

EFFICIENCY OF BIOLOGICAL TREATMENT AFFECTED BY HIGH STRENGTH OF AMMONIUM-NITROGEN IN LEACHATE AND CHEMICAL PRECIPITATION OF AMMONIUM-NITROGEN AS PRETREATMENT

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ABSTRACT

Leachate samples with high strength of ammonium-nitrogen ($\text{NH}_4^+\text{-N}$) were collected from a local landfill site in Hong Kong. Two experiments were carried out to study (1) the inhibition of microbial activity of activated sludge by $\text{NH}_4^+\text{-N}$ and (2) the chemical precipitation of $\text{NH}_4^+\text{-N}$ from leachate as a preliminary treatment prior to the activated sludge process. The experimental results demonstrated that the efficiency of COD removal decreased from 97.7 to 78.1% and the dehydrogenase activity of activated sludge decreased from 9.29 to 4.93 $\mu\text{g TF/mg MLSS}$ respectively, when the $\text{NH}_4^+\text{-N}$ concentration increased from 53 to 800 mg/l. The experiment also demonstrated that the $\text{NH}_4^+\text{-N}$ in the leachate can be quickly precipitated as $\text{MgNH}_4\text{PO}_4\cdot 6\text{H}_2\text{O}$, after addition of $\text{MgCl}_2\cdot 6\text{H}_2\text{O} + \text{Na}_2\text{HPO}_4\cdot 12\text{H}_2\text{O}$. The $\text{NH}_4^+\text{-N}$ concentration was reduced from 5,618 to 112 mg/l within 15 minutes, when a mole ratio of $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-} = 1:1:1$ was used. The optimum pH to reach the minimum solubility of

$\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ was found to be in the range of 8.5 and 9.0. Attention should be given to the high salinity formed in the treated leachate by using $\text{MgCl}_2 \cdot 6\text{H}_2\text{O} + \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$, which may affect microbial activity in the following biological treatment processes. Using other two combinations of chemicals [$\text{MgO} + 85\% \text{H}_3\text{PO}_4$ and $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O} + \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$] could minimise salinity generation after precipitation, while they were less efficient for NH_4^+ -N removal.

Key words: Ammonium-nitrogen; landfill leachate; chemical precipitation; magnesium ammonium phosphate

INTRODUCTION

In Hong Kong, there are several strategic large landfill sites, where generate a large amount of leachate containing high strength of organic and ammonium-nitrogen (COD=5,000-20,000 mg/l and NH_4^+ -N=3,000-5,000 mg/l). In general these leachate have low ratios of BOD_5/COD and COD/NH_4^+ -N (EPD 1996; Robinson and Luo 1991). The leachate with high NH_4^+ -N content generated at the aged landfill sites has difficulty to be effectively treated by conventional biological treatment processes due to its toxic inhibition to microorganism. The current practice in Hong Kong is that most leachate is biologically treated on-site. To eliminate the microbial inhibition causing by the ammonium in biological reactors, normally a very high ratio up to 300 – 400% of effluent recirculation needs to be applied to maintain a low ammonium concentration in the biological reactors. Although this high recirculation ratio can technically solve the problem, it would greatly reduce the volumetric loading of the reactors and results in a very high treatment cost of more than HK\$100/m³. At the same time, a large space is also required for construction of those bioreactors. To minimise the operating cost, the recirculation ratio in the process should be reduced as much as possible. Therefore, the maximum level of ammonia in the aerobic tanks to generate significant inhibition to activated sludge would be a critical parameter for the improvement of these biological processes.

Chemical precipitation of NH_4^+ -N in its insoluble form of magnesium ammonium phosphate (MAP) is a new option to selectively remove ammonium from liquid and has been studied for treating several wastewaters including tannery effluent (Tünay et al. 1997), digester supernatant (Siegrist et al. 1994; Siegrist 1996), coke and nitrogen wastewater (Zdybiewska and Kula 1991) and also sludge liquor (Schulze-Rettmer 1991). This study was aimed (1) to determine the inhibition of microbial activity of activated sludge causing by ammonium-nitrogen strength in leachate and (2) to investigate the optimum conditions to chemically precipitate ammonium-nitrogen from landfill leachate as a preliminary treatment process prior to biological treatment.

