PO<sub>4</sub><sup>3-</sup> and those ions can be dissolved using an acidic eluate, it was expected that most of the aqueous Ca<sup>2+</sup>, K<sup>+</sup> and PO<sub>4</sub><sup>3-</sup> could be obtained via the elution. Therefore, it seemed plausible that the removal of the aqueous K<sup>+</sup> obtained by the elution of composted chicken manure would result in the formation of calcium phosphates. If calcium phosphates are formed, they can be used for phosphate rock, which also consists of various calcium phosphates. When using 0.1 mol/L HNO<sub>3</sub>, HCl or H<sub>2</sub>SO<sub>4</sub>, the elution behavior of the PO<sub>4</sub><sup>3-</sup> was not dependent on the acids. However, 0.1 mol/L H<sub>2</sub>SO<sub>4</sub> was not sufficient for the elution of Ca<sup>2+</sup>, probably due to the precipitation of the calcium sulfate. The eluted amount of K<sup>+</sup> using 0.1 mol/L HNO<sub>3</sub> was lower than that using 0.1 mol/L of either HCl or H<sub>2</sub>SO<sub>4</sub>. Since the poor elution of K<sup>+</sup> should enrich the concentrations of Ca<sup>2+</sup> and PO<sub>4</sub><sup>3-</sup> in the acidic aqueous solution after the elution, it was suggested that aqueous HNO<sub>3</sub> would be suitable as an eluate in the present system. After the elution of the composted chicken manure, when 0.1 mol/L HNO<sub>3</sub> was used to adjust the solution pH of the acidic aqueous solution to greater than 6, Ca<sup>2+</sup> and PO<sub>4</sub><sup>3-</sup> were precipitated, but K<sup>+</sup> was not. The precipitate was calcium hydroxyapatite, one of the typical components of phosphate rock, which showed that composted chicken manure could be replaced phosphate rock as a new source of phosphorus.

## Introduction

Phosphorus is an element that is essential to humans, and it is widely used in food production as a main component in fertilizers and pesticides (Nakasaka, 2014), and in advanced materials such as artificial bones, catalysts, sensors, fire retardants, glues, food additives, and detergents, (Matsubae-Yokoyama *et al.*, 2009; Sugiyama, 2010). However, it is generally accepted that phosphate rock as a raw material for phosphorus will disappear in the near future (Abelson, 1999). Therefore, our laboratory has reported on the recovery of phosphate from unused resources such as from rivers by using boehmite as an adsorbent, from dephosphorization slag, chemical slag, and used fluorescence tubes using acidic solutions as eluates (Sugiyama *et al.*, 2011, 2012, 2014, 2015). In particular, a phosphate-containing solid was successfully precipitated from the nitric acid extract of dephosphorization slag, simply by adjusting the solution pH to 7.0 (Sugiyama *et al.*, 2014). If calcium phosphates are formed, the precipitation can be used in

place of phosphate rock, which also consists of various calcium phosphates. Since typical phosphorus manufacturing processes are based on phosphate rock, the production of calcium phosphates from unused resources could result in the development of possible substitutes for rare phosphorus. Therefore, the selection of unused resources is important.

In the present study, composted chicken manure was employed as a new unused source of phosphorus. Chicken droppings are good manure and should be a suitable candidate for this purpose since it mainly contains phosphorus, calcium and potassium together with nitrogen (Dikinya and Mufwanzala, 2010). However, the high content of ammonia in fresh chicken droppings burns plants and creates a bad smell. Therefore, it must be composted (Zhang and Lau, 2007; Nakasaki, 2014). The concentration of phosphate in chicken droppings is enriched in chicken manure after in-situ composting, mainly due to the decomposition of organic compounds (Nakasaka *et al.*, 1999; Nakasaki, 2014).

