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Ammonia and phosphorous precipitation through struvite crystallization from swine wastewater with high suspended solid

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ABSTRACT

Anaerobic effluent of swine wastewater contains high concentration of suspended solid (SS), phosphate (PO_4-P), ammonia nitrogen (NH_4^+-N), and heavy metals such as copper (Cu) and zinc (Zn), posing eutrophication and environmental risk if it was discharged into water body. Struvite crystallization is a promising way to simultaneously recover NH_4^+-N and PO_4-P from wastewater. However, the information is very limited for recovering NH_4^+-N and PO_4-P through struvite from wastewater containing high SS and heavy metals. In this study, the precipitation of NH_4^+-N and PO_4-P through struvite process from real swine wastewater was investigated. The results showed that the amount of formed struvite precipitate of NH_4^+-N and PO_4-P from swine wastewater increased as pH increased from 7.5 to 10.0. The recovery of NH_4^+-N and PO_4-P reached the maximum of 71% and 85%, respectively, at pH 9.5. The contents of Cu and Zn in the struvite precipitates were up to 130 mg kg^{-1} and 400 mg kg^{-1}, respectively. When polymeric aluminum (PAC) and polyferric sulfate (PFS) were separately added as coagulants, 23.9%–40.0% and 17.3%–32.4% of SS were correspondingly removed. In the following struvite crystallization process, 79% of NH_4^+-N and 100% of PO_4-P were precipitated. The contents of Cu and Zn in struvite precipitates declined to 53 mg kg^{-1} and 152 mg kg^{-1}, correspondingly. Combined flocculation and struvite crystallization process can effectively remove SS from the liquid, increase the recovery of NH_4^+-N, and decrease content of Cu and Zn in struvite precipitate, reducing the environmental risk of struvite as fertilizer.

Keywords: Swine wastewater; Flocculation; Struvite; Heavy metals; Nutrient recovery

1. Introduction

Anaerobic effluent of swine wastewater contains high concentration of suspended solid (SS), phosphate (PO_4-P) and ammonia nitrogen (NH_4^+-N) [1]. Usually, before discharging, NH_4^+-N is firstly oxidized to nitrate nitrogen (NO_3^--N), then the NO_3^--N is denitrified into nitrogen gas (N_2) through adding external carbon source. Because of the high concentration of NH_4^+-N in the effluent, the aeration and external carbon source result in high operation cost [2]. Therefore, finding a cost-effective treatment for swine wastewater is very important for protecting the pollution from the anaerobic effluent of swine wastewater. Anaerobic ammonium oxidation (Anammox) has been extensively investigated to remove NH_4^+-N in ammonium-rich wastewater [3]. However, when exceeding critical concentration, heavy metals, phosphate, ammonia and organic matters inhibited the Anammox activity [4]. On the other

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hand, nitrogen and phosphorus are important elements for crop growth, and even phosphorus is finite and nonrenewable resource [5,6]. Therefore, before Anammox treatment, recovering nitrogen and phosphorus from swine wastewater is very necessary [7].

To resolve this issue, many methods have been investigated to recover NH_4^+-N and PO_4-P from wastewater, such as adsorption [8,9], electrochemistry [10–12], gas-permeable membrane [13], and struvite crystallization [14–16]. Among them, struvite crystallization has proved to be a promising way to simultaneously recover NH_4^+-N and PO_4-P [17,18]. The factors affecting struvite crystallization has been widely investigated such as pH, N:P:Mg ratio, suspended solid (SS), and heavy metals [19,20]. The effective struvite crystallization occurred at pH > 8 and Mg:P at 0.8–1 [7,21]. However, the information is limited for real swine wastewater with high suspended solid. Due to complicated component in swine wastewater, such as various ions and organic matter, the effect of pH and N:P:Mg ratio on struvite process for real swine wastewater needs further investigation.

Actually, swine wastewater contained not only high content of NH_4^+-N and PO_4-P, but also high concentration of SS, chemical oxygen demand (COD), which would result in serious pollution in receiving water bodies if improper management before discharging [22–24]. Struvite crystallization had been widely investigated at wastewater with low SS, but the crystallization process with high SS had been seldom reported. High concentration of SS can stain the surfaces of carriers and thus affect mass transfer, resulting worse effluent quality [25]. Furthermore, high concentration of SS would slow down struvite formation [15,26]. Therefore, the removal of SS not only benefits Anommox process but struvite crystallization process. Flocculation is often applied for SS removal in wastewater treatment [27,28], and polymeric aluminum (PAC) and polyferric sulfate (PFS) are commonly used flocculants [29].

