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ORIGINAL ARTICLE

Recovery of nitrogen by struvite precipitation from swine wastewater for cultivating Chinese cabbage

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Abstract

This study assessed the fertilizing value of struvite deposit recovered from swine wastewater in cultivating Chinese cabbage. Struvite deposit was compared with commercial fertilizers: complex, organic and compost to evaluate the fertilizing effect of struvite deposit. Laboratory pot test obviously presented that the struvite deposit more facilitated the growth of Chinese cabbage than organic and compost fertilizers even though complex fertilizer was the most effective in growing Chinese cabbage. It was revealed that the growth rate of Chinese cabbage was simultaneously controlled by phosphorus (P) and potassium (K). Also, the nutrients such as nitrogen (N), P, K, calcium (Ca) and magnesium (Mg) were abundantly observed in the vegetable tissue of struvite pot. Specifically, P was the most abundant component in the vegetable tissue of struvite pot. Meanwhile, the utilization of struvite as a fertilizer led to the lower accumulation of chromium (Cr^{6+}) than other pots, except for compost fertilizer pots, and no detection of cadmium (Cd), arsenic (As) and nickel (Ni) in the Chinese cabbage. The experimental results proved that the optimum struvite dosage for the cultivation of Chinese cabbage was 2.0 g struvite/kg soil. On the basis of these findings, it was concluded that the struvite deposits recovered from swine wastewater were effective as a multi-nutrient fertilizer for Chinese cabbage cultivation.

Key words : Swine wastewater, Struvite, Chinese cabbage, Commercial fertilizer, Pot test

1. Introduction

Swine wastewater typically contains the high levels of ammonia nitrogen (NH_4-N). Among many treatment methods for NH_4-N in swine wastewater, struvite precipitation would be attractive due to its high removal efficiency in a very short reaction time, as revealed in many previous studies (Ryu and Lee, 2010; Huang *et al*., 2011; Liu *et al*., 2011; Zhang *et al*., 2012). However, a considerable amount of struvite sludge generated as a by-product during the removal of ammonium has caused another problem of waste disposal. A feasible solution to the problem would be its reuse as a fertilizer because struvite is considered a recyclable, environmentally friendly and preferred fertilizer. Struvite contains 12.6 % P, 5.7 % N and 9.9 % Mg. Specifically, the presence of Mg also made struvite useful for sugar beets (de-Bashan and Bashan, 2004). Moreover, since struvite is slightly soluble in water and soil solutions, slow-release struvite has been found to be a highly effective source of phos -phorus, nitrogen and magnesium for plants through

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both foliar and soil application.

Although struvite has been qualified as a fertilizer, as mentioned above, to the best of our knowledge, the plant availability and fertilizer value of struvite precipitate obtained from swine wastewater was never tested before. A few fertilizer studies using struvite recovered from semiconductor wastewater, poultry manure wastewater and landfill leachate have just been reported (Li and Zhao, 2003; Yetilmezsoy and Sapci-Zengin, 2009; Ryu *et al*., 2012a, 2012b).

The present study was therefore aimed at inves-tigating the feasibility of the plant availability of nutrients recovered from swine wastewater by struvite precipitation. Specifically, objectives of this study were: (1) to characterize precipitated struvite obtained from swine wastewater; (2) to evaluate the fertilizing value of struvite precipitate with pot trial tests for cultivation of Chinese cabbage by comparing it with commercial fertilizers; (3) to determine the optimum dosage of struvite precipitate for cultivation; (4) to analyze the levels of nutrients and heavy metals in vegetables tissue grown with struvite and other fertilizers; and (5) to examine slow-release properties of struvite precipitate.

2. Materials and methods

2.1. Struvite synthesis

The struvite deposit for experiments was artificially synthesized from a swine wastewater. The swine wastewater from a piggery, which is located in Naesu, South Korea, was used for struvite crystallization and it was characterized in Table 1. Note that the swine wastewater had quite high influent concentration levels of ammonia nitrogen

For struvite synthesis, magnesium chloride ($MgCl_2 \cdot 6H_2O$) and potassium phosphate (K_2HPO_4) were used with the concentrations of 70 g Mg/L and 50 g P/L as the alternate sources of magnesium and phosphate, respectively. $MgCl_2 \cdot 6H_2O$ and K_2HPO_4 were simultaneously added into struvite precipitation tank to reach at 1:1:1 in the molar ratio of NH_4-N:Mg:PO_4-P for struvite formation, and then pH adjustment of the wastewater to 9 was achieved by the continuous addition of 5 N NaOH, as illustrated in Fig. 1. According to the study conducted by Kim *et al*. (2007), the NH_4-N removal efficiency in struvite precipitation was optimal at this feeding sequence of chemicals of magnesium, phosphate and buffering reagent. The mixing speed in the struvite reaction

Table 1. Characteristics of raw swine wastewater

Parameter	Average
Suspended solid (SS) (mg/L)	4160 (±2671[a])
Total chemical oxygen demand (TCOD) (mg/L)	5139 (±748)
Total Kjeldahl nitrogen (TKN) (mg/L)	1036 (±288)
NH_4-N (mg/L)	844.5 (±216.8)
NO_2-N (mg/L)	n.d.
NO_3-N (mg/L)	n.d.
Total phosphorus (T-P) (mg/L)	158.6 (±82.8)
PO_4-P (mg/L)	30.6 (±7.0)
TCOD/T-N	5.2 (±1.2)
TCOD/T-P	39.0 (±14.8)
pH	7.9 (±0.4)

[a]Standard deviation; n.d., not detected.

