



Research Article

Struvite Precipitation and Phosphorous Removal from Urine Synthetic Solution: Reaction Kinetic Study

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Abstract

Phosphorus, like oil, is a non-renewable resource that must be harvested from finite resources in the earth's crust. An essential element for life, phosphorus is becoming increasingly scarce, contaminated, and difficult to extract. Struvite or magnesium ammonium phosphate ($MgNH_4PO_4 \cdot 6H_2O$) is a white, crystalline phosphate mineral that can be used as a bio-available fertilizer. The main objective of this research is to indicate the most important operating parameters affecting struvite precipitation by means of chemical reaction kinetics. The present study explores struvite precipitation by chemical method under different starting molar ratios, pH and SSR. It is shown that an increase of starting $Mg:PO_4:NH_4$ with respect to magnesium (1.6:1:1) strongly influences the growth rate of struvite and so the efficiency of the phosphate removal. This was attributed to the effect of magnesium on the struvite solubility product and on the reached Super Saturation Ratio at optimum starting molar ratio and pH. It was also shown, by using chemical precipitation method that the determined Super Saturation Ratio (SSR) values of struvite, at 8, 8.5, 9, 9.5 and 10 are 1.314, 4.29, 8.89, 9.87 and 14.89, respectively. These are close to those presented in the literature for different origins of wastewater streams. The results show that SSR, pH, and starting molar ratio strongly influences the kinetics of precipitation and so phosphorous removal to reach 93% removal percent, 5.95 mg/l as a minimum PO_4 remained in solution, and 7.9 g precipitated struvite from feed synthetic solution of 750 ml. The product was subjected to chemical analysis by means of EDIX-FTIR, SEM and XRD showing conformity with published literature. First-order kinetics was found to be sufficient to describe the rate data. The rates increased with increasing pH and so SSR and the apparent rate constants for the reaction were determined. © 2015 BCREC UNDIP. All rights reserved.

Keywords: Struvite; Solubility Constant; Reaction Kinetics; Crystallization; Human Urine

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1. Introduction

Phosphorus is an important material, making a major contribution to agriculture and industrial development [1,2]. Within conven-

tional wastewater treatment, several pathways are known to remove phosphorus out of the wastewater; the recovery product then being a phosphorus-rich water or sludge stream. Within the concept of wastewater design, we find a stream that is very low in volume, yet very high in phosphorus: human urine. A technique proven to be a successful method to recover phosphorus from strength phosphorus-

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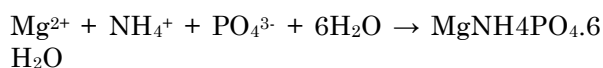
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rich streams is the precipitation of Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) [3].

Human urine is rich of valuable plant nutrients which improves agriculture crops and plant growth. Even though, direct application of human urine as fertilizer can be dangerous to human life due to the presence of some pathogens. The major pathogens excreted in the urine can cause: typhoid, paratyphoid and bilharzias in human beings.

Phosphate can be recovered as struvite from human urine offers combined purpose of removal and recovery of phosphorus. Then further nutrients can be treated and reused after biological treatment of urine solution. Whilst Struvite can be a problem in wastewater treatment plants due to its deposition on the inner surface of pipes, pumps and other equipments in the plants, it is a product of commercial value [4]. Struvite is a known and applied slow-release fertilizer, well applicable to crops in soils with relatively low pH. For plants that need an initial high dose of P followed by a continued slow P release, for example container plants, a product called enhanced struvite is already on the market: struvite treated with phosphoric acid to achieve a modified form of DAP (di-ammonium phosphate, one of the most applied fertilizers in the world) [5, 6]. These researches showed that phosphorus recovery in the form of struvite is technically feasible and economically viable. Chemical conversion to struvite may be seen as a better alternative to these conventional technologies because it would involve removing both ammonia-nitrogen and phosphate-phosphorus in the form of a precipitate that can be recovered as valuable byproduct.

Synthesis of struvite crystals from diluted water solutions of Mg^{2+} , NH_4^+ and PO_4^{3-} ions can be included into modern processes of phosphorus recycling-an important technology currently developed worldwide [7]. The general idea of the technology depends on selective removal of phosphate ions from the complex post-industrial liquid wastes, municipal sewage or manure solution by their precipitation in a chemical form of sparingly soluble complex inorganic salt $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ - magnesium ammonium phosphate (MAP), struvite as shown in the following equation [8, 9]:



Several scientists in previous work [10] focus on the kinetics of struvite formation 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. It was revealed that the control of the magnesium dose initially present in solution is decisive of the speed at which struvite nucleates. It was found [11] also that struvite precipitation kinetics with varying Mg:P ratio reveal that the higher the ratio (in the range of 1.0 -1.6), the better is the ortho-P removal efficiency. The rate constants are found to be 0.942, 2.034 and 2.712 h^{-1} for Mg:P ratios of 1.0, 1.3 and 1.6, respectively. The experimental observations for kinetic study of struvite precipitation with different stirrer speeds clearly show that the mixing intensity used had little effect on the intrinsic rate constants. K values found to be 2.034 and 1.902 h^{-1} for 100 and 70 rpm, respectively. The authors in [12] studied the influence of five process parameters on the kinetics of struvite precipitation in synthetic swine wastewaters. The responses were the kinetics of phosphorus (P) removal, the struvite precipitation rate and the dissolution rate of amorphous calcium phosphates (ACP). This study showed that the P-removal was complete in less than 1 h and was influenced positively by the added MgO and initial $\text{Mg:Ca} > 1.5$ induced a complete dissolution of ACP in 1 h. Synthetic urine was used as a readily available proxy for real urine for determining the factors which affect the recovery of struvite from urine.