Besides, there are many metals with different concentrations in swine wastewater. Among them, copper (Cu) and zinc (Zn) are often used as feed additives [30]. Nevertheless, most of metals are excreted into manure. Heavy metals are great concern in struvite recovery. When struvite crystallization occurred under alkali conditions, metals may precipitate in forms of adsorbed metals or metal deposits. The content of heavy metals in fertilizers is controlled strictly by government [22].

The main objective of this study was to investigate: (i) the effects of pH and N:P:Mg ratio on struvite crystallization from swine wastewater with high SS; (ii) the effect of flocculation on struvite crystallization; (iii) the heavy metals precipitation in the struvite from swine wastewater.

2. Experimental methods

2.1. Characteristics of swine wastewater

Swine wastewater was collected in a lagoon from a pig farm located in Hefei. The wastewater was first filtrated with a 1.0 mm sieve to remove large particles and then stored at 4°C for use. The characteristics of the swine wastewater are listed in Table 1, which has high concentrations of SS, total chemical oxygen demand (TCOD), soluble chemical oxygen demand (SCOD) and NH_4^+-N.

Table 1
Characteristics of the swine wastewater (mg L^{-1})

Item	Concentration
NH_4^+-N	504.9 ± 14.1
PO_4-P	17.82 ± 9.05
NO_3^--N	6.90 ± 0.57
SS	1227.1 ± 257.5
Total solid, TS	3617.5 ± 378.3
Volatile solid, VS	1832.5 ± 258.1
TCOD	2337.9 ± 531.5
SCOD	1155.4 ± 329.9
pH	8.28 ± 0.13

2.2. Experimental setup and procedure

All experiments were carried out in 500 mL glass breakers at room temperature. The experiment was composed of two parts. In the first part, the struvite was crystallized directly with high SS, which was to investigate the influence of pH and the N:P:Mg ratio on the struvite production, SS removal and metal precipitation under high SS conditions. In the second part, the flocculation was applied to remove SS and then subjected to struvite crystallization.

2.2.1. Struvite crystallization with high SS

The influence of pH over the range of 7.5–10.0 on the struvite crystallization was investigated. Subsequently, the ratio of N:P:Mg on the struvite crystallization and SS removal was further investigated, in which, the NH_4^+-N concentration was kept unchanged, and adding $MgCl_2$ and Na_2HPO_4 solution to adjust the mole ratio of N:P:Mg. In addition, a blank contrast without SS was conducted. After adjusting pH to 7.5, 8.0, 8.5, 9.0, 9.5 and 10.0, NH_4Cl^- Na_2HPO_4 and $MgCl_2$ solution were added into pure water in sequence to obtain the N:P:Mg ratio. Magnetic stir was used to improve the struvite formation. Struvite crystallization process lasted 20 min at room temperature for all experiments. The experimental design is listed in Table 2.

2.2.2. Combined flocculation and struvite crystallization process

In this part, flocculation was used to remove the SS, and the supernatant liquid was subjected to struvite crystallization. The coagulants of PAC (10% w/v) and PFS (10% w/v) were tested for the SS removal. The flocculent concentration from 5 to 30 mg L^{-1} was selected. After flocculation, the concentrations of SS, TCOD, SCOD, NH_4^+-N and PO_4-P were determined. Then, 200 mL supernatant liquid was collected for further struvite crystallization at optimal pH and N:P:Mg ratio. After struvite crystallization, the residual NH_4^+-N, PO_4-P, and SCOD in the aqueous phase was determined again. The experimental design is listed in Table 3.

Table 2
The experimental design of struvite crystallization from swine wastewater

Sets	pH	N:P:Mg
1	7.5	1:1:1
2	8.0	1:1:1
3	8.5	1:1:1
4	9.0	1:1:1
5	9.5	1:1:1
6	10	1:1:1
7	9.5	1:1:1
8	9.5	1:0.9:0.9
9	9.5	1:0.8:0.8
10	9.5	1:0.7:0.7
11	9.5	1:0.6:0.6
12	9.5	1:0.5:0.5

Table 3
The experimental design of combined flocculation and struvite crystallization process from swine wastewater

