On precipitation of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$)

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ABSTRACT

Excessive input of N and P into water bodies causes eutrophication which leads to deterioration of aquatic environments and has adverse effects naturally and economically. It is therefore urgent to remove N and P from wastewater prior to disposal into inland and coastal waters. Among the various removal methods, controlled struvite precipitation is preferred. Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) precipitates uncontrollably under the specific conditions producing a scale deposit causing persistent problems in industries and wastewater treatment plants. The scale deposit clogs the piping system and impair plant equipment. On the other hand, thanks to its composition and properties, struvite is a potential fertilizer. In medical field, struvite is a common component of kidney stones. A number of process parameters govern the struvite precipitation. This paper briefly presents these parameters: pH, molar ratios, temperature, mixing, and presence of foreign ions. pH level is considered as the most important variable affecting the precipitation of struvite and the pH level: 9.5 to 10.5 is seen as the optimum. For an effective precipitation the molar ratios of the struvite components, i.e. Mg:N:P should be at least unity. With regard to struvite solubility, the effect of temperature, in the range of 21°C to 49°C, is conflicting, which is probably due to different experimental conditions. Whilst agitation is not regarded as a decisive parameter, the influence of foreign ions, notably divalent metal ions, on struvite morphology and change of crystal phases is significant.

Keywords: pH, phosphate, phosphorus removal, precipitation, struvite

Received: July 31, 2017; **Accepted:** September 10, 2017; **Published online:** October 26, 2017

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Citation: Muryanto, S. (2017). On precipitation of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$). *Journal of Science and Science Education*, 1(2), 21–29.

1. Introduction

Nitrogen (N) and phosphorus (P) are essential nutrients to life and many ecosystems. In excess amounts, however, these nutrients cause eutrophication of water bodies, i.e the reduction of oxygen due to uncontrolled growth of algae. As a result, the aquatic life is threatened and the water bodies becomes unsightly and unhealthy. The wastewater streams, especially those originating from household, agricultural and animal farming, are characterized by a high level of ammonia and P contents (Liu & Qu, 2017; Mpountas *et al.*, 2017; Deng *et al.*, 2006). Therefore, the effluents must be treated before discharging them into water bodies. A number of conventional methods exist to remove N and P from wastewater including biological nitrification and denitrification (Bergmans, 2011), microwave irradiation (Cho *et al.*, 2009) and struvite precipitation (Nelson *et al.*, 2003; Kataki *et al.*, 2016). Struvite precipitation, or struvite crystallization, as the term interchangeably used, has been extensively practiced for the removal of the environmentally harmful ammonium nitrogen ($\text{NH}_4\text{-N}$) and phosphorus (PO_4^-) from wastewater treatment plants, due to its high efficiency, simplicity and environmental sustainability (Darwish *et al.*, 2016; Kurtulus & Tas, 2011).

Struvite is a phosphate mineral that was first described in 1845 by the German chemist Georg Ludwig Ulex (1811–1883), who found this crystalline mineral inside the sewerage system in Hamburg, Germany. The mineral was named struvite by Ulex, in honor of the geographer and geologist Heinrich Christian Gottfried von Struve (1772–1851) of Hamburg (Ulex, 1845). Struvite can form a hard solid phase or scale deposited along the pipelines of the wastewater and sewage sludge treatment plants (Kurtulus & Tas, 2011). Struvite scale formation is a well recognized problem in the piping system of anaerobic sludge digesters, especially at the elbows and the suction side of pumps. Consequences of the struvite scaling are numerous including blockage of pipes, breakdowns of centrifuges, pumps, heat exchangers and other pieces of equipment in contact with anaerobically digested effluents (Doyle & Parsons, 2002; Le Corre *et al.*, 2007). Struvite consists of an equimolar proportion of Mg^{2+} , NH_4^+ and PO_4^{3-} , the essential components for plant growth, plus six molecules of water, with the chemical formula: $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ (magnesium ammonium phosphate hexahydrate). Due to its chemical characteristics many research has been done to investigate struvite with the intention to apply it as a potential fertilizer (Su *et al.*, 2014; de-Bashan & Bashan, 2004; Bergmans, 2011).

Struvite is also a common component of urinary tract calculi or kidney stones, either in human or in animals (Kurtulus & Tas, 2011). The stones are formed as a result of an infection in the urinary tract by microorganisms producing urease (Prywer *et al.*, 2012). Struvite reversibly precipitates in an equimolar concentration of Mg^{2+} , NH_4^+ and PO_4^{3-} at alkaline conditions, according to Eq (1) (Doyle & Parsons, 2002).

In Eq. (1), the values of n are 0, 1, and 2, respectively.

