

# Application of struvite precipitation in treating ammonium nitrogen from semiconductor wastewater

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## Abstract

Struvite precipitation was applied to the removal of  $\text{NH}_4\text{-N}$  in semiconductor wastewater. Batch experiments were conducted to examine the effects of final pH, magnesium and orthophosphate dosages and the initial influent concentrations of  $\text{NH}_4\text{-N}$  and F on the removals of  $\text{NH}_4\text{-N}$  and  $\text{PO}_4\text{-P}$  by forming struvite deposits. pH was an important parameter in the simultaneous removals of ammonium nitrogen and orthophosphate. In struvite precipitation, the amount of orthophosphate in the solution affected  $\text{NH}_4\text{-N}$  removal much more than that of magnesium ions in some cases. It was revealed that the low and high initial concentrations of  $\text{NH}_4\text{-N}$  and F inhibited  $\text{NH}_4\text{-N}$  and  $\text{PO}_4\text{-P}$  removal efficiencies in struvite precipitation, respectively. We also evaluated field-scale treatment plant incorporated by struvite precipitation process. On semiconductor wastewater with an  $\text{NH}_4\text{-N}$  concentration of 155 mg/L, the results obtained showed that the incorporation of the struvite precipitation process brought about a high  $\text{NH}_4\text{-N}$  removal efficiency of over 89% on average.

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**Keywords:** Struvite; Semiconductor wastewater; Fluoride; Ammonium nitrogen removal; Orthophosphate removal

## 1. Introduction

Generally, in a typical semiconductor manufacturing process, a significant amount of wastewater is generated because a large quantity of ultrapure water is consumed in the course of the chemical mechanical polishing (CMP) process which is generally used in the semiconductor industry for the purpose of planarizing the surface of the silicon wafer [1–4]. Semiconductor wastewater commonly contains many refractory chemicals such as organic solvents, acids, bases, salts, heavy metals, fine suspended oxide particles and other organic and inorganic compounds [1,5–9]. Especially, the high levels of ammonia, phosphate and fluoride are presented in semiconductor wastewater.

Among these pollutants, ammonia and phosphate are major concerns. Ammonia and phosphate are formed from ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) and phosphoric acid, which are used as additives in the CMP process [9–11]. Ammonia and phosphate

facilitate  $\text{O}_2$  depletion, eutrophication and red tidal phenomena in water courses [12]. In addition, ammonia has a harmful effect on the local ecology [13,14]. Therefore, they should be removed properly from wastewater before entering into aquatic systems.

However, it may be inadequate to biologically treat semiconductor wastewater containing high concentration of ammonia because semiconductor wastewater has various toxic substances which inhibit active nitrification by nitrifying bacteria [15,16]. To overcome this difficulty, the precipitation of  $\text{NH}_4\text{-N}$  and  $\text{PO}_4\text{-P}$  by forming magnesium ammonium phosphate (Struvite,  $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) would be attractive in semiconductor wastewater treatment. Struvite crystallizes as a white orthorhombic crystalline structure, which is composed of magnesium, ammonium, and phosphate in equal molar concentrations [17].

To the best of our knowledge, the feasibility of struvite precipitation in semiconductor wastewater has not yet been studied. Also, struvite formation in semiconductor wastewater must be studied in advance of any real application.

For this study, batch and field-scale experiments were conducted to determine the effectiveness of struvite precipitation in semiconductor wastewater. In the batch experiments, the evaluations were focused on the following parameters: (1) pH, (2)

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Table 1  
Characteristics of raw semiconductor wastewater

Parameter	Concentration range
Total chemical oxygen demand (TCOD) (mg/L)	221–444
Soluble chemical oxygen demand (SCOD) (mg/L)	67–136
Total Kjeldahl nitrogen (TKN) (mg/L)	106–171
Total phosphorus (T-P) (mg/L)	5–402
pH	2.4–4.3
Cations	
NH <sub>4</sub> -N (mg/L)	80–250
Mg (mg/L)	1.4–2.0
Ca (mg/L)	6.2–11.6
Na (mg/L)	6.0–40.1
K (mg/L)	9.0–49.0
Anions	
PO <sub>4</sub> -P (mg/L)	5–388
F (mg/L)	75–799
SO <sub>4</sub> (mg/L)	3.7–6.4
Cl (mg/L)	6.9–9.9
NO <sub>3</sub> -N (mg/L)	1.6–2.8

magnesium and orthophosphate dosage, (3) initial ammonium nitrogen concentrations, and (4) initial fluoride concentrations. Finally, a field-scale test was performed in a semiconductor wastewater treatment plant for a period of 176 days.

