

# Phosphorus recovery by struvite formation from Al Samra municipal wastewater treatment plant in Jordan

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#### ABSTRACT

Struvite precipitation is a well-known problem in wastewater treatment plants (WWTPs) as it causes clogging in pumps and pipes. However, controlled struvite recovery from sewage sludge can reduce these problems. The special feature of the present study is the pilot-scale reactor applied to study the efficiency of the designed system in struvite recovery from anaerobic sludge supernatant produced at Al Samra WWTP in Jordan. Moreover, various operational parameters effects on struvite formation have been investigated. By adjusting the pH value between 8.5 and 9, the efficiency of phosphorus removal achieved from experimental runs reached about 77%. Best efficiencies were obtained at high mixing speed (300 rpm), low mixing time (0.5 h) and settling time in the range of 8–16 h. Efficiency was found to be directly proportional to mixing speed and inversely proportional to mixing time and settling time. Struvite formation was verified by X-ray diffraction tests.

Keywords: Sludge; Phosphorus recovery; Struvite; Precipitation; Mixing; Settling

## 1. Introduction

Phosphorus recovery from sludge has been a subject of extensive researches and development efforts in Japan, Europe and North America over the past decade [1–3]. There are many primary drivers for researches and commercial development of phosphorus recovery technologies [4–9]. The most important long-term goal is to achieve sustainability in sludge handling and conservation of phosphorus resources [10]. Nutrient recovery from treated sludge is an option to reduce the quantities of sludge accumulated in treatment plants or disposed of at landfills. This in turn reduces the environmental problems caused by uncontrolled sludge disposal. On the other hand, since phosphorus can be regarded as a scarce resource today, there is significant necessity to find a renewable phosphorus resource to substitute the continuous depletion of phosphorus mining resources. Reducing the environmental impact of current phosphate-ore mining processes is another important driving force toward environmental friendly phosphorus recovery.

In this context, it is worth mentioning that even Jordan's phosphate-mining output is ranked fifth in the world, however, Jordan is also classified second in the world in water scarcity and is drastically influenced by climate change [11–16]. Jordan's carrying capacity and infrastructure were adversely influenced by the influx of refugees since mid of 2013 [17–20].

One of the most common methods for phosphorus recovery from sludge is the recovery of struvite after anaerobic sludge treatment process [21–23]. Struvite is the mineral name of magnesium ammonium phosphate hexahydrate (MgNH<sub>4</sub>PO<sub>4</sub>·6H<sub>2</sub>O) [24]. It is a soft phosphate mineral that can be utilized as a valuable source of fertilizer due to its low solubility characteristics, which minimize the danger of

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burning crop roots [25]. Moreover, it is a non-odorous and non-sludgy crystal which releases nutrients slowly [26].

Production of struvite results from a chemical reaction among magnesium, ammonium and phosphate ions with six molecules of water according to the following equation:

$$Mg^{+2} + NH_4^{+} + PO_4^{-3} + 6H_2O \rightarrow MgNH_4PO_4.6H_2O$$
 (1)

In WWTPs using enhanced biological nutrient removal process followed by sludge anaerobic digesters, digester supernatant was found to be rich of phosphate and ammonium [27]. Struvite precipitation in digester supernatant recycling lines, especially at the elbows and the suction side of pumps, is a well-recognized problem in many WWTPs worldwide in general [28], as well as in the largest treatment plant in Jordan at Kherbet Al Samra, in particular. Due to the turbulences caused by the pumps and pipe fittings such as valves and bends, degassing is taking place and consequently pH rises due to CO<sub>2</sub> release at these localized zones [29]. Hence, increasing pH levels increases the potential of struvite crystallization. At certain pH and saturation ratios for magnesium, ammonium and phosphate ions, the chemical reaction described in Eq. (1) starts to produce a quantity of struvite that accumulates and causes operational and maintenance problems [30]. Controlled struvite recovery after anaerobic sludge digestion and sludge dewatering is expected to benefit the treatment plant by reducing the unwanted struvite scale formation in the reject water recycling equipment. However, methods to control struvite scaling may have drawbacks in terms of cost (labor, materials and time) which can make them impractical [31].