In this paper, we study the effect of process parameters as super-saturation ratio (SSR), pH of synthetic urine solution and $\text{Mg:PO}_4 : \text{NH}_4$ molar ratio at different precipitation time and their effect on kinetic rate constant. We give some indications, which parameters affect struvite precipitation. Knowing these effects will facilitate the implementation and understanding of struvite precipitation unit from urine on a bigger scale. The rate law describing a chemical reaction can be deduced from experimentally measured time-concentration data for one or all of the reactants [13] kinetic rate methods, such as differential, integral and initial rate-methods, are commonly used to abstract kinetic parameters for developing rate laws [14, 15].

The ability to predict struvite precipitation potential from human urine is an important-consideration for designers and operators to determine the feasibility and economics of nutrient recovery as struvite, and for the subsequent design and operation of reactors for struvite precipitation. For good quality control, it is essential to know the best conditions under which struvite precipitation is likely to occur.

2. Material and methods

The experiments were performed to determine the effect of various parameters on remained phosphate, magnesium, and ammonium concentration in the supernatant synthetic urine solution.

A synthetic urine solution was prepared by dissolving 2800 mg of potassium dihydrogen phosphate (KH_2PO_4), 1000 mg of ammonium-chloride (NH_4Cl), 652 mg magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), 10000 mg soya broth, 4350 mg potassium chloride (KCl), 1100 mg creatinine, 25000 mg urea, 2300 mg sodium sulfate (Na_2SO_4), 4600 mg sodium chloride (NaCl), and 652 mg $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in one liter of distilled water for 3 hours on a hot plate magnetic stirrer model DRAGON Lab MS7-H550-pro, with a stirring power till 1500 rpm and heat till 550 °C. For all experiments synthetic human urine was used, fully hydrolyzed by storage for 14 days in a KIRIAZI - No Frost freezing unit 98 liters (-6 °C).

Precipitation experiments were carried out in a 1-L Erlenmeyer flask with 3 neck for sampling system, condenser and temperature controller, in which 750 ml of synthetic solution was used. A magnetic stirrer with 50 rpm was used for mixing after addition of needed masses of magnesium chloride, ammonium chloride, and potassium dihydrogen phosphate to adjust ratio between Mg to P. The precipitation pH was varied with precipitation time and the filtrated solution was analyzed for magnesium, ammonium and phosphorous. The pH of the solution was monitored continuously with a pH probe. Solution pH was adjusted to required pH value from 8-10 by adding 5 N NaOH solution drop-wise till required pH was reached. Precipitation was found to occur instantaneously. After reaching the required pH, the solution was stirred at 100 rpm for 60 minutes, and samples were withdrawn at 0, 5, 10, 15, 20, 30, 45, and 60 minutes which instantaneously acidified with a diluted hydrochloric acid solution (0.1 M) to stop the reaction in the samples and to evaluate the remaining phosphate, ammonium and magnesium concentration. All experiments were performed as shown in Figure (1) at constant precipitation temperature of 20 °C. Another set of experiments was performed at constant optimized pH, and the starting precipitating molar ratio between magnesium, ammonium and phosphorous was varied with different precipitating time, and concentration of phosphorous (PO_4) was recorded for all samples.

Then the sample withdrawn (50 ml) was filtered using 0.6 μm Whatman filter paper to recover the precipitate, which was dried in oven at 104 °C for 16 hours and weighed in a sensitive balance AE-ADAM (PW124), $d=0.0001$ gm (Max 120 gm.) made in UK. The filtrate liquid and dried solid crystals were subjected to full chemical analysis. Solid crystals were subjected to high resolution scan electron microscope (SEM) JEOL 5410 scanning electron microscope (SEM) at 20 kV and XRD crystallography model Bruker 8 advance, Cu-K α , Target with secondary mono chromator $\lambda = 40$, $m\text{A} = 40$, made in Germany. Filtrate liquid was analyzed for Mg^+ , NH_4 and PO_4^{3-} using Hardness reagents on spectrophotometer model DR 2000 by (CaCO₃), phenate and stannous chloride-methods and the spectrophotometric measurements were performed at 522 nm, 630 nm, and 690 nm for Mg, NH_4 , and PO_4 , respectively.

The data for phosphate concentration over the course of the reaction were fitted with zero, first-, second-, and third-order decay models. The adequate model is the one that accurately predicts the initial concentration with a high correlation coefficient value.