## 2. Materials and methods

### 2.1. Characteristics of semiconductor wastewater

The raw wastewater samples for both the batch and field-scale studies were obtained from a large semiconductor manufacturer in Cheongju, Korea. The composition of the semiconductor wastewater for the batch and field-scale experiments is shown in Table 1. Significantly high concentrations of ammonia, phosphate and fluoride were observed.

### 2.2. Experimental setup

#### 2.2.1. Batch test

For struvite precipitation, magnesium chloride (MgCl<sub>2</sub>·6H<sub>2</sub>O) was used with a concentration of 30 g Mg/L as the magnesium source. For the phosphate source, a potassium phosphate (K<sub>2</sub>HPO<sub>4</sub>) stock solution containing 30 g PO<sub>4</sub>-P/L was also prepared. Phosphate was only fed if orthophosphate in solution did not reach at a molar ratio of 1:1:1 for NH<sub>4</sub>-N:Mg:PO<sub>4</sub>-P in struvite formation. 5N NaOH was used for pH adjustment. In order to investigate the effects of fluoride on struvite crystallization, actual and synthetic wastewater were used. Synthetic fluoride solutions were prepared with concentrations of 350, 544, 592, 639, 683, 774, and 891 mg F/L, respectively, by adding hydrofluoric acid (HF) to deionized water. In the synthetic solution, the concentrations of NH<sub>4</sub>-N and PO<sub>4</sub>-P were 149.7 mg/L and 162.3 mg/L, respectively. All chemicals used were of analytical grade.

Experiments were carried out at ambient laboratory temperature (20 °C) and were conducted using a jar test apparatus. The paddle at the end of each stirrer shaft had a diameter of 7.6 cm and a height of 2.5 cm. Jars were made of acrylic plastic with dimensions of 11.5 cm × 11.5 cm × 25 cm and held 1.0 L of liquid. In all experiments, the struvite precipitation proceeded by the addition of magnesium and phosphate followed by pH adjustment to 9, as found in our previous study [18]. The mixing speed was 150 rpm and the mixing time was 2 min, followed by settling of 30 min. A syringe was used to withdraw sample from each jar at the end of the settling period. The samples were filtered through membrane filters (Gelman GN-6 with effective pore size of 0.45 μm) prior to analysis.

#### 2.2.2. Field-scale study

On-site monitoring of the struvite precipitation process was performed at a large semiconductor manufacturer in Cheongju, Korea. The semiconductor wastewater manufacturing facility discharged the wastewater with a quantity of 500 m<sup>3</sup>/d. The wastewater discharged from the semiconductor manufacturing facility entered an existing wastewater treatment plant for the purpose of removing fluorides and phosphates by precipitation using CaCO<sub>3</sub>, without any consideration of ammonium nitrogen removal (Fig. 1a).

The wastewater treatment plant was retrofitted by installing a struvite precipitation process, as shown in Fig. 1b. The discharged wastewater was initially pumped to the equalization basin. In chemical mixing tank, MgCl<sub>2</sub>·6H<sub>2</sub>O as an alternate source of magnesium ion was added to reach at 1:1:1 in the molar ratio of NH<sub>4</sub>-N:Mg:PO<sub>4</sub>-P for struvite formation and then, the pH adjustment of the wastewater to 9 was achieved by the continuous addition of 5N NaOH. An external phosphate source was not used for this experiment because the orthophosphate (PO<sub>4</sub>-P) concentration in the wastewater was already high enough to form struvite crystals. The liquid stream then moved to the struvite reaction tank where struvite was formed. The mixing speeds in both of the chemical mixing tank and the struvite reaction tank were 250 rpm. The solution was allowed to settle in the intermediate settler. The settled sludge was recycled to the chemical mixing tank at a rate of 250 m<sup>3</sup>/d in order to provide seeding material. It has been noted in previous works that the addition of pre-formed struvite as seeding material enhanced struvite precipitation in solutions [18,19]. The concentration of suspended solids (SS) in the recycle line was on average about 10000 mg/L. The effluent from the intermediate settler flowed into the fluoride removal tank. Fluoride was finally precipitated with CaF<sub>2</sub> by adding CaCO<sub>3</sub> to final settler. In this study, all experimental data were obtained from the struvite precipitation process. Table 2 summarizes the operational parameters of the retrofitted semiconductor wastewater treatment plant.

