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# Removal of ammonia nitrogen from washing wastewater resulting from the process of rare-earth elements precipitation by the formation of struvite

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## ABSTRACT

This paper presents a study of the removal of ammonia nitrogen ($NH_3$-N) from washing wastewater resulting from the process of rare-earth elements precipitation using struvite precipitation. Experiments were conducted in batches to examine the effect of an excess of magnesium and phosphate on the removal of $NH_3$-N, and to investigate the feasibility of the reuse of the produced struvite for $NH_3$-N removal. Experimental results showed that any separate excess of magnesium and phosphate did not provide significant increase in $NH_3$-N removal. However, a simultaneous excess of magnesium and phosphate can provide an obvious increase in $NH_3$-N removal, with the remaining $NH_3$-N as a minimum of 8.7 mg/L at the Mg:N:P molar ratio of 1.2:1:1.2. A stable $NH_3$-N concentration of effluents (around 15 mg/L) was maintained through recycling struvite for six cycles. (In each cycle, fresh magnesium and phosphate was supplemented for keeping a steady Mg:N:P molar ratio (1.2:1:1.2) and the decomposition residue of struvite was dissolved by acid.) X-ray diffraction (XRD) analysis showed that struvite was the main composition of the precipitates obtained in the sixth reuse cycle. Scanning electron microscopy with energy dispersive X-ray (SEM-EDX) analysis indicated that the struvite crystal was fragmented and its size was irregular, and its surface composition was mainly P, Mg, O. An economic estimation indicated that reusing struvite for six cycles could save 30.8% and 60.9% treatment cost under the conditions of consideration and non-consideration of the struvite value, respectively.

***Keywords:*** Ammonia nitrogen; Rare-earth; Recycling struvite; Washing wastewater

## 1. Introduction

Rare-earth elements are widely applied in many fields, such as electronics, steel, nonferrous metals, petrochemicals, biological medicines, and environmental protection industries [1–4]. Generally, separation of rare-earth elements requires many processes, such as extraction, precipitation, and calcination. In a typical precipitating and separating process for rare-earth elements, a significant amount of ammonium carbonate is used to precipitate the rare-earth elements, resulting in the generation of a large quantity of wastewater containing ammonia nitrogen ($NH_3$-N). High concentration of $NH_3$-N in mother liquor from the precipitating process (>10,000 mg/L) could be removed by evaporation and crystallization [5], but the wastewater from washing the precipitates (WWP) is not suitable for treatment using this process because its

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$NH_3$-N concentration is relatively low (<2,000 mg/L). WWP commonly contains high levels of $NH_3$-N, less organic compounds, and small amounts of heavy metals [5].

Among these pollutants, $NH_3$-N is of major concern. When ammonium is present in receiving waters, it can cause eutrophication of lakes and rivers, resulting in excessive growth of algae and other microorganisms, dissolved oxygen depletion, and toxicity for the fish population [6,7]. Therefore, it is necessary to remove these substances from wastewater to prevent environmental pollution.

Conventional biological processes have been widely utilized for low-cost removal of $NH_3$-N from wastewater [8]. However, these processes might not be very appropriate for treating the WWP because of the lack of organic compounds for electron donors. Struvite precipitation is a promising method of ammonium removal and would prove attractive in solving the problem of WWP treatment. Struvite – magnesium ammonium phosphate hexahydrate ($MgNH_4PO_4{\cdot}6H_2O$, MAP) – is a white crystal with a solubility as low as 2.3 mg per 100 ml $H_2O$ [9] and is composed of magnesium, ammonium, and phosphorus in equal molar concentrations [10]. Its solubility reduces with increasing pH, reaching the minimum at pH 9 and then increases as pH continues to rise above pH of 9 [11]. Because of its high reaction rate and low residual $NH_3$-N concentration after reaction, struvite precipitation is nowadays widely used for the removal of $NH_3$-N from landfill leachate [12,13], swine wastewater [14,15], and industrial wastewater [16,17].

