

RECOVERY OF AMMONIUM-NITROGEN FROM LANDFILL LEACHATE AS A MULTI-NUTRIENT FERTILIZER

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Abstract: The leachates generated in Hong Kong landfill sites contain high strength of ammonium-nitrogen (NH_4^+ -N) in the range of 2,000-5,000 mg l⁻¹, which could be used to produce nitrogen-fertilizer. To recover the NH_4^+ -N from the leachate, a lab-scale study was performed to investigate the efficiency of magnesium ammonium phosphate (MAP) precipitation using three combinations of chemicals, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O} + \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$, $\text{MgO} + 85\% \text{ H}_3\text{PO}_4$ and $\text{MgSO}_4 \cdot 7\text{H}_2\text{O} + \text{Ca}(\text{H}_2\text{PO}_4) \cdot \text{H}_2\text{O}$. The results indicated that the NH_4^+ -N was recovered by 92%, 36% and 70%, respectively using the selected chemicals at pH 9.0 and a molar ratio of Mg:N:P = 1:1:1. The MAP precipitate ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) as struvite was examined by scanning electron microscopy and the results showed that the MAP precipitate had a typical morphology of elongated tubular and short prismatic crystals. The MAP precipitate was also analyzed by energy dispersive spectroscopy and the analytical results indicated its composition of 4.9%N, 8.6%Mg and 16%P, which was similar to the standard struvite composition of 5.7%N, 9.9%Mg and 12.6%P.

To investigate the fertility of the MAP precipitate, a set of pot trial tests was conducted. Four quick-growth vegetables, Chinese flowering cabbage (*Brassica parachinensis*), Chinese

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chard (*Brassica rapa var. chinensis*), water spinach (*Ipomoea aquatica*) and water convolvulus (*Ipomea aquatica, I. reptans*), were planted in completely decomposed granite soil and the MAP precipitate was applied as a fertilizer. The germination and growth of the selected vegetables in the pots with MAP showed significantly greater rates than those in the pots without MAP as control tests. The experiment also demonstrated that over-dosing about 2-8 times of MAP in the soil did not cause any problems with water spinach (*Ipomoea aquatica*) growth, due to its limited solubility in water. MAP application as a fertilizer did not result in more heavy metals in the vegetables than those from soil and model fertilizer, whereas the higher levels of P and Mg in the vegetables would stimulate vegetables' growth. This study confirmed the feasibility of recovering NH₄⁺-N from landfill leachate effectively by chemical precipitation, and also the feasibility of applying the MAP precipitate as a multi-nutrient fertilizer for vegetable growth.

Key words: Landfill leachate, ammonium nitrogen, precipitation, crystallization, magnesium ammonium phosphate (MAP), struvite and vegetables.

INTRODUCTION

Magnesium ammonium phosphate (MAP), struvite, is a naturally occurring crystal, when the combined concentrations of Mg²⁺, NH₄⁺ and PO₄³⁻ exceed its solubility limit. As struvite has a low solubility constant pK_s between 12.60 (Stumm and Morgan, 1996) and 13.26 (Ohlinger et al. 1998), its insoluble form can be easily formed and simply separated from water phase. Controlled MAP crystallization can be used to remove nutrients such as nitrogen and phosphorus from wastewaters. However, the success of the MAP process depends on two main factors: the molar ratio of Mg:N:P and the pH value in the reactor (Münch and Barr, 2001). In most cases, magnesium deficiency in wastewater allows it to be

added to remove all available nutrients such as phosphorus and/or ammonium-nitrogen in water phase. Two types of magnesium, $Mg(OH)_2$ and $MgCl_2$, are generally used in the MAP process. Compared to $Mg(OH)_2$, $MgCl_2$ is more widely used because it dissociates faster than $Mg(OH)_2$ and results in shorter reaction time. If the MAP process is applied to remove ammonium-nitrogen only, phosphate forms of H_3PO_4 or $NaHPO_4$ are usually needed (Siegrist, 1996; Schulze-Rettmer, 1991). As for the required alkaline condition of an elevated pH in the MAP system, it can be achieved by alkaline addition or pre-aeration (Battistoni et al., 1998). A study performed by Fujimoto et al. (1991) has demonstrated that addition of $NaOH$ is more effective than addition of lime or $Mg(OH)_2$.