### 2.3. Analytical procedures

TCOD and SCOD (standard code: 5220 D), Total Kjeldahl nitrogen (standard code: 4500-N B), and PO<sub>4</sub>-P (standard code: 4500-P E) were analyzed by the Standard Methods [20]. NH<sub>4</sub>-N

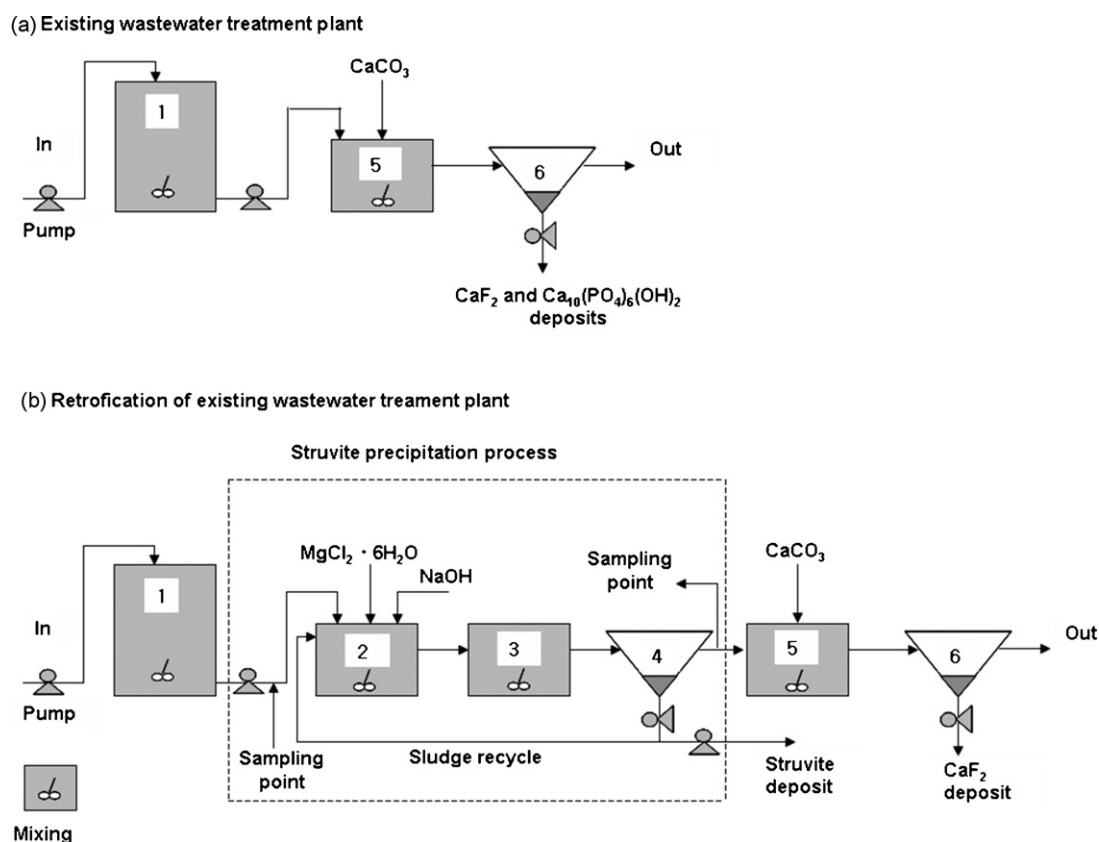


Fig. 1. Schematic diagrams of field plants before (a) and after (b) the installation of struvite precipitation process: (1) equalization basin; (2) chemical mixing tank; (3) struvite reaction tank; (4) intermediate settler; (5) fluoride removal tank; (6) final settler.

and T-P were determined using the HACH Nessler method and the HACH persulfate UV oxidation method, respectively. Mg, Ca, Na, K, F, SO<sub>4</sub>, and Cl were measured using a DX-100 ion chromatograph (Dionex, USA). All the columns used in this study were from Dionex. For the separation of anion an IonPac AG4A-SC guard column (50 mm × 4 mm) and an IonPac AG4A-SC (250 mm × 4 mm) analytical column were used. The eluent was Na<sub>2</sub>CO<sub>3</sub> (1.8 mM)/NaHCO<sub>3</sub> (1.7 mM) at a flow rate of 2 mL/min. For cationic separation, an IonPac CG12 (50 mm × 4 mm) guard column and an IonPac CS12 (250 mm × 4 mm) analytical column were utilized. Methane sulfonic acid (20 mM) was used as the eluent at flow rate of 1 mL/min. The dried deposits produced in the experiments were characterized by X-ray diffraction (XRD, Model DMS 2000 system, SCINTAG). All the samples were analyzed immediately after sampling.