As common for the formation process of crystalline materials, struvite precipitation consists of two basic steps, i.e. nucleation and crystal growth. Nucleation is the formation of stable crystal embryos or nuclei. The nucleation step is subsequently followed by crystal growth where nuclei grow bigger by the deposition of solute molecules or growth units onto the surface of the nuclei. Both nucleation and growth are rate processes and defined as the precipitation kinetics. The kinetics of precipitation determine the ultimate characteristics of crystals and are governed by a variety of parameters. This paper briefly describes the parameters affecting struvite precipitation.

2. Influence of a number of process parameters on struvite precipitation

Precipitation of struvite is subject to a number of physico-chemical parameters: pH (Nelson *et al.*, 2003; Fang *et al.*, 2016), component-ion molar ratios (Nelson *et al.*, 2003; Wang *et al.*, 2005), intensity of mixing (Fang *et al.*, 2016), and presence of foreign ions (Muryanto & Bayuseno, 2014; Saidou *et al.*, 2015).

2.1 Influence of pH

During the precipitation of struvite, proton (H^+) is released (see Eq.1), thus lowering the pH level of the precipitating solution. Consequently, experiments on struvite precipitation are normally carried out at a certain pH range higher than neutral. Researchers have attempted to perform experiments on struvite precipitation at a low pH (Wang *et al.*, 2010; Hao *et al.*, 2008), but the crystals obtained were quite minimal rendering the process impracticable.

Song *et al.* (2007) used a range of pH from 8.0 to 12.0 and found that the optimum pH was between 9.5 and 10.5. Within this pH range the struvite formed was maximum, as indicated by the P removed from the solution as high as 93%. At pH >11.0 the struvite obtained decreased due to the formation of $\text{Mg}(\text{OH})_2$ and the evolution of free NH_3 (Crutchik & Garrido, 2016), thus reducing the Mg^{2+} and NH_4^+ ions (Hao *et al.*, 2008) available for struvite formation.

Experiments carried out by Saidou *et al.* (2009) showed that for the initial solution pH of 10, another phosphate mineral, namely $\text{Mg}_3(\text{PO}_4)_2 \cdot 22\text{H}_2\text{O}$ starts precipitating. Such formation is the results of the dominant species of phosphate ions in the form of PO_4^{3-} in the solution. In fact, varying the pH levels results in different species of phosphate ions: PO_4^{3-} , HPO_4^{2-} , H_2PO_4^- (Crutchik & Garrido, 2016; Bergmans, 2011; Saidou *et al.*, 2009; Andrade & Schuiling, 2001), and consequently generating other phosphate compounds alongside struvite. As observed by Le Corre *et al.* (2005), at pH >8.5 other compounds may precipitate such as Ca_3PO_4 and CaHPO_4 . Thus struvite precipitation is hampered by the presence of Ca. Furthermore, other magnesium compounds may also precipitate, such as Mg_3PO_4 and $\text{Mg}(\text{OH})_2$ (Hao *et al.*, 2008).

As regards the pH levels, precipitation of struvite is better performed within the metastable zone, i.e. between 8.0 and 10.0 (Ali & Schneider, 2006) so that precipitation predominantly occurs in a heterogeneous mode. As is well recognized heterogeneous precipitation promotes larger crystal size and less fines, rendering the process more efficient.

The effect of pH on struvite precipitation can also be observed in the change of the kinetic rates of precipitation. Crutchik & Garrido (2016) investigated the thermodynamic and kinetics properties of struvite precipitation and found that for three pH levels: 8.2, 8.5 and 8.8. the kinetic rates of precipitation increased as $1.21 \cdot 10^{-4}$, $1.25 \cdot 10^{-4}$, and $1.63 \cdot 10^{-4} \text{ mol m}^{-2} \text{ min}^{-1}$, respectively. Thus, the kinetics of struvite precipitation (and consequently its dissolution) are affected by slight variations of pH. As pH increases, the ionic activity product (IAP) also increases which results in the rise of supersaturation; and in turn, causes the rise in kinetics rates.

2.2 Influence of Molar Ratio

As shown in Eq. (1), the precipitation of struvite requires equal molar ratios of its components: Mg, N, and P. Different molar ratios have shown to influence several characteristics. Kozik *et al.* (2011) investigating the recovery of phosphate (V) from wastewater containing organic impurities found that $\text{PO}_4:\text{Mg}$ of 1:1.2 resulting in larger crystals than that of 1:1 ratios. It can be argued that as more Mg is available for the reaction, the more crystal units are generated, hence larger crystals may form. As reported by Merino-Jimenez *et al.* (2017), several studies have applied the ratios of PO_4 and Mg around 1:1.2 for optimum yields. Moreover, the concentration of component-ions has direct correlations with the saturation index (SI) of the solution, hence directly influences the nucleation and growth. Higher initial concentrations of P, keeping all other component concentrations and operating conditions constant, resulted in larger diameter of struvite crystals (Fang *et al.*, 2016). Fang *et al.* (2016) observed an increase of > 300% in diameter of the struvite crystals ($20.5 \mu\text{m}$ to $69.3 \mu\text{m}$) when the initial concentration of P was increased from 155 mg.L^{-1} to 930 mg.L^{-1} .