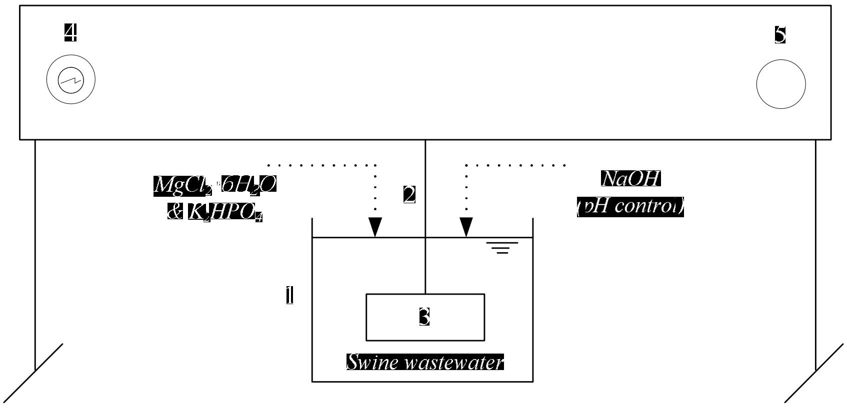

Fig. 1. Schematic of jar test apparatus: (1) jar; (2) stirrer shaft; (3) paddle; (4) power supply; (5) mixing speed adjuster.

tank was 250 rpm. The solution was allowed to settle in the jar for 5 min. In struvite precipitation, the NH_4-N removal efficiency was about 81 % on average. The settled struvite deposit was collected and used as a multi-nutrient fertilizer for cultivating Chinese cabbage in our experiments. The collected struvite deposit was dried in the shade at room temperature for 7 days before being used in pot trial tests.

2.2. Pot tests: evaluation of struvite deposit as a fertilizer

The fertilizing potential of struvite was evaluated by comparing it with that of popular Korean commercial fertilizers: complex, organic and compost. The compositions of struvite deposit and each commercial fertilizer are given in Table 2. The compost fertilizer was consisted of 5 % organic matter, 0.1 % nitrogen, 1.0 % NaCl and less than 50 % of water in weight percent. For the complex fertilizer, N, P, K, and Ca existed in the forms of $(NH_4)_2HPO_4$, K_2SO_4 and $Ca(NO_3)_2$, respectively.

Raw soil samples were taken from a local mountain in Cheongju and dried at room temperature for 15 days. The dried soil was sieved with maximum 1.2 cm prior to filling them in each pot. The soil classification was sandy loam and it was composed of

Table 2. Composition of struvite deposit and commercial fertilizers used in agricultural tests

Elements	Complex fertilizer	Organic fertilizer	Struvite
N	11	5.0	14.8
O	-	-	34.0
Mg	4	9.3	10.2
Si	14	-	-
P	6	0.4	15.6
K	6	0.9	2.0
Ca	20	13.3	-
B	0.1	-	-
C	-	-	26.0
Cl	-	-	3.4

Note: All indicated figures are based on weight percent (wt. %).
Note: In this table, compost fertilizer was not included and its composition can be found in the text.

Table 3. Characteristics of soil used in our experiments

Items	T-N	T-P	K_2O	CaO	MgO	TCOD	TOC[a]
Concentration (g/kg soil)	0.908	0.572	0.151	0.286	0.048	6.9	4.2

[a] TOC indicates total organic carbon.

Table 4. Concentration of heavy metals in soil, commercial fertilizers and struvite

Items	Heavy metals (mg/kg)							
	Cd	Cu	As	Hg	Pb	Cr	Zn	Ni
Soil	0.037	0.201	n.d.	n.d.	0.599	0.216	1.750	0.108
Complex fertilizer	0.038	0.452	0.156	n.d.	0.005	3.064	2.711	4.437
Organic fertilizer	0.036	4.028	n.d.	n.d.	0.157	0.398	8.843	0.229
Compost fertilizer	0.008	0.241	n.d	n.d.	0.022	0.098	0.139	0.044
Struvite	0.011	4.597	n.d.	n.d.	0.034	0.341	14.9124	0.072

n.d., not detected

54.6 % of sand, 33.3 % of silt and 12.1 % of clay. The soil pH was 5.3. Other important soil characteristics are provided in Table 3. The concentrations of heavy metals in soil, commercial fertilizers and struvite are also presented in Table 4.

