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KINETICS OF STRUVITE PRECIPITATION: EFFECT OF THE MAGNESIUM DOSE ON INDUCTION TIMES AND PRECIPITATION RATES

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ABSTRACT

The presence of white deposits in specific areas of wastewater treatment plants is generally the consequence of the spontaneous formation of a mineral called struvite. Struvite forms when the levels of phosphate, ammonium and magnesium naturally available in wastewater effluents reach a minimum molar ratio 1:1:1 under specific conditions of pH, temperature and mixing energy. Originally regarded as a phenomenon to control or eliminate, struvite has been lately identified as an alternative way of removing and recovering P from wastewater effluents and generating a product identified as an excellent base for the production of slow release fertilisers. Chemical and physical principles of struvite precipitation and the development of crystallisation technologies have been widely investigated. However, little interest has been given to kinetics of struvite precipitation. In the present work the kinetics of struvite formation have been investigated at both laboratory and pilot scale in synthetic solutions containing Mg^{2+}, NH_4^+, and PO_4^{3-} ions in a molar ratio 1:2:2 at room temperature. These different tests have used pH measurements to assess the impact of water chemistry on induction times, and more precisely the influence of magnesium levels on kinetic rates. Experimental results and kinetic calculations revealed that the control of the magnesium dose initially present in solution is decisive of the speed at which struvite nucleates.

Key words: struvite, precipitation, kinetics, magnesium dose

INTRODUCTION

Phosphorus (P) removal from wastewater effluents has become a real driver, especially since 1991 when European legislation made compulsory the removal of phosphorus from wastewater discharged in sensitive areas and imposed minimum P concentrations in wastewater effluents

to limit problems of eutrophication [1]. As a result of this strict legislation, numbers of new P removal processes have been developed and include biological removal technologies (Biological Nutrient Removal (BNR), Enhanced Biological Phosphorus Removal (EBPR)), chemical precipitation by metal salts and lately crystallisation of P as either calcium phosphate or struvite [2, 3]. Although the two first methods have been largely applied, the two others remain principally at an experimental stage. However the removal of phosphorus from wastewater effluents by struvite crystallisation is a promising solution as conversely to the other methods it presents the advantage of generating a fully recyclable product, the sales of which could benefit wastewater companies. Indeed, researchers have identified struvite ($MgNH_4PO_4.6H_2O$) as a slow release soil additive highly effective for foliar and soil application and as efficient as traditional mono calcium phosphates (MCP) [4, 5].

Struvite occurs spontaneously in solution when the content in Mg^{2+}, NH_4^+ and PO_4^{3-} reaches a 1:1:1 molar ratio under specific conditions of pH, temperature, mixing energy and presence of foreign ions [6]. Although conditions affecting success of P removal and recovery as struvite and their consequences on thermodynamics of precipitation have been widely investigated [7, 8], knowledge on kinetics of struvite precipitation, that is to say on rates at which struvite forms, is still limited [9]. Struvite crystallisation can be divided in two major steps: nucleation and crystal growth [10]. An important parameter of struvite precipitation is the time lapsed from the start of an experiment until the first particles occur, more commonly known as induction time [11, 12]. Struvite induction time is known to depend on the nucleation process involved in struvite crystal formation and on a multitude of parameters including supersaturation levels, mixing energy and presence of foreign ions [9, 13, 14]. Controlling precisely the effects of all these parameters seems then fundamental in the success of struvite crystallisation.

In the present work the influence of magnesium ions on kinetics of struvite crystallisation in synthetic liquors has been investigated. Initially used to estimate the magnesium impact on struvite induction time of struvite crystallisation, pH measurements have then been used to assess kinetics of struvite precipitation.

MATERIAL AND METHODS

Laboratory scale investigations

Struvite was precipitated from volumes of stock solutions of magnesium chloride hexahydrate ($MgCl_2.6H_2O$/ Fisher Analytical Reagent Grade, UK) and ammonium dihydrogen orthophosphate ($NH_4H_2PO_4$/ Fisher Analytical Reagent Grade, UK) diluted in deionised (DI) water into 250 ml flasks so that the final concentration of Mg after mixing ranged from 1.2 mM to 3.5 mM; the molar ratio Mg:N:P always equalled 1:2:2. Prior to mixing, the diluted solutions were adjusted to pH 9, by addition of sodium hydroxide (NaOH 0.01 N and 2 N / Fisher Analytical Reagent Grade, UK). The two solutions were then simultaneously mixed together in a 600 mL beaker, and the pH was measured periodically over 25 minutes. At the end of each test, the precipitate was filtered through 0.2 µm pore size Whatman membranes (Fisher scientific, UK) and dried at room temperature. The recovered product was characterised via XRD (Powder X-ray Diffractometer D5005, Siemens, Germany) and SEM (Scanning Electron Microscope XL 30 SFEG, Philips, The Netherlands).

