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Thermodynamics and solution chemistry of struvite

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Abstract

The key feature of this paper is the combined removal of magnesium, ammonium and phosphate from a representative synthetic solution of piggery effluent. The byproduct of this recovery process is struvite, commercially used as slow-release fertiliser. A thermodynamic model was developed relating to all the complexes of magnesium, ammonium and phosphate using gPROMS process software. A laboratory-based batch experiment confirmed 80% recovery of phosphate using this crystallization technique. The batch experiment was conducted at constant pH using synthetic solution made up of analytical-grade $MgCl_2$ and $NH_4H_2PO_4$. Borosilicate glass grindings of 5 g were used as seeds in this batch experiment. Basic solution characteristics of piggery effluent are reported in this paper.

Keywords: Struvite, solution chemistry, piggery effluent, thermodynamic model.

1. Introduction

Livestock intensification results in effluent discharge issue. This leads to soil acidification and premature eutrofication of surrounding waterways due to the presence of nutrients such as ammonium and phosphate. Occasionally, strong nutrient in wastewater stream forms solid deposits and clogs water distribution system. This situation of solid encrustation occurs when the solubility limit of nutrients exceeds the saturation limit, which is called supersaturation. In solution chemistry, solubility limit is expressed by saturation level of solution, which can be determined by comparing thermodynamic (absolute) solubility product and conditional solubility product of the reactive solution. The value of minimum struvite solubility product is well documented in the literature [1–4]. The resulting negative logarithmic value of minimum struvite solubility product, pK_{so} ($-\log_{10}K_{so}$), documented by Ohlinger [5] was 13.26 ± 0.04, whereas Snoeyink and Jenkins [4] reported 12.6, which was reportedly used by other researchers [6–9]. Minimum struvite solubility product, proposed by Ohlinger [5], was employed in this thermodynamic modelling.

Solution supersaturation and pH value are the predominant parameters of controlling struvite precipitation. The apparent pH for struvite crystallization has been documented as 7.5–11.0 [4, 5, 10–12] depending upon solution concentration. The thermodynamic study of

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struvite solution explored better understanding on struvite solution chemistry and resultant precipitation. Wide variation of pH for struvite crystallization was observed due to solution supersaturation, which related both solution concentration as well as pH value.

2. Thermodynamic modelling

Solution chemistry of magnesium, ammonium and phosphate in ionic state plays a predominant role in struvite crystallization. In supersaturated solution, struvite forms by chemical reaction among free Mg^{2+}, NH_4^+ and PO_4^{3-} as demonstrated in eqn (1).

$$Mg^{2+} + NH_4{}^{+} + PO_4{}^{3-} \Leftrightarrow MgNH_4PO_4. \tag{1}$$

The key parameters involved in struvite solution chemistry are solution supersaturation, pH, and initial concentration of the reactants. Solution consists of magnesium, ammonium and phosphate remains in complex forms of Mg^{2+}, $MgOH^+$, $MgH_2PO_4^+$, $MgHPO_4$, H_3PO_4, $H_2PO_4^-$, HPO_4^{2-}, PO_4^{3-}, $MgPO_4^-$, NH_3 (aqueous) [1, 7]. The thermodynamic equilibria of different complexes are shown in eqns (2)–(9), in which $\{Mg^{2+}\}$, $\{NH_4^+\}$ and $\{PO_4^{3-}\}$, etc. are the activity-based concentrations. The thermodynamic equilibria of the active solution chemistry including activity-based concentration, $\{i\}$, and reaction quotient, K_i, are shown in Table I.

$$K_{MgOH^+} = \frac{\{Mg^{2+}\}\{OH^-\}}{\{MgOH^+\}} \tag{2}$$

$$K_{NH_4^+} = \frac{\{H^+\}\{NH_3(aq)\}}{\{NH_4^+\}} \tag{3}$$

$$K_{HPO_4^{2-}} = \frac{\{H^+\}\{PO_4^{3-}\}}{\{HPO_4^{2-}\}} \tag{4}$$

$$K_{H_2PO_4^-} = \frac{\{H^+\}\{HPO_4^{2-}\}}{\{H_2PO_4^-\}} \tag{5}$$

$$K_{H_3PO_4} = \frac{\{H^+\}\{H_2PO_4^-\}}{\{H_3PO_4\}} \tag{6}$$

$$K_{MgH_2PO_4^+} = \frac{\{Mg^{2+}\}\{H_2PO_4^-\}}{\{MgH_2PO_4^{2+}\}} \tag{7}$$