METHODOLOGY

Material

Raw leachate samples were collected from a local landfill site in Hong Kong and their composition was characterised in our laboratory.

Experiment I

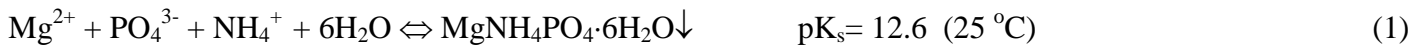
The raw leachate sample with an initial ammonia-nitrogen of 5618 mg/l was diluted by tap water accordingly to achieve variable ammonia-nitrogen strengths in the range of 50-800 mg/l. However, to ensure there was an enough amount of biodegradable organics existing in the tests, a fixed amount of glucose, equivalent to 1 g/l of COD, was added into all the diluted leachate samples. Six reactors with same volume of 2 litres were run in parallel. While one reactor was used for treating the synthetic wastewater with 50 mg/l NH_4^+ -N (Tap water plus NH_4^+ -N only) as a control test, the other five reactors were used for treating the diluted leachate samples with addition of glucose. In addition, phosphorus from K_2HPO_4 and KH_2PO_4 were also added into the diluted leachate samples at a ratio of glucose COD: P = 100:1 by weight, and either NaHCO_3 or HCl was used for adjusting pH to 7.5 - 8.5 in the biological reactors. All reactors were initially filled with the diluted leachate containing mixed liquor suspended solids (MLSS) of 2 g/l and added COD (glucose) of 1 g/l, and continuously operated for 24 hours. In each cycle, the treated wastewater samples were consequently

collected and analysed for COD, $\text{NH}_4^+\text{-N}$, MLSS and TTC-dehydrogenase activity (Eckenfelder et al. 1986; Ford et al. 1966; Weddle and Jenkins 1971). After 24 hours, the COD concentration in the reactors was made up to 1 g/l again by adding glucose stock solution accordingly and the second cycle started. The experiment lasted for 6 days and the operational conditions in all reactors are listed in Table 1.

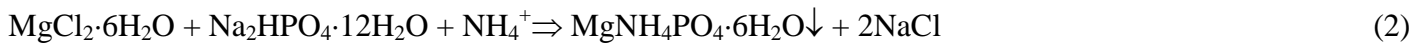
[Table 1]

Experiment II

The formation of MAP can be expressed in the following reaction:



The stoichiometric ratio of different chemicals needed to precipitate $\text{NH}_4^+\text{-N}$ can be calculated from the following equations:



The MAP precipitation tests were carried out in 500-ml beakers with a batch mode and its procedures are outlined below:

- 1) to weigh chemicals and to measure the 500-ml raw leachate for each test;
- 2) to put the measured leachate samples and the weighed chemicals into beakers;
- 3) to start magnetic stirrers at the bottom of beaker and to monitor pH of the samples;
- 4) to react for 15 minutes until a stable pH indicated (equilibrium state);
- 5) to stop magnetic stirrers and to settle for 15 minutes;
- 6) to adjust pH for the minimum MAP solubility with 10 M NaOH solution;
- 7) to collect the supernatant above the MAP precipitate for measuring the required parameters;
- 8) to use mixed liquor and the generated MAP sludge for examining its characteristics.

The 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 $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O} + \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, were respectively tested. Totally 13 tests with different $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ ratios were carried out (11 sets by adding $\text{MgCl}_2 \cdot 6\text{H}_2\text{O} + \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$; the other two by adding $\text{MgO} +$

85% H_3PO_4 and $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O} + \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$). The effect of pH on the remaining $\text{NH}_4^+\text{-N}$ was also investigated in the study. After reaction and sludge sedimentation, supernatant was separated from the settled MAP sludge and collected as supernatant samples. Then 10 M NaOH solution was progressively added into the supernatant to explore the minimum solubility of MAP sludge. The pH and remaining $\text{NH}_4^+\text{-N}$ were carefully monitored during the addition of NaOH.

Analytical methods

The parameters including COD, BOD₅, MLSS, TSS (total suspended solids), VSS (volatile suspended solids), TDS (total dissolved solids) and FDS (fixed dissolved solids) were examined with the Standard Methods (APHA 1995). pH and $\text{NH}_4^+\text{-N}$ were measured with an expandable ion analyser (EA940, Orion Research Incorporation), while DO was monitored with a YSI DO meter. Dehydrogenase activity was employed in this study to indicate microbial activity because of its accurateness and rapidity (Awong *et al.* 1985).