Five sets of three pots, for a total of 15 plastic pots, were prepared. Each plastic pot had 9 cm surface diameter and approximately 8 cm of working depth. 320 g of sieved soil was mixed with respective fertilizer sources and added to each pot. One set of 3 pots was used as control in which no fertilizer source was added. In other four sets, three commercial fertilizers and struvite deposit were added to achieve an equivalent concentration of 110 kg N/ha, respec -tively. This application rate was chosen based on a scientific recommendation made by the National Academy of Agricultural Science of Korea in growing Chinese cabbage. The amount of fertilizer needed to reach 110 kg N/ha was 0.26, 0.57, 28.3, 0.19 g for complex, organic, compost, and struvite deposit, respectively.

The pot tests were performed at room temperature. Fluorescent lightening was continuously supplied to the plants in order to maintain the specific intensity of illumination. Illuminances measured during the experimental period were between 5770 and 5850 lux (lx). Three seeds of Chinese cabbage were planted within the top 1.5 cm of soil in each pot. The room temperature of laboratory was 18.3 ℃ on average with the standard deviation of 1.5 ℃ during the experi -mental period. 25 mL of distilled waster was added in each pot every two days. Length of leaves was periodically recorded in each pot. After 32 days the plants were harvested from each pot and weighed before and after drying (in an oven set at 105 ℃ for 24 hours) to determine their fresh and dry weight. As conducted by Li and Zhao (2003), the Chinese cabbages were sprayed with distilled water to wash the dust off prior to harvesting. The level of heavy metals and nutrients in dry vegetables were also analyzed.

2.3. Pot tests: determination of optimum struvite dosage

The optimum struvite dosage for cultivating Chinese cabbage was determined. Six sets of three pots were filled with 250 g of sieved soil and struvite dosages of 0, 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 g, which are equivalent to concentrations of 0, 0.4, 0.8, 1.2, 1.6, 2.0 and 2.4 g struvite/kg soil, respectively. Three seeds of Chinese cabbage were sowed within the top 1.5 cm of soil in each pot. The room temperature was 19.0 ℃ on average with the standard deviation of 2.0 ℃ during the experimental period. Fluorescent lightening was also supplied to the plants in order to maintain the intensity of illumination. Illuminances were between 5670 and 5780 lux (lx). 25 mL of distilled waster was added in each pot every two days. Length of leaves was periodically recorded for each pot. After 42 days the plants were harvested and weighed to determine their fresh and dry weight.

2.4. Analytical procedures

SS (standard code: 2540 D), Total chemical oxygen demand (TCOD) (standard code: 5220 D), total kjeldahl nitrogen (TKN) (standard code: 4500-N B), NO_2-N (standard code: 4500-NO_2^- B), NO_3^-N (standard code: 4500-NO_3^- B) and PO_4-P (standard code: 4500 -P E) in all samples were analyzed by the Standard Methods (APHA, 2005). NH_4-N and total phosphorus (T-P) were analyzed using DR4000 spectrophoto -meter with relevant Hach regents as provided by the Hach manual (Hach Company, USA). Mg, Ca, Na, K, F, SO_4, and Cl in raw semiconductor wastewater were measured using a DX-100 ion chromatograph (Dionex, USA).

For soil characteristics analysis, 10 g of sieved soil sample was placed in a 100 mL Pyrex tube contain -ing 50 mL of 0.1 N HCl. The mixture was heated at 100 ℃ for 1 h. After the temperature cooled down naturally, 10 mL of 5 % HNO_3 was added into the tube and mixed using a vortex. The mixture was then centrifuged at 1448 g for 15 min. The supernatant was filtered with 0.45 µm membrane filter. Then T-N, T-P, K_2O, CaO, MgO and organic carbon (as COD) were analyzed. T-N (standard code: 4500-N) was determined according to the procedure described in Standard Methods (APHA, 2005). The analyses of K_2O, CaO and MgO were performed by inductively coupled plasma-atomic emission spectrometry (ICP-AES 3300DV, Perkin Elmer, USA).

Heavy metals, which are contained in dry vegetable, were also measured by ICP-AES (Perkin Elmer, USA) after acidic digestion. In digestion, the dry vegetable samples were placed in a Pyrex tube containing 4 mL of conc. HNO_3. The mixture was heated at 200 ℃ until dry. After the temperature cooled down naturally, 20 mL of 5 % HNO_3 was added into the tube and heated at 50 ℃ for 20 min. When the sample temperature was cooled down to room temperature, the solution in each tube was evaluated using ICP-AES. The minimum detection limit of ICP-AES for measuring Cd, Cu, As, Hg, Pb, Cr, Zn and Ni was 0.002, 0.001, 0.0002, 0.0909, 0.004, 0.002, 0.0015 and 0.003 mg/L, respectively.

The compositions of commercial fertilizers and dried struvited deposits were analyzed using energy dispersive analysis (EDS) of X-rays. Additionally, X-ray diffraction (XRD, Model DMS 2000 system, SCINTAG) was used to further characterize the dried struvite deposit obtained from swine wastewater. The crystal morphology of the struvite deposit was also observed using a scanning electron microscopy (SEM, Leica Stereosacan 440).