Sets	pH	N:P:Mg	PAC	PFS
1	9.5	1:0.8:0.8	5	–
2	9.5	1:0.8:0.8	10	–
3	9.5	1:0.8:0.8	15	–
4	9.5	1:0.8:0.8	20	–
5	9.5	1:0.8:0.8	25	–
6	9.5	1:0.8:0.8	30	–
7	9.5	1:0.8:0.8	–	5
8	9.5	1:0.8:0.8	–	10
9	9.5	1:0.8:0.8	–	15
10	9.5	1:0.8:0.8	–	20
11	9.5	1:0.8:0.8	–	25
12	9.5	1:0.8:0.8	–	30

2.3. Analytical methods

The concentrations of PO_4-P, NH_4^+-N, TCOD, SCOD, and SS were determined according to the Standard Methods [31]. Supernatants were taken out immediately into a bottle for the analysis of SS and TCOD after 20 min crystallization, and 2.0 mL supernatant was taken out by adding 8 μL HCl to prevent further struvite crystallization, and then was filtrated with 0.45-μm membrane for the analysis of NH_4^+-N, PO_4-P, and SCOD.

The metal concentration in the swine wastewater and precipitates was measured using inductively coupled plasma – mass spectrometry (ICP-MS, Agilent 7500 quaternary pole mass spectra, Agilent, USA). Fifty milliliter swine wastewater or 0.25 g struvite precipitate was digested with 5.0 mL nitric acid (HNO_3) to 1 mL. If the digested solution is transparent, it was continued to steam to nearly dry. Otherwise, it was continuously digested with more HNO_3 addition until the solution is transparent, and then dried. Then, 1% HNO_3 was added to dissolve the debris to 5.0 mL, and the solution was filtrated by 0.22-μm glass membrane. Finally, the beaker and filter were rinsed with 1% HNO_3, transferred and diluted to 10.0 ml in a volumetric flask for ICP-MS analysis. All of the chemicals used were analytical grade.

3. Results and discussion

3.1. Struvite crystallization with high SS

3.1.1. Effect of pH on SS removal and struvite crystallization

In this study, the influence of pH on struvite crystallization and SS removal in swine wastewater with high SS was investigated. As shown in Fig. 1, the recovery of NH_4^+-N and PO_4-P increased as pH increased from 7.5 to 10.0, as well as the removal of SS, TCOD, and SCOD. Usually, struvite crystallization was composed of two stages: the nucleation stage and crystal growth stage. At lower pH, phosphate mainly existed in the ions of HPO_4^{2-} and $H_2PO_4^-$ in solution [15], resulting in poorer struvite crystallization. With the increase of pH from 7.5 to 9.5, phosphate mainly existed in the ions of HPO_4^{2-}, which improves the formation of struvite, resulting in the increase of recovery efficiency of NH_4^+-N and PO_4-P from 21% and 24% to 73% and 85%, respectively, which are lower than previous report [32], which might be due to the high SS and complex components in the swine wastewater. In the blank control without SS, the recovery of NH_4^+-N and PO_4-P was much higher than that with high SS, as shown in Fig. 1(c). While at pH 10.0, there was slight decline in NH_4^+-N recovery but slight increase in PO_4-P recovery and SS removal, which can be deduced due to the generation of phosphate compounds, such as $Ca_3(PO_4)_2$ or $Mg_3(PO_4)_2$, lowering the generation of struvite [32]. Compared with NH_4^+-N, PO_4-P, and SS, the removal of TCOD and SCOD was less affected by the increasing pH.

As shown in Table 4, the recovery of NH_4^+-N, PO_4-P and Mg through struvite precipitates increased with the increasing of pH from 7.5 to 9.5, but the percentage of them decreased when pH increased from 9.5 to 10. At pH 9.5, the recovery ratio of PO_4-P and Mg was up to 76.15% and 76.45%, however, NH_4^+-N recovery ratio was only 59.68%. It can be speculated that ammonia volatilizes more strongly and other compounds would precipitate as pH is greater than 9.5. In theory, struvite contains 5.7% N, 12.6% P and 9.9% Mg. Compared with the theoretical value, it could be deduced that SS was precipitated into the solid with struvite formation, because N, P and Mg were lower than that in pure struvite.