In this study, batch experiments were conducted for two purposes: one of the purposes is that the remaining $NH_3$-N in effluents is required to be less than the level of $NH_3$-N discharging ($\leq$15 mg/L) due to the fact that conventional biological processes cannot be used for the advanced treatment of the effluents of the WWP. For this purpose, the effect of an excess of magnesium and phosphate on the $NH_3$-N removal from the WWP was investigated. The other purpose is that the treatment cost of the WWP is required to be lowered for the practical application. Therefore, the reuse of regenerated magnesium and phosphate by thermal decomposition of struvite in alkali solution was investigated. In addition, the economic feasibility of using the process was analyzed.

## 2. Experimental materials and methods

### 2.1. Materials

Samples of WWP (the wastewater from the first washing) were obtained from a rare-earth separation plant located in Guangdong, China. The characteristics of the samples are summarized in Table 1. Solid magnesium chloride ($MgCl_2{\cdot}6H_2O$) and di-sodium hydrogen phosphate ($Na_2HPO_4{\cdot}12H_2O$) were used as the magnesium and phosphate sources, respectively. The pH was controlled by the addition of sodium hydroxide (5 M NaOH) or hydrochloric acid (5 M HCl). $H_3PO_4$ (85%) was used as an alternative supplement for phosphate in the reuse of regenerated magnesium and phosphate. All the chemicals used in the experiments were of analytical grade.

Table 1
Characterization of the washing wastewater resulting from rare-earth elements precipitation process

| Parameter | Parameter value |
|---|---|
| pH | 6.7 ± 0.01 |
| TOC (mg/L) | 3.4 ± 0.9 |
| $NH_3$-N (mg/L) | 1556 ± 28 |
| TP (mg/L) | 0.3 ± 0.1 |
| Zn (mg/L) | 5.4 ± 0.9 |
| Ca (mg/L) | 0.1 ± 0.05 |
| Mg (mg/L) | 0.3 ± 0.1 |
| Cu (mg/L) | 0.2 ± 0.2 |
| Ni (mg/L) | 1.2 ± 0.3 |

### 2.2. Experimental methods

#### 2.2.1. Effect of an excess of magnesium and phosphate on struvite precipitation performance

The struvite precipitation tests were carried out in a 1,000-ml beaker placed on a magnetic stirrer at a stirring rate of 500 rpm and ambient laboratory temperature (6–10°C). In all the experiments, $Na_2HPO_4{\cdot}12H_2O$ was first added to the wastewater sample of 500 ml. After $Na_2HPO_4{\cdot}12H_2O$ was dissolved, $MgCl_2{\cdot}6H_2O$ was added to the solution. The mixture was stirred for 30 min to enable mixing and reaction. During this period, solution pH was always maintained at a value of 9 (± 0.02) by using 5 M NaOH, as reported in other published literatures [12,18]. When the 30 min reaction time had elapsed, the solution was allowed to be settled for 30 min. Supernatants of 50 ml were removed and filtered using 0.45-μm membrane filters for component analyses.

To determine the effect of a separate excess of phosphate (P) on struvite precipitation, P:N:Mg molar ratios of 1:1:1, 1.1:1:1, 1.2:1:1, 1.3:1:1, 1.4:1:1, and 1.5:1:1 were tested. Similarly, to determine the effect of a separate excess of magnesium (Mg) on struvite precipitation, Mg:N:P molar ratios of 1:1:1, 1.1:1:1, 1.2:1:1, 1.3:1:1, 1.4:1:1, and 1.5:1:1 were also tested. Furthermore, to study the effect of a simultaneous excess of magnesium and phosphate on $NH_3$-N removal, Mg:N:P molar ratios of 1.1:1:1, 1.1:1:1.1, 1.2:1:1.1, 1.2:1:1.2, and 1.3:1:1.3 were adopted.