3. Results and discussion

3.1. Characteristics of struvite deposit

To determine the morphology, the obtained preci -pitated matters were examined by SEM. As illustrated in the SEM micrograph of Fig. 2, the crystal size of struvite deposit was widely distributed in the range 14 ㎛.

XRD analysis was also used to characterize the purity of struvite deposits collected from swine wastewater. The X-ray diffractograms exhibited several peaks indicative of the struvite presence as illustrated in Fig. 3. The XRD pattern generated from the preci

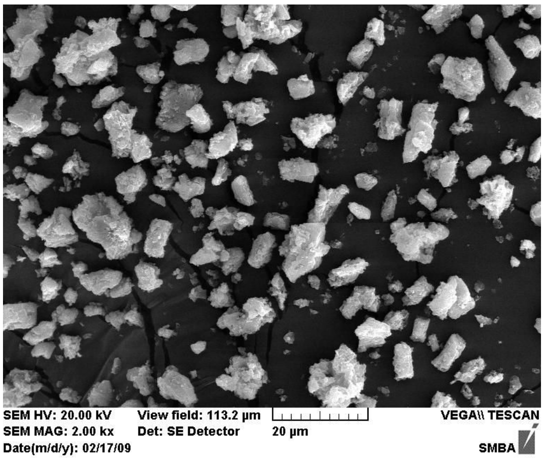

Fig. 2. SEM image (×2000) of the precipitated matters obtained from swine wastewater.

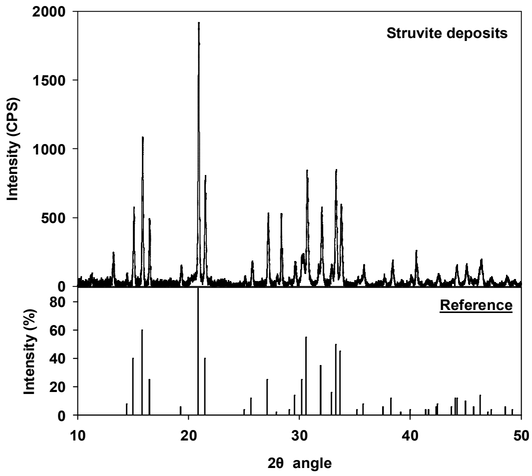

Fig. 3. XRD diffractograms of the precipitated matters

-pitated matters matched with the database model for struvite, i.e., position and intensity of the peaks. The high purity of struvite deposits would be due to the high NH4-N removal of 81 %.

3.2. Fertility evaluation of struvite deposit

The obtained struvite deposits were utilized in culti -vation tests and compared with that of commercial fertilizers to asses its fertility. During the experimental period of 32 days, the tallest leaf in each pot was selected and measured. Fig. 4 illustrates that the Chinese cabbage grew at different rates depending on fertilizing sources. On 32nd days, the average leaf length of Chinese cabbage in control, complex, organic, compost and struvite pots reached 17, 54, 24, 21 and 43 mm, respectively, as presented in Fig. 5a. After 32 days, the plants from each pot were harvested and weighed before and after drying to determine their fresh and dry weights. As evident from Fig. 5b, it was clear that the addition of struvite significantly increased the average fresh and dry weights of Chinese cabbage than control. It is well documented by previous studies that the vegetables grown in struvite pots have had much higher growth rates than control pots (without addition of external nitrogen and phosphorus) (Li and Zhao, 2003; Diwani et al.,

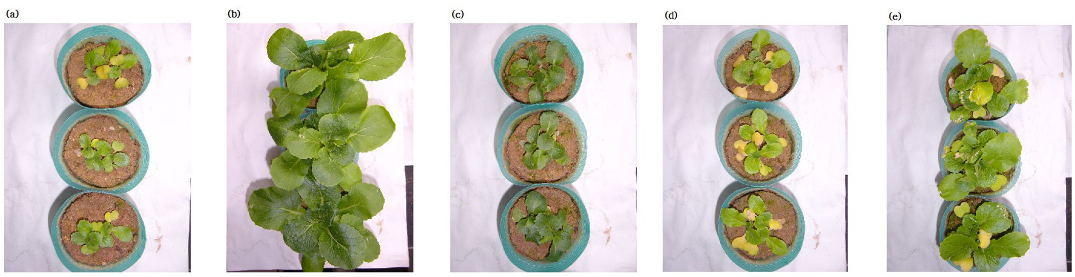

Fig. 4. Growth status of Chinese cabbage on 32 days depending on fertilizing sources: (a) Control; (b) Complex fertilizer; (c) Organic fertilizer; (d) Compost fertilizer; (e) Struvite.