Table 2  
Operational parameters of retorfitted semiconductor wastewater treatment plant

Parameter	Volume (m <sup>3</sup> )	Contact time (h)
Equalization basin	200	9.6
Chemical mixing tank	12	0.6
Struvite reaction tank	12	0.6
Intermediate settler	250	1.2

### 3. Results and discussion

#### 3.1. Batch test: effect of pH

Fig. 2a represents the removal characteristics of ammonium and orthophosphate as a function of pH. The best experimental ammonia nitrogen removal efficiency was observed to be 78% at pH 9.2 and its removal efficiency decreased over pH 9.4. The orthophosphate removal followed a similar trend as that of the ammonia and the minimum supernatant phosphate concentration was observed at pH 10.0. The optimum pH for the removal of ammonia observed in this experiment was in agreement with other studies. Booker et al. [21] reported that pH 9.2 was optimum, whereas Tünay et al. [22] found pH 8.5–9.3 to be the optimal range. Snoeyink and Jenkins [23] illustrated that struvite solubility generally decreases with increasing pH. However, over pH 9.0, struvite solubility begins to increase since the ammonium ion concentration will decrease and the phosphate ion concentration will increase. The fact that the optimum removal of orthophosphate occurred at a higher pH than that of ammonium was probably due to additional precipitation of phosphate ions as hydroxylapatite (Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>) at pH 10.0. It is well known that as the pH value of wastewater increases, calcium ions will then react with phosphate to precipitate hydroxylapatite in the metastable zone. This generally occurs between pH 9 and 10.5 [24–26]. In addition, precipitation

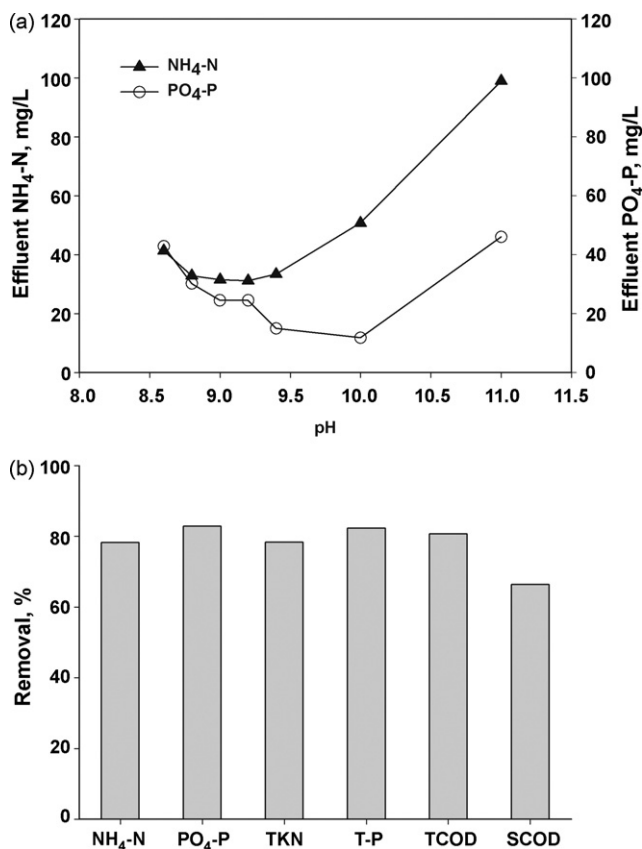


Fig. 2. Effluent concentrations of ammonia nitrogen and orthophosphate versus pH for struvite precipitation (a) and the removal efficiencies of NH<sub>4</sub>-N, PO<sub>4</sub>-P, TKN, T-P, TCOD, and SCOD at pH 9.2 (b): Initial NH<sub>4</sub>-N concentration was 143.5 mg/L; initial PO<sub>4</sub>-P concentration was 142.5 mg/L; initial TKN concentration was 157.4; initial T-P concentration was 160.0 mg/L; initial TCOD concentration was 370.3 mg/L; initial SCOD concentration was 113.2. Magnesium and orthophosphate dosage for struvite precipitation was 1.0:1.2:1.0 in the molar ratio of NH<sub>4</sub>-N:Mg:PO<sub>4</sub>-P.

of magnesium phosphate (Mg<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>) would be expected. Lee et al. [17] found that bittern addition to the standard solution (a mixture of NH<sub>4</sub>Cl and KH<sub>2</sub>PO<sub>4</sub>) led to precipitation of significant concentrations of struvite in the range of pH 7.5–10 in model prediction using MINTEQA2 in order to indicate the mechanism of removals of ammonium and orthophosphate. Magnesium phosphate was precipitated in the pH 8.5–11 range. The differing optimum pH levels for the removal of ammonium and phosphate were also reported by Booker et al. [21]. They reported that maximum ammonium removal was found at pH 9.2, whereas maximum phosphate removal was observed at pH 9.8.