3. Results and Discussions

3.1. Reaction Kinetic Study for the Effect of pH on Concentration of Remained Phosphorous

The effect of different pH on struvite precipitation represented as remained phosphorous in filtrate solution was recorded at different reaction time in Figure (2). It shows that the increase in pH strongly assisted reaction conversion and so struvite precipitation. The minimum reached phosphorous concentration was 33.32 mg/l representing only 60.15 % removal at pH of 8 and reaction time of 20 minutes. On the other hand, 8.58 mg/l as phospho-



Figure 1. Experimental Set-Up

rous were remained at pH of 9.5 giving about 89.73% removal or precipitation as struvite. This removal percentage represents the amount of phosphates precipitated as struvite and the rest remained still dissolving in solution. As the pH was increased to reach 10, the amount of phosphates remained in solution decrease to reach 7.943 mg/l which shows that economically the added chemicals to reach a pH of 10 are not compensated with the increase in precipitated amount of struvite from 89.73% to be 90.5%. It was obvious from this family of curves that the increase in precipitation time above 20 minutes has an adverse effect on remained phosphorous as it shows slight increase in its concentration after reaching a minimum value due to phosphate dissolution after being precipitated.

It was shown from the above figure, that the variation of phosphorus concentration in the

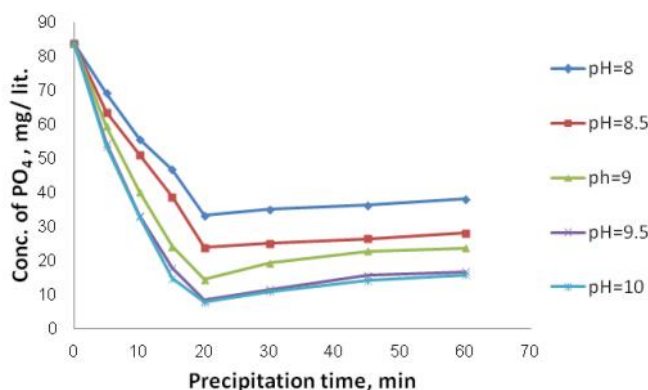


Figure 2. Variation of phosphate concentration with the precipitation time

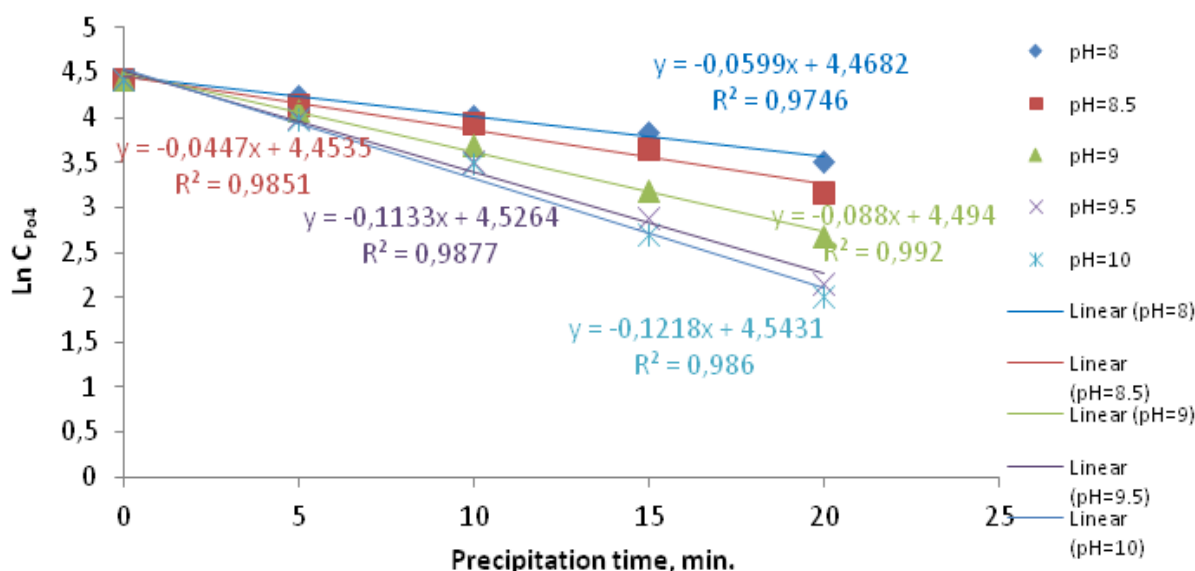


Figure 3. Linearization of the first order kinetic according to Equation (3) for determining the kinetic constants for synthetic solution with different pH values in the 1st interval.

filtrate with time. Typical exponential curves were obtained in all cases. A rapid diminution of phosphorus concentration was achieved during the first 20 min. As demonstrated in the literature, first order kinetic model represents the struvite formation in aqueous solutions.