Studies showed that phosphorus recovery from the anaerobic digested sludge centrate was large since the supernatant may contain up to 70% of phosphorus load of the plant [32]. Recovery efficiency can reach up to 90% of total phosphorus removed depending on sewage treatment process design, digestion process parameters, struvite crystallization method design and other operational conditions [22,33,34].

Various process parameters such as supersaturation ratio, pH, Mg:P molar ratio, etc., are likely to affect the struvite precipitation process; however, these parameters were studied in many researches. On the other hand, few studies discussed the effect of mixing and settling periods. Thus, the main objective of the present study is to investigate the effect of factors on the phosphorus recovery from treated sewage sludge centrate in the form of struvite. These factors are mixing speed, mixing time and settling time. Moreover, the present study aims also at assessing the efficiency of the designed reactor; verifying the struvite formation through controlled experiments; and studying the effect of the variable parameters on the efficiency of phosphorus recovery.

## 2. Materials

#### 2.1. Study area

Wastewater samples were taken from Al Samra WWTP which was selected for this study as it is the largest wastewater treatment plant in Jordan (350,000 m<sup>3</sup>/d). According to Al Samra WWTP's laboratory records,  $PO_4$  concentration is reduced from around 30–50 mg/L at influent to

10–15 mg/L approximately at effluent, which meets the Jordanian Standards for Reclaimed Domestic Wastewater (JS 893/2006). As the quantity of removed phosphorus is transferred to the sludge stream, it was important to assess the sludge treatment process.

Sludge removed from secondary settling tanks after biological treatment process is thickened in diffused aeration floatation thickeners, then it is treated in full mixed anaerobic digesters. According to the mass balance of the treatment process, the quantity of digested sludge produced is approximately 3,100 m<sup>3</sup>/d, and its content of total phosphorus (TP) is around 3,000 kg/d. After digesters, the sludge is dried in solar drying beds.

The operation staff of Al Samra WWTP started to search for other mechanical sludge dewatering alternatives due to the operational problems experienced in the solar drying beds. Therefore, a new decanter (sludge centrifuge) of 40 m<sup>3</sup>/d has been installed to test the viability of this option. More decanters are intended to be installed at a later stage to reduce the load on solar dewatering basins. The supernatant separated from the decanter is re-circulated to the inlet works of the treatment plant because of its high content of BOD, TSS and nutrients, which make it impossible to be discharged to the environment without treatment.

Al Samra WWTP has advanced biological nutrient removal process. In the process, a portion of anaerobic digested sludge is dewatered using a decanter (centrifuge) that separates sludge into a solid phase to be disposed of, and a liquid phase (supernatant). The struvite accumulation problem appears in the pumping and piping systems that convey this supernatant to the inlet works of the wastewater treatment plant. Every 2 weeks, the decanters have to be shut down and pumping system has to be stopped and cleaned, hence, around 2 kg of pure solid struvite are being removed. Flushing by water is not enough for cleaning sometimes due to the hardness of struvite scale, so hammering is needed. With time, some pipes and fittings have to be replaced as cleaning fails. Remediation is often not practical and expensive in terms of labor, materials and system downtime.

#### 2.2. Wastewater samples

Samples from the sludge supernatant produced from sludge decanters at Al Samra WWTP were used to run the experiments of the present study. Since all pipes and fittings that recirculate the supernatant to treatment plant's inlet works are subsurface, the only possible source to collect samples is the drain valve of the centrifuge supernatant discharge.

Sludge supernatant (liquor) was collected in containers of 20 L capacity. They were completely filled and transported from Al Samra WWTP to the pilot-scale reactor in the lab. Some samples of solid struvite precipitated and caused clogging in sludge supernatant pipes at Al Samra WWTP were also taken from the site for analyses and benchmarking.

