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To cite this article: Athanasius P. Bayuseno & Wolfgang W. Schmahl (2019): Thermal decomposition of struvite in water: qualitative and quantitative mineralogy analysis, Environmental Technology, DOI: [10.1080/09593330.2019.1615558](https://doi.org/10.1080/09593330.2019.1615558)

To link to this article: <https://doi.org/10.1080/09593330.2019.1615558>



Accepted author version posted online: 04 May 2019.
Published online: 10 May 2019.



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Thermal decomposition of struvite in water: qualitative and quantitative mineralogy analysis

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ABSTRACT

Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is a potential fertilizer mineral that can be obtained from wastewaters. When the ambient temperature changes, struvite may decompose in water and other phosphate-bearing minerals form instead. The wet decomposition may include complex mineralization, as the struvite crystal structure releases both water molecules and ammonia. An in-situ x-ray measurement for the wet transformation of the struvite is needed to get insight into the mineral formed and into the influence of the water temperature on the decomposition/remineralization. In this study, the X-ray diffraction (XRD) sample holder containing struvite and water in a sealed condition was heated to temperatures of 55 to 120°C for 24 h. Later the still sealed sample holder was exposed to the X-ray beam with the Debye-Scherrer transmission technique, and the diffraction pattern was analyzed by the XRD Rietveld method. With increasing temperature (<100°C), struvite first dehydrated to dittmarite ($\text{MgNH}_4\text{PO}_4 \cdot \text{H}_2\text{O}$). Moreover, a decomposition of struvite into an amorphous form of magnesium hydrogen phosphate has occurred as the XRD background increased dramatically and showed a structured profile with very broad intensity maxima. Furthermore, struvite transforms into dittmarite, newberyite, and bobierite when the sample was heated above 100°C. The outcome of this work is expected to add knowledge on the instability of struvite, which may occur in the fields of the wastewater treatment and in the bio-mineralization in the urine of animals and humans.

ARTICLE HISTORY

Received 27 June 2018
Accepted 29 April 2019

KEYWORDS

XRD method; thermal decomposition; struvite; dittmarite; newberyite

1. Introduction

Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is a common mineral that contains important components needed as fertilizers in agriculture. It can precipitate from wastewater, where precipitation can be induced by so-called MAP (magnesium, ammonium, and phosphate) crystallization techniques, where two main factors are controlled: the molar ratio of Mg: N: P and the pH value [1]. In many cases, the wastewater may contain an insufficient concentration of magnesium, such that magnesium needs to be added from external sources, such as $\text{Mg}(\text{OH})_2$ and MgCl_2 . The latter is commonly employed because of its high solubility in water. The presence of sufficient magnesium ions allows reacting with the available nutrients in the wastewater such as phosphorus and ammonium to form struvite as a precipitate. Furthermore, struvite precipitation requires an alkaline condition, which is controlled by the addition of alkali hydroxides (KOH or NaOH) or by pre-aeration to degas CO_2 in the MAP solution [1,2]. Correspondingly, struvite crystallization can occur in a broad diversity of environments that link to the processing method [3–6].

Further to struvite supersaturation [7,8], crystallization in the specific environment could be influenced by temperature [9], the rate of stirring [10,11], nature of phosphate materials [12], and the presence of foreign ions including calcium and copper [10,13,14] or sulfate [15]. The thermal stability of struvite [9] in water is related to the release of volatile components. Accordingly, struvite can decompose into other phosphate-bearing minerals, and this decomposition occurs depending upon the favourable conditions for the gradual release of water molecules and ammonia [16]. For instance, different phases may result from the struvite decomposition in nitrogen gas atmosphere or moist air, while the partial pressure of oxygen in urine may also control the decomposition of struvite [17]. Correspondingly, the transformation of struvite in saturated solutions also depends upon the rate of heating [18]. It was reported previously that struvite decomposition could be observed in ‘kidney’ stones, which had been heated slowly at temperatures below 40°C [16]. More rapid heating of struvite to temperatures around 80°C was also shown to promote struvite decomposition [18].