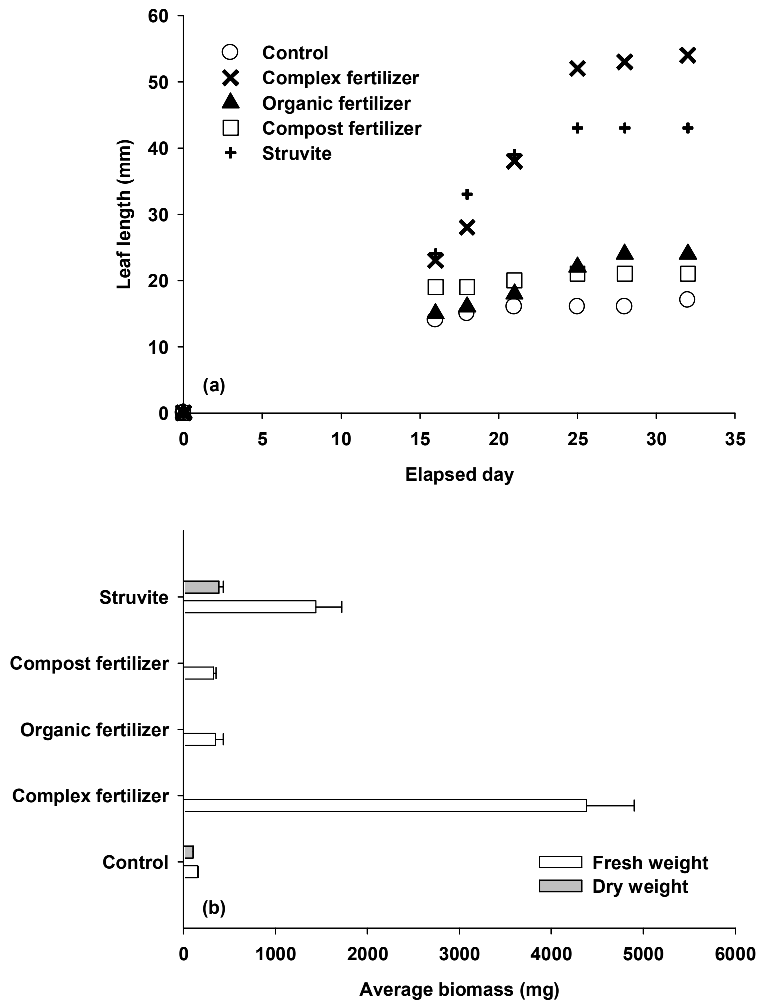

Fig. 5. Temporal variation of leaf length (a) and fresh and dry weight of lettuce after 32 days growth (b) depending on fertilizing sources: error bars indicate standard deviation.

2007; Ganrot et al., 2007; Yetilmezsoy and Sapci-Zengin, 2009). Also, the average fresh and dry weights of Chinese cabbage in struvite pots ranked second in the experimental group. The fresh and dry weights of Chinese cabbage decreased in order of complex fertilizer > struvite > organic fertilizer > compost fertilizer > control pot. This finding is further supported by data in Fig. 5a, where the longest leaf was also found in the same order. The difference of growth level according to the kind of fertilizer would be due to the amount of nutrients applied to the soil. Fig. 6 clearly illustrated that the growth rates of leaves were determined by the concentration product of phosphorus (P), potassium (K) and magnesium (Mg) applied to the soil. Specifically, they were more affected by the concentration product of P, K and Mg than by that of P and K (see Fig 6a and 6b). Mean -while, a correlation between the leaf growth rate and concentration product of K and Mg or P and Mg was weak (see Fig. 6c and 6d). It indicates that the leaf