The fertilizing property of MAP ($MgNH_4PO_4$) as struvite has been already demonstrated in the 1960s in Germany and the United States as summarized by Schuiling and Andrade (1998). Because it is only slightly soluble in water and soil solutions, slow-release struvite has been found to be a highly effective source of phosphorus, nitrogen and magnesium for plants through both foliar and soil application. When properly granulated, struvite can be applied to soil at rates greatly exceeding those of conventional fertilizers without danger of burning plant roots. Since less frequent application of struvite is required, struvite has been used commercially for container plants and is also appropriate for use on turf, tree seedlings, ornamentals, vegetables, and flower boards (Münch and Barr, 2001). Improved growth of grass, fruit and various high-value crops was observed when they were fertilized with struvite, compared with conventional soluble fertilizers (Rothbaum and Rohde, 1976). Its use may be particularly advantageous when soluble fertilizers are inefficient, low soluble salts are required in the root zone, or a long residual effect is required.

The leachates generated in Hong Kong landfill sites contain high levels of ammonium-nitrogen in the range of 2,000-5,000 mg l^{-1} . Currently, aerobic biological processes are

engaged for leachate treatment at the main landfill sites in Hong Kong, but no consideration of ammonium-nitrogen recovery at all. Actually the ammonium-nitrogen in the landfill leachate can be recovered by MAP precipitation a fertilizer. So far, only limited studies on this technique have been reported (Ghosh et al., 1996; Goto, 1998). This study was aimed at investigating the feasibility of generating MAP precipitate and applying this precipitate as a potential fertilizer. The specific objectives are to (1) precipitate ammonium-nitrogen from leachate using different chemical sources; (2) characterize the MAP precipitate; and (3) evaluate the fertilizing value of MAP precipitate using plant studies.

MATERIAL AND METHODS

MAP precipitation tests

Raw leachate samples were collected from the North East New Territory (NENT) landfill site in Hong Kong, where up to $800 \text{ m}^3 \text{ d}^{-1}$ of leachate generated from the 37 Mm^3 Landfill contains COD in excess of $10,000 \text{ mg l}^{-1}$, and ammoniacal-N up to $4,000 \text{ mg l}^{-1}$. The raw samples were fully characterized prior to the experiment. The experiments of MAP precipitation were carried out in 500-ml beakers in a batch mode with the following experimental procedure: 1) the leachate sample was first put into the beakers and then the chemicals were added as required (magnesium was added before phosphorous); 2) a magnetic stirrer at the bottom of beaker was switched on and the pH of the leachate sample was monitored using a pH meter; 3) MAP reaction lasted for about 15 min until an equilibrium state reached as indicated by a stable pH ; 4) the magnetic stirrer was switched off to allow the solution to settle for another 15 min, and then the supernatant sample above the precipitate was taken for analysis; 5) pH was then adjusted to a desired level by adding 10M NaOH solution into the leachate sample with mixing and the second reaction and settling as same as above were followed; 6) the supernatant sample was collected and analyzed again; 7)

A MAP precipitate sample was also collected from the bottom of the beakers and characterized.