In this present study, the kinetics of struvite precipitation was investigated. Zero-, second-, and third-order models and it did not give satisfactory fit to the experimental data with a reasonable *R*-square value. The kinetic data were fitted to a slightly-modified, first-order kinetic model, as proposed by researchers [16]. The model expresses the relationship between the disappearance of a reactant ($-dC/dt$) and its concentration (Equation (1)). A constant of proportionality, *k*, referred as the rate constant, is introduced. By mathematical manipulation of Equation (1) and integration, it generates the linear form of the first-order reaction kinetics (Equation 3). As described by the first order reaction kinetics, a plot of $\ln(C)$ against time generates a straight line with slope $-k$ and intercept of $\ln(C_0 - C_{eq})$.

$$-dC/dt = k(C) \quad (1)$$

By integrating Equation (1) and ordering the terms, the following linear form of the first order rate equation was obtained (Equation 2):

$$-\ln(C) = k t + \text{constant} \quad (2)$$

$$-\ln C = k t + \ln C_0 \quad (3)$$

where C_0 is the initial concentration of the reactant, assuming first-order reaction kinetics, a plot of $-\ln[(C-C_e)/(C_0-C_e)]$ against time should

give a straight line with slope of K [15] and intercept equal to zero. According to Equation (1), the value of C_e can be obtained from the intercept with the abscissa at $-dC/dt$ equals zero when this parameter is plotted versus the phosphorus concentration remaining in the filtrate [17]. By integrating Equation (2), it is possible to obtain the production of struvite as a function of the reaction time becomes Equation (4).

$$(C_0 - C_e) - (C - C_e) = (C_0 - C_e) - (C_0 - C_e) e^{-Kt} \quad (4)$$

The process for struvite formation through phosphorous concentration shows two different

intervals, the first was the precipitation of struvite presented by C_{PO_4} remained in solution. This interval from 0 - 20 min was called precipitation time, and the interval from 20 - 60 min was nominated as dissolution of phosphorous. The rate of precipitation and dissolution are shown in Figures (3) and (4), respectively.

It was clear from Figure 3 that the values of K was 0.059 min^{-1} at pH of 8 and then it increases to 0.088 min^{-1} at pH of 9 and further increase to be 0.113 min^{-1} which shows that the optimum pH will be 9.5 as the rate constant shows slight increase corresponding to pH of 10. On the other hand, it was clear that almost all the above data at both intervals fit to the

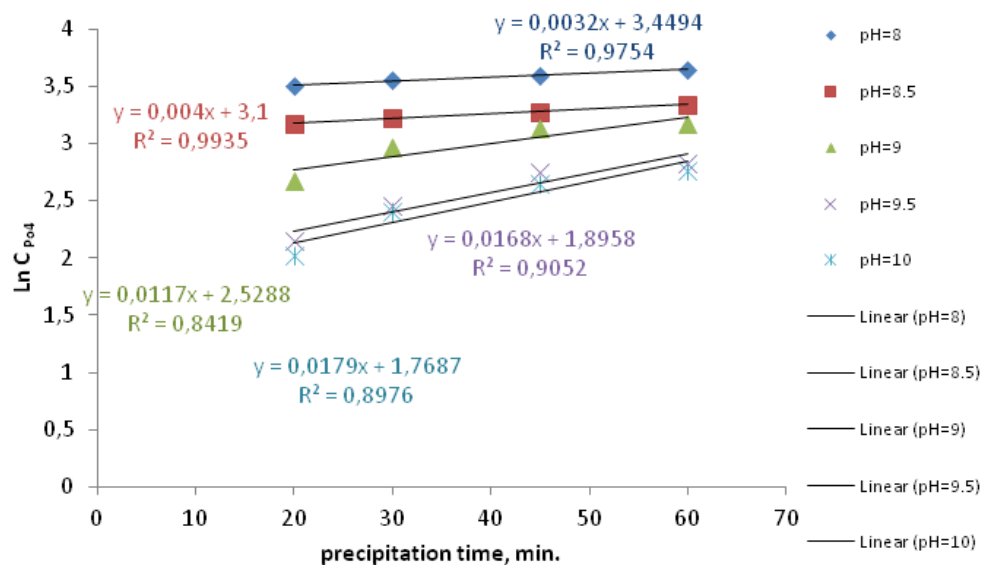


Figure 4. Linearization of the first order kinetic according to Equation (3) for determining the kinetic constants for synthetic solution with different pH values for the 2nd interval.

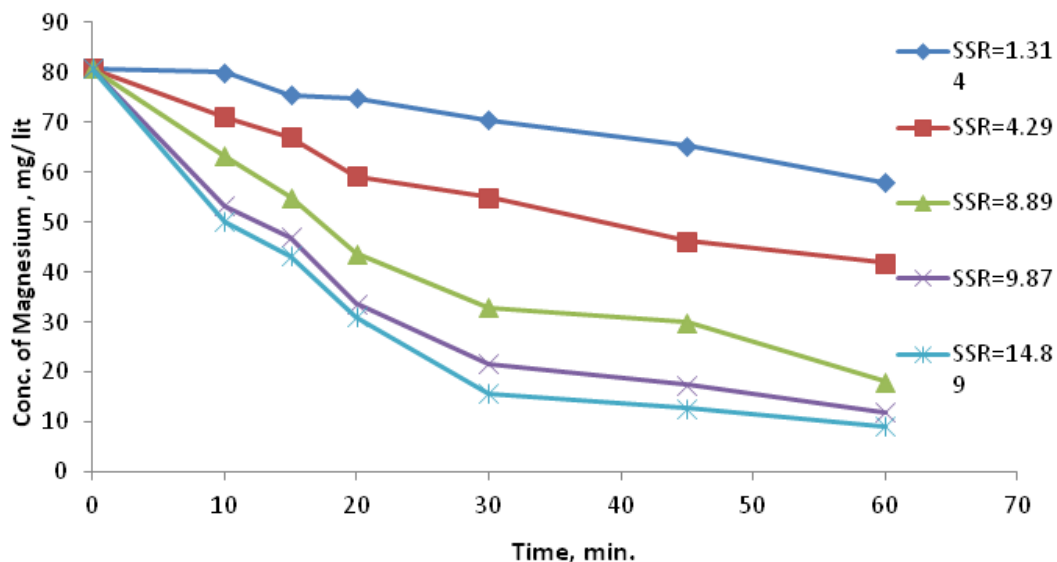


Figure 5. Effect of precipitation time on remained magnesium concentration at different Super Saturation Ratio (SSR)

first-order reaction kinetics model and shows a regression of about 0.9 which is a good fit.