Pilot scale investigations

A pilot scale reactor has been designed and developed at Cranfield University (Figure 1) to study struvite crystallisation on both synthetic and real liquors.

The process was composed of a 10 l reactor with three side sampling ports and a drain valve. A peristaltic pump simultaneously injected solutions of ammonium dihydrogen orthophosphate ($NH_4H_2PO_4$ / Fisher Analytical Reagent Grade) and magnesium chloride hexahydrate ($MgCl_2.6H_2O$ / Fisher analytical Reagent Grade) prepared by the dissolution of the corresponding solid compounds in deionised (DI) water.

Prior to mixing in the reactor, each solution was adjusted to pH 9 with sodium hydroxide (NaOH 2N/ Fisher analytical reagent grade) in stirred buckets. Both solutions were transferred in the reactor up to 9 L so that the final molar ratio Mg:N:P always equalled 1:2:2. A pH probe introduced in the reacting zone controlled the pH during the crystallisation process. The pH was recorded in the column shaped zone of the reactor every minute, over 1 hour of experiments.

An upward air-flow and a liquid recirculation were used and adjusted (10 to 5 l min^{-1} for air, and 1.5 to 1.35 l min^{-1} for liquid) to ensure the mixing and growth of the particles formed so that they were kept in suspension during the experiment. Struvite crystal harvesting was achieved through a drain valve situated at the bottom of the reactor. All standard experiments were conducted at room temperature over 60 minutes and repeated 3 times.

Kinetics of struvite formation

As struvite precipitates it releases hydrogen ions in solution causing a drop in pH, directly linked to rate of struvite formation and to the consumption of magnesium ions reacting to form struvite. In the system studied here, struvite formed according to the following equation:

$$Mg^{2+} + NH_4^+ + H_2PO_4^- + 6H_2O \rightarrow MgNH_4PO_4.6H_2O + 2H^+ \quad \text{(i)}$$

From equation (i), the rate of struvite occurrence can then be expressed as:

$$\frac{d[MgNH_4PO_4.6H_2O]}{dt} = \frac{1}{2}\frac{d[H^+]}{dt} = -\frac{d[Mg^{2+}]}{dt} = -\frac{d[NH_4^+]}{dt} = -\frac{d[H_nPO_4^{3-n}]}{dt} = Kr \quad \text{(ii)}$$

where: K is the reaction kinetic constant and r is the rate of crystallisation in mol l^{-1} s^{-1}.

The rate of disappearance of Mg^{2+} (k_{Mg}) ions can then be defined as:

$$-\frac{d[Mg^{2+}]}{dt} = k_{Mg}[Mg^{2+}] \text{ or } -\frac{d[Mg^{2+}]}{[Mg^{2+}]} = k_{Mg}dt \quad \text{(iii)}$$

And the rate of appearance (k_H) of H^+ ions is:

$$\frac{1}{2}\frac{d[H^+]}{dt} = k_H[H^+] \text{ or } \frac{1}{2}\frac{d[H^+]}{[H^+]} = k_H dt \quad \text{(iv)}$$

In the current work, mixing speed as well as $Mg:NH_4:PO_4$ ratios were kept constant and the initial pH set to 9 to leave Mg as the main parameter influencing the precipitation rates of struvite. Consequently, the rate of disappearance of Mg^{2+} ions equalled the rate of appearance of H^+, so combining equation (iii) and (iv) lead to the following expression

$$-\frac{d[Mg^{2+}]}{[Mg^{2+}]} = \frac{1}{2}\frac{d[H^+]}{[H^+]} \quad \text{(v)}$$