The metal concentration in the swine wastewater was detected and is listed in Table 5. Part of metals was precipitated in the solid during struvite crystallization under alkaline conditions. The metal concentration in the struvite precipitates at different pH values is shown in Fig. 2. The concentration of Cu and Zn in struvite precipitates was up to 130 mg kg^{-1} and 400 mg kg^{-1}, respectively. It could be found that the concentrations of Cu and Zn in struvite precipitates decreased with the increasing

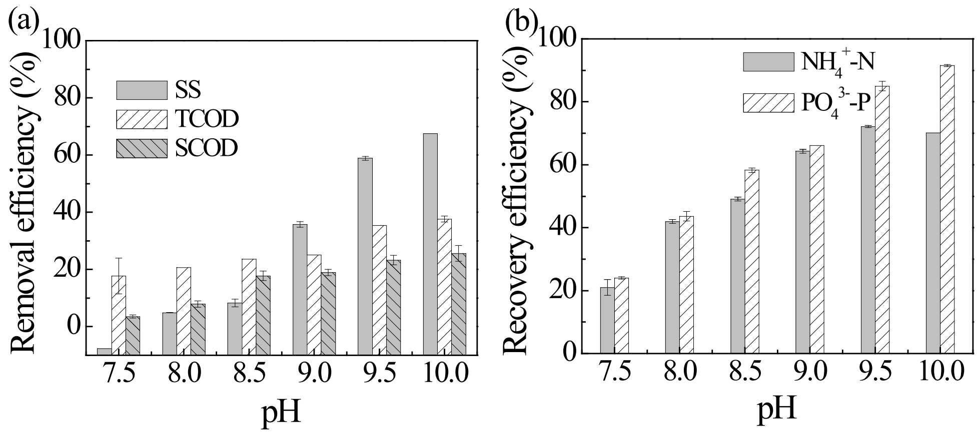

Fig. 1. Effect of pH on (a) the removal of SS, SCOD, TCOD, and (b) the recovery of NH_4^+-N, PO_4-P during struvite crystallization with high SS.

Table 4
The percentage (%) and recovery ratio (%) of NH_4^+-N, PO_4-P and Mg^{2+} in struvite precipitates

pH	NH_4^+-N		PO_4-P		Mg^{2+}	
	Percentage	Recovery	Percentage	Recovery	Percentage	Recovery
7.5	3.21	24.37	7.32	25.14	6.44	28.56
8.0	4.45	41.79	9.24	39.21	7.44	40.83
8.5	4.03	44.38	10.25	50.91	8.96	57.50
9.0	4.81	55.30	12.00	62.33	10.35	69.45
9.5	4.81	59.68	13.60	76.15	10.57	76.45
10.0	3.69	53.08	13.00	84.58	10.04	84.39

Table 5
The concentration of metals in the swine wastewater (mg L^{-1})

Item	Concentration
Mg	2.95 ± 0.83
Cu	1.68 ± 0.82
Zn	5.08 ± 2.63
Na	10.64 ± 4.08
K	53.22 ± 25.80
Ca	10.87 ± 6.07
Chromium, Cr	0.02 ± 0.02
Manganese, Mn	1.00 ± 0.66
Iron, Fe	0.63 ± 0.47
Nickel, Ni	0.06 ± 0.03
Arsenic, As	0.016 ± 0.0042
Cadmium, Cd	0.0002 ± 0.0003
Plumbum, Pb	0.0063 ± 0.0067

pH. It might be because Cu and Zn had been nearly completely precipitated at pH 8.0, and more precipitates were formed at higher pH. Therefore, the relative content of metal concentration in the precipitates decreased. It was reported that the concentration of heavy metals in soil can increase by excessive fertilizer, which means that the struvite recovered from swine wastewater cannot be quantified as fertilizer directly [33]. The high concentrations of potassium (K) and sodium (Na) were also detected with the increasing pH, and approximately 1200 mg kg^{-1} of K and 900 mg kg^{-1} of Na were detected at pH 10. It might be due to the generation of K-struvite and Na-struvite precipitates instead of struvite at higher pH [16,34]. Besides, calcium (Ca) presented high levels in the precipitates and it might be from insoluble phosphate compounds under alkaline conditions. Other heavy metals in the precipitates were also detected, but were at very low concentrations.

Considering the struvite production, SS removal and heavy metals together in the precipitates, pH 9.5 was selected as optimal pH in the following experiment. Other researchers also confirmed that pH 9.0–9.5 displayed large aggregate sizes and high stability for struvite precipitation [35].