In case of struvite decomposition, the finding of new phases could be linked to the concentration of free ions (Mg^{2+} , NH_4^+ and PO_4^{3-}) within the mother solutions (artificial wastewater or urine) [2,10]. Accordingly, magnesium phosphates such as $\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$ (newberyite), $\text{Mg}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ (bobierite) and $\text{Mg}_3(\text{PO}_4)_2 \cdot 22\text{H}_2\text{O}$ (cattiite) could be formed [4]. Correspondingly, struvite (S) and newberyite (N) could be grown through the control strategy of their respective supersaturation ratios [7]. When the supersaturation ratio of N/S is less than 2, struvite will be the first crystalline phase to form. After struvite, however, newberyite will crystallize when the supersaturation ratio reaches $\text{N/S} > 2$.

Furthermore, the transformation of struvite to dittmarite ($\text{MgNH}_4\text{PO}_4 \cdot \text{H}_2\text{O}$) has been reported to occur in air at a temperature of 103°C [19] and in water at lower temperatures of 60°C [20]. In the thermal decomposition of what was originally struvite at ambient conditions, the remaining water and NH_4 is released at 235°C , which is followed by the formation of the pyrophosphate at 575°C [21,22], which becomes the most stable phase found in the high temperature. Nevertheless, an evolution sequence involving different minerals may be found between 235°C and 575°C . Furthermore, the temperature range of struvite decomposition and the obtained mineralogical phase depends on the atmosphere: nitrogen gas, air, or water [22].

More recently, the thermal decomposition of struvite has been investigated through material characterization methods, including thermoanalytical techniques (e.g., Raman microprobe spectroscopy, Infrared absorption spectroscopy) [2,22]. It has been demonstrated by thermoanalytical techniques that struvite decomposition occurred by the loss of five water molecules and subsequently loss of 1 water molecule during heating. Lastly, the process was followed by the loss of one water molecule together with 0.5 water molecules [2,22]. The resultant phases of its decomposition were largely dependent upon the partial pressure of water and heating rate. However, so far, the thermal decomposition of struvite in water has not been examined in detail by *in-situ* XRD in order to determine the forming phases [2,22]. The use of the conventional XRD method has a limitation in that some X-ray diffraction peaks in the diffractogram of multiple mineral phases could not be accurately identified. The drawbacks of superimposed peaks in traditional XRD for mineralogical quantitative evaluation could be solved by the use of the XRD Rietveld technique [23,24]. Most importantly, high-resolution powder diffraction using a Debye-Scherrer transmission geometry can provide better analytical XRD data on the phase transformation in struvite as a function of temperature. Moreover, use of the XRD Rietveld

method is critical to analyze the struvite stability and phase transitions in the complex water systems. Here, the Rietveld method relies on the availability of crystal structure data in a reliable database for phases of natural and synthetic materials [23].

The present research examines the stability and decomposition of struvite in water-sealed condition during heating to varying temperatures. The *in-situ* XRD analysis was aimed to add knowledge on the susceptibility of struvite to heat-induced decomposition, which is commonly found in wastewater treatment and the urinary system. Further, a direct mineralogical characterization (qualitative and quantitative) of magnesium-ammonium-phosphate in the water-sealed sample was required to understand the struvite stability and wet decomposition process that may similarly happen in a complex hydrothermal system, urinary tracts, and kidneys. Additionally, the quantitative mineralogical analysis was required for effective control in the phase composition of minerals in association with struvite decomposition. Accordingly, these characterization results would provide valuable insights into the mechanisms of the struvite decomposition at different natural and laboratory conditions. Eventually, this will improve our understanding of the ammonia and phosphorus recovery in wastewater and it has implications for the dissolution of kidney stones in the urine by thermal decomposition of struvite.