### *2.2.2. Reuse of struvite decomposition residues*

The struvite intended for reuse were generated by adding $MgCl_2{\cdot}6H_2O$ and $Na_2HPO_4{\cdot}12H_2O$ to the WWP of 500 ml according to the optimum conditions obtained from Section 2.2.1. The struvite generated were collected using a 0.45-μm membrane filter and washed three times using ultra-pure water, and then were decomposed in alkali solution under the following conditions: temperature, 90°C; $NH_4^+$:$OH^-$ ratio, 1:1; and heating duration, 4 h (with possible loss of ammonia exceeding 96%) [9]; the ammonia released was absorbed by HCl solution. Because a portion of the added magnesium and phosphate remained with the supernatant after reaction, before each reuse of the regenerated magnesium and phosphate (struvite residues after decomposition), the residues were supplemented with fresh $H_3PO_4$ and $MgCl_2{\cdot}6H_2O$ reagents to maintain the optimum Mg:N:P molar ratio of the reaction, and 5 M HCl of 10 ml was added to dissolve the decomposed solid residue, and then this mixture was stirred for 10 min. The resulting material was added to a subsequent batch reaction. This process was repeated six times. After each reuse experiment, supernatants of 50 ml were filtered with a 0.45-μm membrane filter and analyzed for the components ($NH_4^+$, $Mg^{2+}$ and $PO_4^{3-}$).

All the experiments were performed in triplicate.

### *2.3. Analytical methods*

The wastewater samples were analyzed according to Standard Methods [19]. Ammonia nitrogen and orthophosphate were measured colorimetrically using a spectrophotometer (752N, China). The pH of the samples was measured with a pH meter (pHs-3D, China). The precipitates collected were washed three times with ultra-pure water and dried in an oven at 30°C for 48 h; the dried precipitates were analyzed using scanning electron microscopy with energy dispersive X-ray (SEM-EDX) (FEI Quanta 400, America), X-ray diffraction (Philips Model PW1830).

## 3. Results and discussion

### *3.1. Effect of an excess of magnesium and phosphate on struvite precipitation performance*

The experimental results of the effect of an excess of magnesium and phosphate are shown in Figs. 1(a)–(c) and Table 2. From Fig. 1a and Table 2, it is observed that as phosphate dose increased, the concentration of the remaining $NH_3$-N in solution decreased from 138.6 mg/L at P:N:Mg molar ratio of 1:1:1 to 73 mg/L at P:N:Mg molar ratio of 1.1:1:1 and remained stable at 80 mg/L within a P:N:Mg range of 1.2:1:1–1.5:1:1. However, any separate excess dose application for P resulted in a significant amount of remaining $PO_4$-P in solution, which is not economical for additional $NH_3$-N removal. In separate consideration of P, the optimum P:N:Mg molar ratio for $NH_3$-N removal was 1.1:1:1. Herein, it is necessary to note that at pH 9, the volatilization of ammonia might contribute to the removal of $NH_3$-N. Nevertheless, in this study, the contribution rate was less than 1% because of the low remaining $NH_3$-N concentration and the low laboratory temperature.

In Fig. 1b and Table 2, it is found that with the increase of Mg:N:P molar ratio, the remaining $NH_3$-N concentration in solution decreased from 138.6 mg/L at Mg:N:P ratio of 1:1:1 to 124.5 mg/L at Mg:N:P ratio of 1.1:1:1, and then increased slowly from 124.5–189 mg/L at Mg:N:P range of 1.1:1:1–1.5:1:1. Since amorphous magnesium phosphate ($Mg_3\ (PO_4)_2$) may form in a alkaline media of a pH range of 8.5–11 [20,21] and its amount formed could increase with the increase of the dose of magnesium [10,22,23], this causes the reduction of the $PO_4$-P available for struvite crystallization. Hence, this may be a reason for the fact that remaining $NH_3$-N increased in Mg:N:P range of 1.1:1:1–1.5:1:1. Nevertheless, the remaining $PO_4$-P rapidly decreased from 93.4 to 4.1 mg/L with the increase of Mg:N:P molar ratio in the range of 1.1:1:1 –1.5:1:1:1. In separate consideration of Mg, the optimum Mg:N:P molar ratio for $NH_3$-N removal was 1.1:1:1.