#### 2.3. Experimental set up

As shown in Fig. 1, a pilot-scale reactor designed for this study is simple and more compatible with the structures of treatment plants. It mainly consists of mixing tank and settling tank which are locally manufactured from carbon steel and coated with chemical resistant epoxy coating (Carboline, Phenoline® 373) to sustain high pH. A locally manufactured galvanized carbon steel impeller with 20 mm shaft diameter was used for mixing. The speed of mixer was measured using a digital contact type tachometer with  $\pm$  0.05% accuracy. Portable pocket size pH meter manufactured by Hanna Instruments, Michigan, USA (pHep, HI 98107, accuracy  $\pm$ 0.1 pH) was used to check the value of pH through the different steps of the experiment. A sewage pump of 0.5 horse power manufactured by River Garden Pumps – China, was used to lift the liquor from the containers to the mixing tank.

Struvite precipitation unit consists of two tanks: cylindrical mixing tank of sludge supernatant (100 L capacity, 0.5 m diameter, 0.5 m side depth and flat bottom) with a base to initiate crystals nucleation, which is also connected via 2 inch pipe with another settling tank (0.5 m diameter, 0.5 m side depth and sloped hopper [30° inclination from horizontal] to collect settled solids and crystals from the bottom of the tank via 2 inch valve). The liquor flows from mixing tank to settling tank by gravity force due to the difference in levels.

## 3. Methods

Efficiency of orthophosphate  $(PO_4)$  removal was used as an indicator for the efficiency of struvite formation; Eq. (2) was used in calculations:

$$\eta = \frac{PO_{4IN} - PO_{4INF}}{PO_{4IN}} \times 100\%$$
(2)

where  $\eta$ : efficiency percentage of PO<sub>4</sub> removal; PO<sub>4 IN</sub>: initial concentration of PO<sub>4</sub> ions in liquor before mixing and pH adjustment (mg/L); PO<sub>4 F</sub>: final concentration of PO<sub>4</sub> ions in liquor after settling (mg/L).

## 3.1. Design of experiments

The experiments of the present study were designed to study the effect of three variables: mixing speed, mixing time and settling time on struvite formation by adjusting the pH from 8.5 to 9. Three mixing time durations (0.5, 1.0 and 2.0 h) were selected. At constant mixing duration, three mixing speeds (100, 200 and 300 rpm) were used, however, for each experiment, three settling times (8, 16 and 24 h) were also applied. Experiments were numbered T1, T2, T3, etc., as shown in the following array (Table 1).

The volume of sludge supernatant used to run the experiments was approximately 60 L per trial. All experiments were carried out at a room temperature in the range of 15°C–22°C. Fig. 2 summarizes the procedures of the experiments.

Sludge supernatant liquor was received from the treatment plant with pH value of 7.5–8.0. By adding NaOH



Fig. 1. Struvite precipitation pilot-scale reactor main components.

## Table 1

Matrix of experiments performed at variable parameters

Mixing time	0.5 h				1 h			2 h		
Mixer speed		100 rpm	200 rpm	300 rpm	100 rpm	200 rpm	300 rpm	100 rpm	200 rpm	300 rpm
Settling time	8 h	T1	T2	T3	T4	T5	T6	T7	T8	T9
	16 h	T10	T11	T12	T13	T14	T15	T16	T17	T18
	24 h	T19	T20	T21	T22	T23	T24	T25	T26	T27

317



Fig. 2. Struvite precipitation experiment flow chart.

(normal 6) manually to the supernatant in the mixing tank at the beginning of the mixing process, pH was adjusted to 8.5–9.0 for all experiments. The value of pH was checked throughout different stages of the same experiment to assure the stability of pH value.

After the mixing process was performed for a certain mixing time and at a certain mixing speed, the liquor was left to settle in the second tank. The proposed settling times are supposed to give sufficient time for struvite crystals to grow and precipitate at the hopper bottom of the tank with other solids that already existed in the liquor.

#### 3.2. Laboratory tests

At the beginning of each run, before mixing and pH adjustment, an initial sample from the raw sludge supernatant was taken to determine the concentration of orthophosphate (PO<sub>4</sub>). After the completion of settling duration, final sample was taken from the settling tank and specifically from the centrate at the surface of the liquor. For both samples, orthophosphate (PO<sub>4</sub>) was analyzed using Vanadomolybdophosphoric Acid Colorimetric Method (Test Method 4500 P-C).