2. Materials and Method

2.1. Sample preparation

Struvite crystals were grown by a simple precipitation method using chemicals of analytical grade, namely $0.025\text{ M NH}_4\text{H}_2\text{PO}_4$ (VWR Chemicals) and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (Fluka). For the stock solution, these powder materials were dissolved in distilled water with equimolar ratio of MAP reactants (1:1:1). A solution pH of 8.0 was set-up by dropwise addition of 50% w/w KOH (Merck) in a glass beaker followed by mechanical stirring for 20 minutes. This resulted in a white colloidal suspension. Subsequently, the suspension of solid particles was filtered through a $0.22\ \mu\text{m}$ membrane paper filter, and the precipitate obtained on the filter was then dried for 24 h in air at room temperature.

For the *in-situ* XRD measurement, the fine struvite powder (about 1 mg) was dispersed in a small amount of water. The substances were vigorously mixed and dispersed into the specially built XRD sample holder for transmission X-ray measurement. The XRD sample holder consisted of two sealable circular stainless steel plates with a central hole of 1 cm diameter for X-ray transmission. The bottom plate was covered with a

high-density polyethylene foil to protect the stainless steel from the chemicals. The top plate was covered by a polyimide film.

Before XRD measurement, the sealed XRD sample holder was placed in a 50-ml glass beaker of water, and the beaker was heated from room temperature to final temperatures of 55, 70, 80, 90, 100, 110 and 120 °C for 24 h in a Fisher Scientific Isotemp Muffle Furnace. These temperatures were selected according to the expected phase changes as a function of heating [22]. Samples were subsequently removed from the beaker and directly measured in the x-ray diffractometer. In this way, the samples were allowed to cool to room temperature during mounting on the diffractometer.

2.2. Characterization of mineral phase using XRD

The X-ray diffraction pattern was collected by an STOE-STADIP diffractometer in transmission (Debye-Scherrer) geometry using Mo-K α_1 (radiation $\lambda=0.70926$ Å) and a generator of 50 kV/30 mA. Data were recorded between 3 and 50 2θ with step size 0.01°. For better accuracy in counting statistics, the same aforementioned procedure was repeated eight times in the sample across to the same 2θ range, with a total recording time of 16 hours. The eight diffractograms were summed up and then utilized for the qualitative and phase quantitative analysis by the Rietveld method. Identification of crystalline substances was initially performed by the MATCH software package. The identified crystalline phases of the search match method were then verified by the Rietveld refinement method with Program X'Pert plus 1.0 (Philips Analytical B.V.). The crystal structure model for the Rietveld refinement was obtained from the literature (AMCSD-American mineralogist of crystal structure database) [25].

In the Program of X'Pert plus, profile fitting routines to get a calculated pattern of different phases can be systematically adjusted by a user-friendly interface. The developed artificial model was subsequently employed for a better match with the observed XRD profile of the sample under analysis. Up to 7 different phases could be integrated into the study. After obtaining a good profile fitting between the measured and calculated pattern, the scale factor derived from the refinement process was then used to calculate the proportion of the different crystalline phases or minerals present. In this study, only the identified crystalline minerals were quantified, while the amount of amorphous material was not determined. In some instances, the signal from the amorphous phase was subtracted directly from the data of any sample. This procedure was required for confident identification of a particular mineral because the relatively high amorphous signal in the background

tends to obscure minor peaks. With the XRD Rietveld method, the amounts of all crystalline phases present in the sample could be quantified simultaneously. In this method, the weight percentages of the phases were estimated using the X'Pert plus 1.0 software, on which the obtained scale factor of each phase was used to calculate the weight fraction of each crystalline phase. Accordingly, the major and minor crystalline phases could be identified by this method. The total of the weight percentages of the crystalline phases would be 100%. The detailed discussion of the Rietveld refinement method is presented elsewhere [23,26].