$$K_{MgHPO_4} = \frac{\{Mg^{2+}\}\{HPO_4^{2-}\}}{\{MgHPO_4\}} \tag{8}$$

$$K_{MgPO_4^-} = \frac{\{Mg^{2+}\}\{PO_4^{3-}\}}{\{HPO_4^-\}} \tag{9}$$

The total concentration of the constituents of Mg, NH_4 and PO_4, denoting $C_{T,Mg}$, C_{T,NH_4}, C_{T,PO_4}, are the add-up ionic concentrations of their complexes and free ions as illustrated in eqns (10)–(12).

$$C_{T,PO_4} = [H_3PO_4] + [H_2PO_4^-] + [PO_4^{3-}] + [MgH_2PO_4^+] + [MgHPO_4] + [MgPO_4^-] \tag{10}$$

$$C_{T,Mg} = [Mg^{2+}] + [MgOH^+] + [MgH_2PO_4^+] + [MgHPO_4] + [MgPO_4^-] \tag{11}$$

$$C_{T,NH_4} = NH_3(aq) + [NH_4^+]\,. \tag{12}$$

The described thermodynamic equations incorporate pH value of the solution due to the presence of H^+ and OH^- when equilibrium constant of water ($K_w = 10^{-14}$) is known (eqns (13–14)).

$$[H^+] = 10^{-pH} \tag{13}$$

$$K_w = [H^+][OH^-] \tag{14}$$

provided the concentration of each component (C_i) and their respective valency (Z_i), bulk fluid ionic strength (I) and activity coefficient (g_i) can be determined by eqns (15) and (16). It is worthwhile pointing out that Davies equation was used to calculate activity coefficient [14, 15].

$$I = \frac{1}{2}\sum C_i Z_i^2 \tag{15}$$

$$-\log g_i = A{Z_i}^2\left[\frac{I^{1/2}}{1+I^{1/2}}\right] - 0.3I \tag{16}$$

where I is the ionic strength in mol/l, Z_i, the valency of the corresponding elements and A, the DeBye-Hückel constant, 0.493, 0.499, 0.509, 0.519 at 5, 15, 25, and 35°C.

Demonstrating the ionization fraction of Mg^{2+}, NH_4^+ and PO_4^{3-}, as $\alpha_{Mg^{2+}}$, $a_{PO_4^{3-}}$, $a_{NH_4^+}$, it can be defined as the quotient of free ion concentration and total concentration of each component (eqns (17–19)).

$$a_{Mg^{2+}} = \frac{[Mg^{2+}]}{C_{T,Mg}} \tag{17}$$

$$a_{PO_4^{3-}} = \frac{[PO_4^{3-}]}{C_{T,PO_4}} \tag{18}$$

$$a_{NH_4^+} = \frac{[NH_4^+]}{C_{T,NH_4}}\,. \tag{19}$$

Two types of solubility products can express the solubility of struvite crystals, which include conditional solubility product (P_{cs}) and absolute solubility product (P_{so}). P_{so} relates to solution properties such as ionization fraction (a_i), activity constant (g_i) and minimum struvite solubility product (K_{so}), whereas conditional solubility product relates to total concentrations (C_i). Solution of higher conditional solubility product than absolute solubility product ($P_{cs} > P_{so}$) refers to supersaturation. Equal numerical value of P_{cs} and P_{so} characterize the saturated condition, whereas $P_{so} < P_{cs}$ demonstrates undersaturation. The negative logarithmic value of minimum struvite solubility product (pK_{so}) applied in this thermodynamic modelling was 13.26 [1].

$$P_{so} = \frac{K_{so}}{a_{\mathrm{Mg}^{2+}} g_{\mathrm{Mg}^{2+}} a_{\mathrm{NH}_4^+} g_{\mathrm{NH}_+^-} a_{\mathrm{PO}_4^{3-}} g_{\mathrm{PO}_4^{3-}}} \tag{20}$$

$$P_{cs} = C_{\mathrm{T,Mgm}} C_{\mathrm{T,PO_4m}} C_{\mathrm{T,NH_4m}}. \tag{21}$$