Fig. 2b summarizes the removal efficiencies of NH<sub>4</sub>-N, PO<sub>4</sub>-P, TKN, T-P, TCOD, and SCOD at pH 9.2, the reported optimum pH for ammonia removal. Those were 78, 83, 78, 82, 81, and 66%, respectively. It is speculated that the high removal of organic carbon was due to an excessive dose of magnesium. As is well known, magnesium ions are used as flocculants to remove particulate organic matters in semiconductor wastewater. Considering that the semiconductor wastewater used in this experiment contains a high concentration of TCOD, it is important to note that an excessive magnesium dose would be necessary to achieve high removal of both ammonia and TCOD.

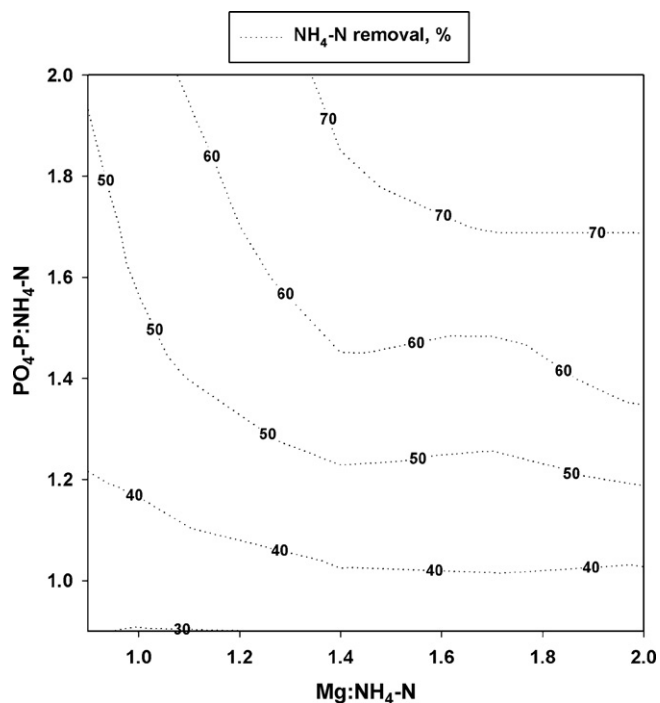


Fig. 3. Effect of magnesium and orthophosphate dosage on NH<sub>4</sub>-N removal: initial NH<sub>4</sub>-N concentration was 79.8 mg/L; initial PO<sub>4</sub>-P concentration was 4.8 mg/L. Final pH for struvite precipitation was 9.2.

### 3.2. Batch test: effect of magnesium and orthophosphate dosage

It has been noted in previous studies that ammonium and phosphate removal was generally affected by the amount of magnesium added to the struvite precipitation [17,27–29]. Especially, Stratful et al. [27] reported that magnesium ions are a limiting factor to struvite precipitation. However, our study showed that the amount of orthophosphate affected NH<sub>4</sub>-N removal much more than that of magnesium ions in some cases. Fig. 3 illustrates the effect of both the magnesium and phosphate dosages on NH<sub>4</sub>-N removal by adding alternate magnesium and orthophosphate sources to the wastewater. It was found that NH<sub>4</sub>-N removal was affected more by the amount of orthophosphate added than magnesium in some cases. In the NH<sub>4</sub>-N removal line of 70%, the NH<sub>4</sub>-N removal efficiency depended totally on the molar ratio of PO<sub>4</sub>-P:NH<sub>4</sub>-N when magnesium was injected above the molar ratio of 1.7:1.0 for Mg:NH<sub>4</sub>-N. In other words, no amount of magnesium could achieve an NH<sub>4</sub>-N removal of 70% if the PO<sub>4</sub>-P dosage was less than 1.69:1.0 in the molar ratio of PO<sub>4</sub>-P:NH<sub>4</sub>-N. Similar results were also observed in the NH<sub>4</sub>-N removal lines of 40%, 50%, and 60%, respectively. In the NH<sub>4</sub>-N removal lines of 40%, 50%, and 60%, the amount of PO<sub>4</sub>-P added heavily affected NH<sub>4</sub>-N removal efficiencies if magnesium ions were added above 1.4:1.0 in the molar ratio of Mg:NH<sub>4</sub>-N. This means that even with the addition of more magnesium ions, the removal efficiency of NH<sub>4</sub>-N cannot be enhanced if PO<sub>4</sub>-P dosages are below the molar ratios of 1.03:1.0, 1.23:1.0 and 1.43:1.0 for PO<sub>4</sub>-P:NH<sub>4</sub>-N in each removal line, respectively.

