

Chemical equilibrium model of struvite precipitation from anaerobic digester effluents

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Abstract

This project investigated the usefulness of a chemical equilibrium model to predict the extent of ammonia removal as struvite from the effluent of anaerobic digester treating molasses-based industrial wastewater. The chemical equilibrium model was established by taking into account the activity coefficient and thermodynamic properties of potential compounds that may coprecipitate. It was considered that struvite was the only potential compound that precipitated under the conditions studied. pKs value of struvite was estimated by fitting the model predictions to the experimental results and found to be between 11 and 12 when the temperature is kept at 37 °C, at different $Mg^{2+}:NH_4^+:PO_4^{3-}$ ratios and pH values which are lower than those reported in the literature. This indicates that struvite is more soluble in the wastewater studied here. The analysis of the precipitate and model predictions suggest that there is another compound present in the precipitate in addition to struvite, which is most likely $Mg_3(PO_4)_2 \cdot XH_2O$.

Key Words: Ammonia recovery, Struvite, Magnesium ammonium phosphate, Chemical equilibrium model, Solubility product

1. Introduction

The increasing stringent discharge limits for nitrogen (N) compounds present in industrial and domestic wastewaters constitute a great challenge to reduce concentrations to acceptable levels. Their presence in excess quantities results in deterioration of water quality, causing imbalance in the N cycle. In the absence of anthropogenic activities the transfer of unreactive N to reactive and back again is almost balanced. During the last decades, N pollution has greatly increased concomitant with human-caused activities and resulted in the accumulation of reactive N compounds in the terrestrial system (Galloway et al., 1995). There are a number of physicochemical and biological techniques available in order to remedy the negative effects of reactive N compounds in nitrogen containing wastewaters. The techniques such as biological nitrification/denitrification and breakpoint chlorination reduce N compounds to dinitrogen gas. However, alternative technologies exist to convert ammonia (NH₃)

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into a reusable and saleable useful product, thus contributing to the overall N cycle. One such technology is the recovery of NH_3 as struvite, which has potential as a fertilizer.

Struvite or magnesium ammonium phosphate (MAP) precipitates in the presence of magnesium (Mg^{+2}), ammonium (NH_4^+), and phosphate (PO_4^{3-}) according to the reaction (1) when the thermodynamic solubility product, K_s , is exceeded:



According to reaction (1), Mg^{2+} , NH_4^+ , and PO_4^{3-} are required in equimolar quantities to form $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$. However, because of the common ion effect, other chemical species in solution will form Mg^{2+} , NH_4^+ , and/or PO_4^{3-} compounds. This effect requires that additional Mg^{2+} and/or PO_4^{3-} be needed to precipitate the maximum mass of NH_4^+ .

Using an anaerobic digester to treat a molasses-based industrial wastewater, Çelen and Türker (2001) found that the optimum $\text{Mg}^{2+}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratio for NH_3 removal as struvite was 1.2:1:1.2. Jaffer et al. (2002) achieved only a 3% increase in P removal when the molar Mg:P ratio increased from 1.05 to 3.5, during pilot plant trials using magnesium chloride (MgCl_2) as a Mg^{2+} source.

Struvite precipitation reaction is highly dependent on pH. Struvite precipitates in basic conditions, with pH values ranging from 6.5 to more than 10 (Bowers, 2004). Because the typical animal wastewaters are near neutral pH, the pH can be adjusted upward by either adding a base or by aeration to strip CO_2 from the solution. Battistoni et al. (1997) showed that stripping out CO_2 increased pH from 7.9 to between 8.3 and 8.6 in the supernatant of anaerobically digested sludge in 150 min.

In the literature, several papers addressed the recovery of NH_3 or PO_4^{3-} as struvite from industrial, domestic, and animal wastewaters (Battistoni et al., 2000; Çelen and Türker, 2001; Parsons et al., 2001; Ueno and Fujii, 2001; Jaffer et al., 2002; Suzuki et al., 2004; Çelen et al., 2007; Türker and Çelen, 2007; Çelen, 2009). The precipitation of struvite may present some problems in wastewater treatment plants, causing deposits in pipe walls (Snoeying and Jenkins 1980; Loewenthal et al., 1994). However, struvite has potential use as a fertilizer. It has been shown to be a highly effective source of N, Mg^{+2} , and phosphorus (P) for plants and can be used as a slow release fertilizer at high application rates without damaging plant roots (Priestley et al., 1997; Gaterell et al., 2000).