Solution thermodynamic property specifies the state of saturation, amount of free ion concentrations, presence of ion complexes and state of precipitation. Precipitation of struvite occurs in supersaturated solution with the influence of pH value and initial reactant concentration [17]. The active concentration of each reactant (free ion concentration) and the rate of chemical reaction depends upon the thermodynamic driving force (b). When the free ion concentrations $[\mathrm{Mg}^{2+}]$, $[\mathrm{NH}_4^+]$ and $[\mathrm{PO}_4^{3-}]$ are identified from equilibrium thermodynamic relations, the thermodynamic driving force (b) can be figured out with known values of number of reactants (n), ion activity product (IP) and activity solubility product (K_a). The mathematical expressions of ion activity product (IP) and activity solubility product (K_a) are demonstrated in eqns (23) and (24), incorporating activity constants (g_i) and struvite solubility product (K_{so}).

$$b = nLn\left(\frac{IP}{K_a}\right)^{1/n} \tag{22}$$

$$\mathrm{IP} = g_{\mathrm{Mg}}[\mathrm{Mg}^{2+}] g_{\mathrm{PO_4}}[\mathrm{PO}_4^{3-}] g_{\mathrm{NH_4}}[\mathrm{NH}_4^+] \tag{23}$$

$$K_a = (g_{\mathrm{Mg}} g_{\mathrm{NH_4}} g_{\mathrm{PO_4}})^{1/n} K_{so} \tag{24}$$

Table I
Values of reaction constant for different complexes

Variables	Values	References
K_{MgOH^+}	$10^{-2.56}$	[21]
$K_{\mathrm{NH}_4^+}$	$10^{-9.25}$	[2]
$K_{\mathrm{HPO}_4^{3-}}$	$10^{-12.35}$	[22]
$K_{\mathrm{H_2PO}_4^-}$	$10^{-7.20}$	[22]
$K_{\mathrm{H_3PO_4}}$	$10^{-2.15}$	[23]
$K_{\mathrm{MgH_2PO}_4^+}$	$10^{-0.45}$	[23]
$K_{\mathrm{MgHPO_4}}$	$10^{-2.91}$	[23]
$K_{\mathrm{MgPO}_4^-}$	$10^{-4.80}$	[23]

Table II
Nutrient concentration (mg/l) of different piggery effluent ponds

Location of pond	Sample site details	pH	Mg	$\mathrm{NH_4^-N}$	$\mathrm{PO_4^-P}$	TP
Belconnen	Single pond	7.8	12.2	77.9	77.9	93.7
Byculla	Primary pond	7.9	20.3	314.2	10.6	53.4
Byculla	Secondary pond	8.0	17.7	292.5	9.2	34.3
Byculla	Final irrigation pond	8.3	50.7	34.5	4.4	20.5
Cabarlah Park	Primary pond	7.38	21.0	199.7	22.1	281.4
Cabarlah Park	Final irrigation pond	8.12	14.9	89.5	9.9	74.3
CEFN	Primary pond	7.7	32.0	224.1	8.3	110.0
CEFN	Secondary pond	7.9	36.2	228.3	7.3	50.4
CEFN	Final irrigation pond	8.4	33.0	196.5	5.7	51.1

$\mathrm{NH_4^-}$ N = Ammonia-nitrogen: $\mathrm{PO_4^-}$ P = Orthophosphate: TP = total phosphate.

A computer model was developed based on solution chemistry of magnesium, ammonium and phosphate. Solution pH and concentration of representative field effluent were used as model input. A trial and error and successive ionic approximation was applied to estimate saturation level. Presence of base (such as NaOH) facilitated increasing free ions of phosphate (eqn 25), thereby increasing the supersaturation [17].

$$PO_4^{3-} \Leftrightarrow HPO_4^{2-} \Leftrightarrow H_2PO_4^{-} \Leftrightarrow H_3PO_4 \,. \tag{25}$$

3. Field survey

As part of this research, Queensland Department of Primary Industry (QDPI)-Toowoomba conducted field survey on effluent characteristics of different pig farms in Queensland (Table II).