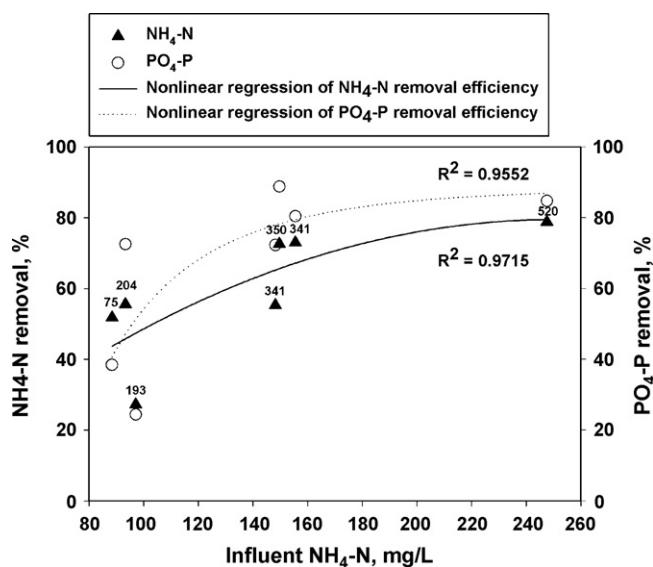


Fig. 4. Effect of initial NH<sub>4</sub>-N concentration on removals of NH<sub>4</sub>-N and PO<sub>4</sub>-P: magnesium and orthophosphate dosage for struvite precipitation was 1.0:1.2:1.0 in the molar ratio of NH<sub>4</sub>-N:Mg:PO<sub>4</sub>-P. Final pH for struvite precipitation was 9.2. Figures above symbols are initial F in solution.

It must be noted in Fig. 3 that the ammonium nitrogen removal efficiency of 37% was very low when compared to the 78% results shown in Fig. 2b when the magnesium and orthophosphate dosages for struvite precipitation was 1.0:1.2:1.0 in the molar ratio of NH<sub>4</sub>-N:Mg:PO<sub>4</sub>-P. The difference between these two results is assumed to be due to the applied initial ammonium nitrogen concentration. The details of this are given in the Section 3.3.

### 3.3. Batch test: effect of influent ammonia nitrogen concentration

According to the experimental data shown in Fig. 4, it was found that ammonium and orthophosphate removal relied heavily on the initial ammonium nitrogen concentration. At low initial NH<sub>4</sub>-N concentrations in semiconductor wastewater, NH<sub>4</sub>-N and PO<sub>4</sub>-P removal was relatively low. However, the opposite was found for high initial NH<sub>4</sub>-N concentrations. Struvite crystals seem to show limited growth at low ammonia nitrogen concentrations in the wastewater because the transportation of ammonium ions to the crystal surface is restricted due to the scarcity of ammonium ions in solution [30]. It is speculated based on the result obtained from Fig. 3 that at low initial NH<sub>4</sub>-N concentrations, an excess of magnesium and orthophosphate relative to NH<sub>4</sub>-N would be necessary to enhance the removal of ammonium and orthophosphate through struvite precipitation.

### 3.4. Batch test: effect of influent fluoride concentration

The effect of fluoride concentrations on the percentage removal of the ammonium and orthophosphate of actual semiconductor wastewater is shown in Fig. 5. The experimental results obtained from actual wastewater were compared to the