For most of the experiments, ammonium  $(NH_4)$ , magnesium (Mg) and calcium (Ca) ion concentrations in initial and final samples were also analyzed. Preliminary distillation followed by Titrimetric Method (Test Method 4500 NH<sub>3</sub>-B&C) was used for ammonium. Magnesium was tested by Direct Air Acetylene Flame Atomic Absorption Method (Test Method 3111B) and calcium by Direct Nitrous Oxide Acetylene Flame Atomic Absorption Method (Test Method 3111D).

The above-mentioned tests were performed at Royal Scientific Society, Environmental Research Center's laboratories, according to Standard Methods for the Examination of Water and Wastewater issued by American Public Health Association, American Water Works Association and Water Environment Federation.

Samples of solids taken from the hopper bottom of the settling tank for most of the trials were dried using an oven of 40°C–45°C temperature, and then tested by X-ray diffraction (XRD). The aim of this test was to verify the formation of struvite. XRD analyses were also performed at the Royal Scientific Society.

#### 4. Results and discussions

## 4.1. Initial concentrations of PO<sub>4</sub>, NH<sub>4</sub> and Mg

The initial concentration of the three main struvite components (PO<sub>4'</sub> NH<sub>4</sub> and Mg) in raw sludge supernatant liquor is one of the essential parameters that affect struvite crystallization and precipitation. Therefore, the raw liquor was analyzed for most of the experiments. After determining the concentrations of the three main components, the molar ratios have been calculated in order to determine the limiting component.

Several researches showed that in most of similar experiments for struvite precipitation, the limiting ion was magnesium [35–37]. Therefore, the addition of external source of magnesium was necessary. However, the present

study shows that the limiting ion is  $PO_4$ , since the molar ratios  $NH_4$ :PO<sub>4</sub> and Mg:PO<sub>4</sub> had values larger than 1. The molar ratio Mg:PO<sub>4</sub> values were in the range of 1.6 to 3.7, so that there was no need to add external source of magnesium.

#### 4.2. Initial concentration of calcium and molar ratio Ca:Mg

The presence of calcium in sludge supernatant liquor has a significant effect due to the competition between calcium and magnesium that may occur during the experiment and affect the efficiency of struvite formation.

Initial concentrations of calcium were analyzed for a number of experiments and molar ratios Ca:Mg were calculated. It is noticed that the molar ratios Ca:Mg were in the range of 1.0 to 2.0, which leads to predict a competition between calcium and magnesium.

## 4.3. Final concentrations and removal ratios of PO<sub>4</sub>, NH<sub>4</sub> and Mg

The initial concentrations of  $PO_4$ ,  $NH_4$  and Mg in liquor, before mixing and pH adjustment, were given the subscript (<sub>IN</sub>). The final concentrations of  $PO_4$ ,  $NH_4$  and Mg after settling were given the subscript (<sub>F</sub>). The difference between initial and final value represents the removed quantity of each ion.

As stated before, struvite formation results from the reaction between equal molecules of each component  $(PO_4, NH_4 \text{ and } Mg)$ . Theoretically, both molar ratios  $NH_4:PO_4$  and  $Mg:PO_4$  for ions consumed in reaction should have the value 1:1. Since  $PO_4$  is the limiting ion in this study, the actual molar ratios of removed  $NH_4:PO_4$  and  $Mg:PO_4$  with values larger than or equal 1 ( $\geq$ 1) can indicate that all removed  $PO_4$  was consumed in struvite formation.