The objective of the present research was to identify the extent of ammonia removal as struvite from anaerobic digester effluents based on the chemical equilibrium model.

2. Theory

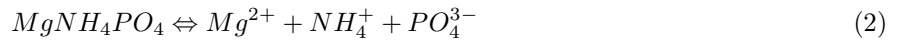
2.1. Chemical equilibrium model

In order to predict the extent of NH_3 removal, a chemical equilibrium model was established using thermodynamic equations representing the interactions among the species present in solution. The potential compounds that may co-precipitate during struvite precipitation were identified using the pK values shown in Table 1 and literature data. There are a number of calcium (Ca^{+2}) compounds that may precipitate. However, Ca^{+2} concentration in the solution is very low compared to Mg^{+2} and PO_4^{3-} . Therefore, Ca^{+2} and its compounds were omitted from the model.

Table 1. The potential compounds that may precipitate and their pK values (adapted from Musvoto et al., 2000).

Mineral Solubility equilibria	Reaction	Stumm and Morgan 1981 pK _s at 25 °C	Nordstrom et al., 1990 pK _s at 25 °C	JESS (Murray and May, 1996) pK _s at 25 °C	Other Sources pK _s at 25 °C
Magnesite	$MgCO_3 \Leftrightarrow Mg^{2+} + CO_3^{2-}$	7.46; 8.2		7.46; 8.2	5.9; 7.9
Nesquehonite	$MgCO_3 \cdot 3H_2O \Leftrightarrow Mg^{2+} + CO_3^{2-} + 3H_2O$	5.19		4.67; 5.19	
Struvite	$MgNH_4PO_4 \cdot 6H_2O \Leftrightarrow Mg^{2+} + NH_4^+ + PO_4^{3-} + 6H_2O$	12.6		13.16	13.2; 13
Newberyite	$MgHPO_4 \cdot 3H_2O \Leftrightarrow Mg^{2+} + HPO_4^{2-} + 3H_2O$			5.8	5.8
Bobierite	$Mg_3(PO_4)_2 \cdot 8H_2O \Leftrightarrow 3Mg^{2+} + 2PO_4^{3-} + 8H_2O$			25.2	25.2
Trimagnesium phosphate	$Mg_3(PO_4)_2 \cdot 22H_2O \Leftrightarrow 3Mg^{2+} + 2PO_4^{3-} + 22H_2O$				23.1

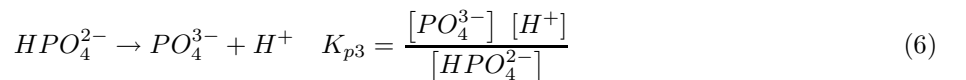
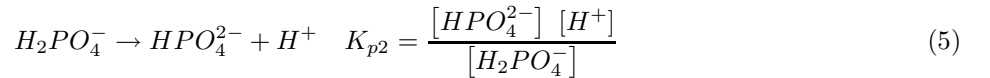
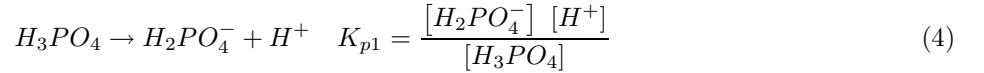
Thermodynamic data from the literature suggest that a number of Mg⁺² compounds may precipitate from the solutions containing, NH₄⁺, and PO₄⁻³ ions: MAP, magnesium hydrogen phosphate trihydrate or newberyite (MgHPO₄·3H₂O) and trimagnesium phosphate in 2 states of hydration, Mg₃(PO₄)₂·22H₂O and Mg₃(PO₄)₂·8H₂O (bobierite). Several researchers have identified 3 regions for precipitation of these compounds (Abbona et al., 1982, 1986, 1988). Struvite precipitates at neutral and higher pH. Newberyite precipitates significantly at lower pH (pH < 6). Trimagnesium phosphate has never been observed in the pH range of 6-9 and is reported to have a low precipitation rate (Mamais et al., 1994). The forms of magnesium carbonate, magnesite and nesquehonite, may also precipitate in the pH range studied in this research. However, their solubility products are much higher than those of struvite. Therefore, they are also removed from the model. As a result, the equations 2 to 10 were considered to describe the system:



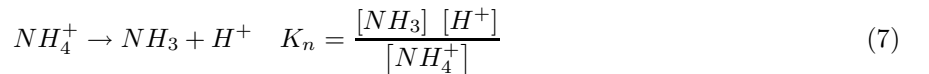
the thermodynamic solubility constant. K_s :

$$[Mg^{2+}] [NH_4^+] [PO_4^{3-}] = K_s \quad (3)$$

Phosphate System:



Ammonia System:



Mass Balance:

$$M_T = [Mg^{2+}] + S \quad (8)$$

$$P_T = [H_3PO_4] + [H_2PO_4^-] + [HPO_4^{2-}] + [PO_4^{3-}] + S \quad (9)$$

$$N_T = [NH_4^+] + [NH_3] + S \quad (10)$$

2.2. Ionic strength

Ionic strength (I) of wastewaters may vary from 0.0 to 0.2, which differs according to the dissolved solids concentrations and is given by Eq. (11):

$$I = \frac{1}{2} \sum z_i^2 C_i \quad (11)$$

where z_i is valency of the ion and C_i is its concentration. If the composition of the wastewaters is not exactly known, the ionic strength can be approximated by multiplying the dissolved solids (mg/L) by 2.5×10^{-5} (Sawyer and McCarty, 1978). In the system, total dissolved solids (TDS) were determined to be approximately 7600 mg/L. Therefore, the ionic strength was calculated as 0.19. Debye-Hückel method, indicated in equation 12, for activity correction was selected because of its frequent use in the research literature (Çelen et al., 2007). In Eq. (12), log and γ stand for logarithm and mean ionic activity coefficient, respectively.

$$\log \gamma = -0.5Z^2 \frac{\sqrt{I}}{1 + \sqrt{I}} \quad (12)$$

3. Materials and methods

In order to validate the results of the chemical equilibrium model, the effluent of anaerobic digester treating molasses-based industrial wastewater was used as a physical model. Struvite precipitation experiments were carried out in a batch reactor with a volume of 200 mL mixed with magnetic stirrer. Since the anaerobic digester effluent temperature was 37 °C, all precipitation experiments were performed at that temperature. A thermostatic controller was used to control the temperature of the reactor (Haakel, Model 001.3568 Germany). The pH measurements were made with pH meter E588 and pH was adjusted either with HCl or NaOH solutions. The chemicals used were commercial grade and their contents were determined before the experiments. All analyses were carried out according to Standard Methods (Standard Methods for the Examination of Water and Wastewater, 1998). After each experiment, the supernatants and the precipitants were analyzed for Mg^{+2} , NH_4^+ , and PO_4^{-3} content to check the consistency of experimental results and the mass balance. The species used in the model were calculated by MATLAB program by entering total Mg^{+2} , P, N, and hydrogen (H^+) values. By using these values, the program calculated Mg^{+2} , NH_3 , NH_4^+ , H_3PO_4 , $H_2PO_4^-$, HPO_4^{2-} , PO_4^{3-} , and $MgNH_4PO_4$. Model outputs were compared to the experimental results. In the calculations Ksp values of MAP were 10^{-9} , 10^{-10} , 10^{-11} , 10^{-12} , and 10^{-13} and the pH ranges were between 6 and 9.

3.1. The composition of wastewater

The wastewater used in this work to recover NH_3 is the effluent of anaerobic digester treating industrial wastewater, the chemical composition of which is given in Table 2.

Table 2. The approximate composition of wastewater.

Parameter	Concentration (mg/L)
NH_4^+	~ 1400
Mg^{2+}	21.4
PO_4^{3-}	24
Ca^{2+}	21.2
K^+	2150
TDS	7600
pH	7.9

The effluent contains approximately 1400 mg/L NH_4^+ , 24 mg/L PO_4^{3-} , 21.4 mg/L Mg^{+2} , and 21.2 mg/L Ca^{+2} . Therefore, PO_4^{3-} and Mg^{+2} were added in stoichiometric quantities at different molar ratios in struvite precipitation studies.