3.1.2. Effect of N:P:Mg ratio on SS removal and struvite crystallization

The molecular formula of struvite is $MgNH_4PO_4 \cdot 6H_2O$, and the theoretical mole ratio of N:P:Mg is 1:1:1. While in real swine wastewater, the mole concentration of NH_4^+-N was always higher than that of PO_4-P and Mg^{2+}. Higher SS affected struvite formation. Therefore, the effect of

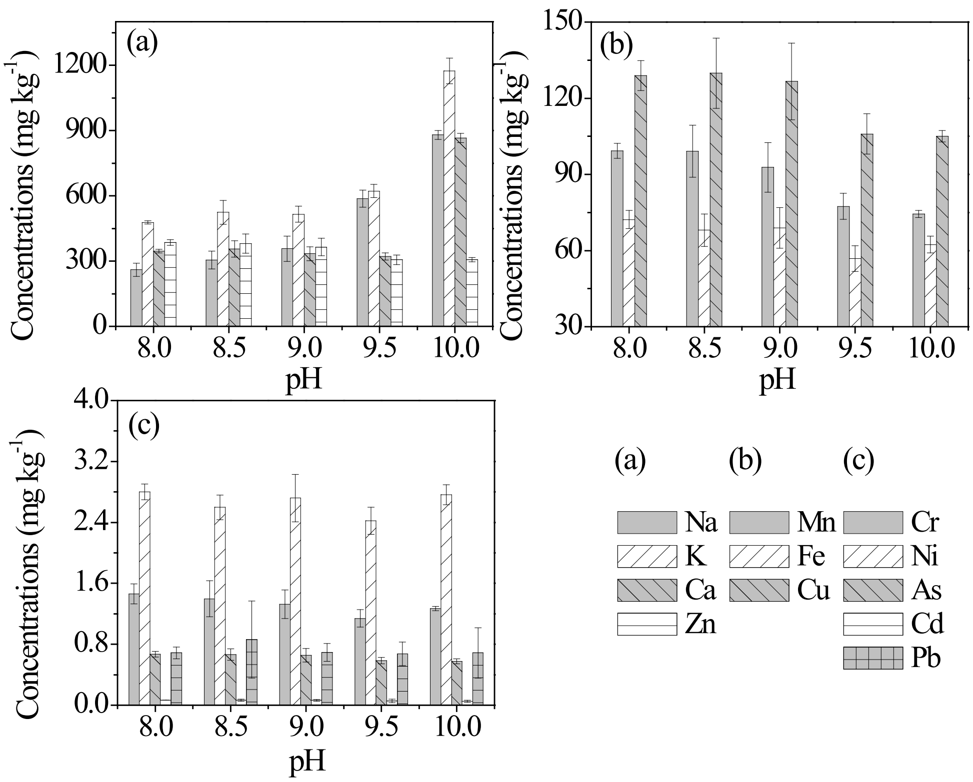

Fig. 2. Metals concentrations in unit struvite precipitate obtained at different pH.

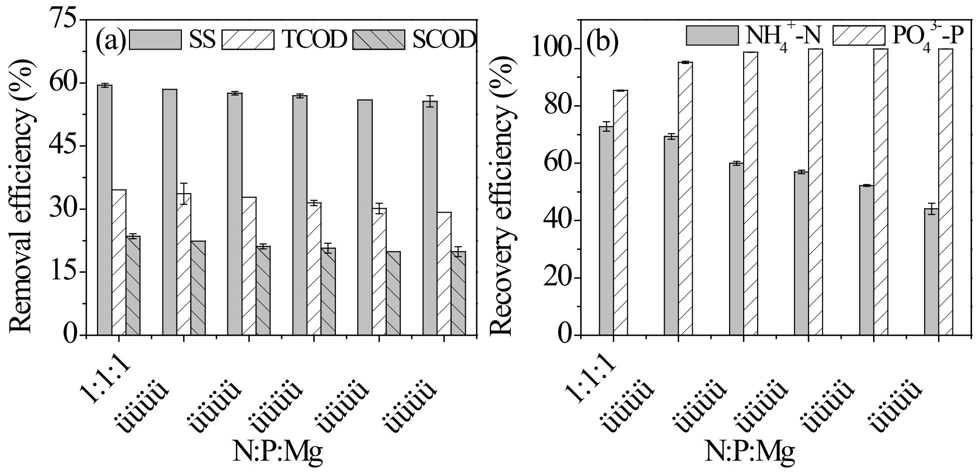

Fig. 3. Effects of N:P:Mg mole ratio on (a) the removal of SS, SCOD, TCOD and (b) the recovery of NH_4^+-N and PO_4-P during struvite precipitation (pH 9.5) with high SS.