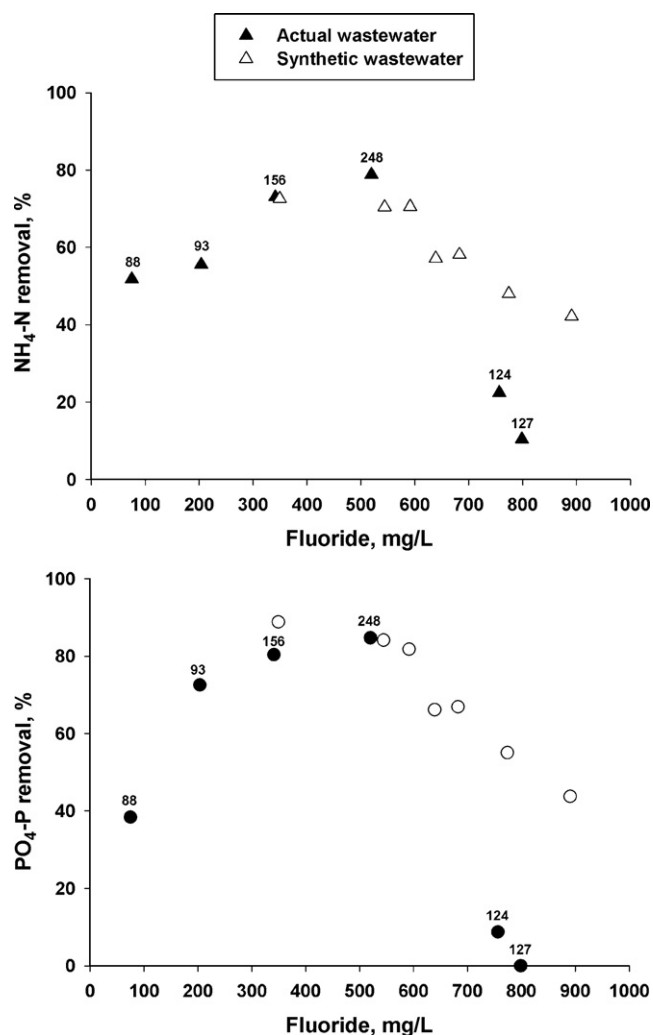


Fig. 5. Effect of initial fluoride concentration on removals of NH<sub>4</sub>-N and PO<sub>4</sub>-P: magnesium and orthophosphate dosage for struvite precipitation was 1.0:1.2:1.0 in the molar ratio of NH<sub>4</sub>-N:Mg:PO<sub>4</sub>-P. Final pH for struvite precipitation was 9.2. Figures above symbols are initial NH<sub>4</sub>-N in solution.

synthetic fluoride solutions. In Fig. 5, it was clearly observed that high fluoride concentrations inhibited the removal of NH<sub>4</sub>-N and PO<sub>4</sub>-P by forming struvite. In the actual semiconductor wastewater, NH<sub>4</sub>-N and PO<sub>4</sub>-P removal efficiencies drastically decreased when there was a fluoride concentration of over 700 mg/L. For 75 and 204 mg/L of initial fluoride concentrations, low NH<sub>4</sub>-N removal was also observed. This effect is assumed to be due to low initial NH<sub>4</sub>-N concentrations, as mentioned in the previous section. In the synthetic solutions, the inhibition of struvite precipitation was also observed at high fluoride concentrations. When the initial fluoride concentration was less than about 592 mg/L, the NH<sub>4</sub>-N and PO<sub>4</sub>-P removal efficiencies were over 70% and 80%. However, as initial fluoride concentration increased to over 600 mg/L, the NH<sub>4</sub>-N and PO<sub>4</sub>-P removal efficiencies gradually decreased. It is speculated that fluoride inhibits struvite crystallization at high concentrations. Fluoride may react with magnesium, forming magnesium fluoride. The probability of such a reaction increases as the fluoride concentrations in wastewater increase. Therefore, it is



suggested that wastewater be treated for fluoride prior to struvite precipitation to enhance the removal performance of ammonium and orthophosphate by struvite precipitation. If this is not possible, an excessive dose of magnesium would be necessary to increase the ammonia removal performance.

### 3.5. Field-scale study: ammonium nitrogen removal performance

The struvite precipitation process for ammonium nitrogen removal was applied to a large semiconductor manufacturer in Cheongju, Korea. Raw semiconductor wastewater with an influent ammonium nitrogen concentration of 154 mg/L on average was continuously introduced to the retrofitted treatment plant suggested in Fig. 1b. An external orthophosphate source was not used because, in most cases, there was already an excessive proportion of orthophosphate present in solution for reaction with  $\text{NH}_4\text{-N}$ . During the 176 day operational period, the effluent ammonium nitrogen concentration was about 17 mg/L and its removal efficiency was 89% on average with the standard deviation of 6% as shown in Fig. 6. In this experiment, orthophosphate removal performance was not evaluated because the residual orthophosphate not treated in struvite precipitation can be later removed in the fluoride removal process shown in Fig. 1b.

The struvite deposits were sampled and analyzed by the X-ray diffractograms shown in Fig. 7. The X-ray diffractograms exhibited several peaks indicative of the presence of struvite.