We can conclude from Figure (4) that the increase in phosphorous concentration with the increase in precipitation time was of great concern for pH from (9-10). But for pH of 8 and 8.5, the concentration of phosphorous above 20 minutes were not varied over reaction extent. This can be attributed to precipitation of struvite was enhanced by the increase of medium alkalinity above pH of 9. In other words, the rise in concentration of phosphorous due to decomposition of formed struvite is taken into consideration as the medium pH was raised above 8.5. This can be explained with the fact in almost all literature that the rise in reaction conversion takes place as the alkalinity was increased to range from pH 9-10. So the decomposition also occurs obviously for pH 9-10. The above interpretation was strongly assisted by the rate constants shown in Figure (4) which increases from 0.003 to 0.017 min⁻¹ with a good fitting regression around 0.9.

3.2. Influence of Super-saturation Ratio on Magnesium Ion Concentration Remained in Solution

Super-Saturation Ratio (SSR) is defined as the ratio of conditional solubility product (P_s) to the equilibrium conditional solubility product (P_{seq}). Conditional solubility product represents the solubility product in actual conditions and equilibrium counterpart means its value at equilibrium state. Therefore, $SSR = P_s / P_{seq}$ and the conditional solubility product is expressed as: $P_s = [C_{TMg}] [C_{TPO_4}] [C_{TNH_3}]$.

An increase in the concentrations of the any constituent ions would increase the conditional solubility product, whereas at a higher pH, the values of P_{seq} would decrease. So the prevailing SSR in the process fluid can be increased by increasing the concentration of struvite constituent ions or by increasing the pH of the bulk solutions. In this study, the SSR of the reaction solutions were changed by adjusting the pH value. Magnesium is another important constituent ion for struvite precipitation. Hence, it also affects the supersaturation level and the progress of precipitation process. To investigate the effects of SSR on the formation of struvite, the disappearance of the Mg ions were monitored during the experimental run. As shown in Figure (5), the results clearly indicate that SSR plays an important role in the struvite precipitation kinetics. With increasing SSR, the removal of Mg increases with the equilibrium being achieved faster for the SSR

ranging from 9.87 to 14.89.

It can be concluded from Figure (5) that reached equilibrium in remained magnesium concentration was greatly influenced by SSR as the precipitation time increased over the first 30 minutes. But after 30 minutes, a slight decrease was achieved as the reaction progressed. A minor decrease in starting magnesium ion concentration and so struvite precipitation was shown at lower SSR values ranging from 1.314 to 8.89. The minimum concentration of magnesium reached at SSR 9.48 was 21.6 mg/l which gives about 73% removal as struvite at 30 minutes precipitation time. This percentage was increased to reach 81% removal at SSR 14.89 and precipitation time 30 minutes. In this work, operating conditions were optimized by combining Figures (2) and (5) to show that phosphorous recovery through struvite precipitation will be achieved at pH of 10, SSR of 14.98, and 20 minutes precipitation time. Although from Figure (4), it was clear that 30 minutes shows a minimum in Mg ions concentration but in Figure (2) any further increase above 20 minutes at pH from 9 to 10 will decompose struvite giving rise in PO₄ ions remained in solution. The same for pH and SSR, It was obvious that Figure (2) recommends pH 9.5 as an optimum compared to minimum PO₄ ions remained at pH of 10, but for Figure (5), the reached removal percent in Mg ions was remarkably increased from 73.5 to 81%.

The predicted initial value of Mg ions concentration deviates from the experimental value by indicating from the values of the correlation coefficient [18, 19], the first-order model was found to fit the experimental data the best (see Figure (6)). It gives reasonable prediction of the initial concentration value of Mg of about 76 mg/L. The observed first-order rate constants, half-lives and the corresponding experimental conditions are given in Table (1).

The time required for half of the original concentration of magnesium ion to be converted into struvite was approximately 144, 60, 27, 18 and 15 min at SSR 1.31, 4.29, 8.89, 9.87 and 14.89, respectively. The results indicate that the rate of reaction increased as the SSR increased and the reaction rate was slowly to reach 143 minutes half life for SSR 1.3.

3.3. Influence of Starting Precipitation Ratio on Recovered Struvite as Phosphorous Concentration

The starting precipitating ratio between phosphorous, ammonium and magnesium was proposed in literature to be one of the control-

ling parameters affecting struvite precipitation. The effect of precipitation time on the concentration of remained phosphorous in solution after struvite precipitation was studied as shown in Figure (7). The effect of starting molar ratios was studied at the small range recommended in literature [10-12].