Dehydrogenases are the oxidation-reduction enzymes, which participate in the mainstream of electron transport from organic substrate to molecular oxygen. Dehydrogenase activity assays were determined with a Japanese standard examination method (Japan Association 1984) with the following procedure: add 1-mL of triphenyl tetrazolium chloride (TTC, 5 g/l) and 3 drops of NaSO_3 solution (5 g/l) in a 15-ml tube containing 10 ml of mixed liquor from the reactor and then put the tube in dark place at 20°C for 1 hour; centrifuge the tube 5 minutes at 3000 rpm, discard the supernatant and add 10 ml of ethanol into the tube for extraction of colour product of triphenyl formazon (TF) from cells, then centrifuge again and finally measure the concentration of the produced TF at 480 nm by spectrophotometer.

To substantiate the results of microbial activity expressed in dehydrogenase activity, specific oxygen uptake rate (SOUR) was also determined for each reactor according to the following procedure: take 300 ml of mixed liquor out from the respective reactor and put it into 6 BOD bottles respectively to allow for the sludge settling for 30 minutes; siphon the supernatant out, put magnetic stirrer in it and refill the bottles with pre-aerated

synthetic wastewater with initial COD of 500 mg/l; start mixing and record the depletion of dissolved oxygen in each bottle with DO meter; and finally calculate SOUR expressed in $\text{mgO}_2/\text{gMLSS}\cdot\text{hr}$.

RESULTS AND DISCUSSION

Characteristics of landfill leachate sample

The collected leachate sample was characterised and its analytical results are shown in Table 2.

[Table 2]

Inhibition of microbial activity by ammonia-nitrogen in the diluted leachate

In this study, the diluted leachate samples with addition of glucose had COD concentration in the range of 1 – 2 g/l and variable NH_4^+ -N concentration from 50 to 800 mg/l, and were tested to determine the inhibition of microbial activity an activated sludge process. The activated sludge collected from a local sewage treatment works was used and acclimated in the 6 reactors, which were operated with same initial MLSS of 2 g/l. One reactor was continuously fed with the synthetic wastewater containing 50 mg/l NH_4^+ -N as a control and the other five reactors were used for treating the diluted leachate samples with increasing NH_4^+ -N concentrations from 50 to 800 mg/l. After every 24 hours operation, samples from each reactor were collected for the determination of remaining COD and dehydrogenase activity. To study the efficiency of activated sludge process affected by NH_4^+ -N strength, the average value of COD and dehydrogenase activity at each day are calculated and plotted in Figure 1 and Figure 2 respectively.

[Figure 1]

[Figure 2]

In the experiment, it had been assumed that the initial glucose COD of 1 g/l was completely biodegradable while the COD in the diluted leachate was partially biodegradable. The biodegradable portion of COD in the diluted leachate was estimated by the equation of $\frac{0.22 \cdot COD_{measured}}{1 - e^{-0.22 \cdot t}}$, because of the ratio of BOD₅/COD in the leachate = 0.22 based on the data in Table 1. Therefore, the values of remaining COD concentrations plotted in Figure 1 had been converted into the biodegradable COD by the subtraction of non-biodegradable COD in the leachate from the measured COD. The operating conditions and experimental results in 6 reactors are summarised in Table 3.

[Table 3]

[Figure 3]

The experimental data demonstrated that average COD removal decreased from 97.7 to 78.1% (95.7% for the control) and the dehydrogenase activity decreased from 9.29 to 4.93 µg TF/mg MLSS (10.46 µg TF/mg MLSS for the control) respectively, when the ammonia-nitrogen concentration increased from 50 to 800 mg/l progressively. Microbial inhibition could also be substantiated by the decrease of SOUR from 67.4 to 45 mgO₂/g MLSS (70 mgO₂/g MLSS for the control) accordingly as shown in Figure 3. All results imply that the high strength of ammonia-nitrogen in leachate needs to be eliminated to a level of less than 100 mg/l, either by a pretreatment such as air-stripping or chemical precipitation prior to biological treatment or recirculation of the treated effluent in the biological treatment process.