To confirm the most favorable dissolution of  $\text{Ca}^{2+}$  and  $\text{PO}_4^{3-}$  from the manure, we followed a procedure established in our previous papers (Sugiyama *et al.*, 2012) by first examination of the elution behavior of composted chicken manure using aqueous  $\text{HNO}_3$ ,  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$  to form an aqueous complex mixture containing calcium and phosphate. Aqueous  $\text{HNO}_3$  was the most suitable eluate, so we examined the separation of calcium phosphates as a precipitation from the nitric acid extract via pH-adjustment using aq.  $\text{NH}_3$  to promote an alkaline condition. For the elution of phosphate species from phosphate-containing waste, a widely used sequential fractionation procedure that was developed by Hedley *et al.* (1982) for elution from soil was also used by Frossard *et al.* (1994) for work on urban sewage sludge. Ito *et al.* (2010) and Hashimoto *et al.* (2014) have employed the sequential fractionation procedure for the elution of phosphate species from various forms of manure and concluded that eluted phosphate species from chicken manure are unfavorable for direct employment in the growth of plants. Based on this information, a one-step elution followed by the precipitation of calcium phosphates was examined in the present study.

## 1. Experimental

We used a batch method to examine the elution behaviors of phosphate and various cations from composted chicken manure using various concentrations of aq.  $\text{HNO}_3$ ,  $\text{HCl}$ , and  $\text{H}_2\text{SO}_4$  (Wako Pure Chemical Industries, Ltd.) as an eluate. The composted chicken manure was prepared using the same treatment reported earlier (Nakasaki, 2014) and was used in the present study after drying at 353 K for 2 d, followed by milling for 30 minute. The composition of the composted chicken manure was analyzed using X-ray fluorescence

(XRF; EDX-800, Shimadzu Corp.), as described in **Table 1**. Phosphate will hereafter be referred to as P. The P and Ca in the manure were approximately estimated at 12 and 40 wt.%, respectively. The present study was focused mainly on the cations described in Table 1.

The batch method began with the addition of 100 mL of an aqueous acidic solution of either  $\text{HNO}_3$ ,  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$  to a flask (200 mL), followed by the addition of 1.0 g of the composted chicken manure, which was dried for 2 d at 353 K, then milled to a particle size of less than 0.85 mm for 0.5 h, unless otherwise stated. The mixture was stirred at 130 rpm and 298 K in a constant-temperature bath. The solution was filtered after a scheduled time of stirring using filter paper (4  $\mu\text{m}$  of pore size) and a membrane filter (0.45  $\mu\text{m}$  of pore size), then it was analyzed by ICP-AES for all cations except  $\text{K}^+$ , as described in Table 1, and by ion-chromatograph (PIA-1000, Shimadzu Corp.) for  $\text{K}^+$ .

In order to precipitate the solid from the nitric acid extract, the pH of the nitric acid extract was increased using an aq.  $\text{NH}_3$  solution. The solid samples thus obtained were dried and analyzed via X-ray diffraction (XRD; RINT 2500X, Rigaku Corp.).

Composition [mol%]									
P	Ca	K	Mg	Fe	Mn	Zn	Al	Si	Others
11.1	50.9	17.5	3.04	1.43	0.303	0.275	1.25	3.41	10.8

## 2. Results and Discussion

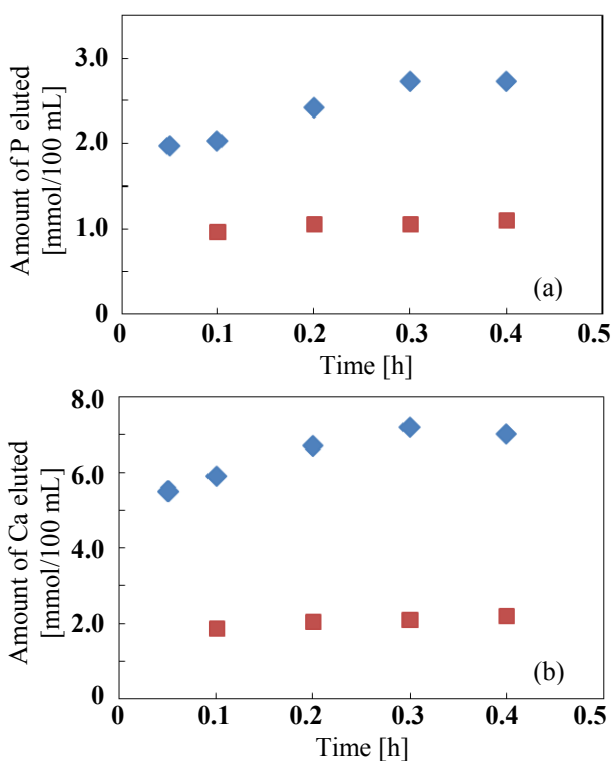
### 2.1 Elution behavior from the composted chicken manure using various concentrations of $\text{HNO}_3$