4. Results and discussion

4.1. Chemical equilibrium model and laboratory results

The precipitation potential of struvite was predicted by solving Eqs. (2)-(10) using the values in Tables 3 and 4 under different environmental and initial conditions. Also, the conditions that were modelled by chemical equilibrium model were tested in the laboratory and compared with the experimental results (Table 5). When the $\text{Mg}^{+2}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratio was 1.2:1:1.2 and pH 9, the highest NH_3 removal was obtained as 95.4%. However, when the $\text{Mg}^{+2}:\text{NH}_4^+:\text{PO}_4^{3-}$ molar ratio was 1:1:1 and pH 8, the lowest NH_3 removal was obtained as 78.7%.

Table 3. Values of pK and their temperature dependencies for weak acid bases (Loewenthal et al., 1994).

	pK = (A/T) - B + C.T, where T is Kelvin				
	pK	25 °C	A	B	C
Water	pK_w	14.000			
Phosphate	pK_{p1}	2.148	799.3	4.5535	0.01349
	pK_{p2}	7.198	1979.5	5.3541	0.01984
	pK_{p3}	12.023		not given	
Ammonium	pK_n	9.245	2835.8	0.6322	0.00123

In Figure 1, the concentrations of NH_4^+ in supernatant were estimated and compared to experimental values which are shown in Table 5 by assigning different values of K_{sp} of MAP. In the literature, a number of different values for pKs values are listed in Table 6. In Figure 1(a-d), the pKs of MAP was determined between 11 and 12, which was lower than the commonly reported values. This result indicates that struvite was more soluble under prevailing digester conditions than pure water. However, a few researchers reported rather lower values (Borgerding (cited in Andrade and Schuling 1999), Abbona et al. 1982 and Boorum et al. (cited in Andrade and Schuling 1999)). In Figure 1d, the experimental supernatant concentrations are available between pH 5.5 and 9.0. In this case, pKs value of 12 fits the experimental results best.

Table 4. Equilibrium and solubility products of compounds used in the model (Stumm and Morgan, 1996).

Species	Equation	Logarithmic constant, pK
$MgNH_4PO_4 \cdot 6H_2O$	$K_s = [Mg^{2+}][NH_4^+][PO_4^{3-}]$	to be determined
H_3PO_4	$K_{p1} = \frac{[H_2PO_4^-][H^+]}{[H_3PO_4]}$	2.21
$H_2PO_4^-$	$K_{p2} = \frac{[HPO_4^{2-}][H^+]}{[H_2PO_4^-]}$	7.18
HPO_4^{2-}	$K_{p3} = \frac{[PO_4^{3-}][H^+]}{[HPO_4^{2-}]}$	12.23
NH_4^+	$K_n = \frac{[NH_3][H^+]}{[NH_4^+]}$	8.9
H_2O	$K_w = [H^+][OH^-]$	14