MAP precipitation with MgCl₂·6H₂O + Na₂HPO₄·12H₂O

After adding MgCl₂·6H₂O and Na₂HPO₄·12H₂O into the raw leachate samples, white colour precipitant was rapidly settled as soon as stirring stopped. The results indicated that NH₄⁺-N was efficiently decreased from

5,618 to 210 mg/l with a $Mg^{2+}:NH_4^+:PO_4^{3-}$ mole ratio of 1:1:1. Overdosing either Mg^{2+} or PO_4^{3-} by 10% further lowered the remaining NH_4^+-N to 112 mg/l or 158 mg/l respectively and then any more overdosing of Mg^{2+} or PO_4^{3-} did not achieve additional NH_4^+-N removal, as shown in Figure 4.

[Figure 4]

From an engineering point of view, it may be practical to control the $Mg^{2+}:NH_4^+:PO_4^{3-}$ mole ratio at 1:1:1. Correspondingly, pH dropped from 8.86 to 6.7 during the precipitation. To determine the optimum pH of the precipitation, some supernatant liquid were separated from the settled sludge and further added with the 10M NaOH solution to raise pH to 10.5, as shown in Figure 5.

[Figure 5]

At the initial stage of the experiment, while NaOH solution was being added in the supernatant, pH increased gradually and remaining NH_4^+-N decreased sharply. When pH reached 8.64, the lowest NH_4^+-N concentration was found to be 65 mg/l. Further addition of NaOH solution resulted in an increase of remaining NH_4^+-N again. Therefore, the pH between 8.5 - 9.0 may be determined as the optimum range to achieve the minimum MAP solubility. This result was similar to the optimum pH of 8 to 10 found in treating other types of wastewaters (Zdybiewska and Kula 1991; Schule-Rettmer 1991). However, it should be concerned that high salinity in the treated leachate was found due to the formation of NaCl as a byproduct. According to Equation 2, an equivalent amount of 8.375 g NaCl would be generated to precipitate 1 g NH_4^+-N from leachate.

MAP precipitation with other chemicals

To minimise high salinity produced by the precipitation, other chemicals including $MgO + H_3PO_4$ and $Ca(H_2PO_4)_2 \cdot H_2O + MgSO_4 \cdot 7H_2O$ were employed with the mole ratio of 1:1:1. The experimental results shown in Figure 6 demonstrate the efficiencies of ammonium removal and the salinity of the treated leachate with different chemicals respectively. It can be indicated that the $MgCl_2 \cdot 6H_2O + Na_2HPO_4 \cdot 12H_2O$ was the most efficient chemicals to remove ammonium but generated the highest salinity (conductivity, TDS and FDS) in the treated leachate.

[Figure 6]

[Figure 7]

On the other hand, the experimental results indicate that using either $\text{MgO} + 85\% \text{H}_3\text{PO}_4$ or $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O} + \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ generated much less dissolved salts in the treated leachate, but their efficiency to remove ammonium were not as good as using $\text{MgCl}_2 \cdot 6\text{H}_2\text{O} + \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$.

CONCLUSIONS

The characterisation of the leachate from the local landfill site indicated that it belonged to the methanogenic site with a low BOD_5/COD ratio of 0.22 and high strength of ammonia-nitrogen around 5 g/l. The operation of biological reactors receiving both synthetic wastewater and mixed wastewater of glucose and leachate demonstrated that the high levels of ammonia-nitrogen could significantly inhibit the microbial activity of activated sludge, which were indicated by the declination of COD removal efficiency, dehydrogenase activity and SOUR. The experimental results imply that the high strength of ammonia-nitrogen in leachate needs to be eliminated to a level of below 100 mg/l, either by a pretreatment such as air-stripping or chemical precipitation prior to biological treatment or recirculation of the treated effluent in the biological treatment process.