Moreover  $NH_4$ :PO<sub>4</sub> ratios have high values much larger than 1 which means that in addition to the amount of  $NH_4$ used for struvite formation, there is another large amount removed during the experiment. This can be explained by the chemical equilibrium between ammonium and ammonia, where ionized ammonium ( $NH_4^+$ ) tends to convert into free ammonia gas ( $NH_3$ ) when pH increases, according to the following reaction:

$$NH_4^{+} \rightarrow NH_3 + H^+$$
(3)

## 4.4. PO<sub>4</sub> removal efficiency

The efficiency of  $PO_4$  removal can be considered as an indicator for struvite formation efficiency, so it was calculated by dividing the amount of  $PO_4$  removed by the initial concentration as shown in Eq. (2). The results show that the highest efficiencies obtained were between 74.3% and 76.8% under 0.5 h mixing time and 300 rpm mixing speed.

Fig. 3 shows that the effect of changing the mixing time using 100 rpm speed mixer had a slight effect on efficiency. The effect clearly appeared at 200 and 300 rpm mixing speed where efficiency had an inverse relationship with mixing time. Highest efficiency values were obtained at 0.5 and 1.0 h mixing time with 300 rpm mixing speed.

When 16 h settling time was used (Fig. 4), increasing the mixing time from 0.5 to 1.0 h, caused the efficiency to decrease at all mixing speeds. The values of efficiency then became approximately constant when mixing time increased to 2 h. At this point, the effect of mixing speed disappeared and all efficiency values were approximately similar. Highest efficiency values were obtained using 300 and 200 rpm mixing speed at 0.5 h mixing time.

Although Rahaman et al. [38] found that the effect of mixing speed on struvite formation is negligible, the experimental observations of this study clearly indicated that mixing intensity has a significant effect on the struvite precipitation for the range of mixing speed tested at 8 h and 16 h settling time. It is important to notice that Rahaman et al. [38] used two mixing speeds (70 and 100 rpm) and 1.5 L jar test. However, the volume used in the present study is relatively much larger (60 L) and the range of mixing speed used is higher.

As stated before, in the precipitation process, crystal formation has two different stages: nucleation and growth. Induction time is defined by the time required for struvite crystals to nucleate. At the optimum pH, the induction time is affected by the fluid turbulence. Therefore, the induction time decreases as the mixing speed increases and the nucleation of struvite becomes rapid [31]. Once the nuclei are formed, struvite crystal growth is mainly controlled by the mixing energy [38]. If the mixing time is prolonged, the applied mixing energy might become in excess of the optimum value for the process parameters in the experiment,



Fig. 3. Efficiency of  $\mathrm{PO}_4$  removal at variable mixing time, variable mixing speed and 8 h settling time.



Fig. 4. Efficiency of  $PO_4$  removal at variable mixing time, variable mixing speed and 16 h settling time.

which might cause the break down or the dissolution of the initially formed crystals, and thus reduce the settling ability and the efficiency of phosphorus removal.

The high efficiencies obtained at high mixing speed (300 rpm) and short mixing time (0.5 h), as well as, the decreasing in efficiency by increasing the mixing time, indicate that the high mixing energy provided by the high speed within the first half hour is better for the nucleation to start rapidly and more efficiently. On the other hand, the extension of mixing for longer time is in excess and causes the efficiency to decrease due to the destruction of the formed crystals.

In Fig. 5, constant efficiency values were obtained using 24 h settling time, especially at 1 and 2 h mixing times regardless of the mixing speed. At 0.5 h mixing time, there is a slight variance in efficiency values at different speeds, but the general trend of the curves showed that high settling time neutralized the effect of changing the mixing time or the mixing speed. The values of efficiency obtained at 24 h settling time were in the range of 40%–55%. Results do not show high efficiencies at 300 rpm and 0.5 mixing speed if compared with Figs. 4 and 5.

The results obtained at long settling time (24 h), which showed negligible effect of mixing time and mixing speed on  $PO_4$  removal efficiency, and the decrease in efficiency at the conditions on which highest efficiency values were obtained at shorter settling times (300 rpm mixing speed and 0.5 h mixing time), can be explained by some possibilities. Long time of settling may become in excess which causes the formed struvite crystals to re-dissolve. The other possible cause is the change in  $PO_4$  concentrations of the final samples by the release of phosphorus to the liquor, or its uptake from the liquor by the active microorganisms, or by the decay of the dead cells present within the digested solid particles when long settling time was applied.