Zhang et al. [24] reported that $NH_3$-N in landfill leachate could be treated by struvite precipitation and the optimum molar ratio of P:N:Mg was 1.1:1:1 (in separate consideration of P) and the optimum molar ratio of Mg:N:P was 1.15:1:1 (in separate consideration of Mg). On comparing the results in Figs. 1a and 1b, it is seen that $NH_3$-N removal was affected much more by the amount of phosphate added than magnesium. This concurs with the results reported by Ryu et al. [21].

In order to decrease the remaining $NH_3$-N in solution, further work was undertaken to evaluate the impact of simultaneous excess of magnesium and phosphate on struvite precipitation performance. The results shown in Fig. 1c demonstrate that the remaining $NH_3$-N in solution decreased with simultaneous increase in doses of Mg and P reagents. With Mg:N:P present at a molar ratio of 1.2:1:1.2, the remaining $NH_3$-N concentration at the end of the reaction reached

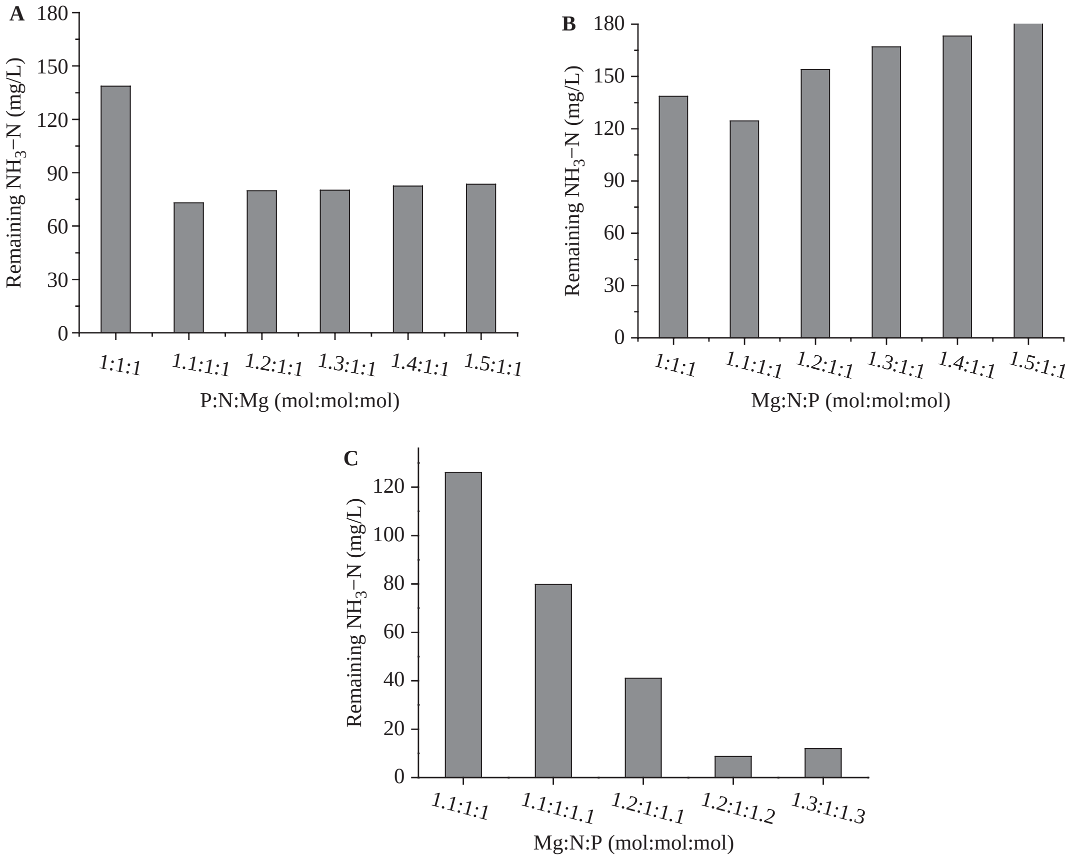

Fig. 1. Effects of the doses of Mg and P on $NH_3$-N removal: (a) a separate excess of P, (b) a separate excess of Mg, and (c) a simultaneous excess of Mg and P.

the lowest concentration of 8.7 mg/L, and the remaining $PO_4$-P was 119.5 mg/L. In view of the efficiency of $NH_3$-N removal, the optimum Mg:N:P molar ratio for $NH_3$-N removal was 1.2:1:1.2. Huang et al. [25] reported that the optimum Mg:N:P molar ratio for $NH_3$-N removal from saponification wastewater by struvite precipitation was 1.2:1:1.2. Since simultaneous overdose of magnesium and phosphate was not economical for $NH_3$-N removal, in order to lower the treatment cost of the WWP, subsequent experiments focused on the reuse of struvite.