### 3.6. Field-scale study: basic economic analysis for the operation of the struvite process

The chemical cost effectiveness of the retrofitted wastewater treatment plant (Fig. 1b), in which the struvite precipitation process was incorporated, was compared to the previous set-up of semiconductor wastewater treatment plant (Fig. 1a). The chemical costs were calculated by analyzing the inflow rates and the per unit price of chemicals. Depending on these factors, the cost of

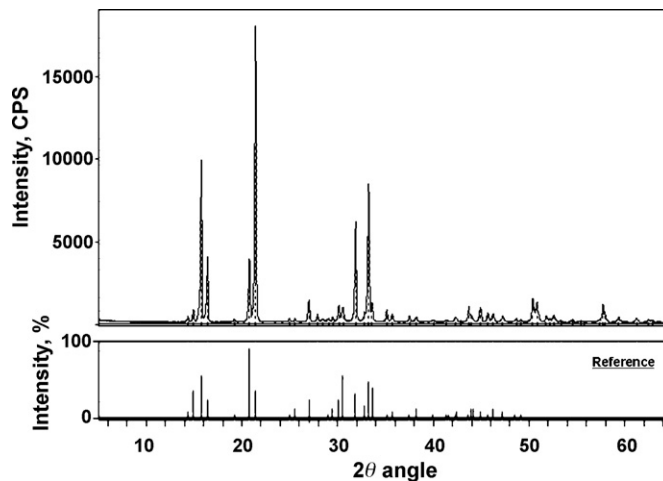


Fig. 7. XRD diffractograms of the precipitated matters.

calcium was calculated to be 186 US\$/d for the treatment of fluoride in the operation of the semiconductor wastewater treatment plant prior to being retrofitted. For the retrofitted wastewater treatment plant, the costs (996 US\$/d) of magnesium and NaOH were added due to the treatment of ammonium and orthophosphate by struvite precipitation. If struvite product can be sold at a favorable price, it may become economically viable to operate retrofitted semiconductor wastewater plant for the removal of ammonium and orthophosphate.

## 4. Conclusions

Struvite precipitation was applied to the removals of ammonia and phosphate in semiconductor wastewater. Batch tests were performed to investigate the effects of operational parameters on struvite precipitation. Furthermore, the effectiveness of struvite precipitation for nitrogen removal was evaluated in a field-scale plant. Based on the results of experimental tests, the following conclusions can be drawn:

- (1) pH was an important parameter in the simultaneous removals of ammonium nitrogen and orthophosphate. Optimum reaction for ammonium nitrogen removal was clearly observed at pH 9.2.
- (2) Excess dosages of magnesium and orthophosphate source were highly beneficial to struvite formation. In some cases, the concentration of orthophosphate in the solution affected  $\text{NH}_4\text{-N}$  removal much more than that of magnesium ions.
- (3) Ammonium and orthophosphate removal heavily depended on initial ammonium nitrogen concentration.
- (4) High fluoride concentrations inhibited the removal of  $\text{NH}_4\text{-N}$  and  $\text{PO}_4\text{-P}$  by forming struvite crystals. As fluoride concentrations increased to over 600 mg/L,  $\text{NH}_4\text{-N}$  and  $\text{PO}_4\text{-P}$  removal efficiencies gradually decreased.
- (5) In the field-scale study, the ammonium nitrogen removal efficiency was 89% on average during the 176 day operational period. Consequently, struvite precipitation was very effective for the removal of ammonium nitrogen in semiconductor wastewater.

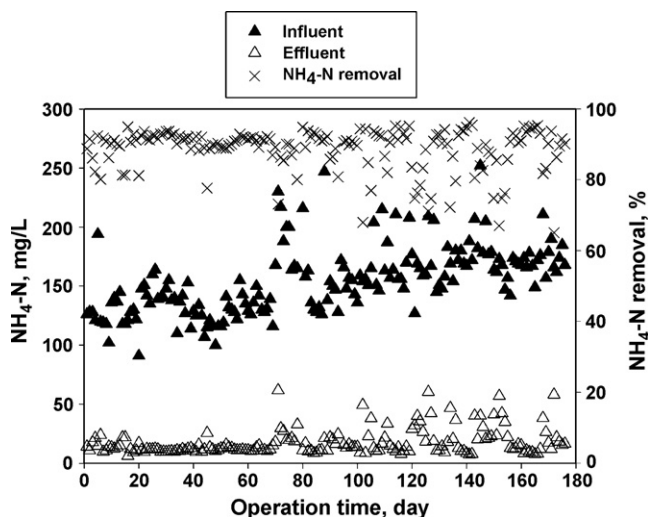


Fig. 6. Field-scale study of the semiconductor wastewater treatment by incorporating struvite precipitation process.

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