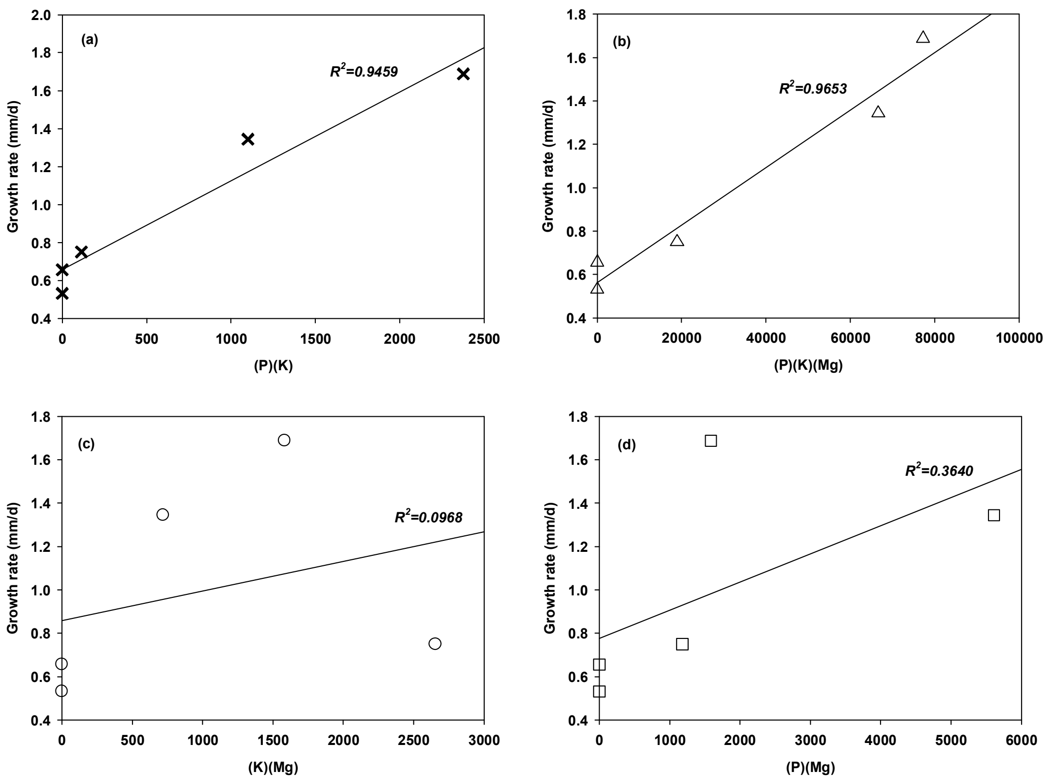

Fig. 6. Effect of concentration product on leaf growth rate of Chinese cabbage.

growth rate of Chinese cabbage was simultaneously affected by both P and K. When considering these facts, it is crystal clear that the lower growth of Chinese cabbage in struvite pots than in complex fertilizer pots was not due to the lack of Mg. It is inferred that K found in struvite deposits would be present as a potassium magnesium phosphate ($KMgPO_4 \cdot 6H_2O$, struvite-K) which can be formed as a by-product material in the process of struvite precipi -tation. The previous studies also support our experi -mental finding. From a previous study by Wilsenach *et al.* (2007), it was reported that struvite as well as struvite-K were successfully precipitated from source separated urine through struvite precipitation. Furthermore, Sun *et al.* (2010) also reported that the simultaneous precipitation of struvite and struvite-K was observed in goats during onset of urolithiasis.

3.3. Heavy metals in vegetable tissue

The heavy metal levels were also compared to evaluate the accumulation degree of heavy metals in Chinese cabbage tissue. Fig. 7 illustrates that heavy metals including copper (Cu), mercury (Hg), chromium (Cr^{6+}) and zinc (Zn) were contained in all samples, whereas cadmium (Cd), arsenic (As) and nickel (Ni) were not detected. Similarly, no detection of Cd and As in application of struvite to vegetable cultivation was reported in the previous study in which struvite recovered from landfill leachate was used in growing vegetables (Li and Zhao, 2003). For Cu and Hg, their concentrations in struvite pots were slightly lower than in complex fertilizer pots. For Cr^{6+}, its level in struvite pots was lower than in other pots, except for compost fertilizer pots. In case of lead (Pb), struvite

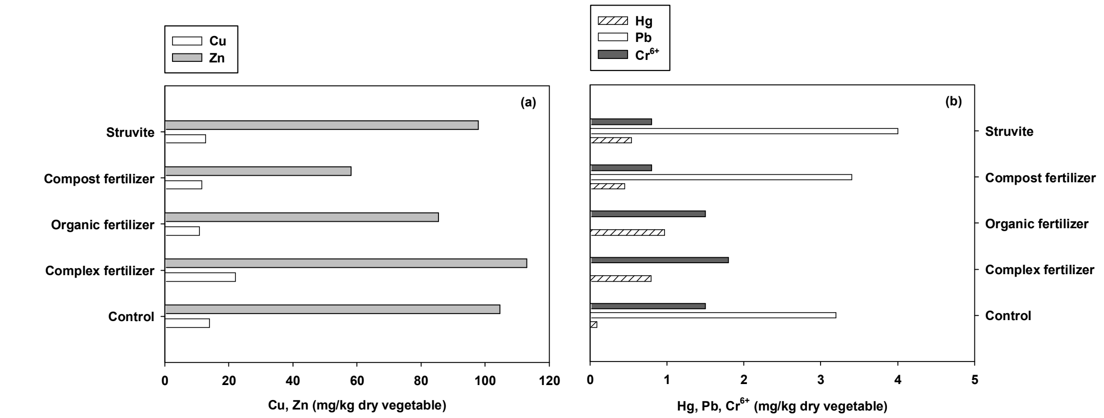

Fig. 7. Concentration of heavy metals in dried Chinese cabbage: (a) Cu and Zn, (b) Hg, Pb and Cr^{6+}.

pots had the highest concentration in all tested pots, although there was no significant difference between struvite pots and complex fertilizer pots. In summary, Chinese cabbage tissue of struvite pots did not have such high level of heavy metal concentration compared to other commercial fertilizer. Also, the test data of accumulated heavy metal in Chinese cabbage indicates that the cultivation of Chinese cabbage with struvite deposit recovered from swine wastewater could be safe.