Precipitation of MAP was technically effective to remove high strength of $\text{NH}_4^+\text{-N}$ over 5,000 mg/l from municipal landfill leachate. When $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ were employed at $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-} = 1:1:1$, a very satisfactory removal of $\text{NH}_4^+\text{-N}$ was achieved, in which $\text{NH}_4^+\text{-N}$ was quickly reduced from 5,618 to 112 mg/l within 15 minutes. pH to achieve the minimum MAP solubility was found to be between 8.5 and 9.0. High salinity was formed in the precipitation by using $\text{MgCl}_2 \cdot 6\text{H}_2\text{O} + \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$. $\text{MgO} + 85\% \text{H}_3\text{PO}_4$ and $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O} + \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ were conducive to generate lower salinity, but they also demonstrated a lower efficiency of $\text{NH}_4^+\text{-N}$ removal.

ACKNOWLEDGMENT

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Table 1. Operating conditions of activated sludge process in 6 reactors

ID of reactor	pH	COD in leachate (mg/l)	Glucose COD added (mg/l)	Initial MLSS (mg/l)	Initial NH ₄ ⁺ -N (mg/l)
#0 (Tap water+NH ₄ ⁺ -N+ glucose)	7.92	0	1000	2000	50
#1 (Diluted leachate+glucose)	8.13	65	1000	2000	50
#2 (Diluted leachate+glucose)	7.79	131	1000	2000	100
#3 (Diluted leachate+glucose)	8.51	262	1000	2000	200
#4 (Diluted leachate+glucose)	8.27	524	1000	2000	400
#5 (Diluted leachate+glucose)	8.52	1047	1000	2000	800

Table 2. Characteristics of leachate from the local landfill site in Hong Kong

Parameter	Value	Parameter	Value
<i>Physical</i>		Total PO ₄ ³⁻ -P	16.3 mg/l
Colour	7800 Hazen (Peaty Brown)	Alk.(as CaCO ₃)	13195 mg/l
Odour	Slightly ammoniacal	Chloride	3032 mg/l
pH	8.22	VFA	420 mg/l
Conductivity	37000 µS/cm	Potassium	3920 mg/l
Turbidity	4100 NTU	Sodium	2505 mg/l
<i>Chemical</i>		Calcium	13.7 mg/l
Total COD	7439 mg/l	Magnesium	93 mg/l
Soluble COD	6446 mg/l	Iron	3.811 mg/l
BOD ₅	1436 mg/l	Manganese	0.182 mg/l
TOC	2535 mg/l	Nickel	0.365 mg/l
TSS	784 mg/l	Copper	0.120 mg/l
VSS	654 mg/l	Zinc	1.155 mg/l
TDS	12352 mg/l	Chromium	0.553 mg/l
FDS	9420 mg/l	Cadmium	0.103 mg/l
Ammonium-N (as N)	5618 mg/l	Lead	<0.01 mg/l

Table 3. Summary of the operating conditions and experimental results in 6 reactors

Reactors	pH (Med.)	Init. Sol. COD ⁺ (mg/l)	Init. Biod. COD ⁺⁺ (mg/l)	Resid. Biod. COD ⁺⁺⁺ (mg/l)	Initial NH ₄ ⁺ -N (mg/l)	Resid. NH ₄ ⁺ -N (mg/l)	Deh. Activity (μg/mg)	Final MLSS (mg/l)	SOUR (mgO ₂ /g MLSS)
#0	7.92	1000	1000	43	50	0.19	10.46	2477	70
#1	8.13	1065	1023	28	50	0.30	9.29	2467	68
#2	7.79	1131	1046	33	100	46	8.80	2554	64
#3	8.51	1262	1091	59	200	143	8.71	2521	65
#4	8.27	1524	1182	177	400	353	7.12	2583	56
#5	8.52	2047	1364	299	800	718	4.93	2564	45

Note: COD^+ = Glucose COD + COD measured in leachate
 COD^{++} = Glucose COD + Biodegradable COD in leachate
 COD^{+++} = Biodegradable COD in effluent