3. Results and Discussion

3.1. Thermal decomposition of struvite

Before undertaking an analysis of the heated samples, X-ray diffraction patterns of the pure struvite (S), and the empty sample holder were examined (Figure 1). The X-ray diffraction pattern of the S powder sample

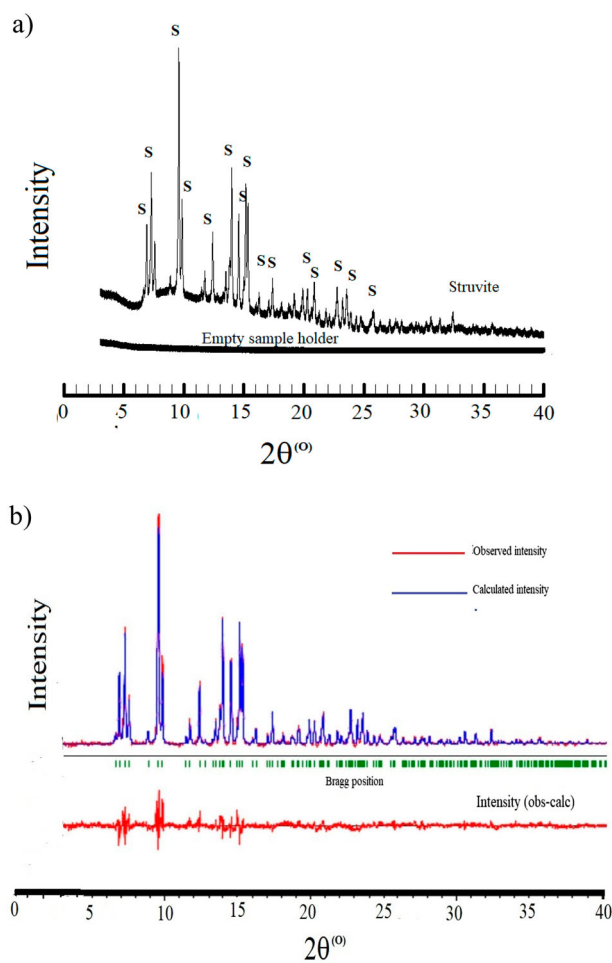


Figure 1. (a) X-ray diffractograms of struvite (S) (top) and empty sample holder (bottom); (b) XRD Rietveld refinement plot of struvite.

shows the characteristic peaks corresponding to those of the struvite standard pattern in the powder diffraction file [PDF#71-2089]. The results of the XRD Rietveld analysis also confirmed that the X-ray profile pattern of the crystalline solid fits very well with that of the struvite model as shown in Figure 1(b). Struvite crystallizes in the orthorhombic system (space group $Pm2_1n$) with the lattice parameters $a = 6.9335 \text{ \AA}$, $b = 6.1422 \text{ \AA}$, and $c = 11.1784 \text{ \AA}$ [27]. Moreover, the Kapton® Polyimide films of the empty sample holder shows no significant signals of amorphous phase [28]. After collecting the X-ray diffraction patterns of the unheated samples, the heated samples were directly measured by XRD and the diffraction patterns are presented in Figure 2.

The XRD patterns in Figure 2 indicate a transition of struvite to an amorphous state at some temperature between 55 and 70°C (Figure 2(a)). Here, there was a prominent, broad hump of background intensity on which the sharp Bragg peaks of the crystalline phases are superimposed. The amorphous phase appears to show a