The effect of starting precipitation ratio was studied at constant optimized parameter of pH 10, SSR of 14.89 against PO₄ concentration remained in solution to govern its probable dissolution once again to minimize struvite decomposition. In this study, one can conclude that the effect of starting precipitating ratio plays a minor role on struvite precipitation and so phosphorous removal as shown in Figure (7). The remained PO₄ ions at optimum precipita-

tion time 20 min was 5.95 mg/l for Mg:PO₄:NH₄ = 1.6:1:1 which gives about 93% removal as struvite as shown in Figure (9) for XRD confirming that all precipitates are about 100 % struvite. This aforementioned ratio was decreased to reach 90.54%, 89.93% at starting ratios of Mg:PO₄:NH₄ = 1:1.1:1 and 1:1:1.

A considerable decrease in concentration was observed during the first 20 min of reaction, as shown in Figure (7) while the phosphorus concentration increased slowly after this time. Straight lines were obtained with linear regression coefficients higher than 0.90 as shown in Figure (8).

Moreover, the increase in starting molar ratio with respect to magnesium molar ratio contributed to the increase in the phosphorous re-

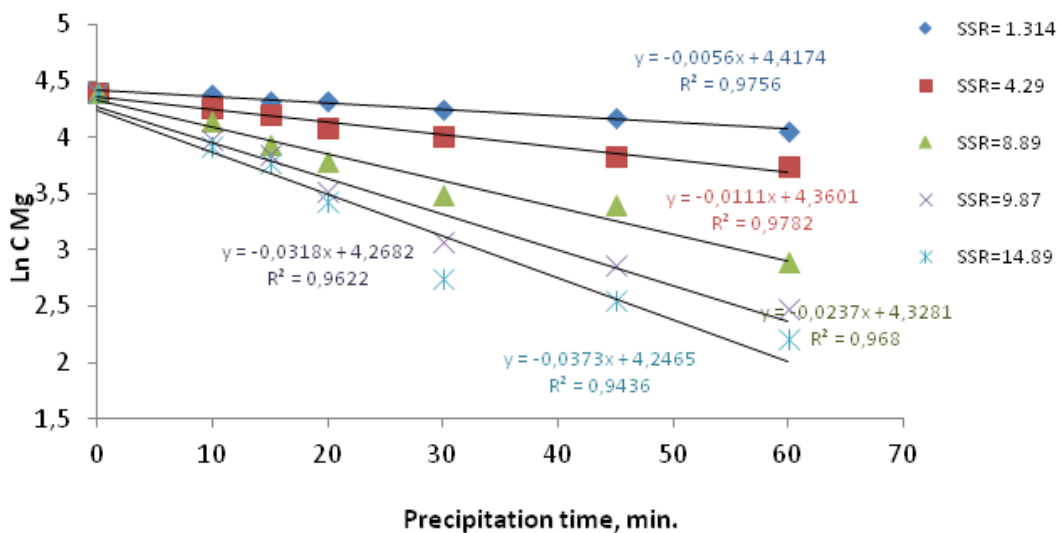


Figure 6. Linearization of the first order kinetic for determining the kinetic constants for synthetic solution with different SSR values with Mg concentration.

Table 1. Values for rate constants, half-lives, and experimental conditions

Rate constant, min ⁻¹	Half-lives, min.	Experimental condition
0.005	144	SSR=1.31, Mg:PO ₄ :NH ₄ =1:1:1
0.011	60	SSR=4.29, Mg:PO ₄ :NH ₄ =1:1:1
0.023	27	SSR=8.89, Mg:PO ₄ :NH ₄ =1:1:1
0.031	18	SSR=9.87, Mg:PO ₄ :NH ₄ =1:1:1
0.037	15	SSR=14.89, Mg:PO ₄ :NH ₄ =1:1:1

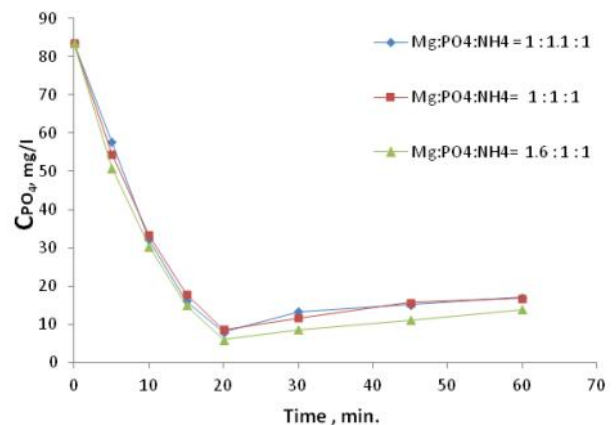


Figure 7. Effect of precipitation time on remained phosphorous concentration in supernatant synthetic urine solution.

moval rate. Scientists [20] proposed second order kinetic reaction for the precipitation of Struvite at different reaction parameters. While for this study, the data shows a good fitting with the first order reaction kinetics with accepted regression coefficients. The rate constant K increases with the increase of starting molar ratio to be 0.023 min^{-1} compared with 0.016 min^{-1} at equimolar starting ratio.