Pot trial tests

Sandy clay (Red Earth) samples as raw soil were taken from an upland area in Hong Kong and sieved with a maximum aperture size of 3 mm prior to filling each pot ($\phi 170$ mm \times 100 mm). The soil was slightly acidic (pH 6.2), nutrient-poor, redundant and low in organic carbon (1.96%). To evaluate the fertilizing value of the MAP precipitate, four species of quick-growth vegetables including Chinese flowering cabbage (*Brassica parachinensis*), Chinese chard (*Brassica rapa var. chinensis*), water spinach (*Ipomoea aquatica*) and water convolvulus (*Ipomea aquatica, I. reptans*) were selected for the pot trial tests. Each of the plants was growing in 3 different conditions: 1) raw soil with addition of KCl only as control; 2) raw soil with addition of KCl and MAP precipitate; and 3) raw soil with addition of KCl, NH_4NO_3 and $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ as a model fertilizer. It was suggested that an optimum dosage to add fertilizer in soil for Chinese vegetable growth in soil was hydrolyzed nitrogen >70 mg kg^{-1} , exchangeable potassium $>100-150$ mg kg^{-1} and instant phosphorus $>60-80$ mg kg^{-1} respectively (He, 1994). If the lower limits were applied, the required dosage of KCl (99.8%), NH_4NO_3 (99.5%) and $\text{Ca}_2(\text{H}_2\text{PO}_4) \cdot \text{H}_2\text{O}$ in 1 kg of soil were 191 mg, 201 mg and 265 mg respectively. If the MAP precipitate were used as fertilizer, its dosage on the basis of a 70 mg kg^{-1} nitrogen requirement would be 1.4 g kg^{-1} in soil. Therefore, 2.8 g of MAP precipitate was applied in each pot, since each pot contained 2 kg of soil. Other experimental conditions are summarized in Table 1. To investigate the influence of MAP dose on plant growth, water spinach (*Ipomoea aquatica*) was planted in four pots with different amounts of MAP addition as one time (1 \times), two times (2 \times), four times (4 \times) and eight times (8 \times).

[Table 1]

The vegetable cultivation was proceeded as follows: 1) fill each pot with 2 kg of sieved CDG as base soil; 2) remove the top layer of 1-2 cm and evenly mix with the pre-weighed chemicals as required in each test; 3) add 200 ml of water into each pot and evenly lay the vegetable seeds directly on the exposed surface of the soil; 4) cover the seeds with the removed soil; 5) place the pots outdoor and irrigate as required.

Analyses and measurement

Both pH and NH_4^+ -N were determined by an expandable ion analyzer (EA940, Orion Research Corporation). Conductivity was analyzed by a conductivity meter (ES-14E, Horiba Ltd., Japan). Metals such as K, Na, Fe, Mn, Mg and Ca in leachate were determined by an atomic absorption spectrometer (AAS3300, Perkin Elmer). The parameters including total dissolved solids (TDS), fixed dissolved solids (FDS), chemical oxygen demand (COD), biochemical oxygen demand (BOD), and alkalinity were measured with the standard methods (APHA, 1995). The MAP precipitate samples were filtered through a 0.45 μm filter and then dried in an oven below 60 °C to minimize water loss (Ando et al., 1968). The crystal morphology of the MAP precipitate was observed using a scanning electron microscopy (SEM, Leica Stereoscan 440). The analyses of MAP composition were conducted by energy dispersive spectroscopy (EDS, Oxford Model 5526), with an accelerating voltage of 10.0 kV.

Total P and metals including Cd, Cr, Cu, Ni, Pb, Zn, Co and Mo were analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES 3300DV, Perkin Elmer) after acid digestion. In which both soil and vegetable samples were dried at 70 °C and ground prior to digestion, and then 0.25 g of soil sample was placed in a Pyrex tube (150 mm × 18 mm) containing 4 ml of 65% HNO_3 (AR) and 1 ml of 60% HClO_4 . The mixture was heated

with a gradient program of 50 °C for 3 h, 75 °C for 0.5 h, 100 °C for 0.5 h, 150 °C for 3 h and 175 °C until dry. After the temperature cooled down naturally, 10 ml of 5% HNO₃ was added into tube and mixed using a vortex. The mixture was then centrifuged at 3500 rpm for 10 min and the metal concentrations of the supernatant were determined. For the vegetative samples, a mild heating program started at 60 °C for 3 h, 100 °C for 1 h, 120 °C for 1 h and 190 °C for 2 h respectively. After drying and cooling, 10 ml of 5% HNO₃ was added into the tube and heated at 50 °C for 15 min. When the sample temperature was back to room temperature, the solution in each tube was ready for determination of metals concentration as well as phosphorus by ICP-AES.