By integrating equation (v) between the initial time (t=0) and the final time (t= 25min or 60 min at pilot scale), it was then possible to link the magnesium concentration to the pH value recorded over time as by definition pH=-Log $[H^+]$ as followed:

$$-\int_0^t \frac{d[Mg^{2+}]}{[Mg^{2+}]} = \frac{1}{2}\int_0^t \frac{d[H^+]}{[H^+]}$$

$$Ln\,[Mg^{2+}]^t = Ln\,[Mg^{2+}]_0 - \frac{1}{2} Ln\left(\frac{[H^+]^t}{[H^+]^o}\right)$$

$$Ln\,[Mg^{2+}]^t = Ln\,[Mg^{2+}]_0 - \frac{1}{2} Ln\left(\frac{10^{-pHt}}{10^{-pHo}}\right)$$

$$[Mg^{2+}]^t = Exp\left(Ln[Mg^{2+}]_0 - \frac{1}{2}\left(\frac{10^{-pH_t}}{10^{-pH_0}}\right)\right) \quad \text{(vi)}$$

This expression was then used to estimate the evolution of magnesium concentration over time using the different pH values recorded during the various laboratory and pilot scale experiments. The kinetic constants of struvite formation were then assessed by fitting a first order kinetic model to the different sets of data, as according to previous studies struvite kinetics of reaction were found to follow this model [9, 15]. For pilot scale tests only the concentrations calculated in the initial stage of reaction were used to fit the model to compare them to lab-scale experiments and ensure that the constant determined corresponded to precipitation kinetics. The linear form of the modified expression of a first-order kinetics model given by Nelson *et al.* [9] has been used here. The form of the rate expression is:

$$Ln(C-C_{eq}) = -kt + Ln(c-C_0) \quad \text{(vii)}$$

where: C is the concentration of the reactant concentration at a moment t, Ceq the reactant concentration at equilibrium, C_0 the initial reactant concentration, and k the kinetic constant.

In the present study, if the rate of Mg^{2+} reaction follows this model, a plot of $Ln([Mg]-[Mg_{eq}])$ versus time should be linear with a slope of –k.

RESULTS AND DISCUSSION

Induction time

Initial experiments at both laboratory and pilot scale used changes in solution pH to indicate the start and rate of struvite precipitation, hence to determine induction time for different initial magnesium concentrations. The effects of varying the concentration of Mg ions in solution on struvite precipitation are shown in Figures 2a and 2b. The plots of tangents to pH curves obtained for both laboratory and pilot scale experiments illustrate that when the concentration in Mg ions increase, the rate at which the pH drops also increases, indicating a strong influence of the Mg dose on the reaction kinetics. In his study of struvite crystallisation kinetics, Nkansah [16] also concluded that rates at which struvite precipitation reached a steady state were closely linked to the concentration of solutions in Mg from which it was precipitated, with induction times decreasing from 150 to 15 seconds for respective Mg concentrations increasing from 2.337 to 5.827 mM in molar ratios Mg:N:P 1:2:2.

Determination of the period of time preceding the occurrence of first crystals at laboratory scale showed that an increase in initial magnesium concentration from 1.2 mM to 2.3 mM resulted in an approximately 10 fold reduction of the induction time (Table 1). These results are in accordance with previous studies that have demonstrated that the induction time for struvite crystallisation was mainly reaction controlled (*i.e.* pH and supersaturation dependant) [13]. For instance, Bouropoulos and Koutsoukos [11] also demonstrated the dependence of the induction time on solution supersaturation, hence Mg dose. For precipitation tests in synthetic solutions ($MgCl_2.7H_2O + NH_2H_2PO_4$) at pH 8.5 they reported similar trends as for an increase of Mg from

2.75 mM to 4 mM, the relative induction period decreased from 125 min to 6 min. However, times taken for struvite crystals to occur in the present system are higher than that observed by Bouropoulos and Koutsoukos [11]. These variations can be attributed to the difference in pH of precipitation and conditions of precipitation (i.e. composition of synthetic solutions, mixing energy, temperature and agitation). Indeed Nelson *et al*. [9] illustrated the effects of pH on struvite precipitation rates in anaerobic swine lagoon effluents by showing that rate constants varied significantly from 3.7 h^{-1} to 12.3 h^{-1} between pH 8.4 and 9.

At pilot scale, induction times were less affected by variations in magnesium concentrations than for laboratory tests (Table 1). For example, at the Mg concentration of 1.2 mM, the solution became cloudy after 12 minutes of reactor operation, while at 2.3 mM the first particles were visible only 6 minutes after the filling up start.