**Table 2** shows the elution behaviors of various cations from the composted chicken manure using 0.01, 0.1, 0.5, and 1.0 mol/L  $\text{HNO}_3$ , respectively, for various stirring times. Since, among the cations described in Table 1,  $\text{Si}^{4+}$  was not eluted, data for  $\text{Si}^{4+}$  is not included

$\text{HNO}_3$ [mol/L]	Stirring time [h]	Concentration eluted [mmol/100 mL]							
		P	Ca	K	Mg	Fe	Mn	Zn	Al
0.01	0.1	0.393	0.992	0.988	0.343	0	0.003	0	0.031
	0.2	0.420	1.05	1.00	0.349	0	0.003	0	0.031
	0.3	0.439	1.12	1.05	0.362	0	0.003	0	0.032
	0.4	0.461	1.16	1.06	0.373	0	0.003	0	0.033
0.1	0.1	0.970	1.870	0.856	0.495	0	0.017	0.024	0.063
	0.2	1.06	2.05	0.860	0.515	0	0.018	0.019	0.067
	0.3	1.06	2.09	0.845	0.521	0	0.017	0.018	0.072
	0.4	1.10	2.22	0.833	0.516	0	0.018	0.018	0.076
0.5	0.1	1.34	2.82	0.799	0.628	0.024	0.020	0.022	0.093
	0.2	1.32	2.85	0.803	0.617	0.004	0.020	0.022	0.095
	0.3	1.33	2.81	0.786	0.629	0.013	0.020	0.024	0.099
	0.4	1.26	2.71	0.785	0.610	0.020	0.019	0.020	0.104
1.0	0.1	1.24	2.53	0.849	0.526	0.071	0.015	0.013	0.110
	0.2	1.18	2.48	0.879	0.530	0.077	0.014	0.012	0.117
	0.3	1.16	2.39	0.817	0.527	0.081	0.014	0.012	0.123
	0.4	1.17	2.38	0.813	0.527	0.078	0.014	0.013	0.122

in Table 2. This indicated that  $\text{Si}^{4+}$  could have been completely separated from these  $\text{HNO}_3$  extracts via the elution process. The elutions of six cations, with the exception of Fe and Zn, shown in Table 2 were detected even after a short stirring time of 0.1 h using four concentrations of  $\text{HNO}_3$  solutions, and the elution behaviors of these cations were similar after 0.4 h of stirring. The elution of Ca and P using 0.01 mol/L  $\text{HNO}_3$  was evidently improved using 0.1 mol/L  $\text{HNO}_3$ , while further increases in the concentration of  $\text{HNO}_3$  resulted in little influence on the elution process. The elutions of K and Mg were insensitive to the concentration of  $\text{HNO}_3$ .