5. Modelling technique

This thermodynamic model was developed using process simulation software ‘gPROMS’ using Cabarlah Park Primary Pond data for model run-1. Field survey showed the molar concentration of magnesium, ammonium and phosphate concentration of 0.0000875, 0.011094 and 0.000233 M. To determine the effect of initial concentration (run-2), magnesium and phosphate concentration of 0.0001083, and 0.000359 M were used as model input; however, ammonium concentration remained the same as run-1. This technique was employed due to the limiting nature of magnesium and phosphate in the field effluent.

6. Batch experiments

Batch experiments were conducted using magnesium and phosphate concentration of 669.80 and 518.0 mg/l, respectively. Magnesium and phosphorus were analysed by ICP method. Ammonium analysis was ignored due to its volatile nature and nonlimiting field concentration (excess molar concentration with respect to the other two components). Analytical-grade $MgCl_2$ and $NH_4H_2PO_4$ of 2.44 and 3.36 g were used to make up synthetic solution using distilled water. Constant pH of 8.0 ± 0.2 was maintained using NaOH solution of 0.25 M concentration. Samples were collected and filtrated by 0.45 μm filter paper and thereafter preserved at 4°C temperature. Each of the batch experiments was conducted using 5.0 g of seeds of borosilicate glass grindings. Impeller speed of 50 rpm was applied for uniform mixing of solution. After each experiment, solution was kept undisturbed for 1 h to allow the settlement of struvite crystal. Thereafter, filtered struvite was dried at 60°C temperature for 24 h and dry struvite mass was measured using an electronic balance.

6. Results and discussion: Modelling

Solution of magnesium, ammonium and phosphate exists in very complex form and free ion concentration of specified components depends on the solution pH and concentrations. Figures 1 and 2 demonstrate the ionization fraction of struvite components. Ionization fraction of Mg^{2+} and NH_4^+ interpreted negative trend with respect to pH, whereas ionization fraction of PO_4^{3-} showed a positive trend. A fraction of the total components remains in free ion form, ($a_{Mg} \approx 1$), whereas a negligible portion of the total magnesium remained as $MgOH^-$

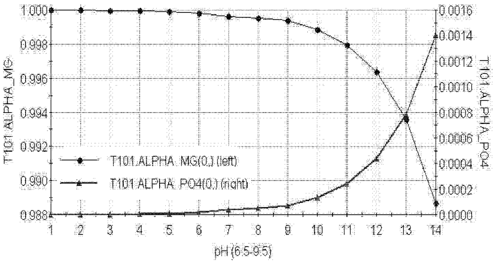

FIG. 1. Ionization fraction of free Mg^{2+} and PO_4^{3-} ions in solutions.

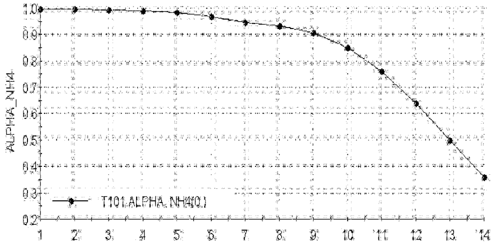

FIG. 2. Ionization fraction of free NH_4^+ ion in solutions.

ion in the specified range of pH (6.5–9.5). The free ion concentration of PO_4^{3-} is reasonably diminutive compared to Mg^{2+} ion, since total phosphate remains in complex form of $MgH_2PO_4^+$, $MgHPO_4$, H_3PO_4, $H_2PO_4^-$, HPO_4^{2-}, PO_4^{3-}, and $MgPO_4$. Major portion of ammonium remains as NH_4^+ below pH 9.0 (Fig. 2). The presence of different phosphate ions in basic solution, demonstrated in Fig. 3, showed an increase of free PO_4^{3--} ion due to deprotonation of HPO_4^{2-}, $H_2PO_4^-$ and H_3PO_4 followed by successive transformation of H_3PO_4 (eqn 25 [17]). The ionic concentration of Mg^{2+} decreased in basic phase; however, any significant increase of $MgOH^-$ concentration is merely small (Fig. 4). At or below pH 9.0, most of the ammonium remains as ionic NH_4^-, and the transformation of NH_4^- to NH_3 has a potential in basic solution (Fig. 5). The formation of $MgH_2PO_4^-$, $MgHPO_4$ and $MgPO_4^-$ were not observed in model response, since mathematically their possible molar concentrations were found to be pessimistic. A trial-and-error method of the simulation run found that $MgH_2PO_4^+$, $MgHPO_4$ and $MgPO_4^-$ did not exist in struvite solution system in the specified range of pH with illustrated component concentrations.