From the previous observations it is clear that the intensity of the drop in pH is linked to the concentration in magnesium initially present in solution and is characteristic of the speed at which the first crystals of struvite occur, consequently to the rate of struvite formation. Finding a relationship between pH and Mg concentration against time could then help to predict kinetics of struvite precipitation and determine rate constants of struvite formation under specific condition of precipitation.

Kinetics of precipitation

Equation (vi) has been used to assess the evolution of the magnesium concentration versus time from the different pH measurements shown in Figures 2a and 2b. Based on the literature [9, 15], the struvite formation reaction has been assumed to obey a first-order kinetic model with respect to Mg and H ions.

Figure 3 illustrates plots of Ln ($[Mg^{2+}]$-$[Mg^{2+}]_{eq}$) versus time for the different experimental conditions tested at laboratory scale. The different plots revealed that calculated Mg concentrations fit well to a first kinetic order model, where correlation coefficients (R^2) ranged from 0.92 to 0.99 over the range of initial Mg concentrations tested.

At pilot scale, good correlations of the magnesium concentration calculated to the first-order kinetic model were also obtained with R^2 values 0.92 to 0.98 over the range of initial Mg concentrations tested (*i.e.* 1.2mM, 1.64mM, 2.3mM and 3.5mM). The rate constants obtained from each plot are reported in Table 2.

Kinetic constants seem to follow a trend in that the higher the initial Mg concentration, the higher the rate constant and so the faster the precipitation. To illustrate, kinetic constants obtained at laboratory scale ranged from 9.7 to 15.7 h^{-1} for Mg increasing from 1.2 to 3.5 mM, while at pilot scale kinetic constants ranged from 2.4 to 9.6 h^{-1} between 1.2 and 2.3 mM.

Comparison of the laboratory and the pilot tests for similar initial concentrations showed that rate constants at pilot scale were significantly lower than those determined at laboratory-scale, especially for low concentrations in Mg. For an Mg concentration of 1.2 mM at both laboratory and pilot scale, kinetic constants were respectively 9.2 h^{-1} and 2.4 h^{-1} while at a Mg concentration of 2.3mM kinetic constants were 13.4 h^{-1} and 9.6 h^{-1} (Table 2). Nelson *et al*. [9] reported for anaerobic swine lagoon that lower kinetics constants are influenced by the time taken for the crystallisation to start, or induction time. As induction times observed for precipitation tests in the reactor were significantly longer than at lab scale (Table 1), this would explain the lower rate constants determined at pilot scale. Furthermore, although previous studies have minimised the influence of mixing energy on struvite crytallisation kinetics [13],

mixing energy in small volumes of solution such as in jar tests are thought to be higher than in the 10 l reactor agitated with air, hence accelerating the kinetics of precipitation.

When compared to rate constants observed in the literature, the values obtained here are in the range of those reported for struvite precipitation in real liquors. For example, in their study of struvite kinetics using sludge lagoon supernatants in a pilot fluidised bed reactor, Ohlinger *et al.* [17] determined a rate constant of 4.2 h^{-1} at a precipitation pH of 8.3, whereas Nelson *et al.* [9] found at laboratory scale a rate constant of 3.7 h^{-1} for struvite precipitated in similar media at pH 8.4 and initial Mg concentrations varying from 0.8 to 1.2 mM. Constant rates determined by Nkansah [16] for identical conditions of precipitation as the one presented here (*i.e.* Mg:N:P molar ratio 1:2:2/ identical stock solutions, mixing apparatus and speed) were similar to the one calculated here at the same Mg concentrations. To illustrate, for initial Mg concentrations of 2.3 mM and 3.5 mM the constant determined here were respectively of 13.4 and 15.7 h^{-1} while Nkansah [16] values were respectively of 11.3 and 13.3 h^{-1}.

Furthermore, by applying equation (vi) used here to predict the evolution of Mg concentration with time to the set of pH data reported by Nkansah [16], the results confirmed the use of a first-order kinetics model to describe struvite precipitation kinetics and indicated an excellent correlation between the concentration calculated at equilibrium and actual concentrations in Mg measured at equilibrium by Inductively Coupled Plasma Emission Spectroscopy (Table 3). To illustrate, for an initial Mg concentration of 2.3 mM the present model predicted an equilibrium concentration of 0.916 mM while Nkansah [16] measured an equilibrium concentration of 0.922 mM.