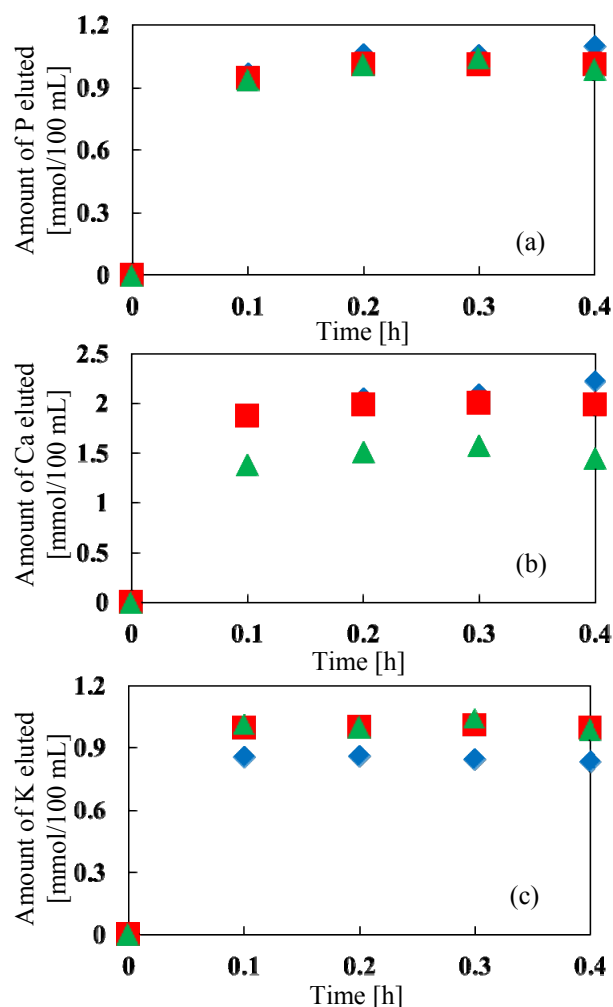
It is noteworthy that the elution of Fe was not detected using 0.1 mol/L  $\text{HNO}_3$ , although sufficient elutions of Ca and P were observed. As shown in our previous paper (Sugiyama *et al.*, 2014), the elution of Fe is not suitable for the production of calcium phosphate from nitric acid extract. The elutions of Mn and Zn were also insensitive to  $\text{HNO}_3$ , particularly for concentrations greater than 0.1 mol/L. With an increase in the concentration of  $\text{HNO}_3$ , however, the elution of Fe was increased. Based on these results, 0.1 mol/L  $\text{HNO}_3$  was employed for the remainder of the present study.



**Fig. 1** Elution behavior of P (a) and Ca (b) from 1.0 (■) and 5.0 g (◆) of the composted chicken manure using 0.1 mol/L  $\text{HNO}_3$

During the examination of the elution behaviors using 1.0 g of the composted chicken manure, the weight of the recovered solid and the solution pH were certainly influenced by the elution time, and 1.0 g seemed insufficient to obtain clear results on both

factors. Therefore, the elution behaviors particularly on P and Ca were re-examined using 5.0 g samples of the manure. As shown in **Figure 1**, in which the elution behaviors using 1.0 and 5.0 g of the manure is described, most of the elution of P and Ca was completed with 0.05 h elution, indicating that the solution reached elution equilibrium during a rather short elution time even when using 5.0 g samples. This was further supported by the similar weights of the recovered solid for each of the elution times (4.4, 4.5, 4.4, 4.3, and 4.3 g at 0.05, 0.1, 0.2, 0.3, and 0.4 h of the elution, respectively), indicating again that the elution of the manure reached elution equilibrium at 0.05 h. The solution pH obtained after elutions at 0.05, 0.1, 0.2, 0.3, and 0.4 h were 1.8, 1.9, 2.3, 2.6, and 2.6, respectively. Therefore, the pH shift from 1.0 to greater values resulted in the suppression of the elution from the manure, as shown in Figure 1.



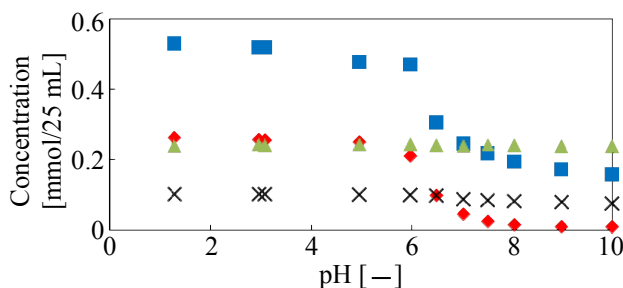
**Fig. 2** Elution behavior of P, Ca, and K from the composted chicken manure using (a) 0.1 mol/L  $\text{HNO}_3$  (◆), (b) 0.1 mol/L  $\text{HCl}$  (■), and (c) 0.1 mol/L  $\text{H}_2\text{SO}_4$  (▲)