$$H_3PO_4 \overset{H^+}{\leftrightarrow} H_2PO_4^- \overset{H^+}{\leftrightarrow} HPO_4^{2-} \overset{H^+}{\leftrightarrow} PO_4^{3-} . \tag{26}$$

Theoretically, crystallization of struvite requires the solution to be supersaturated since the formation of nuclei and organization into definite structure is an energy transformation process from supersaturated solution [4]. In alkaline solution, absolute solubility product of struvite (P_{so}) decreased along with promoted value of ionization fraction product $(a_{Mg^{2+}} a_{PO_4^{3-}} a_{NH_4^+})$ and activity constant product $(g_{Mg^{2+}} g_{PO_4^{3-}} g_{NH_4^+})$, as described by eqn (27). The resultant cause of the above-mentioned promoted values is due to increase of free PO_4^{3--}; however, slight declination of free Mg^{2+} and NH_4^+ was also observed.

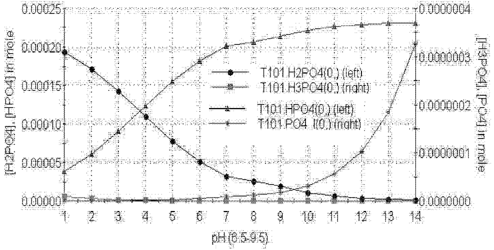

FIG. 3. Variations of concentration of different phosphate ions with pH in solution.

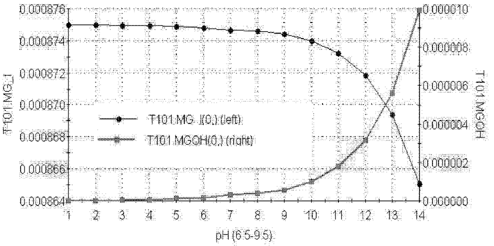

FIG. 4. Variations of concentration of different magnesium ions with pH in solution.

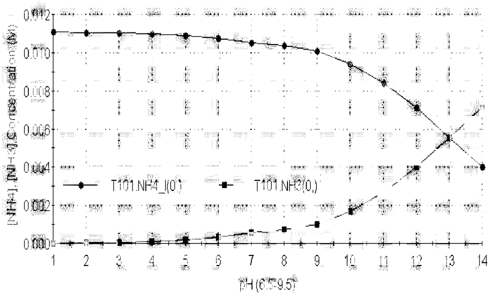

FIG. 5. Variations of concentration of different ammonium ions with pH in solution.

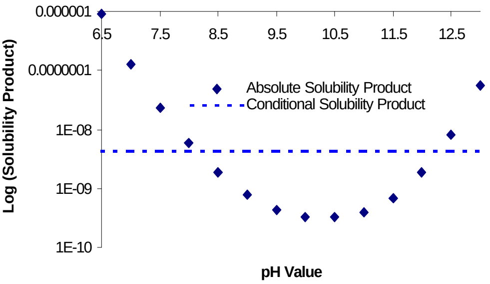

FIG. 6. Comparison of solution solubility products at different pH values.

$$P_s = \frac{K_{so}}{(a_{\mathrm{Mg}^{2+}} a_{\mathrm{PO}_4^{3-}} a_{\mathrm{NH}_4^+})(g_{\mathrm{Mg}^{2+}} g_{\mathrm{PO}_4^{3-}} g_{\mathrm{NH}_4^+})}. \tag{27}$$

Based on the model response (Figs 6 and 7), struvite crystallization occurred at pH 8.1, possessing lower value of absolute solubility product (P_s) than conditional solubility product (P_{cs}). For struvite solution, the effective concentrations that induce precipitation are free Mg^{2+}, NH_4^+ and PO_4^{3-} ions. The resultant increase of free Mg^{2+}, NH_4^+, PO_4^{3-} ion causes the decrease of other ion complexes in the system. The model predicted the pH for minimum solubility of struvite (10.5) was validated and confirmed by existing data [4, 5, 10, 12, 19]. Apparent pH of minimum solubility of struvite has been documented (9.0–11) in the quoted literature.