N:P:Mg ratio under higher SS conditions on the recovery efficiency of NH_4^+-N and PO_4-P was investigated. As shown in Fig. 3, the recovery efficiency of NH_4^+-N and the removal of SS, SCOD, and TCOD declined with the decrease of N:P:Mg ratio, but the recovery efficiency of PO_4-P was not obviously affected, which was because the mole concentration of NH_4^+-N was higher than that of PO_4-P, and PO_4-P had been reacted with NH_4^+-N to generate struvite at higher N:P:Mg ratio. As shown in Fig. 3b. The NH_4^+-N recovery efficiency obtained at N:P:Mg ratio of 1:1:1 and 1:0.5:0.5 were 72% and 45%, respectively. At N:P:Mg ratio of 1:1:1, the NH_4^+-N recovery of 75% was far lower than theoretical value of 100%, and the recovery of PO_4-P was 85%, indicating that about 10% PO_4-P might be precipitated in the formation of $CaPO_4$, $Mg_3(PO_4)_2$, etc. SS removal had no obvious decrease with the decrease of N:P:Mg ratio, about 60%. Furthermore, SS in swine wastewater may stain on struvite and prevent struvite formation, resulting decline in SS removal and struvite production [15]. Therefore, it was an important step to remove SS before struvite precipitation. With N:P:Mg ratio varying, COD removal efficiency had little change, TCOD removal increased from 25% to 40%, and SCOD from 16% to 28%, respectively.

Metals content in the precipitates after struvite crystallization at pH 9.5 and different N:P:Mg ratio are displayed in Fig. 4. Cu and Zn concentrations in struvite precipitates increased with N:P:Mg ratio changing from 1:1:1 to 1:0.5:0.5. It might be because the generated struvite at 1:0.5:0.5 was less than that at 1:1:1. It has been reported that Cu and Zn in wastewater formed complexes with organic colloids, thus could be removed by coagulation [36]. This result indicated that Cu and Zn could be removed before struvite process through coagulation. K could replace NH_4^+-N location during struvite process at low ammonium concentration [34]. In this experiment, ammonium concentration was high, so K concentration in the precipitates had no obvious change.

3.2. Combined flocculation and struvite crystallization process

Flocculation process was applied to remove SS for facilitating subsequent struvite process [32]. Two common flocculants, PAC and PFS were tested. The results of flocculation and post-struvite are presented in Fig. 5. In flocculation process, removal of SS and TCOD were highly influenced by the dosage of flocculants [37]. 23.9%–40.0% and 17.3%–32.4% of SS were removed when PAC and PFS were separately added from 5 to 30 mg L^{-1}. TCOD was removed from 5% to 25% by PAC or PFS. It can be obtained that PAC was more suitable than PFS for the SS removal for the real swine wastewater. However, SCOD and NH_4^+-N were not affected by the flocculants addition because of the characteristic of high solubility for SCOD and NH_4^+-N.

Fig. 5b and Fig. 5d shows the combined results of flocculation and struvite crystallization together. At PAC dosage of 15 mg L^{-1}, 79% of NH_4^+-N was recovered, which increased about 6% than that without flocculation. With More PAC added, more SS was removed and higher NH_4^+-N was recovered. Almost complete PO_4-P removal was achieved by flocculation-struvite process. When 15 mg L^{-1} PAC was added, 79% of SS and 46% of TCOD were removed after struvite crystallization, increased 20% and 10% respectively than struvite without flocculation.

Metals content in the precipitates after struvite process pre-treated with different concentration of flocculants are displayed in Fig. 6. Pre-treating with flocculation had significant effect on Cu and Zn removal. Cu and Zn in precipitates were lower than struvite without flocculation, about 62 mg kg^{-1} of Cu and 163 mg kg^{-1} of Zn at PAC dosage of 5 mg L^{-1}. At PAC dosage of 25 mg L^{-1}, the contents of Cu and Zn in precipitates declined to 50 mg kg^{-1} and 141 mg kg^{-1}, respectively. Other heavy metals in the precipitates also decreased as the increase of PAC dosage.