## 2.2 Elution behavior from the composted chicken manure using 0.1 mol/L $\text{HNO}_3$ , $\text{HCl}$ and $\text{H}_2\text{SO}_4$

In order to confirm the suitability of  $\text{HNO}_3$ , other acids,  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$ , were also used as an eluate. In **Figure 2**, the elution behaviors of the three major cations in the composted chicken manure (P, Ca and K) are described using 0.1 mol/L  $\text{HNO}_3$ ,  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$ . The amount of P eluted was essentially the same regardless of the acids employed (**Figure 2 (a)**). However, the amount of Ca using 0.1 mol/L  $\text{H}_2\text{SO}_4$  was evidently smaller than when using 0.1 mol/L  $\text{HNO}_3$  and  $\text{HCl}$  (**Figure 2 (b)**), probably due to the formation of barely soluble compounds such as  $\text{CaSO}_4$ . The elution amount of K when using  $\text{HNO}_3$  was smaller than when  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$  were used (**Figure 2 (c)**). The small amount of Ca in the extract when using  $\text{H}_2\text{SO}_4$  was not suitable for the precipitation of calcium phosphate from this extract. Furthermore, a small amount of K in the extract when using  $\text{HNO}_3$  was suitable for the precipitation of calcium phosphate. Therefore aqueous  $\text{HNO}_3$  is suitable for use as an eluate in the present system.

### 2.3 Recovery of calcium phosphate from nitric acid extract

As shown in Table 2, the elutions of P, Ca, K and Mg are evident when using various concentrations of  $\text{HNO}_3$  as an eluate. In order to precipitate calcium phosphates from the  $\text{HNO}_3$  extract, the precipitation of P and Ca together with the remaining K and Mg in the extract would be required. In the present study, to precipitate a phosphate-containing compound, the solution pH of 25 mL of the 0.1 mol/L  $\text{HNO}_3$  extract obtained after 0.1 h stirring was controlled by adding an aq.  $\text{NH}_3$  solution. As expected, precipitation was confirmed for solutions with a pH greater than 6.5. The concentrations of P, Ca, K and Mg in the extract after adding aq.  $\text{NH}_3$  were plotted against the solution pH (**Figure 3**).

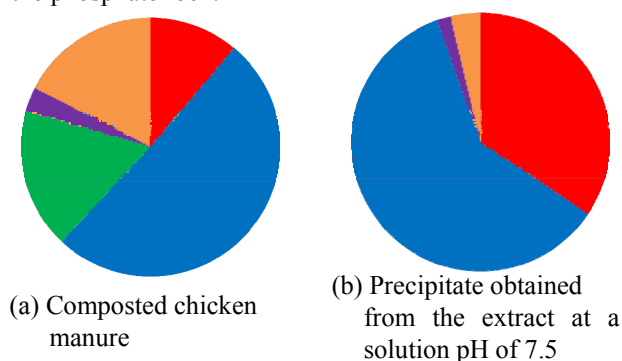


**Fig. 3** Effect of pH on the concentration of P (●), Ca (■), K (▲), and Mg (×) in the nitric acid extract

The concentrations of P and Ca in the extract were decreased when the solution pH was greater than 6.5. The concentration of P could barely be detected at a solution pH of approximately 10, while the precipitation of Ca was almost completed at the same solution pH. It is noteworthy that the concentrations of K and Mg were similar at solution pH values between 1.3 and 10.0.

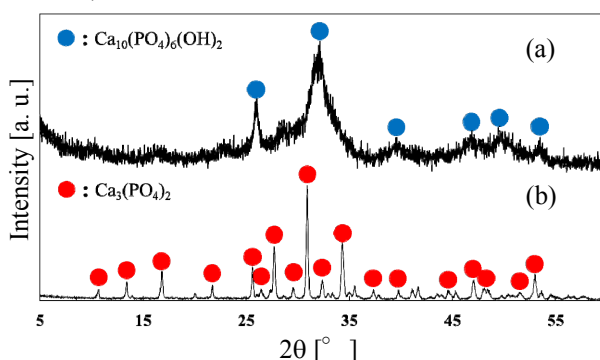
Therefore, K and Mg were not contained in the precipitate. This may indicate that the precipitation consists mainly of P and Ca.