### *3.2. Reuse of struvite decomposition residues*

The objective of this work was to study low-cost removal of $NH_3$-N from the WWP, using recycled magnesium and phosphate sources. Some authors had reused the residues of struvite decomposed under alkali condition as magnesium and phosphate sources. He et al. [9] found that struvite decomposed by heating under alkali conditions could form amorphous magnesium sodium phosphate ($MgNaPO_4$), $Na^+$ in which might be less stable than $NH_4^+$. Therefore, $Na^+$ in $MgNaPO_4$ could be replaced by $NH_4^+$ to form more stable struvite, which makes it possible to reuse struvite decomposition residues for $NH_3$-N removal. However, with the increase of reuse cycles, a portion of the recycled magnesium and phosphate would remain with the effluents, and the recycled magnesium and phosphate becomes more inactive because of the increase of $Mg_3(PO_4)_2$ and $Mg_4P_2O_7$ [26], which have a low capability for $NH_3$-N removal [27], resulting in progressive decrease in removal efficiency. Hence, in order to obtain stability in $NH_3$-N removal in the reuse

Table 2
Residual $PO_4$-P in solution at different Mg:N:P and P:N:Mg molar ratios

| Molar ratio | Residual $PO_4$-P (P:N:Mg) (mg/L) | Residual $PO_4$-P (Mg:N:P) (mg/L) |
|---|---|---|
| 1:1:1 | 93.4 | 93.4 |
| 1.1:1:1 | 182.9 | 30.3 |
| 1.2:1:1 | 484.9 | 6 |
| 1.3:1:1 | 781 | 4.7 |
| 1.4:1:1 | 1003 | 4.3 |
| 1.5:1:1 | 1328 | 4.1 |

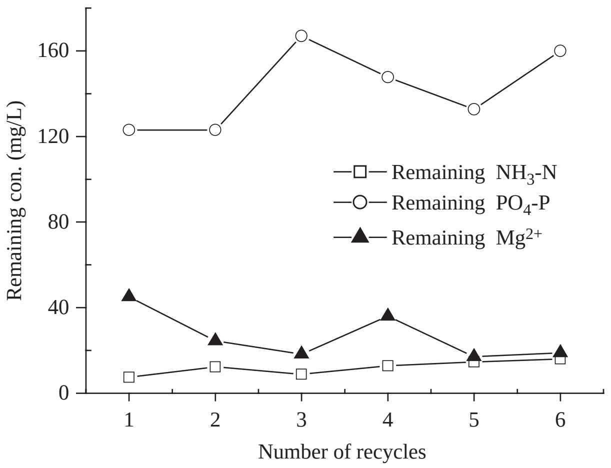

Fig. 2. Plot showing remaining NH$_3$-N in different reuse cycles (in each cycle, Mg:N:P = 1.2:1:1.2, pH = 9).