Struvite precipitation from agricultural wastewater is also known as a method to remove nitrogen (N). Yetilmezsoy & Zengin (2009) conducting the precipitation of struvite to assess the recovery of N from a poultry wastewater treatment observed a lesser N and COD removal when the ratio of Mg

applied was less than unity, i.e. not in equimolar ratio to the other two component-ions: NH_4 and PO_4 . Further, the removal increased as the Mg ratio was increased to 1.0, 1.1, and up to 1.2. It was therefore concluded that for an effective removal of N from wastewater through struvite precipitation, $\text{Mg}:\text{NH}_4:\text{PO}_4$ should be at least unity (Rahaman *et al.*, 2011). In most cases, agricultural wastewater is deficient in Mg, and consequently Mg must be added (Zamora *et al.*, 2017; Kataki *et al.*, 2016). MgCl_2 is preferred since this chloride salt has higher solubility compared to other magnesium compounds such MgO and $\text{Mg}(\text{OH})_2$ (Zamora *et al.*, 2017; Kataki *et al.*, 2016).

2.3 Influence of Temperature

Temperature mildly affects the reaction rates of struvite. Fang *et al.* (2016), investigating an unseeded precipitation of struvite using synthetic wastewater in a fluidized bed reactor, found that the average size of crystals increased steadily (from 65 μm to 69 μm) as the temperature was elevated from 21°C to 49°C. These findings seem contradictory to the established concept that higher temperatures would cause higher solubility with the implication that struvite is more difficult to precipitate (Le Corre *et al.*, 2009). It can be argued, therefore, that temperature affects the relative rates of diffusion as well as surface integration of particles or component-ions in a positive manner (Le Corre *et al.*, 2005). Hence, higher temperatures lead to an increase in crystal growth of struvite and subsequently its size.

Temperature is also known to affect the morphology and habit of struvite. As observed by Boistelle *et al.* (1983), struvite precipitated at 25°C assumed rectangular and prismatic shape, while at 37°C square and thick crystals were dominant. The change in morphology of struvite as affected by temperature was later confirmed by Babić-Ivančić *et al.* (2002) who indicated that at 37°C struvite transformed faster into newbryte ($\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$) than at 25°C. The change in shape, morphology and phases may influence the tendency of the struvite crystals to agglomerate and subsequently to form scale deposit. It is envisaged that struvite with rectangular and prismatic shape is more difficult to agglomerate than those with square and thick morphology.

Moussa *et al.* (2011) demonstrated that temperature affected induction time, t_{ind} , of struvite, i.e. "the time lapse between the mixing of the solutions containing struvite component-ions and the first measurable detection of struvite nuclei." Crystal nuclei are formed through aggregation of ions, hence it is obvious that higher temperatures shorten the t_{ind} , since at elevated temperatures ions may collide among themselves more readily and subsequently promote the agglomeration. The findings of Moussa *et al.* (2011), however, may go against this basic concept. At 14.5°C, t_{ind} was 15 min, but it was constant at 20 min for three different temp: 20, 25, and 35°C. A possible reason for the unfamiliar findings could be the experimental conditions selected.

2.4 Influence of Mixing

Fang *et al.* (2016), investigating struvite precipitation in a fluidized bed reactor, stated that "agitation influences the inter-solute mass transfer and the width of metastable zone." Thus, mixing affects mass transfer. If the mixing is slow enough, it may encourage the transfer of ions or crystal units from the solution onto the crystal surface. On the other hand, if the mixing is too high, the solutes may have difficulty to attach to the crystal surface. Hence, mixing may negatively correlate with the crystal growth. It can be concluded that the particle size decreases with the increase in agitation. In addition, higher agitation rates yield higher number of crystals but smaller in size due to the breakage of the crystals caused by the collision among the crystals or between the crystals and the agitator.