The precipitate obtained at a solution pH of 7.5 was analyzed using XRF, and this was compared with the composition of the composted chicken manure (**Figure 4**). After the composted chicken manure was eluted and the precipitate was obtained, K and Si (not shown in Figure 3) were completely removed from the precipitate, and the composition of the other cations, with the exception of P, Ca, K, and Mg, was improved from 17.5 to 2.8 mol%. The molar ratio of P against Ca in the precipitate (0.57) was within the range of the ratios for  $\text{CaHPO}_4$  (0.5),  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$  (0.6), and  $\text{Ca}_3(\text{PO}_4)_2$  (0.67), which are the main components in phosphate rock. Therefore, it was evident that the precipitate consisted of the main components found in the phosphate rock.



**Fig. 4** Composition [mol%] of P (■), Ca (■), K (■), Mg (■), and others (■) in the chicken manure and the precipitate obtained from the extract at a solution pH of 7.5

XRD was used to characterize the precipitate. As shown in **Figure 5 (a)**, broad signals were detected from the precipitate and were assigned to calcium hydroxyapatite ( $\text{CaHA}_p$ ;  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ; JCPDS 721243).



**Fig. 5** XRD patterns of the precipitate obtained at pH = 7.5 (a), and after the calcination at 1,073 K for 5 h (b)

Following the calcination of the precipitate at 1,073 K for 5 h, sharp signals were detected, as shown in **Figure 5 (b)**, and were matched to the reference peaks for

Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> (JCPDS 90348). Since it is generally accepted that CaHAP can be converted to Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> via calcination (Sugiyama *et al.*, 1999), we concluded that elution of the composted chicken manure followed by precipitation would result in the formation of calcium phosphate.

As shown in Figure 5 (a), XRD revealed that the precipitation obtained from the extract with a solution pH of 7.5 mainly consisted of Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>. Therefore, the weight percent of CaO and P<sub>2</sub>O<sub>5</sub> in the precipitation was estimated to be approximately 55.8 and 42.2 wt.%, based on Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub> (= 10CaO + 3P<sub>2</sub>O<sub>5</sub> + H<sub>2</sub>O). In this case, the corresponding molar ratio of P against Ca in the precipitate (0.60) was similar to that estimated from XRF (0.57) (Figure 4 (b)). Since CaO and P<sub>2</sub>O<sub>5</sub> in natural phosphate rocks are generally 49-55 and 34-39 wt.% (Akiyama, 2009), it can be concluded that the present precipitation consists of almost same components as that of natural phosphate rock.

## Conclusions

Phosphate rock is the raw material that is used in the production of phosphorus, and this raw material is expected to disappear in the near future. In the search for a substitute material, the elution of composted chicken manure followed by precipitation was carried out. As an eluate for the dissolution of calcium and phosphate from composted chicken manure, 0.1 mol/L HNO<sub>3</sub> was suitable. The precipitation of calcium and phosphate from the HNO<sub>3</sub> extract was suitable when the solution pH was adjusted to approximately 7.5 using aq. NH<sub>3</sub> solution. Since a higher purity of calcium phosphate was obtained from the composted chicken manure using this simple treatment procedure, it can be concluded that composted chicken manure can replace phosphate rock as a source of phosphorus. The proposed procedure in the present study consists of the composting of chicken droppings, the drying of the composted manure, an acid treatment process of the dried compost, and a precipitation process leading to calcium phosphates. Among these four processes, the drying is an expensive process that can be excluded since the proposed procedure employs an aqueous system in the subsequent acid treatment process. It should be noted that previous reports have shown that the acid treatment is more expensive than the alkali treatment, although the elution using the former treatment is superior to that using the latter one (Yasuike and Shimogaki, 2011). Further examination to overcome those problems for practical use is now in progress.

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