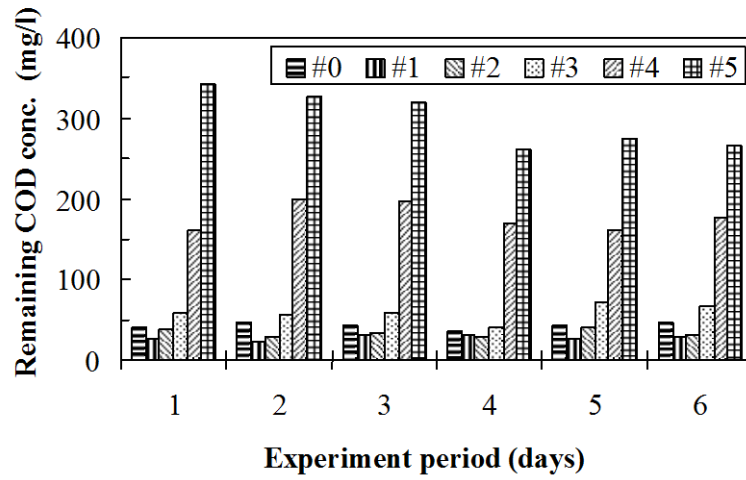


Figure 1. Remaining COD conc. at different experimental period

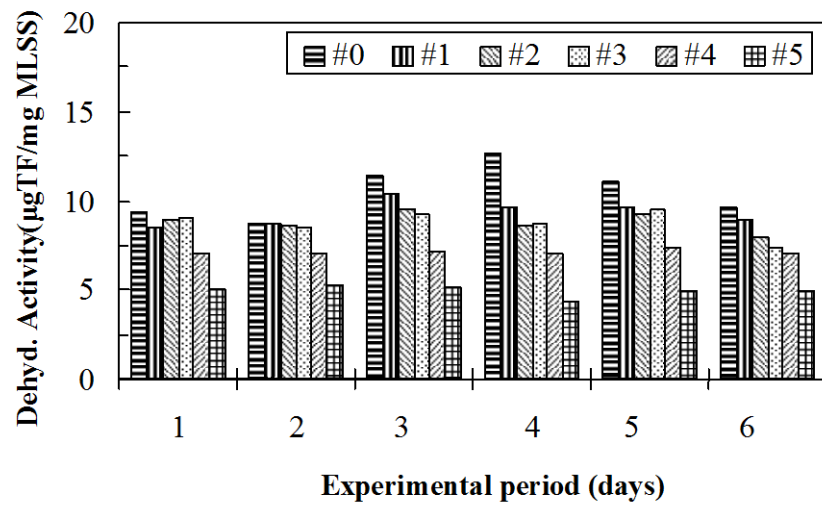


Figure 2. Dehydrogenase activity of microbe at different experimental period

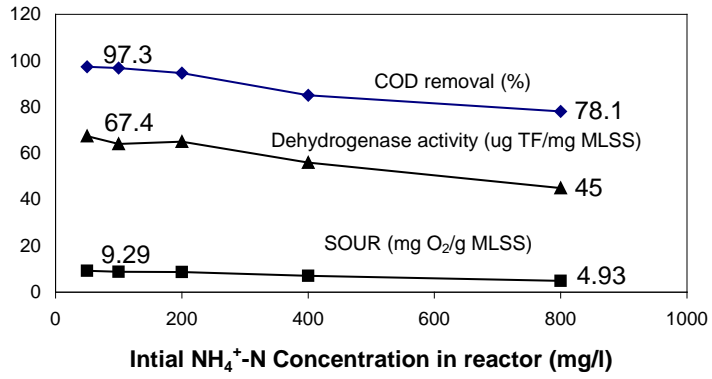


Figure 3. Variation of COD removal, Dehydrogenase activity and SOUR affected by $\text{NH}_4^+\text{-N}$ concentration

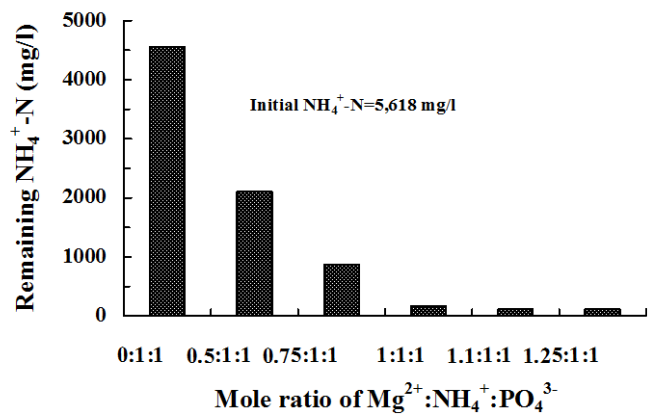
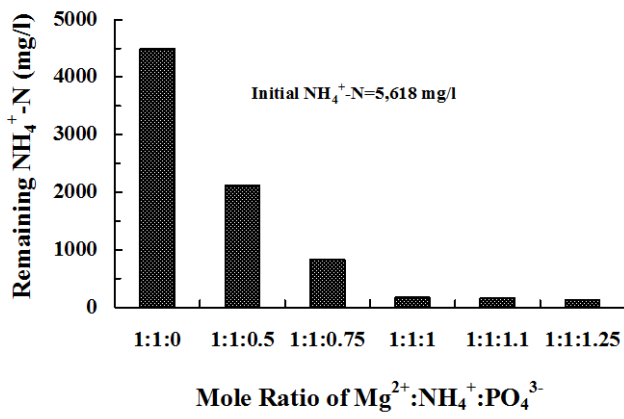