cycles, it is necessary to enhance solubility of $Mg_3(PO_4)_2$ and $Mg_4P_2O_7$ by adding acid (because the removal of ammonium with $Mg_3(PO_4)_2$ and $Mg_4P_2O_7$ proceeds through the dissolution–precipitation mechanism [27]), and maintain an optimum Mg:N:P molar ratio by supplementing with fresh magnesium and phosphate sources.

On the basis of the results obtained from the above experiments, the struvite precipitate used for reuse was generated by adding Mg and P reagents at a Mg:N:P molar ratio of 1.2:1:1.2. The Mg:N:P molar ratio at optimum level of 1.2:1:1.2 (± 2%) was always maintained in all reuse cycles. The experimental results are shown in Fig. 2, which suggest that the reuse of struvite decomposition residues, with the supplementation of fresh Mg and P reagents and the dissolution of the decomposition residues using HCl could achieve a stable removal of NH$_3$-N. The remaining PO$_4$-P and $Mg^{2+}$ concentrations varied within the ranges of 120–170 and 15–45 mg/L, respectively, in the reuse cycles. The remaining NH$_3$-N concentration increased slightly with the increase of reuse cycles. The accumulation of some metal ions in the wastewater samples might be responsible for this phenomenon; however, since the contents of the metal ions such as $Zn^{2+}$, $Cu^{2+}$, and $Ni^{2+}$ in WWP were low, its effect on NH$_3$-N removal was limited compared to that of landfill leachate in the study of He et al. [9]. The remaining NH$_3$-N was 16 mg/L in the sixth cycle, whereas the values for the first to the fifth cycles were lower than 15 mg/L, achieving the national standards specified for wastewater discharge of NH$_3$-N in China. The precipitate obtained in the sixth cycle was analyzed by SEM-EDX and XRD (Figs. 3a, 3b and 3c). The SEM picture shown in Fig. 3a shows that the precipitate is fragmentized and its size is irregular (10–30 μm). The EDX spectrum in Fig. 3b shows that the surface composition of the precipitate mainly contains Mg, P, and O and traces of C, Na, Si, and Cl. The X-ray diffractogram shown in Fig. 3c indicates that the characteristic peaks of the precipitate is close to that of the pattern for the struvite standard. The main composition of the precipitates was struvite.

### *3.3. Economic analysis of reusing struvite decomposition residues*

Economic analysis of reusing struvite decomposition residues as magnesium and phosphate sources was carried out. Labor costs and maintenance costs were not taken into account. The 2008 market prices of various materials used in the experiments are given in Table 3, and the calculated results are shown in Fig. 4 according to the data in Table 3.

Fig. 4 indicates that the costs for removing 1 kg NH$_3$-N decreases gradually with increase in the number of recycles. The average treatment costs, with and without the consideration of the market value of struvite, are \$3.9 and \$8.7/kg NH$_3$-N, respectively, if recovered struvite are not reused; however, the average treatment costs after reusing the struvite decomposition residues for six cycles decreases to \$2.7 and \$3.4/kg NH$_3$-N, respectively, a saving of 30.8% and 60.9%, respectively. At present, the market for struvite is still not well-established. Furthermore, it was found that the struvite formed contained higher zinc content than the requirement of Chinese Soil Quality Criterion (100 mg/kg; GB 15618-1995). This means that the struvite obtained may not be appropriate for use in agriculture. Therefore, reusing struvite is economical for NH$_3$-N removal from WWP by struvite precipitation.