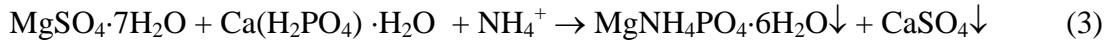
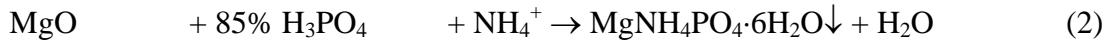
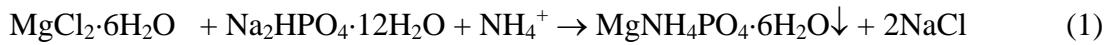
RESULTS AND DISCUSSION

Recovery of ammonium-nitrogen from leachate by MAP precipitation

The leachate sample collected from the NENT landfill site had typical characteristics of an aging methanogenic landfill leachate as summarized in Table 2, which appeared peaty brown in color, slightly ammoniacal, and had high conductivity and alkalinity, a low BOD₅/COD ratio and an elevated ammonia-nitrogen concentration. The leachate sample contained trace amounts of heavy metals including Cr, Cu, Zn and Pb, but Cd and Hg concentrations were below the detection limits.

[Table 2]

To precipitate an approximate NH₄⁺-N concentration of 2,750 mg l⁻¹ effectively from the landfill leachate, three combinations of chemicals including MgCl₂·6H₂O + Na₂HPO₄·12H₂O, MgO + H₃PO₄ and MgSO₄·7H₂O + Ca(H₂PO₄)₂·H₂O were employed in the study. The principal chemical reactions for the MAP precipitation are shown below:



Based on the stoichiometric calculation, the stipulated dose of chemicals to precipitate NH_4^+ -N is listed in Table 3, when the Mg:N:P molar ratio of 1:1:1 was employed.

[Table 3]

The experimental results, as shown in Fig. 1, illustrate the NH_4^+ -N concentrations in the leachate supernatant after precipitation at different pH levels. In these tests, it was found that the initial pH 8.5 in the leachate sample dropped immediately to 6.75, 6.16 and 5.69 respectively as soon as the chemicals were added. To minimize the NH_4^+ -N residual concentration in the leachate, the pH was raised by adding 10M NaOH solution to a final pH 11. The MAP precipitate was formed rapidly and settled quickly at the bottom of beakers after stopping stirring. When pH was increasing from 7 to 9, the NH_4^+ -N residual concentration decreased significantly, and when the pH was further rising from 9 to 11, the NH_4^+ -N concentration only decreased slightly.

[Fig. 1]

Among the three combinations of chemicals, it was found that the addition of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O} + \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ was the most efficient in terms of the NH_4^+ -N removal from the leachate, while the addition of $\text{MgO} + \text{H}_3\text{PO}_4$ achieved the poorest efficiency as shown in Fig. 1. These phenomena were similar to that in our previous work for treating the leachate from the West New Territory (WENT) landfill site (Li et al., 1999). Since MgO has a limited solubility in the water phase, its very slow dissolution in the leachate sample

resulted in a low efficiency of the MAP precipitation. To achieve the maximum recovery of ammonium-nitrogen from leachate, the addition of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O} + \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ might be the best option. Therefore, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ were used in the following tests, in which a final pH of leachate samples was adjusted to 9. The MAP precipitate generated in the tests was fully characterized to determine its physical and chemical properties.