As mentioned previously, Etter [39] used a stirred tank reactor of 50 L volume to recover struvite from urine separated at source. Etter [39] obtained 80%–90% PO<sub>4</sub> removal efficiency by applying 15 min of mixing and minimum settling time of 24 h. These high efficiencies obtained are due to the use of urine instead of sludge supernatant, which is much richer in phosphate ions and its content of other solid particles and microorganisms is much less. Also, the mixing time used (15 min) were less than the minimum used in this study. As discussed previously, better efficiencies can be obtained by reducing the mixing time. However, high settling times



Fig. 5. Efficiency of  $PO_4$  removal at variable mixing time, variable mixing speed and 24 h settling time.

such as the 24 h used is not practical as it increases the volume of the settling tank specially if applied at full scale.

Since Figs. 4–6 show different trend at variable settling times, it was impossible to determine the general effect of the interaction of variables on  $PO_4$  removal efficiency. Therefore, Yates's Algorithm analysis was used as shown in the next section.

## 4.5. X-ray diffraction analysis

At early stages of the experimental work of the present study, it was necessary to test the nature of the scale material collected from Al Samra WWTP and caused clogging of digested sludge supernatant pipes by XRD in order to determine its chemical composition. XRD analyses were also used to verify the formation of struvite during experimental runs. All samples tested by XRD were compared with the characteristics of struvite identified in terms of the d-spacing at various  $2\theta$  angles. It was ensured that the scale material collected from Al Samra WWTP was pure struvite as shown in XRD graph in Fig. 6.

Samples of solids collected from the hopper of the settling tank were dried at 40°C–45°C and tested by XRD to verify that  $PO_4$  removed during the experiment was consumed in struvite formation. Figs. 7–9 show the resulted graphs obtained for selected experiments at different mixing speeds, mixing times and settling times.

XRD analyses show that the tested solids mainly consisted of struvite and calcite (CaCO<sub>3</sub>). Struvite peaks obviously appeared. The figures also demonstrated that when mixing speed was increased from 100 to 300 rpm in Figs. 7(a) and (b). The same results were noticed when the mixing speed increased from 100 to 200 rpm at a constant mixing time and settling time, the peaks on which struvite appeared were higher, while calcite peaks remained the same.

This indicates that struvite formation was enhanced either by quality or by quantity. Quality refers to the shape and size of struvite crystals relative to calcite crystals, while quantity refers to the amount of struvite crystals in the tested sample compared with that of calcite crystals.

The effect of mixing time was found negative on the efficiency of  $PO_4$  removal, and was also verified by XRD analyses. It was verified by XRD that struvite peaks decreased when 0.5 h mixing time was doubled at 16 h settling time and 100 rpm mixing speed. Same results were obtained when 300 rpm mixing speed was used. However, increasing the mixing time has a negligible effect on struvite peak. This was noticed at 24 h settling time while the XRD graphs show a very slight increase in struvite peaks resulted from increasing the mixing speed.

#### 4.6. Calcium competition

The concentrations of calcium ions were analyzed for initial and final samples for a number of experiments. Percentages of calcium removal were calculated and high values up to 96% were obtained. It is important to verify that the presence of calcium does not affect the formation of struvite by a competition between calcium and magnesium on PO<sub>4</sub> ions. It was confirmed by XRD analysis that the only compounds found in the tested samples were struvite and



Fig. 6. XRD graph for pure struvite sample collected from Al Samra WWTP.



Fig. 7. XRD graph struvite formation at (a) 1.0 h mixing time, 100 rpm, 8 settling time, (b) 1.0 h mixing time, 300 rpm, 8 settling time.

calcium carbonate (calcite), and there was no other compound that contains calcium and phosphorus together, as shown in Fig. 8. Thus, the calcium which was removed had been used in calcite formation and did not compete with magnesium in struvite.