He et al. [9] recycled struvite decomposition residues to remove the NH$_3$-N in landfill leachate for three cycles, saving about 48% chemical costs, but the NH$_3$-N removal efficiency, which was less than 60% in the sixth cycle, declined gradually with the increase in reuse cycles. Türker and Çelen [26] reused struvite decomposition residues for five cycles, the NH$_3$-N removal efficiency progressively declined from the initial 92% to 77% in the fifth cycle; whereas when the struvite residues were reused six cycles by the process presented, the NH$_3$-N removal efficiency almost remained a stable value. As a result, removing NH$_3$-N from the WWP using this process appears attractive.

## 4. Conclusions

Experiments with struvite precipitation were conducted for the purpose of NH$_3$-N removal from the WWP. Some conclusions are:

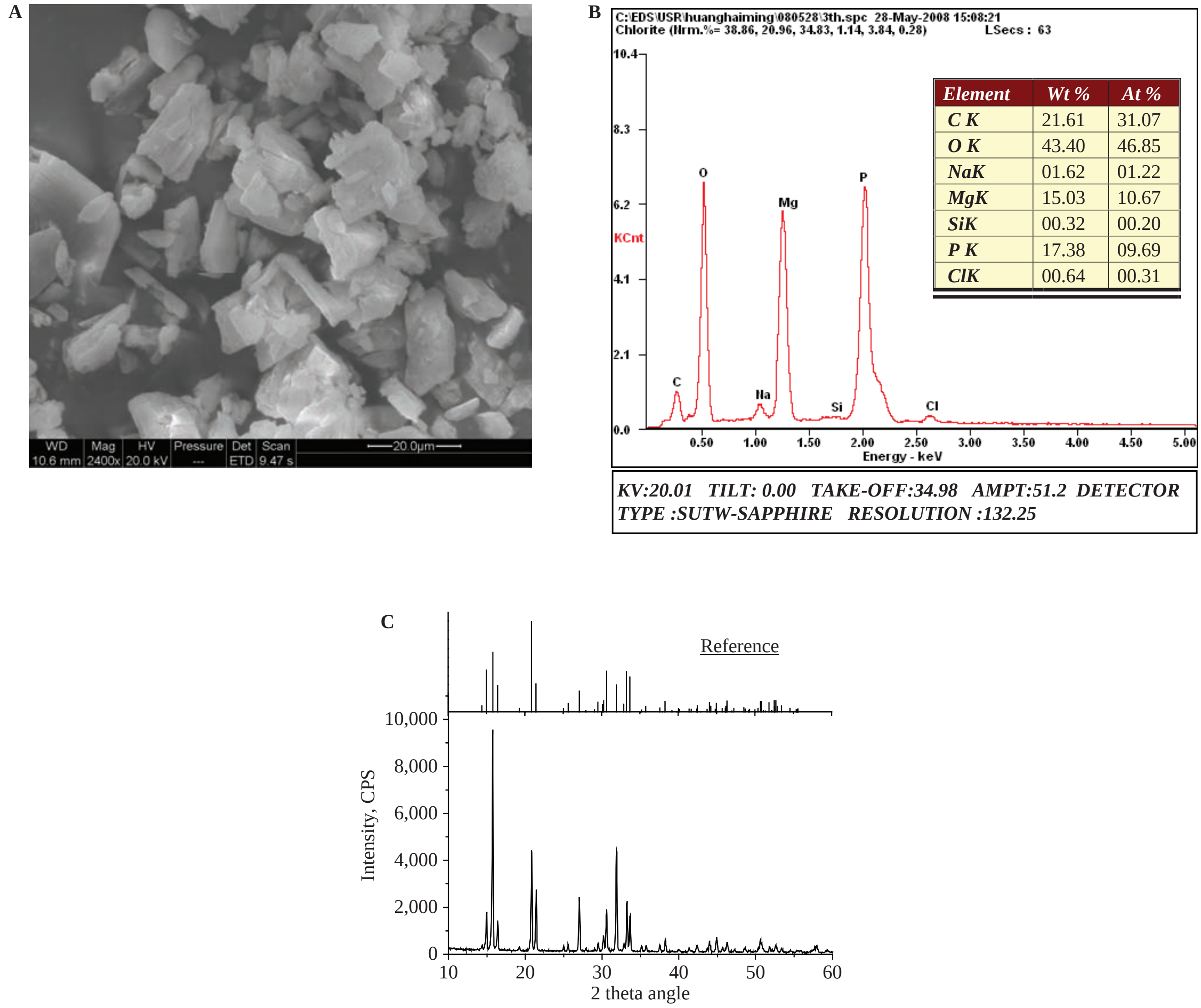

Fig. 3. SEM-EDX and X-ray analysis of the precipitates obtained in the sixth cycle: (a) SEM picture, (b) EDX spectrum, and (c) X-ray diffraction pattern.

1. A separate excess of magnesium and phosphate did not provide a significant increase in $NH_3$-N removal.
2. A simultaneous excess of magnesium and phosphate could markedly decrease the remaining $NH_3$-N concentration in solution. The optimum Mg:N:P was present at a molar ratio of 1.2:1:1.2 and the remaining $NH_3$-N concentration reached the minimum of 8.7 mg/L.