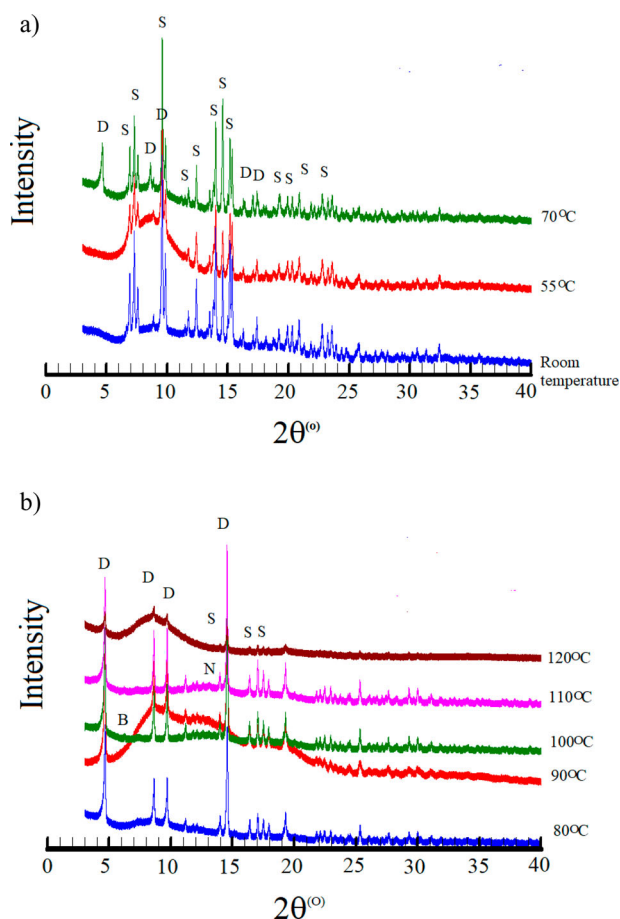


Figure 2. X-ray diffractograms of the mineral formation with different temperatures applied for heating samples; (a) from a room temperature to 70°C, and (b) from a room temperature to 120°C. Notes: dittmarite (D), struvite (S), bobierite (B) and newberyite (N) respectively.

particular short-range order structure similar to the crystalline phases as the crystalline peaks are in the same region as the diffuse hump of intensity from the amorphous phase. In the same temperature range between 55 and 70°C, dittmarite starts to form. The XRD Rietveld quantitative analysis, indicates 81.1 wt% of struvite and 19.9% of dittmarite at 70°C (Table 1). The determination of more precise transition temperatures requires measurement of diffraction patterns in smaller temperature steps and with different treatment times.

Further, an XRD measurement series was performed on the struvite samples heat-treated for temperatures of 80-120°C which were subsequently analyzed to gain a better understanding of its thermal stability. The XRD patterns of all measured samples are presented in Figure 2(b). With increasing temperatures from 70°C, broad peaks of an amorphous phase between 3 and 15° 2θ developed and eventually centred around 6-12° 2θ at the temperature of between 70 and 80°C. At the subsequent temperature of 90°C, only peaks of dittmarite are present. Conversely, with increasing temperature from 90 to 120°C, the characteristic of XRD peaks for struvite, bobierite (B) and newberyite in the range of 5-15° 2θ gradually diminished, whereas the intensities for prominent peaks of dittmarite are shown. The Rietveld analyses confirmed that at 90°C a small amount of dittmarite and mainly amorphous phase are present. At 100°C, the amorphous phase has crystallized to mainly dittmarite (about 92 wt. %) with a small percentage of struvite (3.6%), and bobierite (4.8%) (Table 1). Newberyite occurs at even higher temperatures when dittmarite decomposes (Table 1). We attribute the presence of struvite in the samples treated at 100°C and above to a retrograde reaction during cooling. Also, amorphous phases play a major role in the decomposition of struvite. Struvite decomposition at low temperatures (<55°C) leads to an amorphous phase from which dittmarite crystallizes upon heating. When dittmarite decomposes again an amorphous phase is formed. Further work should be done to complete the XRD quantitative analysis with internal standard to quantify the amorphous phase and SEM analysis of the amorphous phases.

3.2. Significant implications of the study

Struvite could be precipitated in wastewater treatments and also may be formed in urine as 'urine sand', and in urinary tracts or the kidneys as 'kidney stones'. When the ambient temperature changes, struvite may be decomposed into other phosphate-bearing minerals through the continuous discharge of ammonia and water [2]. More recently, the struvite formation and decomposition under wet and dry heating for a

Table 1. XRD quantitative results of crystalline phases from the heated samples at different temperature.