3.4. Nutrients in vegetable tissue

Meanwhile, the availability of nutrients from stru-vite and commercial fertilizers was estimated by investigating nutrients contained in the tissue of Chinese cabbage. Fig. 8 shows that the harvested leaves from struvite pots contained relative high concentration of N, P, K, Ca and Mg. The control pots had the lowest concentration of N, P, K, Ca and Mg compared to other pots. It is interesting to observe that P concentration in struvite pots was much higher than that in commercial fertilizer pots. Much more uptake of P in struvite pots would be explained by the fact that phosphorus amount in struvite deposits was the highest level as found in Table 2. However, it

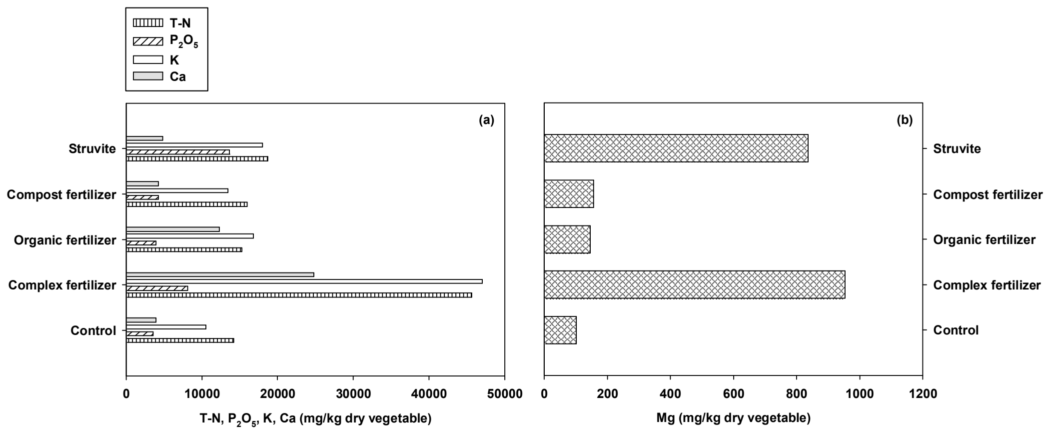

Fig. 8. Concentration of nutrients in dried Chinese cabbage: (a) T-N, P_2O_5, K and Ca, (b) Mg.

should be noted that the amount of N, K, Ca and Mg found in the vegetable tissue of complex fertilizer pots was higher than those of struvite pots. When considering the fact that the leaf growth rate of Chinese cabbage was simultaneously affected by both P and K as shown in Fig. 6, Fig. 8 well explains that K would be the most important element influen -cing the growth rate of Chinese cabbage because the amount of K was the highest in complex fertilizer pots.

3.5. Effect of struvite dosage on Chinese cabbage cultivation

The optimum struvite dosage for cultivating Chinese cabbage was determined. During the experimental period of 42 days, the leaf length of Chinese cabbage was periodically measured. The agricultural tests showed that the Chinese cabbage grew at different rates as a function of applied struvite dosage as illust -rated in Fig. 9. Fig. 10a shows that the leaf length of Chinese cabbage increased as the struvite dosage increased from 0 to 2.4 g struvite/kg soil. However, no additional growth was observed when struvite dosage was increased over 2.0 g struvite/kg soil. Similar trend was also observed in the Chinese cabbage biomass based on fresh and dry weight as shown in Fig. 10b. The test results of wet and dry weight show that increasing struvite dosage up to 2.0 g struvite/kg soil has positive effect on biomass, but ineffective beyond that. These findings were supported by the study of Li and Zhao (2003). They explained that increasing the amount of struvite, which is recovered from landfill leachate, could further stimulate the growth of water convolvulus.

4. Conclusions

Struvite recovered from swine wastewater was applied in cultivation of Chinese cabbage. The ferti