3. While reusing struvite decomposition residues for six cycles, a stable level of $NH_3$-N removal with a remaining $NH_3$-N of approximate 15 mg/L was maintained by supplementing fresh magnesium and phosphate for remaining the steady Mg:N:P ratio (1.2:1:1.2) and adding HCl for dissolving the decomposition residues.

Table 3
Market prices of power ($/kWh) and chemicals ($/kg) used and recovered in the experiments

| Chemicals and power | Price |
|---|---|
| Consumed $MgCl_2 \cdot 6H_2O$ | 0.053 |
| Consumed $Na_2HPO_4 \cdot 12H_2O$ | 0.23 |
| Consumed $H_3PO_4$ (85%) | 0.58 |
| Consumed NaOH | 0.27 |
| Consumed HCl (30%) | 0.08 |
| Power consumption | 0.1 |
| Recovered ammonia (20%) | 0.03 |
| Recovered struvite | 0.28 [28] |

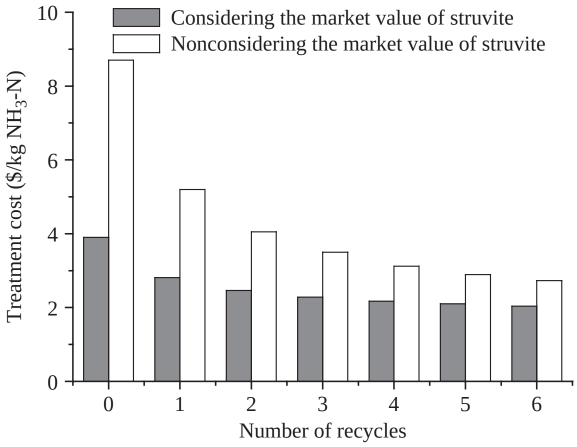

Fig. 4. Plot showing the costs for removing 1 kg NH$_3$-N as a function of the number of cycles.

4. The SEM-EDX analysis indicated the precipitate obtained in the sixth reuse cycle was fragmentized and its size was irregular, the surface composition of the precipitate contained O, P, and Mg and trace of Na, Si, Cl, and C. The XRD analysis demonstrated that the main composition of the precipitate was struvite.
5. Economic analysis showed that no matter whether the value of the struvite was considered or not, a considerable treatment cost can be saved by reusing struvite in the NH$_3$-N removal process.

Based on the results in this study, it is concluded that the method of simultaneously overdosing magnesium and phosphate for the increase of NH$_3$-N removal and reusing struvite for the lowering of treatment cost is suitable for the removal of NH$_3$-N in WWP.

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