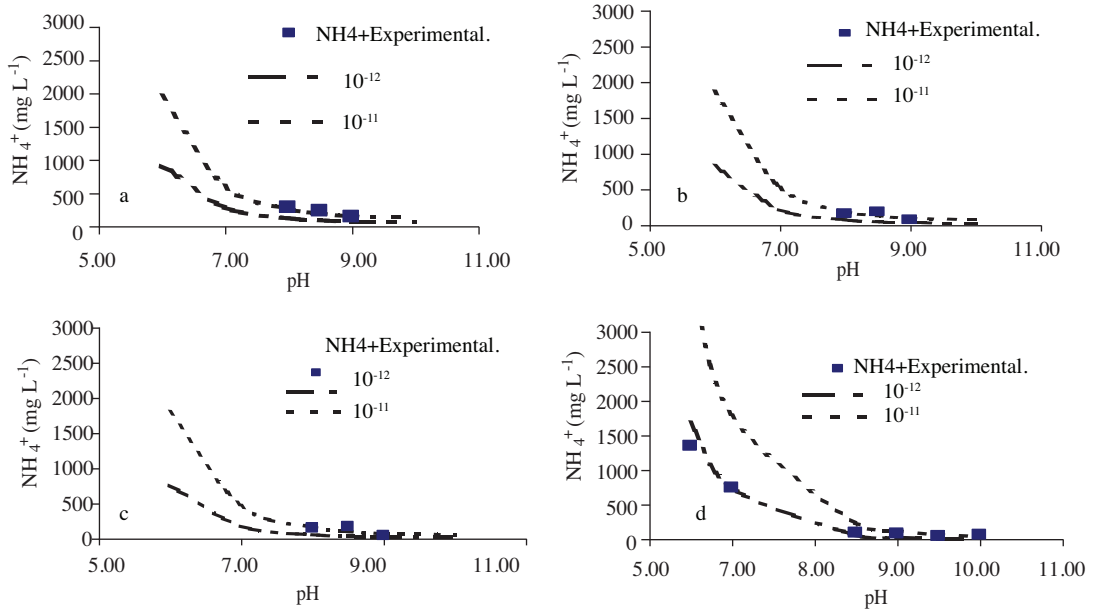


Figure 1. Experimental supernatant NH_4^+ concentrations and model predictions at initial NH_4^+ concentrations of 1350 mg/L: (a) $Mg^{+2}:NH_4^+:PO_4^{-3}$ ratio is 1:1:1; (b) $Mg^{+2}:NH_4^+:PO_4^{-3}$ ratio 1.2:1:1; (c) $Mg^{+2}:NH_4^+:PO_4^{-3}$ ratio 1.4:1:1; (d) $Mg^{+2}:NH_4^+:PO_4^{-3}$ ratio 1.2:1:1.2.

In Figure 2(a-d), Mg^{+2} and in Figure 3(a-b), PO_4^{-3} concentrations in the supernatant were estimated from the chemical equilibrium model and compared to experimental results. In both cases, Mg^{+2} and PO_4^{-3} concentrations were overestimated using the pKs values that fit NH_4^+ results. This could be due to the fact that while NH_4^+ is fixed as MAP, Mg^{+2} and PO_4^{-3} may form other precipitate. This was justified by the analysis of precipitants. In Table 7, the molar ratios of the species removed from the solutions were presented. These results indicate that molar ratios of Mg^{+2} and PO_4^{-3} were higher than that of NH_4^+ . It means that Mg^{+2} and PO_4^{-3} compounds may have coprecipitated in addition to MAP. If the average for 1.2:1:1.2 and 1:1:1.2 were taken, average compositions of precipitates as 1.31:1:1.19 would be obtained. Also the molar ratio of Mg^{+2} and PO_4^{-3} in the non-struvite compound is 3:2, indicating that the most likely by-product formed during MAP

Table 5. Percentage of NH_4^+ removal at different pH and molar ratios.

M:N:P Molar Ratio and pH	NH_4^+ Removal (%)
1:1:1 and 8.0	78.7
1:1:1 and 8.5	83.4
1:1:1 and 9.0	90.2
1.2:1:1 and 8.0	88
1.2:1:1 and 8.5	86
1.2:1:1 and 9.0	94.5
1.4:1:1 and 8.0	88.5
1.4:1:1 and 8.5	87.4
1.4:1:1 and 9.0	96.9
1.2:1:1.2 and 7.5	93.2
1.2:1:1.2 and 8.0	94.3
1.2:1:1.2 and 8.5	97
1.2:1:1.2 and 9.0	95.4
1:1:1.2 and 7.5	81.6
1:1:1.2 and 8.0	82
1:1:1.2 and 8.5	82.6
1:1:1.2 and 9.0	83.8

Table 6. Literature survey for pK_s values of Struvite.

pK_s	T (°C)	Origin	Reference
12.60	38	urine	*Elliot et al., 1959
12.33	38	water	*Jonhson, 1959
13.15	25	aqueous solution	*Taylor et al., 1963
12.6	25	*Bube,	1964
13.12	25	water	*Burns and Finlayson, 1982
12.97	35	water	*Burns and Finlayson, 1982
12.94	38	water	*Burns and Finlayson, 1982
12.84	45	water	*Burns and Finlayson, 1982
12.6	20	simulation	Loewenthal et al., 1994
11.84	25	simulation	*Booram et al., 1975
12.76	25	water	Webb and Ho., 1992
12.36		simulation	*Buchanan et al., 1994
9.41	20	water	*Borgerding, 1972
9.94	25	aqueous	solution Abbona et al., 1982
13.26	25	digester effluent	Ohlinger 1998
11-12	37	digester effluent	this work

*adapted from Andrade and Schuling (1999).

precipitation is $\text{Mg}_3(\text{PO}_4)_2\text{XH}_2\text{O}$ despite the fact that it is claimed that it may not form between pH 6 and 9 as proposed by Mamais et al. (1994). Booker et al. (1999) also reported the composition of precipitates under

different conditions and claimed that the relative ratio of Mg^{+2} and PO_4^{-3} to NH_4^+ is higher than 1. This finding also supports the results presented in the present paper.