Properties of the MAP precipitate

In general, the standard struvite as $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ is a white crystalline powder. However, in our experiment, the MAP precipitate generated from the leachate samples was light yellow in color. This off-color may have originated from co-precipitation with peaty brown side-products in the raw leachate. Some physical properties of the MAP precipitate from the leachate of local landfill sites were studied in our previous work, which included the capillary suction time (CST) of 41.4 s, solids content of 27% and dry density of $2,050 \text{ kg m}^{-3}$ (Li et al., 1999). It was indicated that the MAP precipitate had a high sludge density with a fast settling velocity similar to sand or grit and a short CST allowed it to be more easily dewatered than biosludge or flocculent sludge if dewatering is required.

To determine the morphology, the MAP precipitate generated from the NENT landfill site by adding $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ was examined by SEM in this study and the SEM micrograph is shown in Fig. 2. In general, the MAP precipitate was a mixture of elongated thick tubular and short prismatic crystals dominated with struvite. The crystal size of MAP precipitate was ranged to 10 to 50 μm . The morphology of developed crystal normally depended upon the supersaturation of the solution together with the concentration of impurities. As revealed by Abbona and Boistelle (1979) and reviewed by Durrant et al.

(1999), very high levels of supersaturation, (i.e. conditions of high growth kinetics, high Mg^{2+} and NH_4^+ concentrations and $pH > 8$) promoted the formation of bidimensional and three-dimensional twinned crystals. At high supersaturation conditions ($pH 7$), tubular crystals formed, however crystal morphology altered from a tubular formation to an increasing elongation as supersaturation decreased. Regulation of pH remained the most important role in preventing side reactions such as magnesium hydroxide ($pK_s = 11.16$) or magnesium hydrogen phosphate ($pK_s = 5.8$) precipitation. Both of them formed more quickly than slow crystallizing struvite due to their amorphous nature (Schulze-Rettmer, 1991). These indicated that the precipitate could be a mixture of struvite and the above-mentioned side-products.

[Fig.2]

The chemical properties of the MAP precipitate were also analyzed by EDS. The analytical results indicated that it had a composition 4.9%N, 8.6%Mg and 16%P, when a molar ratio of $Mg:N:P = 1:1:1$ was applied. This result was close to the theoretical composition 5.7%N, 9.9%Mg and 12.6%P of standard struvite (Münch and Barr, 2001; Anonymous, 1998).

Fertilizing potential of the MAP precipitate

After seeding, the vegetables sprouted for about 5 days. It was observed that the seedlings in the 3 pots of control, MAP fertilizer and N&P model fertilizer looked very similar, but the vegetables in the 3 pots grew at very different rates. The vegetables in the MAP pots and N&P pots grew much faster than those in the control pots due to poor N and P nutrient status, but there was no significant difference between the vegetable growth between the MAP pots and N&P pots as illustrated in Fig. 3.

[Fig. 3]

To study vegetable growth, the four vegetables were harvested on the 33rd day after sowing. Before the harvest, the vegetables in the pots were sprayed with water to wash the dust off. After drying in ambient conditions, the 5 tallest vegetable individuals in each pot were selected to measure either the longest leaves (Fig. 3a and Fig. 3b) or the longest stalks (Fig. 3c and Fig. 3d), and then their growth rates were compared as shown in Fig. 4. After measuring the height, the cut vegetable samples were weighed before and after drying at 60 °C. Their wet weight and dry weight were recorded and compared as shown in Fig. 5. On the basis of growth rate of both wet weight (Fig. 5a) and dry weight (Fig. 5b), it was obvious that the vegetables growing in the control pots had much lower growth rates due to the lack of both N and P application, compared to the vegetables growing in the MAP pots and N&P pots. Since the growth rates in the MAP pots and N&P pots were quite close, it indicated that the MAP precipitate played a similar role as the model fertilizer of NH_4NO_3 and $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ chemicals.