Different operating conditions yield and phosphorous concentration at optimum SSR, and precipitation time for different starting precipitation ratio are expressed in Table (2). It was obvious that the maximum struvite yield

Table 2. Struvite yield obtained at optimum operating parameters.

SSR	pH	Precipitation time, min	Minimum PO ₄ , mg/l	PO ₄ : Mg: NH ₄	Yield of struvite, g in 750 ml feed stream
14.89	10	20	8.58	1:1:1	4.98
14.89	10	20	5.92	1:1.6:1	7.9
14.89	10	20	7.908	1.1:1:1	5.57

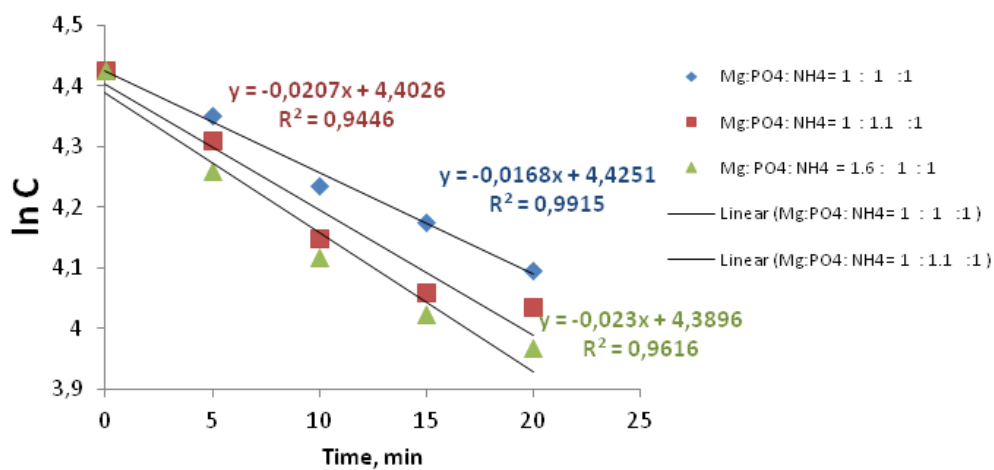


Figure 8. The first order kinetic reactions and constants determined for synthetic solution with different starting molar ratios with PO₄ concentration.

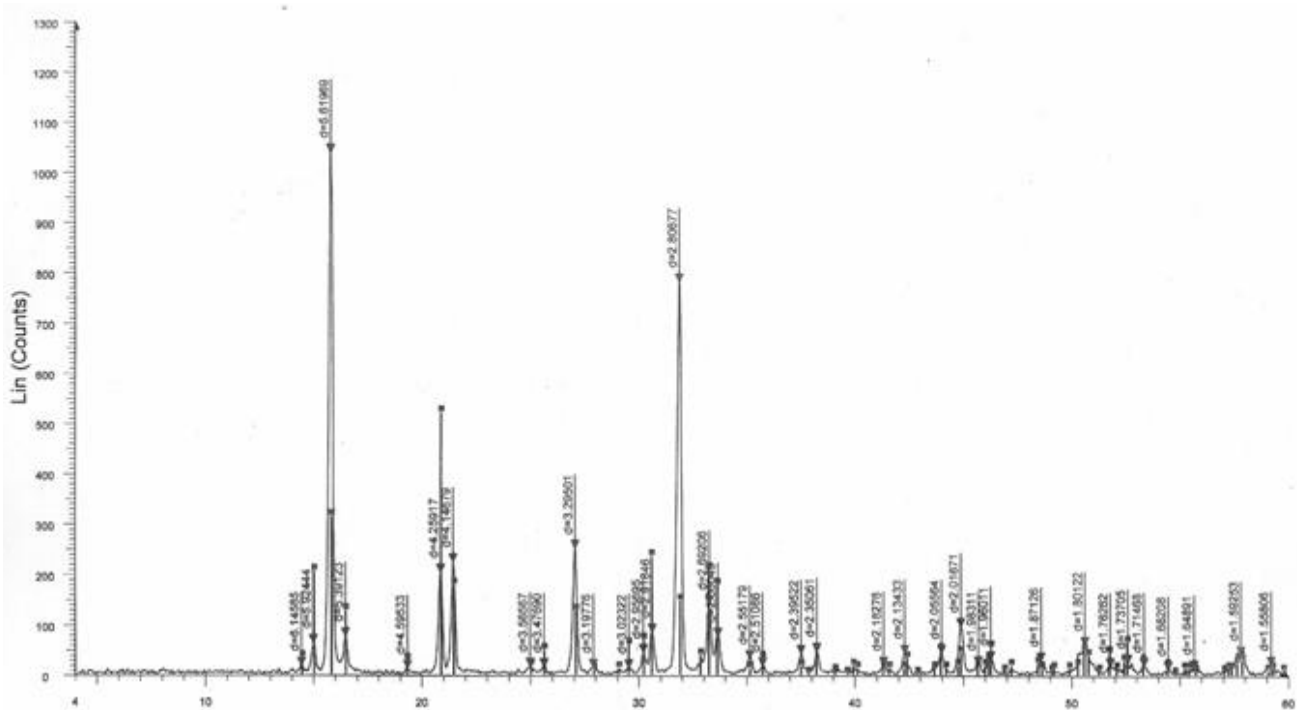


Figure 9. Struvite precipitated at ratio (1.6:1:1) MgNH₄PO₄·6H₂O

reached at increased Mg ratio compared with the equimolar and increased phosphorous ratio.