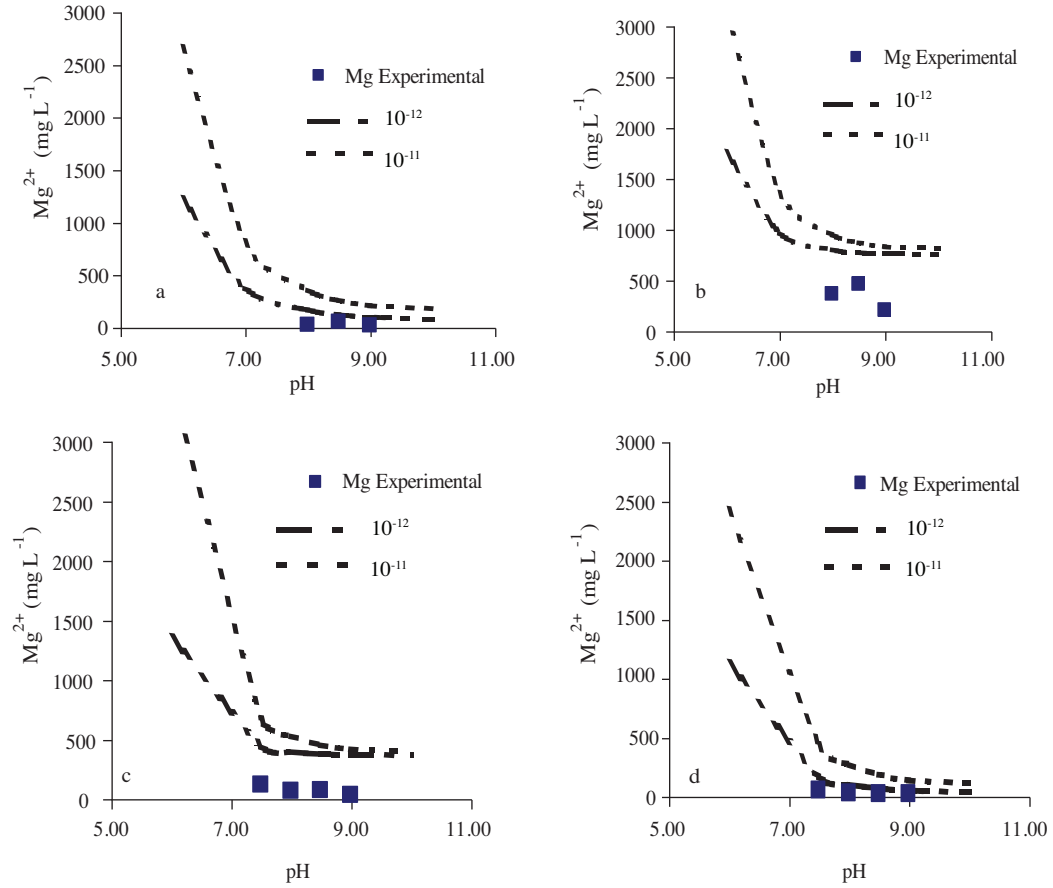


Figure 2. Experimental supernatant magnesium concentrations and model predictions: (a) $\text{Mg}^{+2}:\text{NH}_4^+:\text{PO}_4^{-3}$ ratio is 1:1:1 and initial $\text{Mg}^{+2} = 1798$ mg/L; (b) $\text{Mg}^{+2}:\text{NH}_4^+:\text{PO}_4^{-3}$ ratio 1.4:1:1 and initial $\text{Mg}^{+2} = 2575$ mg/L; (c) $\text{Mg}^{+2}:\text{NH}_4^+:\text{PO}_4^{-3}$ ratio 1.2:1:1.2 and initial $\text{Mg}^{+2} = 2187$ mg/L; (d) $\text{Mg}^{+2}:\text{NH}_4^+:\text{PO}_4^{-3}$ ratio 1:1:1.2 and 1676 mg/L.