The other way of describing solution supersaturation is the thermodynamic driving force. The resulting thermodynamic force relating to pH in Fig. 7 (run-1) shows positive numerical value. Literature documented the formation of struvite at pH 8.1 from synthetic solution [5, 11]. It largely depends on the concentration and presence of other soluble components. Figure 7 (run-2) shows, respectively, the model response with total input concentration of ammonium, magnesium and phosphate of 0.011094, 0.0001083 and 0.000359 M. Field survey of different piggery effluent ponds (Table I) suggests that magnesium and/or phosphate are the limiting solution species in most of piggery effluent ponds. Comparison of runs-1 and 2 in Fig. 7 shows that the solution concentration significantly influences the thermodynamic driving force. A different supersaturation was observed due to the alteration of solution properties, more precisely due to the formation of different amounts of complexes of Mg^{2+}, NH_4^+ and PO_4^{3-}.

8. Results and discussion

Experiments conducted at constant pH of 8.0 ± 0.2 over a duration of 6 h recovered 80% of the Mg^{2+} and P concentration by way of struvite crystallization. The concentration of magnesium was reduced from 518 mg/l to 105 mg/l and that of phosphate from 669.8 to 88.7 mg/l during the experiment. The accumulated mass of dry struvite and seeds was 7.96 g. A second batch of experiments was conducted with initial pH of 8.1, with the same con centration and seeds mentioned above. The change of pH and its equilibrium state is demonstrated in Fig. 8.

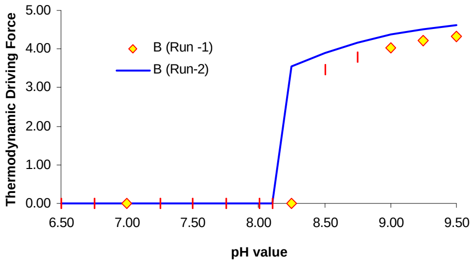

FIG. 7. Thermodynamic driving force of solution at different pH.

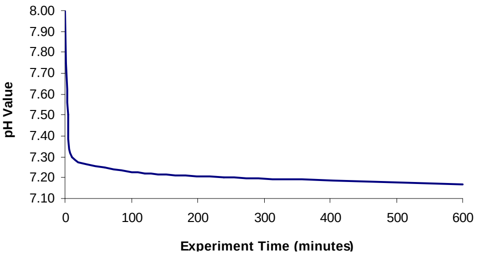

FIG. 8. Change of pH over the batch experiment.

The deposited dry mass of struvite was 0.66 g when the pH was not controlled at constant level, whereas 2.96 g of dry mass of struvite was deposited at constant pH. The significant difference of struvite mass was due to the difference of effective free ions of the system. Reduction of pH throughout the course of uncontrolled crystallization (Experiment-2) depleted free Mg^{2+}, NH_4^+ and PO_4^{3-} concentration, which reduced precipitation rate.

8. Conclusion

Solution chemistry plays an important role in struvite precipitation. Experimental investigation showed the possibility of recovering nutrients using crystallization technique when the solution is supersaturated. Thermodynamic model response shows that both solution pH and free ion concentration of dissolved species (magnesium, ammonium and phosphate) play most significant role in struvite crystallization.

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Nomenclature used in figures

ALPHA_MG	Ionization fraction of magnesium
ALPHA_PO4	Ionization fraction of phosphate
ALPHA_NH4	Ionization fraction of ammonium
[H2PO4]	Concentration of $H_2PO_4^-$ complex
[HPO4]	Concentration of HPO_4^{2-} complex
[H3PO4]	Concentration of H_3PO_4 complex
[PO4]	Concentration of PO_4^{3-} complex
[MG_I]	Free ion concentration of magnesium
[MGOH]	Concentration of $MgOH^+$ complex
[NH4]	Free NH_4^+ ion concentration
[NH3]	Concentration of aqueous NH_3
T101	Name of the developed process model