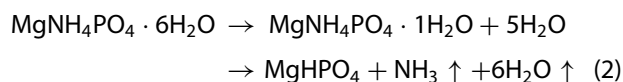
Mineral	Wt. %	Heating temperature °C						
		55	70	80	90	100	110	120
Struvite		100	81.1(3) ^a	22.0(7)		3.6(4)	2.2(5)	18.5(3)
Dittmarite			19.9(2)	78.0(4)	100	91.6(5)	92.9(3)	63.8(2)
Newberyite							2.9(5)	12.9(3)
Bobierite						4.8(3)	2.0(2)	4.9(8)

^aFigures in parentheses indicate the least-squares estimated standard deviation (esd) referring to the least significant figure to left.

prolonged time has been discussed in detail [16]. Many phosphate minerals could be possibly formed as a result of struvite decomposition with the ambient temperature changing. This experiment has demonstrated that struvite begins to decompose already at temperatures of 55°C during 24 h, where the thermal decomposition of struvite was controlled by the water dehydration and release of ammonia. This result agrees very well with the previous work reported in the literature [2,16]. Accordingly, the following equation has been proposed for the struvite decomposition reaction (Eq. 1) [16]:



Based on the TGA and DTGA data for synthetic struvite, about 51% of the original mass was reported to be lost by heating [16]. This mass loss could be related to the formation of amorphous MgHPO_4 , which subsequently decomposed to $\text{Mg}_2\text{P}_2\text{O}_7$. Furthermore, the loss of crystal water (theoretical mass loss 11.6%) may correspond to the formation of dittmarite and amorphous MgHPO_4 according to;



Presumably, the temperature of the solution influenced the release of ammonium and water, thereby resulting in a coexistence of struvite and other mineral phases; dittmarite, newberyite, and bobierite during struvite decomposition in the solution [16]. However, many contradicting experimental results have been reported in the literature regarding the effect of temperature on the struvite decomposition [29,30]. The temperature is suggested to have a negligible effect on ammonia removal from struvite between 25 and 40°C, while the temperature of 30°C was reported for a maximum struvite solubility. However, we observed a significant effect of temperature on struvite decomposition at temperatures of 55°C and above [16].

Because the heating of struvite was carried out in the sealed condition, most of the ammonia was possibly changed into NH_3 species at these high temperatures. Correspondingly, most of the ammonia is supposed to be recovered into struvite and dittmarite, while the remaining water molecule could be transferred

through the formation of newberyite, bobierite and the amorphous phases (possible MgHPO_4) at the temperature studied [28]. Newberyite is a phosphate mineral containing no ammonium. This mineral is commonly formed in association with other phosphates (e.g., struvite and hannayite $[(\text{NH}_4)_2\text{Mg}_3\text{H}_4(\text{PO}_4)_4 \cdot 8(\text{H}_2\text{O})]$). Also, the favourable formation of this newberyite mainly occurs at lower pH ($\text{pH} < 6$) [31,32]. This experiment has shown that a minor amount of newberyite could be produced. However, newberyite could be formed at higher temperatures, where the transformation reaction from struvite to newberyite may occur through a solution-mediated process, especially below a neutral pH [33–35]. A small amount of newberyite was obtained during this study, most likely because the solution conditions and the system used was most favourable for dittmarite formation. The formation of X-ray amorphous phases can also be inferred from the strong and structured background intensity found in this study.

It has been shown that the heating of struvite in the water-sealed condition at temperatures of 50, 60 and 80°C, lead to the transformation reaction from struvite to bobierite as a consequence of a gradual release of ammonium ions from the struvite structure [2]. In this way, ammonia could be released, resulting from the continuation of the hydrolysis of the magnesium ammonium salt with precipitation of newberyite, and finally, as the more stable bobierite [36]. However, the heating of the struvite in the closed system in the present study gave a simultaneous release of water molecules and some ammonia from the struvite structure. The XRD of the sample heated at 90°C for 24 h, in a small amount of water, indicates that dittmarite and an amorphous phase reach a maximum amount at this temperature, while newberyite and bobierite peaks become invisible with increasing temperature (Figure 2). The results suggested that the available phosphate in the limited water would influence the extent of struvite formation and, hence, recovering ammonia from the struvite structure. In general, the exact heating temperature obtained in the study would contribute to the discussion regarding the early stages of struvite decomposition. This correct temperature is also required for dissolution of kidney stone [22,37]. Eventually, the result of the study may add the knowledge on designing novel struvite-based fertilizers.