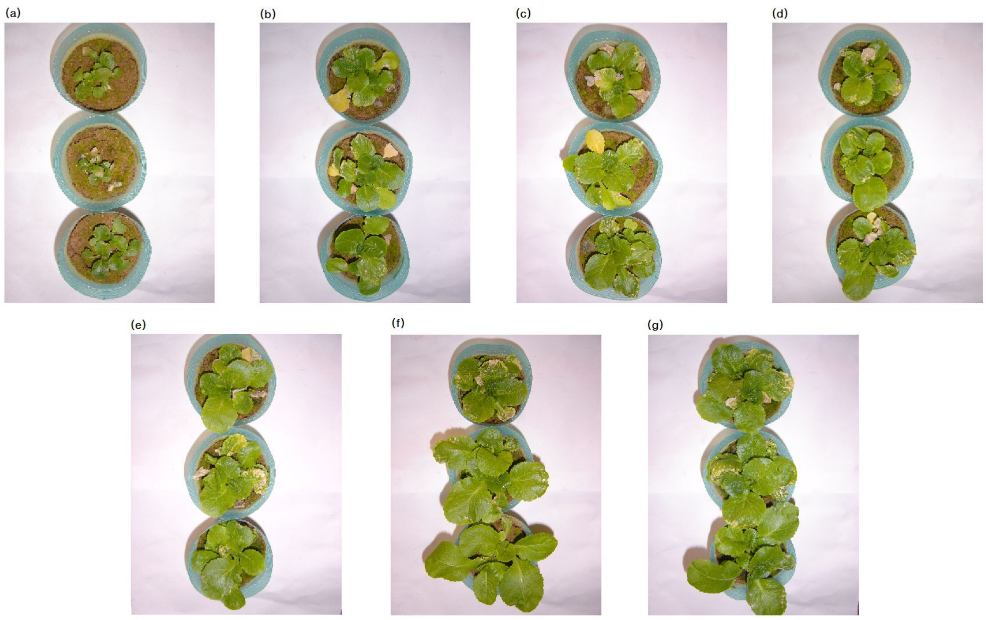

Fig. 9. Growth status of Chinese cabbage on 42 days as a function of applied struvite dosage: (a) 0 g struvite/kg soil; (b) 0.4 g struvite/kg soil; (c) 0.8 g struvite/kg soil; (d) 1.2 g struvite/kg soil; (e) 1.6 g struvite/kg soil; (f) 2.0 g struvite/kg soil; (g) 2.4 g struvite/kg soil.

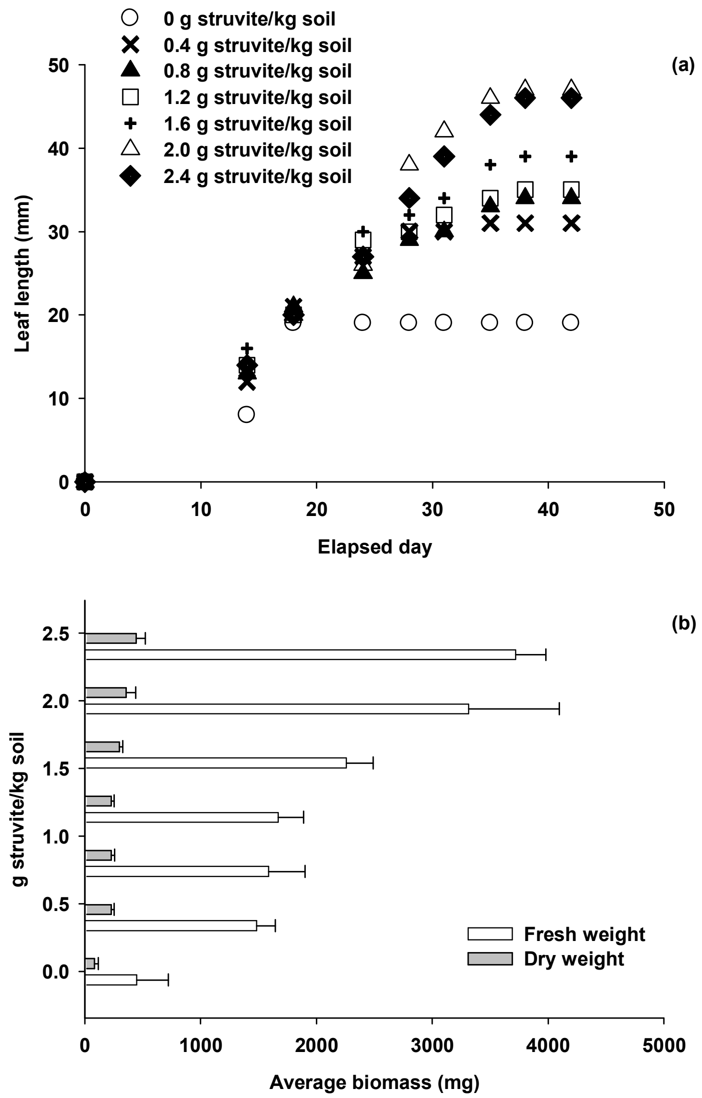

Fig. 10. Temporal variation of leaf length (a) and fresh and dry weight of lettuce after 42 days growth (b) as a function of applied struvite dosage: error bars indicate standard deviation.

-lizing value of struvite was evaluated by comparing it with commercial fertilizers by pot trial tests. Further -more, the optimum struvite dosage was determined. Based on the experimental results, the following conclusions were drawn:

(1) The capability of struvite as a fertilizer far surpassed other fertilizers except for complex fertilizer during the experimental period of 32 days. Also, it was revealed that the growth rate of Chinese cabbage was simultaneously controlled by the amount of phosphorus (P) and potassium (K).

(2) In the investigation of heavy metal effects on the Chinese cabbage growth, the concentrations of Cu and Hg in struvite pots were lower than in complex

fertilizer pots. Moreover, the concentration of Cr^{6+} in struvite pots was lower than in other pots, except for compost fertilizer pots.

(3) It was found that the struvite source provided the essential crop nutrients of N, P, K, Ca and Mg for Chinese cabbage as much as other commercial ferti -lizers. Specifically, much more amount of P was observed in the vegetable tissues of struvite pots.

(4) It was revealed that the Chinese cabbage growth rate increased as struvite dosage increased. The optimum dosage was 2.0 g struvite/kg soil and any additional dosage over the optimum amount didn't cause more growth of Chinese cabbage.

(5) Consequently, it was proved that struvite deposits, recovered from swine wastewater were effec -tive as a multi-nutrient fertilizer in cultivating Chinese cabbage.

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