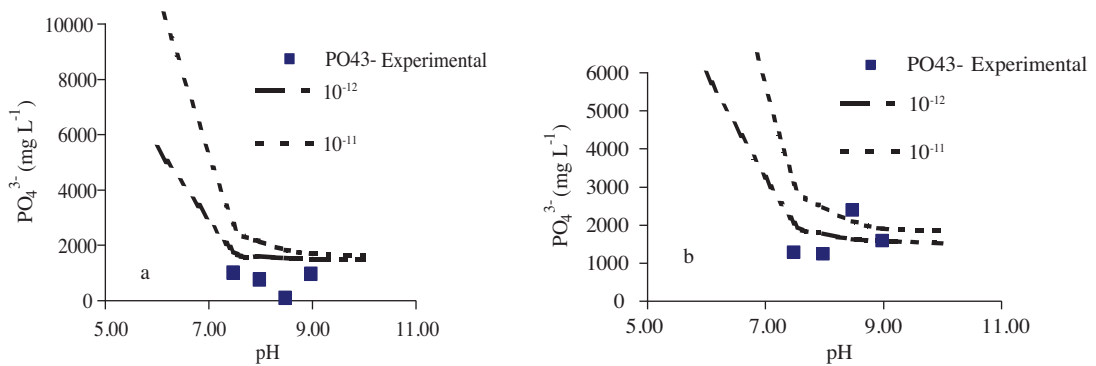


Figure 3. Experimental supernatant phosphate concentrations and model predictions: (a) $\text{Mg}^{+2}:\text{NH}_4^+:\text{PO}_4^{-3}$ ratio is 1.2:1:1.2 and initial $\text{PO}_4^{-3} = 8558$ mg/L, (b) $\text{Mg}^{+2}:\text{NH}_4^+:\text{PO}_4^{-3}$ ratio 1:1:1.2 and initial $\text{PO}_4^{-3} = 7873$ mg/L.

Table 7. The composition of precipitates under different $Mg^{+2}:NH_4^+:PO_4^{-3}$ and pH values.

$Mg^{+2}:NH_4^+:PO_4^{-3}$	pH	$Mg^{+2}:NH_4^+:PO_4^{-3}$ in precipitation
1:1:1	8.0	1.3:1:NM*
	8.5	1.37:1:NM
	9.0	1.33:1:NM
1.2:1:1	8.0	1.36:1:NM
	8.5	1.39:1:NM
	9.0	1.38:1:NM
1.4:1:1	8.0	1.36:1:NM
	8.5	148:1:NM
	9.0	1.47:1:NM
1.2:1:1.2	7.5	1.32:1:1.17
	8.0	1.40:1:1.12
	8.5	1.28:1:1.24
	9.0	1.40:1:1.19
1:1:1.2	7.5	1.23:1:1.29
	8.0	1.29:1:1.13
	8.5	1.27:1:1.19
	9.0	1.30:1:1.22

*not measured

5. Conclusions

The following conclusions can be drawn from the present study:

1) The pKs values of MAP were determined between 11 and 12 and close to 12. In the experiments, the temperature is kept at 37 °C and the M:N:P ratios are 1:1:1 at pH 8.0, 8.5, 9.0, 1.2:1:1 at pH 8.0, 8.5, 9.0, 1.4:1:1 at pH 8.0, 8.5, 9.0, 1.2:1:1.2 at pH 7.5, 8.0, 8.5, 9.0, 1:1:1.2 at pH 7.5, 8.0, 8.5, 9.0.

2) A simple thermodynamic model can predict the extent of ammonia removal as shown in Figure 1.

3) The model suggests struvite and a by-product which is most likely $Mg_3(PO_4)_2XH_2O$

will be formed in the precipitate. Also by using chemical analysis results M:N:P ratios were determined other than 1:1:1, which indicated only MAP formation. If the average for 1.2:1:1.2 and 1:1:1.2 were taken, average compositions of precipitates would be obtained as 1.31:1:1.19. The model data agreed with the experimental data.

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