Most significantly, this experiment has demonstrated that the struvite effectively decomposed in the wet conditions employed by heating it to temperatures of 55–120°C for 24 h. It is supposed in the present study that these characterization results could contribute to the understanding of struvite decomposition behaviour for ammonia and phosphorus recovery in the area of wastewater treatment using a hydrothermal method. It was demonstrated that the hydrothermal method containing hot waters at varying temperatures from 50 to 120°C along with their cooling methods could be used to dissolve mineral from ammonia and phosphate concentrations in the hydrothermal solution [38].

Also this study provides the experimental approach and technical insights for evaluation of phosphorus recovery in the area of wastewater treatment through struvite crystallization and the possible reuse of struvite as a fertilizer. In particular, the thermal instability, phase transition reactions and decomposition of the minerals of the struvite system could be related to the particular temperature conditions. In the anaerobic digestion process of wastewater, for example, digester effluent must be treated at the temperature range of 50–60°C, for the thermophilic digestion of sludge. In Europe, the subsequent treatment of sludge, including an optional pasteurization system is commonly performed at 70°C for over 30 minutes before it is returned to mesophilic conditions (36–38°C) [39]. Thus, the design of the crystallizer requires a comprehensive approach to define the temperature for struvite stability and purity. Instead of struvite, dittmarite could be precipitated at high temperature conditions, as it also contains the MAP nutrients, and it can be converted to struvite by immersing in water or storage at moist conditions at room temperature. Additionally, the use of struvite or dittmarite as a fertilizer should have the capability of providing sufficient nutrients for the plant without building up a concentration, and it may be susceptible to leaching or fixation, which is mainly dependent upon in situ temperature, available nutrients, and water.

Additionally, effective treatment of struvite stones in humans or animals requires a comprehensive approach to eliminate all stone fragments and prevent stone recurrence. Importantly, the proposed treatment may rely on data on temperatures at which struvite could be eradicated. Our experimental results also offer significant insights into ways of relieving from the pain of kidney stones by inducing warm heating of the kidney region. In particular, struvite stability in the urine environment is likely a function of temperature. This experiment has shown that the struvite decomposition started at a low temperature of 55°C, and possibly lower, and largely depended upon the heating rate. Both ammonia and

water molecules are gradually released from the struvite structure as a function of temperature, rather than as a distinct step. Correspondingly, the relief from the pain of kidney stones may potentially be carried out through warming in the kidney region for a particular time. In this way, the proposed treatments for nonsurgical candidates of a kidney stone may combine treatments, in which warm heating of struvite is applied, followed by dissolution therapy [40].

4. Conclusion

The decomposition of struvite wet conditions yielded the release of both ammonia and water molecules from the crystal structure of struvite with increasing temperature and resulted in the formation of X-ray amorphous and crystalline phases other than struvite. The heating of struvite below 100°C in the presence of liquid hydrous solution yielded the release of five water molecules from the struvite structure, and the solid phase into the monohydrate, dittmarite. In this way, dittmarite was obtained as the only crystalline phase, next to an amorphous solid, at a temperature of 90°C. Advantage could be taken of the temperature conditions for the decomposition of struvite to dittmarite to intentionally form this latter phase in the field of P- or MAP recovery from wastewater.

Moreover, struvite heated at elevated temperatures (>100°C) in the presence of originally a small amount water, for 24 h, released ammonia from the solid phase, which was partially transformed into newberyite, and bobierrite. A more exact determination of struvite and dittmarite decomposition temperatures in water or hydrous atmosphere could help to improve the phase-purity of the phosphate bearing-mineral resulting from the decomposition. Temperature is also an essential parameter in the application of struvite or dittmarite as a fertilizer, relating to their ability to provide the plant with sufficient nutrients without forming a concentration susceptible to leaching or fixation.

Acknowledgments

The study was supported by the KAAD (*Katholischer Akademischer Austausch Dienst*) Bonn, Germany for a sabbatical on leave study program at LMU Munich. The authors also wish to thank Miss. Stefanie Hoser for helping to perform lab experiments.

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