[Fig. 4]

[Fig. 5]

In another set of experiment, water convolvulus (*Ipomea aquatica*, *I. reptans*) was cultivated in four pots in parallel with different amounts of MAP addition as 1×, 2×, 4× and 8×MAP. The experiment lasted for 35 days before a harvest. It was observed that there was no significant difference among the sprouts in different pots at the beginning stage and the vegetable in all pots grew quite well during the 35 days period as shown in Fig. 6. The experiment also demonstrated that there were no burning effects on the growth of water

convolvulus. During the experiment period, water convolvulus was harvested on the 25th and 35th day after sowing respectively and the growth rates based on height and wet weight are shown in Fig. 7. In contrast to causing the burning effects, more MAP addition could obviously stimulate the growth of the vegetable tested.

[Fig. 6]

[Fig. 7]

Heavy metals could be a great concern in agronomic field if the recovered MAP is applied as a fertilizer. The heavy metals in both the raw soil and MAP precipitate were analyzed and the results are compared in Table 4. The results showed that the contents of Zn and Cu in the MAP precipitate were slightly higher than those in the raw soil, while the contents of other metals in the MAP precipitate were much lower than those in the raw soil. These results indicated that there should not be any concern of heavy metal contamination if the MAP precipitate is applied into the soil.

[Table 4]

To further investigate the heavy metal effects on the vegetable growth, the heavy metal levels in vegetables tissue were also analyzed and the results are compared in Table 5. The data show heavy metals including Pb, Zn, Ni and Cu present in all vegetable samples with the levels of the vegetable growth in the N&P pots being slightly higher than those growing in the MAP pots. The Cu content was an exception. In general, it seemed that the two sets of data were in a same grade. Compared to the metals in the vegetables growing in the control pots, MAP application as a soil conditioner didn't contribute more metals into the

vegetables, especially the contents of Cd and As in all the vegetables were below the detection limits of ICP analysis in this study.

[Table 5]

Other elements relevant to vegetable growth were also analyzed and the results are listed in Table 6. Phosphorus is a major component of nucleoprotein and lecithin, and plays an important role in the bio-metabolism. Magnesium is a component of chlorophyll and the activating agent of enzyme production. Magnesium has a close relationship with P transfer. The analytical results indicated that the vegetables in the MAP pots uptook more P and Mg than those in the control and N&P pots, which might be affected by a different pH condition in the MAP pots. However, it was clear that higher levels of P and Mg in the vegetables would be beneficial to their growth as observed in this study.

[Table 6]

CONCLUSIONS

Based on the experimental results in this bench-scale study, some specific conclusions might be drawn as follows:

1. Ammonium-nitrogen in the local landfill leachate was effectively recovered as MAP precipitate by adding the chemicals of $MgCl_2 \cdot 6H_2O + Na_2HPO_4 \cdot 12H_2O$, $MgO + 85\% H_3PO_4$ and $MgSO_4 \cdot 7H_2O + Ca(H_2PO_4) \cdot H_2O$ respectively. The most effective chemical was $MgCl_2 \cdot 6H_2O + Na_2HPO_4 \cdot 12H_2O$ with a recovery efficiency of 92% at pH 9.0 and a molar ratio of Mg:N:P = 1:1:1.

2. The SEM micrographs of the generated MAP precipitate show a typical morphology of elongated tubular and short prismatic crystals, while the EDS results indicated that MAP precipitate consisted of 4.9%N, 8.6%Mg and 16%P.
3. The four quick-growth vegetables, Chinese flowering cabbage (*Brassica parachinensis*), Chinese chard (*Brassica rapa var. chinensis*), water spinach (*Ipomoea aquatica*) and water convolvulus (*Ipomea aquatica*, *I. reptans*) in the CDG soil pots amended with the MAP grew very well. The experiment also demonstrated that the sprout and growth of water spinach (*Ipomoea aquatica*) was not affected by over-dosing MAP in the soil, and MAP application as a fertilizer didn't contribute more heavy metals into the selected vegetables than those of CDG soil with the model fertilizer.

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