Although the kinetic model (i.e. first order model) observed in a scaled up reactor operated under similar chemical conditions of precipitation (Mg concentrations and associated Mg:N:P ratios) should be consistent with the one observed here, according to previous results a

significant increase in the time taken for struvite precipitation to occur should result in even lower kinetic constants justified by the time taken in a larger reactor volume for the solutions to become homogenised [18]. A scaling up of the type of reactor described here would then imply even longer reaction time when using liquors with comparable characteristics. However the development of a design tool based on the kinetic rates found here which would also take into consideration reactor volumes and composition of liquors would then allow predicting for a specific reactor the parameters that need adjustment to optimise struvite precipitation rates for a given size of reactor.

Overall the understanding of struvite kinetics of crystallisation is the key to the development and the implementation of full scale struvite crystallisation reactors to be used as alternative solutions to technologies currently operated for the removal of phosphorus from sludge liquors. In biological processes such as EBPR systems, when sludges are treated by anaerobic digestion some polyphosphates can be released via hydrolysis and the phosphorus contained in liquors can lead to an increase of phosphorus being re-injected in the activated sludge process [19]. In that case, struvite crystallisation can be used as an additional step prior to the recycling of sludge liquors, hence allowing removal of a significant part of the phosphorus recycled back to the process and generating a recyclable by-product as already achieved in Japan [20] conversely to chemical precipitation of P with metal salts which produces P rich sludge that can not be recycled industrially [21].

CONCLUSION

The result presented here showed that pH measurement can be used for the prediction of the kinetics of struvite precipitation. The drop in pH typically observed during struvite precipitation reflects effectively rates at which struvite crystals nucleate. The magnesium concentration as expected has a major impact on struvite kinetics, with higher concentrations leading to shorter induction times, hence faster rates of precipitation leading to production of fine crystals. The present paper also showed that pH measurements correlated well with magnesium concentrations, especially at laboratory scale. However, investigations at both laboratory and pilot scale demonstrated that struvite precipitation in synthetic liquors corresponded to a first order kinetic model as observed in previous studies, confirming then the reliability of investigating kinetics from pH measurements.

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Table 1. Struvite induction time at bench and pilot scale

	Induction time in beakers (min)		Induction times in reactor (min)	
	Eye visible	**Estimated from pH graphs**	**Eye visible**	**Estimated from pH graphs**
1.20 mM	5	5	12	12
1.64 mM	2	1.8	6.8	7
1.96 mM	< 1	0.8	6.7	7
2.30 mM	< 0.5	< 0.5	6	6

Table 2. Rate constants calculated for struvite solutions of various initial Mg concentrations

	Lab scale	Pilot scale
Initial [Mg] (mM)	**k (h^{-1})**	**k (h^{-1})**
1.20	9.4	2.4
1.35	10.7	
1.52	12.2	
1.64	12.5	7.4
1.76	13.1	
1.96	12.7	8.4
2.30	13.4	9.6
3.50	15.7	

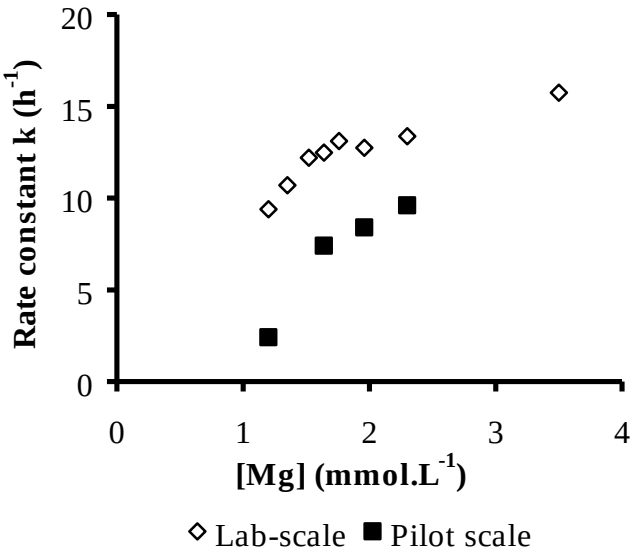

Table 3. Comparison between equilibrium concentrations measured by Nkansah [16], and the corresponding concentrations predicted.

	Nkansah [16]	**Current work**	
Initial [Mg] (mM)	Measured [Mg] at equilibrium (ICP analyses/ mM)	[Mg] calculated with the current model (equation7)	Error (%)
1.2	0.666	0.683	2.5
2.3	0.922	0.916	0.7
3.5	1.010	1.009	0.1

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