The increased yield of struvite as confirmed by XRD, SEM analysis shown in Figures (9) and (10) to reach 7.9 g as precipitated struvite from a feed stream of synthetic urine solution of 750 ml at higher magnesium concentration of 1.6 shows an increase in yield over that expressed in literature [21] with an accepted reached purity in Figure (11) which shows the EDX analysis of the struvite produced.

4. Conclusions

The feasibility of phosphorous removal from urine synthetic solution with struvite precipitation which was proved and maximized to reach around 90% of phosphorus removal percentage. It is essential to know the best conditions under which struvite precipitation reactors is likely to be designed for optimum phosphorous removal percentage and struvite yield. It is shown that an increase of starting Mg: PO₄: NH₄ with respect to magnesium (1.6:1:1) strongly influences the growth rate of struvite especially when compared with increase in starting phosphate (1: 1.1:1) and so on the efficiency of the phosphate removal. The results show that SSR, pH, and starting molar ratio strongly influences the kinetics of precipitation and so phosphorous removal to reach 93% removal percent, 5.95 mg/l as a minimum PO₄ remained in solution, and 7.9 g precipitated struvite from feed synthetic solution of 750 ml compared with only 4.98 g precipitated at starting ratio of 1:1:1 which was proposed in previous

literature. Indeed, the temperature elevation of the effluent is not required to promote struvite precipitation because it increases its solubility. The kinetics of struvite formation indicates that the reaction is apparent first order with respect to free phosphate concentration. The rate of the reaction decreases with the increase of precipitation time with respect to minimum phosphorous and magnesium concentration. At 14.5 °C precipitation temperature, (1.6:1:1) starting Mg:PO₄:NH₄ molar ratio, SSR of 14.98 which corresponds pH of 10 and 20 minutes as precipitation time; a maximum achieved phosphorous removal percentage (93%) was reached with increased amount of precipitated struvite crystals (7.9 g) when compared with that

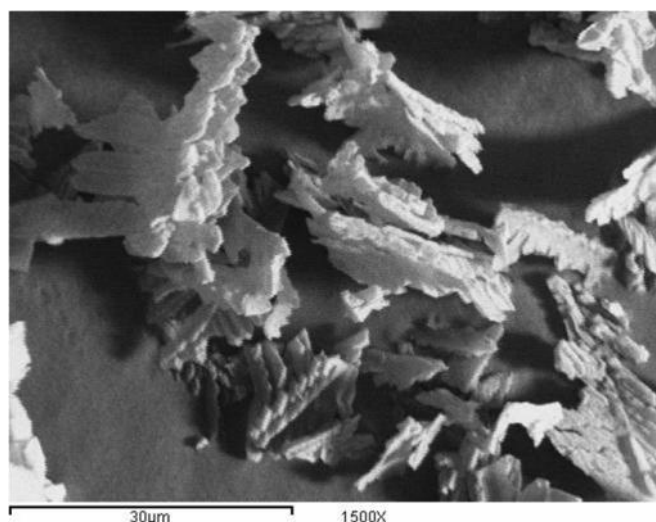


Figure 10. Struvite crystal morphology prismatic rectangular shaped

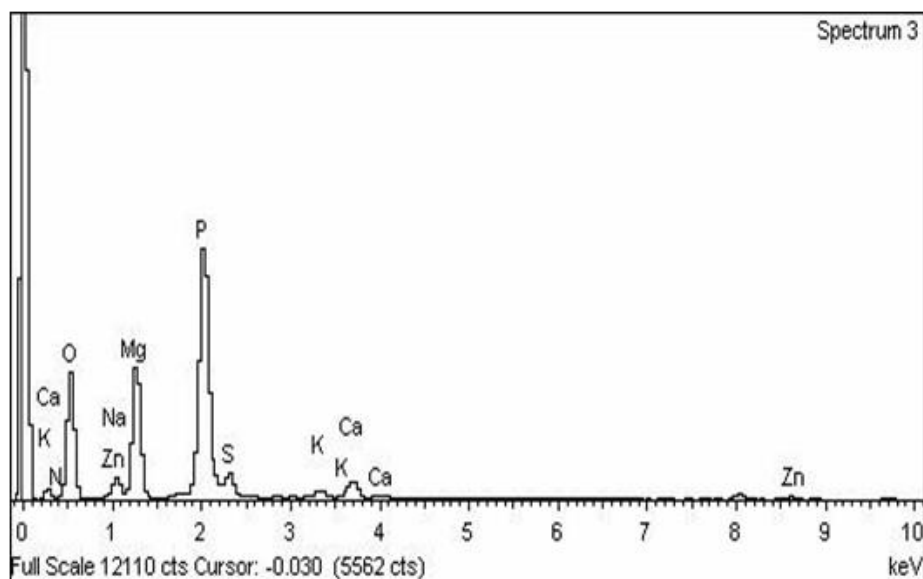


Figure 11. EDX pattern of precipitated struvite

reached amount at lower starting molar ratio.

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