Mixing is required to obtain a more homogeneous precipitation solution that further promotes the precipitation; and this can be achieved through air bubbling (Saidou *et al.*, 2009) or mechanical agitation (Muryanto & Bayuseno, 2014). In a seeded precipitation of struvite, mixing may increase the operating cost compared to an unseeded precipitation (Batistoni *et al.*, 2005). This is understandable since for the effectiveness of seeds as “nucleating agents”, they have to be homogeneously distributed within the solution, thus evidently need agitation. In contrast, an unseeded precipitation needs no homogenizing of seeds.

Using the air bubbling method, Saidou *et al.* (2009) tested the effect of air flow rate on struvite precipitation. The air flow rates were varied from 10 L.min⁻¹ to 40 L.min⁻¹, and the amount of struvite precipitated increased by about 26%. It is apparent that higher flow rates assumed higher mixing and accelerated the mass transport which subsequently promoted the precipitation.

In most cases, however, agitation is not considered as a crucial parameter for struvite precipitation. Agitation is simply to ensure the homogeneity of the solution and accordingly to encourage faster reactions. Care should be taken that the agitation does not cause the breakage of the crystals (Muryanto & Bayuseno, 2014; Le Corre *et al.*, 2007; Suzuki *et al.*, 2007).

2.5 Influence of Foreign Ions

The presence of foreign ions, even in ppm amounts, has a significant effect on the precipitation of struvite. The effect can be evident, e.g. on the alteration of morphology (Saidou *et al.*, 2015; Muryanto & Bayuseno, 2014), crystal phases (Saidou *et al.*, 2015), as well as precipitation rates (Kabdasli *et al.*, 2017). Two divalent metal ions: Cu²⁺ and Zn²⁺, at 5.0 ppm each, individually affected the precipitation rate and morphology of struvite significantly (Muryanto & Bayuseno, 2014). Saidou *et al.* (2015) studied the effect of Cd²⁺, Al³⁺, and SO₄²⁻ on struvite precipitation using the degassing method. They showed that higher Cd²⁺ concentrations (≥ 10 ppm) enhanced the formation of an amorphous phase alongside struvite (Figure 1). As shown in Figure 1, with the addition of Cd²⁺ ≥ 10 ppm, the crystallinity of struvite reduces to amorphous phase. Thus, Cd²⁺ in ppm amounts could be an effective inhibitor for struvite precipitation. The addition of Cd²⁺ at 100 ppm is considered maximum for the following reasons. Firstly, the presence of metal ions such as Cd²⁺ in wastewater is regarded as impurities and seldom higher than a few ppm. Secondly, Cd²⁺ is added into the precipitating solution with the intention to effectively prevent struvite precipitation. Hence, adding Cd²⁺ in higher amounts is inefficient.

Through scanning electron microscope (SEM), the effect of divalent metal ions Cu²⁺ on the morphology of struvite is evident (Figure 2—Muryanto and Bayuseno, UNTAG and Diponegoro universities, joint research, 2012). As can be seen, the characteristic needle-like struvite appears jagged and rougher under the influence of Cu²⁺ (Figure 2b). Cu²⁺ is adsorbed onto the struvite crystal surface. Such adsorption occurs irregularly due to different energy level required to attach to the surface (Muryanto & Bayuseno, 2014). Thus irregular surface growth of the crystals may arise which results in the crystals being jagged and rough. Moreover, incorporation of foreign atoms such as Cu²⁺ into the crystal lattice could produce surface defects which then appear as a rough surface. The associated energy dispersive spectroscopy (EDX) also shows traces of Cu²⁺ (Figure 2b).

Figure 1. XRD pattern of struvite precipitated with varying Cd^{2+} amounts. Higher concentrations of Cd^{2+} (≥ 10 ppm) bring about more amorphous phase (Saidou *et al.*, 2015—an open access source).

Figure 2. SEM-EDX micrographs of struvite precipitated: (a) without Cu^{2+} ; (b) with 5.0 ppm Cu^{2+} . Under Cu^{2+} , the surface of the struvite crystals is rough and jagged (b).

3. Summary

A brief review on struvite precipitation allows the following conclusions. Firstly, excessive N and P input should be prevented to avoid the eutrophication of natural waters: both fresh water and marine. Secondly, uncontrollable struvite precipitation may take place under the specific conditions

giving rise to a hard scale deposit and subsequently causing concerns for industries and wastewater treatment plants. Thirdly, whilst struvite can be detrimental to plant equipment, it has the promising use as a fertilizer. Fourthly, a number of process parameters: pH, molar ratios, temperature, agitation rate, and presence of foreign ions govern the precipitation of struvite. Fifthly, pH level is considered as the most important variable affecting the precipitation of struvite, but agitation rate does not significantly affect the precipitation. Finally, morphology and crystalline phases of struvite is significantly influenced by divalent metal ions, such as Cu^{2+} and Cd^{2+} .

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