Figure 4. Determination of optimum mole ratio to precipitate $\text{NH}_4^+\text{-N}$ from the leachate

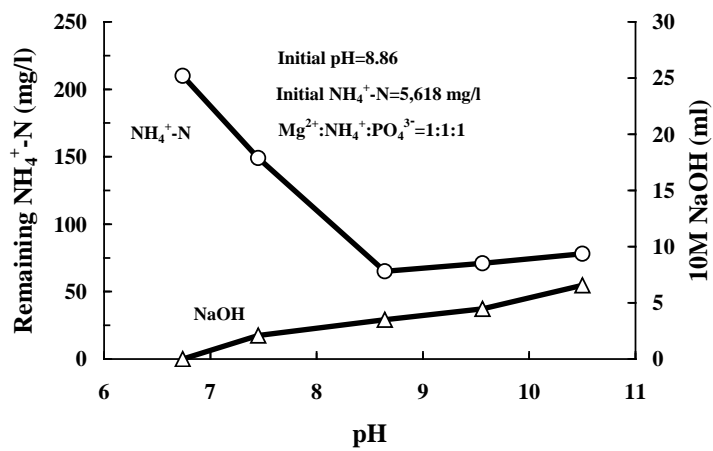


Figure 5. Further precipitation of MAP in supernatant against pH

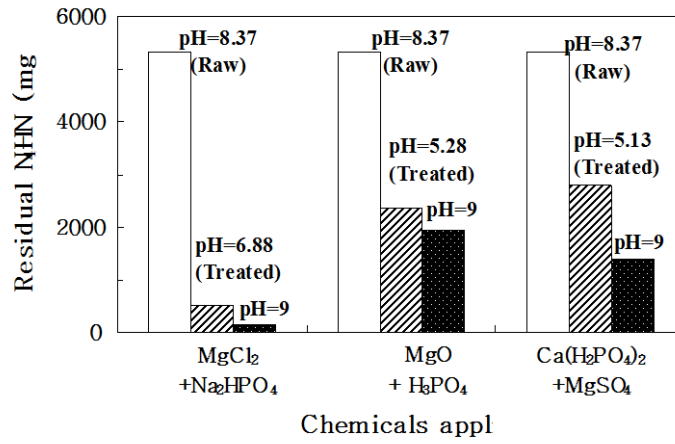


Figure 6. Efficiency of NH_4^+ -N removals using different chemicals

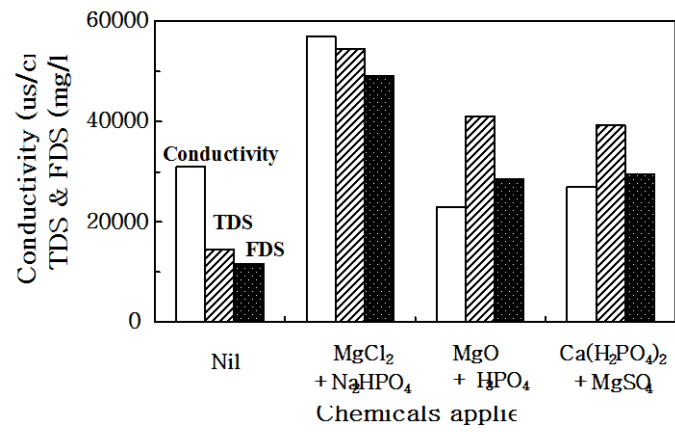


Figure 7. Salinity in treated leachate using different chemicals