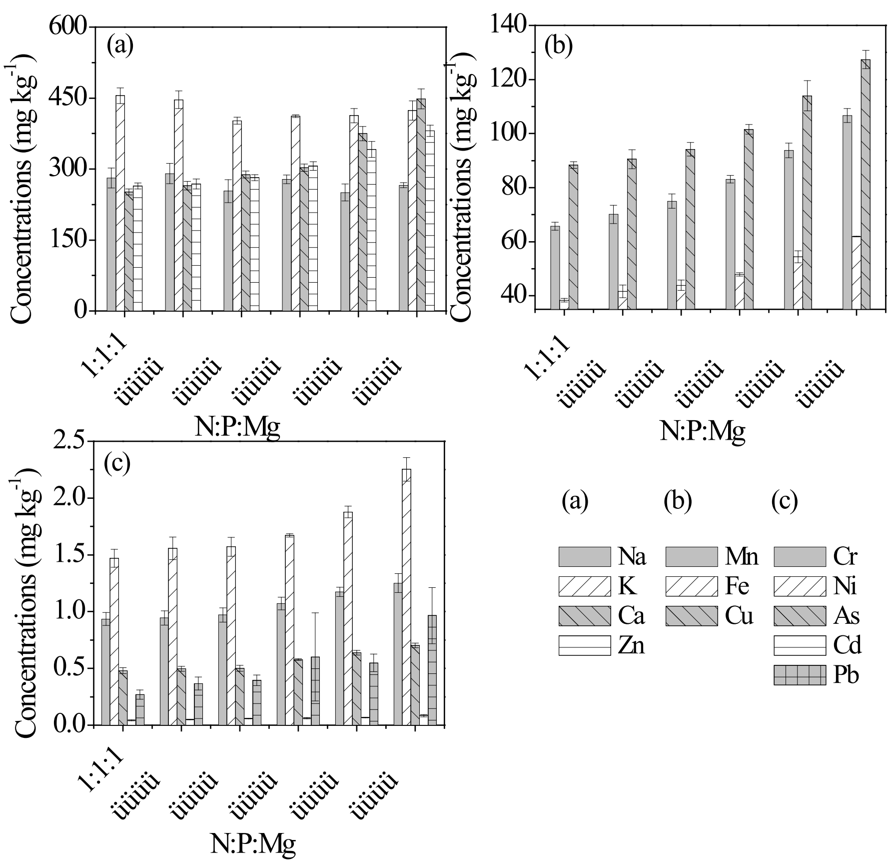

Fig. 4. Metals concentrations in unit struvite precipitates obtained at pH 9.5 and different N:P:Mg ratios.