## 4.7. Calcium carbonate (calcite) formation

The appearance of high peaks of calcite in the XRD graphs for samples of solids settled at the end of experiments necessitated the investigation regarding the timing of the precipitation. In other words, it was necessary to know if calcite formation and precipitation occurred during the struvite precipitation experiments or at prior stage. Therefore, a sample of raw sludge supernatant liquor received from the WWTP was dried and tested by XRD. The results showed that an amount of calcite exists in the raw sample before starting struvite crystallization experiment as shown in Fig. 9(a). However, Fig. 9(b) shows that the peak of calcite increased after finishing the struvite crystallization experiment, which means that calcite precipitation was also enhanced during the experiment.



Fig. 8. XRD analyses showing the presence of struvite and calcite only in the tested sample for (a) 1.0 h mixing time, 300 rpm, 8 h settling time, and (b) 1.0 h mixing time, 300 rpm and 24 h settling time.

Moreover, testing the scale material precipitated in sludge supernatant pipes at Al Samra WWTP supported that all precipitate was a pure struvite without any presence of calcite.

The clear relation between calcite precipitation and CO<sub>2</sub> degassing explains the presence of calcite in raw sludge liquor samples. When samples were collected from the drain valve of sludge centrifuge at Al Samra WWTP, the liquor transferred from closed system to open system and

the prolonged  $CO_2$  degassing caused an amount of calcite to precipitate. This amount increased during struvite crystallization experiment, in which mixing caused more degassing and pH was raised.

Calcite precipitation does not only depend on  $CO_2$  degassing and pH value, but also on other operational parameters such as temperature and pressure. In addition, its equilibrium depends on the system type; open or closed system [40]. This may explain why calcite precipitation did not take



Fig. 9. XRD analyses for (a) raw sludge supernatant liquor before starting struvite crystallization experiment, and (b) settled solids after finishing struvite crystallization experiment.

place in the wastewater treatment plant's sludge liquor pipes (which are considered as a pressurized closed system), where struvite precipitated in large quantities, although they both tend to precipitate at approximately the same conditions of CO, degassing and pH rise.

#### 5. Conclusions

The overall results obtained from this study, which aimed to investigate the potentiality of phosphorus recovery from treated sewage sludge in Jordan, proved that phosphorus recovery in the form of struvite is achievable. The suggested design of the pilot-scale struvite precipitation reactor, which consisted of mixing tank and settling tank, was found to be effective in removing up to 77% of phosphate from the supernatant of the anaerobic digested sludge generated at Al Samra WWTP. Since most of the phosphate removed was consumed in struvite formation, high values of phosphate removal efficiencies indicated that significant amounts of struvite were being produced. Struvite production was verified by XRD.

Mixing speed, mixing time and settling time were considered to be effective operational conditions for phosphate removal efficiency and struvite precipitation process. By adjusting the value of pH between 8.5 and 9, better performance of the system was found when mixing time and mixing speed were 0.5 h and 300 rpm, respectively, and when settling time was between 8 and 16 h.

Experimental results demonstrated that at the interval of 8 to 16 h settling time, phosphate removal efficiency was

directly proportional to mixing speed but inversely proportional to mixing time. Increasing the settling time from 16 to 24 h neutralized the effect of changing mixing time or mixing speed. It was also found that at this interval, there is an inverse relationship between settling time and phosphate removal efficiency.

The suggested struvite recovery process can be easily integrated into existing wastewater treatment plants since it is simple and easy to operate. The controlled recovery of struvite from digested sludge supernatant directly after dewatering process is believed to be the most feasible solution to reduce clogging problems in pipes. Moreover, it reduces the nutrients load that will be added by recycling the supernatant to the wastewater treatment plant without such recovery. However, some modifications are recommended to produce pure struvite since the struvite precipitated in the pilot scale reactor was mixed with calcite and digested sludge particles.

Furthermore, it was found that most of the calcite precipitated immediately after discharging the supernatant liquor from the closed pressurized system (the sludge decanters) to the open system (the filling containers), as a result of pressure release. Therefore, if a primary settling or filtering process is used upstream the mixing tank in the designed reactor, the initially formed calcite and the digested sludge solids will be removed before mixing and pH adjustment. This is expected to enhance the struvite crystallization and to facilitate the filtering requirements to yield pure struvite.

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