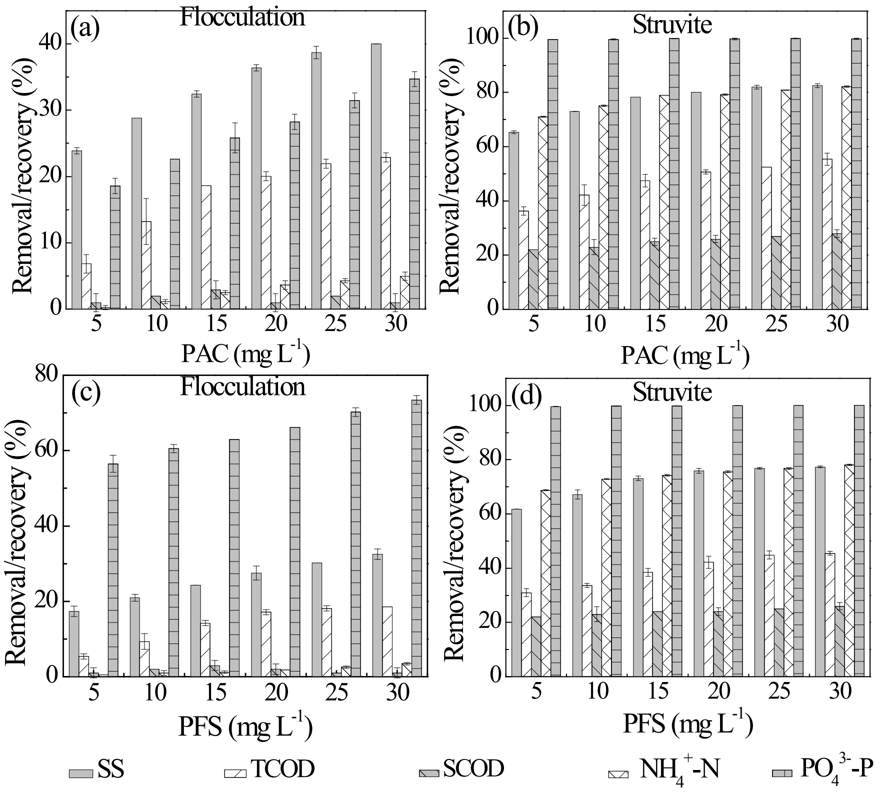

Fig. 5. Effects of (a) PAC dosage during flocculation, (b) PAC dosage post flocculation-struvite precipitation, (c) PFS dosage during flocculation, and (d) PFS dosage post flocculation-struvite precipitation on the removal of SS, SCOD and TCOD, and recovery of NH_4^+-N and PO_4-P at pH 9.5 and N:P:Mg mole ratio of 1:0.8:0.8.

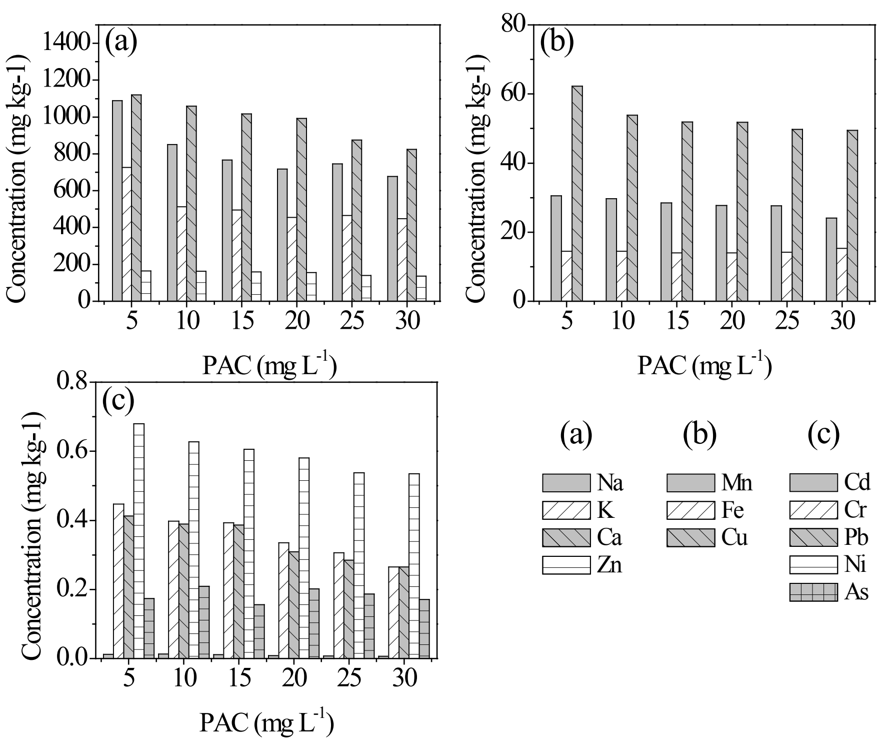

Fig. 6. Metals in unit struvite precipitates obtained at different PAC concentration with flocculation and struvite process.

4 Conclusions

In this study, struvite crystallization of swine wastewater from real swine wastewater with high SS was investigated. Under high SS conditions, the recovery of NH_4^+-N and PO_4-P increased as pH increased from 7.5 to 10.0, as well as the removal of SS, TCOD, and SCOD. The highest of 71% NH_4^+-N and 85% PO_4^{3}-P was recovered. The recovery efficiency of NH_4^+-N declined with the decrease of N:P:Mg ratio, but the recovery efficiency of PO_4-P was not obviously affected by the ratio. High SS in swine wastewater prevented the struvite formation. The concentration of Cu and Zn in struvite precipitates was up to 130 mg kg^{-1} and 400 mg kg^{-1}, respectively. When 5–30 mg L^{-1} of PAC and PFS was separately added as coagulants, 23.9–40.0% and 17.3–32.4% of SS were correspondingly removed. Almost complete PO_4-P was precipitated in flocculation-struvite process, and the highest of 79% NH_4^+-N was recovered. The concentration of Cu and Zn obviously declined to 53 mg kg^{-1} and 152 mg kg^{-1} in the struvite, respectively at PAC dosage of 15 mg L^{-1}. Therefore, the use of coagulants in the anaerobic effluent of swine wastewater can remove SS from the liquid, increase the recovery of NH_4^+-N, and decrease the content of Cu and Zn in struvite precipitate.

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